



# **ARCI**

## **PERFORMANCE REPORT**

### **2014-15**



ARCI is an autonomous R&D institute of Department of Science and Technology (DST), Government of India, set up with a mission to develop unique, novel and techno-commercially viable technologies in the area of advanced materials and subsequently transfer them to industries.

## THRUST AREAS

**Nanomaterials**

**Engineered Coatings**

**Ceramic Processing**

**Laser Materials Processing**

**Fuel Cells**

**Sol-Gel Coatings**

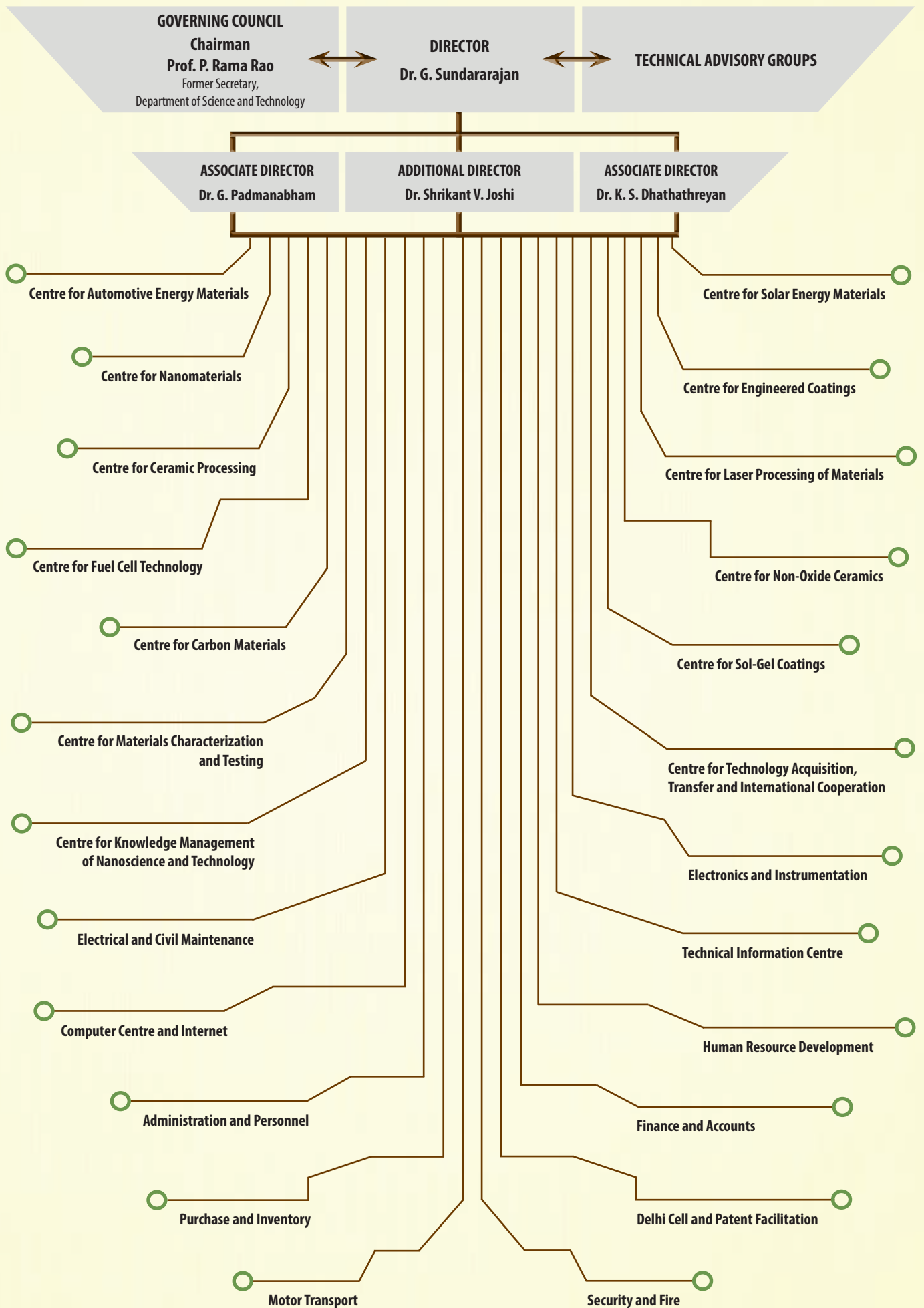
**Solar Energy Materials**

**Automotive Research**

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# ORGANIZATIONAL STRUCTURE





# International Advanced Research Centre for Powder Metallurgy & New Materials (ARCI)

## Governing Council

**Prof. P Rama Rao (Chairman)**

*Former Secretary, Department of Science and Technology*

**Dr. T Ramasami (till April 30, 2014)**

*Secretary, Department of Science and Technology*

**Prof. K Vijay Raghavan (from May 1, 2014 to January 8, 2015)**

*Secretary, Department of Science and Technology*

**Prof. Ashutosh Sharma (from January 9, 2015)**

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*Director - National Institute of Advanced Studies, Bengaluru*

**Dr. Amol Gokhale**

*Director*

*Defence Metallurgical Research Laboratory*

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*Chairman and Managing Director*

*Mishra Dhatu Nigam Limited*

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*Professor, Electrical and Electronics Department*

*Indian Institute of Technology-Bombay*

**Mrs. Anuradha Mitra (till December 14, 2014)**

*Joint Secretary & Financial Adviser*

*Department of Science & Technology*

**Shri J B Mohapatra (from December 15, 2014)**

*Joint Secretary & Financial Adviser*

*Department of Science & Technology*

**Dr. Arabinda Mitra**

*Head, International Division*

*Department of Science & Technology*

**Dr. G Sundararajan (Member Secretary)**

*Director, ARCI*

**Dr. Shrikant V Joshi (Non-Member Secretary)**

*Additional Director, ARCI*



# International Advanced Research Centre for Powder Metallurgy & New Materials (ARCI)

## Technical Advisory Groups

### Chairman and Members of Technical Advisory Group (TAG) of each Centre of Excellence

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**Dr. R. Muralidharan (Chairman)**  
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**Prof. S. Ram**  
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Indian Institute of Technology - Kharagpur*

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Mahindra Reva Electric Vehicles Pvt. Ltd., Bengaluru*

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Indian Institute of Technology - Bombay*

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*Department of Mechanical Engineering  
Indian Institute of Science, Bengaluru*

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*Head - Department of Solid State Physics  
Indian Association for the Cultivation of Science, Kolkata*

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*Principal Scientist - Inorganic & Physical Chemistry Division  
Indian Institute of Chemical Technology, Hyderabad*

#### Centre for Nanomaterials & Centre for Carbon Materials

**Prof. Ashutosh Sharma (Chairman) (till January 8, 2015)**  
*Institute Chair Professor, Department of Chemical Engineering  
Indian Institute of Technology - Kanpur*

**Prof. Sundara Ramaprabhu**  
*Alternative Energy & Nanotechnology Laboratory (AENL)  
Department of Physics, Indian Institute of Technology - Madras*

**Prof. G. U. Kulkarni**  
*Jawaharlal Nehru Centre for Advanced Scientific Research, Bengaluru*

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*Department of Metallurgical Engineering & Materials Science  
Indian Institute of Technology - Bombay*

**Prof. Ashok K. Ganguli**  
*Director  
Institute of Nano Science & Technology, Mohali*

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*Department of Physics  
Indian Institute of Technology - Madras*

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*Chief Scientist  
National Aerospace Laboratories, Bangalore*

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*Emeritus Professor  
Department of Metallurgical & Materials Engineering  
Indian Institute of Technology - Madras*

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*OS & Head, Laser & Plasma Technology Division  
Bhabha Atomic Research Centre, Mumbai*

**Dr. Subroto Mukherjee**  
*Head-FCIPT Division  
Institute for Plasma Research, Gandhinagar*

#### Centre for Ceramic Processing & Centre for Non-Oxide Ceramics

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*Chairman - Department of Materials Engineering  
Indian Institute of Science, Bangalore*

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*INAE Distinguished Professor, Department of Ceramic Engineering  
National Institute of Technology, Rourkela*

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*Head Department of Materials Science  
Sardar Patel University, Vallabh Vidyanagar*

**Prof. Parag Bhargava**  
*Department of Metallurgical Engineering & Materials Science  
Indian Institute of Technology - Bombay*

**Dr. M. Vijaykumar**  
*Scientist-G  
Defence Metallurgical Research Laboratory, Hyderabad*

#### Centre for Laser Processing of Materials

**Prof. Indranil Manna (Chairman)**  
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Indian Institute of Technology - Kanpur*



**Dr. G. Madhusudan Reddy**  
*Scientist "G" & Group Head, Metal Joining Group  
Defence Metallurgical Research Laboratory  
Hyderabad*

**Prof. Ashish Kumar Nath**  
*Department of Mechanical Engineering  
Indian Institute of Technology, Kharagpur*

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*OS, Laser & Plasma Technology Division  
Bhabha Atomic Research Centre, Mumbai*

### **Centre for Fuel Cell Technology**

**Dr. J. Narayana Das (Chairman)**  
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Indian Institute of Technology - Madras*

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*Senior General Manager (R&D), Mahindra & Mahindra Ltd.  
Mahindra Research Valley, Chennai*

**Prof. Prakash Chandra Ghosh**  
*Department of Energy Science & Engineering  
Indian Institute of Technology - Bombay*

### **Centre for Sol-Gel Coatings**

**Prof. D. Chakravorty (Chairman)**  
*Emeritus Professor  
Indian Association for the Cultivation of Science, Kolkata*

**Dr. Goutam De**  
*Chief Scientist & Head, Nano-structured Materials Division  
Central Glass & Ceramic Research Institute (CGCRI), Kolkata*

**Dr. K.G.K. Warriar**  
*Emeritus Scientist, Materials & Minerals Division  
National Institute for Interdisciplinary Science & Technology  
Trivandrum*

**Dr. Dibyendu Ganguli**  
*Rtd. Head, Sol-Gel Division  
CGCRI, Kolkata*

### **Centre for Materials Characterization and Testing**

**Prof. Indradev Samajdar (Chairman)**  
*Dept. of Metallurgical Engg. & Materials Science  
Indian Institute of Technology - Bombay*

**Prof. B. S. Murty**  
*Dept of Metallurgical & Materials Engineering  
Indian Institute of Technology - Madras*

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*Associate Professor, Department of Materials Engineering  
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**Prof. Sundararaman Mahadevan**  
*Visiting Professor, Indian Institute of Technology - Madras*

### **Centre for Technology Acquisition, Transfer and International Cooperation & Centre for Knowledge Management of Nanoscience and Technology**

**Dr. D. Yogeswara Rao (Chairman)**  
*Scientist 'G' and Head of Office  
O/o Principal Scientific Adviser to the Govt. of India, New Delhi*

**R. R. Hirwani**  
*Head - CSIR Unit for Research and Development of Information  
Products, Pune*

**Dr. Premnath Venugopalan**  
*Head, NCL Innovations & Intellectual Property Group  
National Chemical Laboratory, Pune*

**Dr. Krishna Tanuku**  
*Executive Director  
Wadhvani Centre for Entrepreneurship Development (WCED)  
Indian School of Business, Hyderabad*

**Prof. Rishiksha T. Krishnan**  
*Director & Professor of Strategic Management  
Indian Institute of Management, Indore*



# Director's Report

I am pleased to present to you the Annual Report of the International Advanced Research Centre for Powder Metallurgy and New Materials (ARCI) for the year 2014-15. This report reviews the various accomplishments of ARCI during the year on the basis of exhaustive and wide ranging R&D efforts undertaken by the various Centres of Excellence (COEs) of ARCI. The important technology achievements are highlighted below:



- A pilot-plant for manufacture of Li-ion battery, suitable for electric vehicles and electric two-wheelers, has been successfully established.
- A pilot-plant facility for fabrication of CIGS photovoltaic module is nearing completion and is expected to be operational in the next year.
- Oxide dispersion strengthened steels have been developed for supercritical thermal power plant application.
- Technology for production of Zn-S parts using CVD process has been transferred to an industry.
- Final field trials of aerogel based insulation technology is under way and it is expected that this technology will be transferred to the industry in the coming year.
- TiO<sub>2</sub> based photocatalytic self-cleaning technology for textiles has been developed and transferred to industry.
- An improved version of detonation spray coating system, without moving parts and having higher productivity, has been designed and developed and is likely to be launched in the coming year.
- Design and development of a laboratory-scale unit of Micro Arc Oxidation coating has been completed and these units are expected to be sold to a number of academic and educational institutions not only in India but also abroad.
- The design and development of a portable version of the Cold Spray Coating technology is nearing completion.
- Development of a number of new electrode materials for the cathode and anode of Li-ion battery are in progress.
- Soft magnetic alloy based on Fe-P with magnetic properties superior to the non-oriented silicon steel is nearing completion and is undergoing trials in the industry.
- Maximum energy conversion efficiency of 8.1% has been demonstrated in the case of pervoskite based PV using an ambient atmospheric process. The use of the same process, carried out under controlled atmosphere, is expected to give higher efficiencies.
- Solar absorbent, anti-reflective and self-cleaning coatings using cost-effective processes are under development for PV and CSP applications.
- A state-of-the-art T-Sapphire based micro machining system has been established. Microsurface structuring of automotive engine component materials, fabrication of micro heaters etc. are being carried out using this facility.
- Repair and refurbishment of worn out parts through laser based additive manufacturing has been demonstrated.
- A number of joining technologies for aluminium, steel and plastic in different dissimilar combinations have been developed in collaboration with the Fraunhofer institutions.
- A 5kW-48V DC Lab, powered by PEM fuel cell, has been demonstrated for 200 hours of operation without any degradation in performance.
- Continuous hydrogen generation technology required for the viability of PEM fuel cell programmes is also under intensive investigation and a number of novel catalysts and electrolytes have been designed for the above purpose.
- Organic-inorganic hybrid nano composite coatings, developed using sol-gel technique, have been successfully demonstrated for applications like decorative coatings on glass for architectural applications, chrome-free, self-healing and corrosion resistant coatings on aluminium and its alloys and anti-tarnish coatings on noble metals.
- Some of the new facilities added during the year include Microfocus X-ray diffraction unit, nano indenter and creep testing facilities. All these equipment have been installed, commissioned and are under use.

The performance of ARCI, as evidenced by numerous indicators presented in the next page, has been excellent.

As in the past, I would like to place on record the continuous support provided to ARCI by DST and the Governing Council of ARCI. I also take this opportunity to thank all the employees of ARCI for their unstinted cooperation and dedicated effort.

*G. Sundararajan*  
( G. Sundararajan )





Dual Beam Microscope [electron beam and focused ion beam (FIB)] at Centre for Materials Characterization and Testing



Inert Gas atomizer for synthesis of powders at Centre for Nanomaterials



Pilot plant for the large scale synthesis of sols at Centre for Sol-gel Coatings



Dr. Tata N Rao received the FAPCCI Excellence Award from Honourable Chief Minister of Andhra Pradesh on July 29, 2011



Dr. G Padmanabham has been conferred with the Andhra Pradesh Scientist Award in 2012



Dr. S. V. Joshi was inducted as a 'Fellow' of the Indian National Academy of Engineering (INAE) on December 06, 2012



Dr. G Sundararajan being honoured the 'Padma Shri' by the President of India



Visit of Dr. APJ Abdul Kalam to ARCI during INDOCIS Exhibition in 1996



Prof. P Rama Rao, Prof. VS Ramamurthy, H.E. Dr. Oleh Semenets (Ambassador of Ukraine) - inauguration of EB-PVD Facility



Parliamentary Standing Committee on S&T at ARCI in December 2001

## Major Personnel Accomplishments

## Unique

## Performance

| Parameters                                                                         | 1996-97          |
|------------------------------------------------------------------------------------|------------------|
| No. of Employees                                                                   | 107              |
| No. of Scientists                                                                  | 18               |
| No. of Publications*                                                               | 17               |
| Indian Patents**                                                                   | Granted<br>Filed |
|                                                                                    | Nil<br>6         |
| International Patents**                                                            | Granted<br>Filed |
|                                                                                    | Nil<br>Nil       |
| Scientists with Ph.D.                                                              | 8                |
| Scientists Registered for Ph.D.                                                    | Nil              |
| No. of Deputations Abroad                                                          | 2                |
| No. of Conferences/Seminars/Training Courses (in India) attended by ARCI Personnel | 16               |
| ARCI Fellows***                                                                    | Nil              |
| ARCI Trainees                                                                      | Nil              |
| M.Tech./B.Tech. Project Students****                                               | Nil              |

\* Includes journal publications, conference proceedings, and chapter in books

\*\* Cumulative figures up to end of financial year

\*\*\* ARCI Fellows also include ARCI-IIT Fellows, Post Doctoral Fellows, and Research Scholars

\*\*\*\* M. Tech/ B. Tech Project Students also include M. Sc Students

# Includes same patent granted in multiple countries

@ Includes persons who are continuing from previous years

## Visit of Eminent





Ultrafast Laser Micromachining System at Centre for Laser Processing of Materials



Pilot line for CIGS thin film solar cells at Centre for Solar Energy Materials



Li-ion battery facility at Centre for Automotive Energy Materials, Chennai

## Facilities

### Indicators

| 2013-14 | 2014-15 |
|---------|---------|
| 169     | 172     |
| 69      | 70      |
| 150     | 131     |
| 23      | 24      |
| 55      | 59      |
| 8#      | 9#      |
| 4       | 4       |
| 45      | 47      |
| 14      | 10      |
| 37      | 31      |
| 285     | 395     |
| 54@     | 68@     |
| 48@     | 67@     |
| 50@     | 65@     |

## Technology Collaborations and Transfers



Handing over of Nano-Silver based candle filters to Byrāju Foundation by Prof. V S Ramamurthy, Secretary-DST



Signing of contract with EPG, Germany to set-up a joint demo centre for sol-based coatings



Inauguration of Zoz-ARCI joint demo centre for high energy ball milling by Prof. Henning Zoz, President-Zoz GmbH-Germany



Handing over of the know-how document consequent to transfer of technology on Nano Titania for self-cleaning textiles

## Personalities



Shri Bachi Singh Rawat, Minister for Science and Technology, Govt. of India at ARCI



Dr. T Ramasami, Secretary DST during Governing Council meeting at ARCI



Shri Prithviraj Chavan, Minister for Science and Technology, Govt. of India visited ARCI in 2010



## Centre for Automotive Energy Materials

**C**entre for Automotive Energy Materials (CAEM) was specifically setup at Indian Institute of Technology-Madras (IIT-M) Research Park, Chennai by ARCI with a mission to develop techno-commercially viable technologies in the area of automotive energy saving materials and subsequently demonstrate / transfer them to Indian industries. In the above approach, the Centre at Chennai has set for itself the task of striving to bridge the gap between CAEM and the Automotive industries. Consistent with this overall goal, CAEM has dedicated its research effort towards the following: (i) Development of Li-ion battery materials and cells technology for Electric Vehicle (EV) applications (ii) Demonstration of soft and hard magnets technologies at prototype level for motors and alternator applications (iii) Translating high efficiency thermoelectric materials technology towards waste heat recovery applications and draw their attention to industries. The above three major goals are being executed at the Centre under various sponsored projects.

For many years, nickel-cadmium had been the only suitable battery for portable equipment from wireless communications to mobile computing. Nickel-metal-hydride and lithium-ion emerged in the early 1990s, fighting nose-to-nose to gain customer's acceptance. Lithium is the lightest of all metals, has the greatest electrochemical potential and provides the largest energy density for weight. Today, lithium-ion battery is the fastest growing and most promising battery chemistry for sustainable energy transportation. There is no doubt that lithium-ion cell chemistry offers some of the best options for electrical energy storage for high-power and high-energy applications such as transportation and stationary storage due to their electrochemical potential, theoretical capacity, and energy density. In India, the technology is in the initial stages of its establishment and CAEM, ARCI, Chennai has taken a lead role quickly to move forward in establishing the pilot scale facilities for Li-ion for manufacturing of battery cells of various capacities. The Centre is also focusing in developing new electrode materials in Li-ion chemistry for EV applications. The first prototype Li-ion batteries based on  $\text{LiFePo}_4$  are expected to be developed by the Centre during 2015-2016 for demonstration to the EV manufacturers in the country.

CAEM has made considerable progress in magnet programme during 2014-2015. One of the major accomplishments of the Centre was in developing a new soft magnetic alloy based on Fe-P, with magnetic properties superior to the non-oriented Si-steel. This alloy is now being explored for stator applications in motors and alternators for automotive sectors. The Centre is also focusing in developing rare earth less / free hard magnets as well as high performance hard ferrites for various automotive applications. In 2014, the Centre has initiated a seed project on thermoelectric (TE) materials with an aim to develop high thermoelectric efficiency materials, so that their potential for auto exhaust heat recovery for power conversion can be explored.

The Centre has established a strong collaboration with leading companies from automotive and other relevant sectors. M/s. Lucas TVS, Chennai (a pioneer in manufacturing motors of different capacity) and M/s. Mahindra Reva-Bangalore (EV car manufacturer), BEL-Pune, BHEL-Bangalore and with many other automotive institutes / industries. In coming years, CAEM, will closely interact with industries to fabricate prototype devices and evaluate the performance through various field trials for realizing the technology. The major accomplishments of CAEM during 2014-15 are brought out as independent reports by our Team members in this annual report.

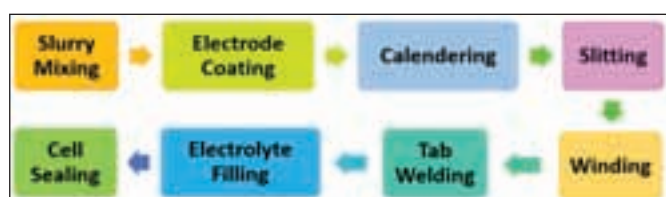


# Fabrication of Prototype Cylindrical Lithium Ion Cell

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Lithium ion battery (LIB) is one of the potential candidates for high energy applications like Electric Vehicles/Hybrid Electric Vehicles. Performance of the LIBs relies on the chemistry of various cathode, anode, and electrolyte used, as well as the cell fabrication process. LIB fabrication process requires humidity controlled atmosphere. Two dehumidified rooms have been established at CAEM. Typically, the rooms have relative humidity of 30% and 0.5% for the fabrication of electrodes and cells, respectively. The cell fabrication process is as given in Scheme 1.



Scheme 1 Flow chart for Li-Ion cell fabrication process

$\text{LiFePO}_4$  cathode and graphite anode slurries were prepared in separate batches using 4-5 kg materials and 5L of N-methyl pyrrolidone. The ratio of active materials, binder (PVDF) and conductive carbon was kept at 90:6:4 for cathode slurry. For anode slurry conductive carbon was not used, thus the ratio of active material to binder was 94:6. The mixing was carried out under static vacuum until homogenous slurry was obtained (~6 h). Rheological measurements were carried out at different stages of mixing process. The slurry viscosity for both cathode and anode was in the range of 15,000-30,000 cP. Slurries were coated on current collectors (Al/Cu) using

coma coating technique and subsequently dried in hot air ovens at 120-150°C. Double side coated electrodes of 150 m length with uniform thickness has been prepared as shown in Figure 1a.

The electrodes were calendered using mirror-finished rollers to decrease the porosity as well as to increase the density and conductivity. After calendering the thickness of the cathode and anode are  $120 \pm 5 \mu\text{m}$  and  $90 \pm 5 \mu\text{m}$ , respectively. The obtained loading per area was  $14 \text{ mg/cm}^2$  for cathode and  $10 \text{ mg/cm}^2$  for anode. The width of the electrodes was slit appropriately for the fabrication of prototype cylindrical cells (3 Ah and 1 Ah). Alternative layers of slit electrodes and separators were wound to form jelly rolls of desired dimensions (Figure 1b). Aluminium and copper tabs were welded to the cathode and anode respectively. The optical micrograph of the wound roll shows uniform winding (Figure 1c). The tabs are welded to the terminals by ultrasonic welding. Resistance between positive and negative terminals was measured to be  $> 4\text{G}\Omega$ , confirming that there is no short-circuit. The wound roll was packed inside a polypropylene container. Cells were evacuated for overnight at 60°C. An appropriate volume of electrolyte ( $\text{LiPF}_6$  in solvent mixture) was filled under inert atmosphere. The cell was then sealed properly and leak-proof test was performed. The cell was kept for equilibration (~48-72 h) until stable open circuit voltage of ~0.2 V was obtained. Currently the electrochemical charge/discharge testing of the prototype cells is in progress.

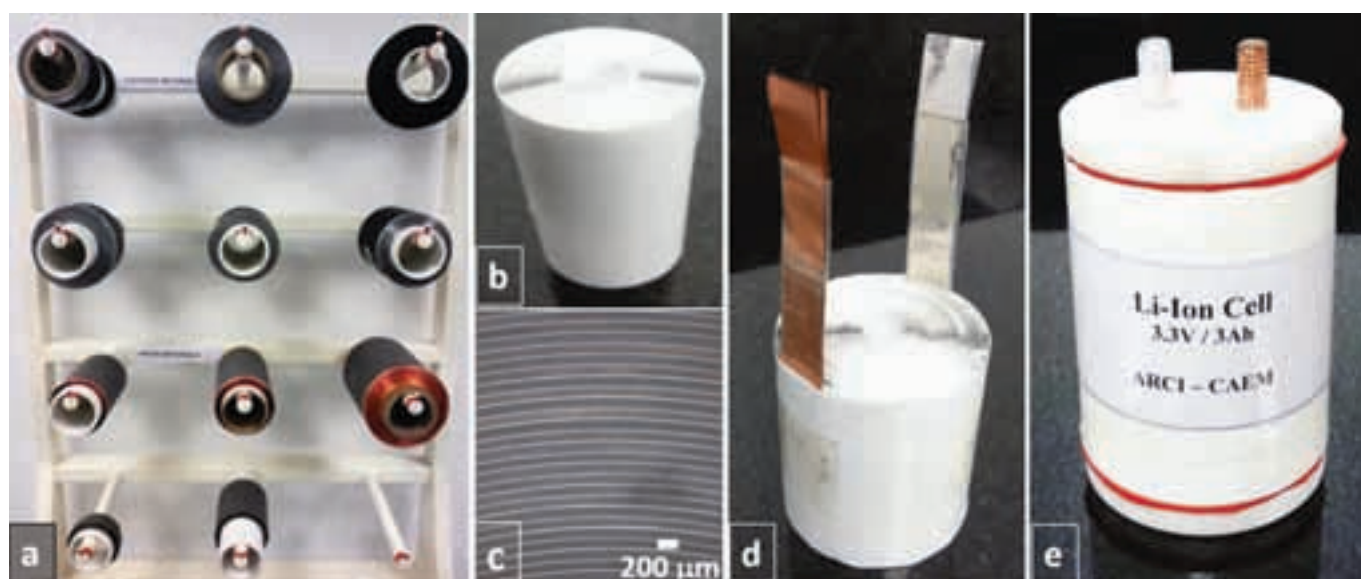


Fig1. Cell fabrication process a) Electrode rolls b) Wound cylindrical jelly roll c) Optical micrograph of winding (top view) d) Jelly roll with tabs e) Final 3.3V/3 Ah Li-Ion prototype cell

Contributors: Sumit R Sahu, K Shanmugam, L Babu, S Jana, A Sivaraj, TP Sarangan, R Vallabha Rao, K Kumari, VVN Phanikumar, S Bhuvaneswari, K Tanuja and R Gopalan



# Effect of Cu and Ni Doping on Thermoelectric Properties of Nanostructured PbTe System

D Sivaprahasam

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PbTe based compounds are one of the widely investigated thermoelectric (TE) materials. The conversion efficiency, which depends on thermoelectric figure of merit  $ZT > 2$  has been demonstrated in bulk PbTe by suitable doping and microstructure engineering.  $ZT$ , is defined as  $ZT = S^2 \sigma / \kappa$  where  $S$  is Seebeck coefficient,  $\sigma$ -electrical resistivity and  $\kappa$ -thermal conductivity.  $ZT$  in general is enhanced by two approaches. Nanostructuring or nanoscale architecture of the materials enhances the phonon scattering resulting in decrease of  $\kappa$  and hence higher  $ZT$ . Improving the power factor ( $S^2 \sigma$ ) by modifying the density of states is another approach. Doping various elements such as Ag, Sb enhanced the  $ZT$  of PbTe system by power factor enhancement. PbTe compounds with hitherto unexplored doping elements such as Cu and Ni are synthesized in the present study and processed into bulk solid compacts for the purpose of fabrication of thermoelectric devices. The Cu and Ni being the interconnect materials used in thermoelectric devices, may alloy with the PbTe legs during the operation of the devices at higher temperatures. Hence, it is important to know their effect on the PbTe system. Nanostructured

alloys of nominal composition  $\text{CuPb}_{18}\text{SbTe}_{20}$  (Figure 1a) and  $\text{NiPb}_{18}\text{SbTe}_{20}$  were prepared by vacuum arc melting, annealing, jet milling (Figure 1b) followed by spark plasma sintering (SPS) of the powder into 30 mm diameter disc of more than 98% relative density using WC/Co die cavity. Both Cu and Ni doping has resulted in n-type PbTe (Figure 2). The Seebeck coefficient and electrical conductivity measured up to 800 K revealed that the Cu and Ni doping resulted in lesser  $ZT$  value in comparison to commonly reported high  $ZT$  materials such as  $\text{AgPb}_{18}\text{SbTe}_{20}$  in this system (Figs. 3 & 4). It is also observed that the thermal conductivity of Cu doped and Ni doped compounds is significantly higher than  $\text{AgPb}_{18}\text{SbTe}_{20}$ . The  $ZT$  obtained in both Cu and Ni doped compounds is around 0.4 at room temperature against 0.5 of  $\text{AgPb}_{18}\text{SbTe}_{20}$ . Interestingly, doping of PbTe with Cu and Ni showed reduction in the fracture toughness of the PbTe, resulted in better structural stability of the legs and the interfaces created between the legs, diffusion barrier layer and the inter connects in the TE device. Studies are in progress to make a device based on the above systems.

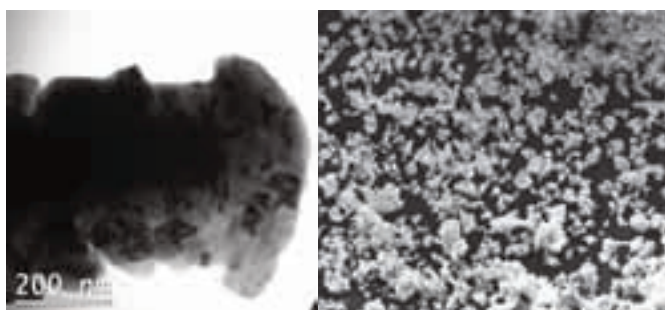


Fig. 1 (a) Nanostructured  $\text{CuPb}_{18}\text{SbTe}_{20}$  (b)  $\text{CuPb}_{18}\text{SbTe}_{20}$  after jet milling



Fig. 2 Spark plasma sintered Cu and Ni doped PbTe samples

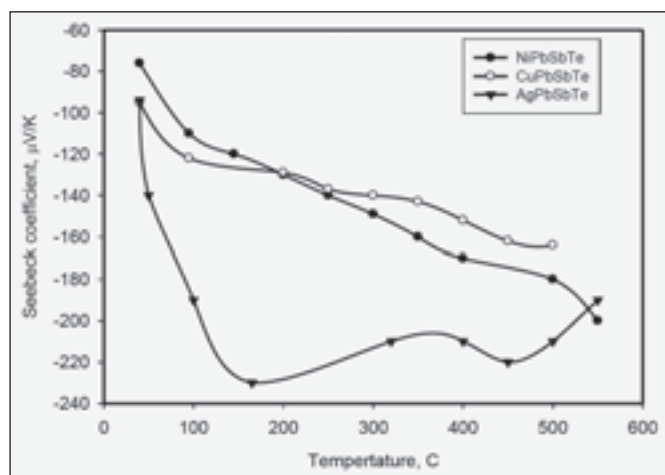


Fig. 3 Seebeck coefficient of Cu, Ni and Ag doped PbTe samples

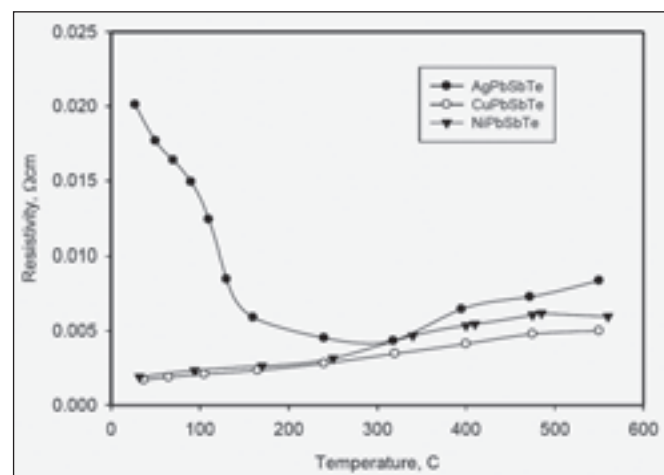


Fig. 4 Electrical conductivity of Cu, Ni And Ag doped PbTe samples

Contributors: R Preethi Venket and R Gopalan

# Fe-P Alloys for Claw Pole Alternators in Automotives

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Alternators are essential components in modern automobiles which generate electricity when the engine is running to charge the battery and to power other electrical systems used in the automobile. Figure 1(a) is a typical claw pole alternator used in an automotive and Fig 1 (b) is the exploded view showing the various components of a typical claw pole alternator. The claw pole is a soft magnetic material requiring high magnetic induction and permeability. The currently used commercial samples have a magnetic induction of 1.6 T and permeability of 2300.

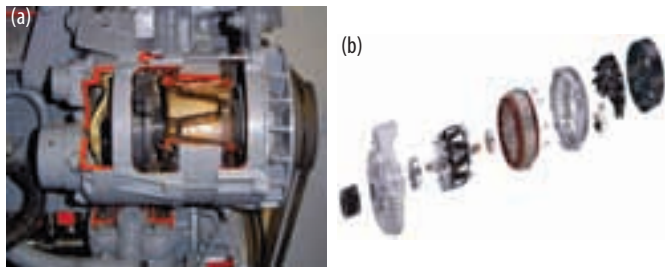


Fig. 1(a) Typical claw pole alternator which is driven by the belt (extreme right) connected to the engine and (b) exploded view showing the claw pole (indicated by arrow) made of soft magnetic material

An alternate material with a higher magnetic induction and permeability will result in higher efficiency and concomitantly save energy. Fe-P-Si based alloy with high induction and low coercivity developed at ARCI is being explored for this application. Fe-P-Si based alloy was produced by melting elemental Fe, with master alloys of  $\text{Fe}_3\text{P}$  and  $\text{FeSi}_3$  in suitable weight ratio in vacuum using an induction melting furnace.

The alloy was forged and rolled to a thickness of 5 mm for the purpose of characterizing these alloys for the magnetic properties. A B-H loop tracer was used for measuring the

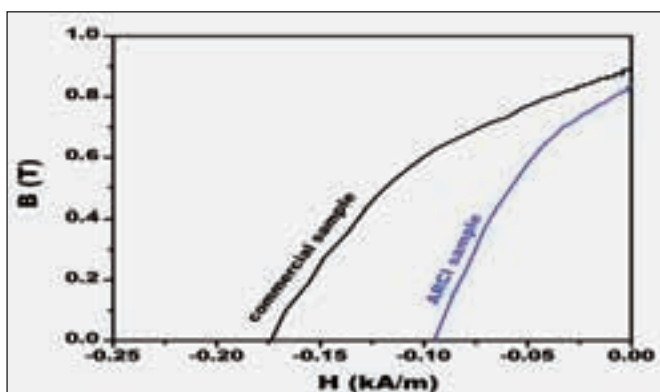


Fig. 2 The second quadrant of the DC loop showing lower coercivity

magnetic properties. For comparison purpose, all the samples were fabricated in the toroid form with outer and inner diameter of 50 and 40 mm respectively with thickness of 5 mm. Currently used commercial sample was also obtained from user agency and was fabricated into toroid of the above dimension for comparing the properties. Figure 2 shows the second quadrant DC magnetic loop with ARCI sample showing lower coercivity than the commercial sample. Figure 3 shows the initial magnetization curves which clearly demonstrate that the ARCI sample has enhanced magnetic induction of 1.65 T and permeability 3500 which are better than the currently used samples.

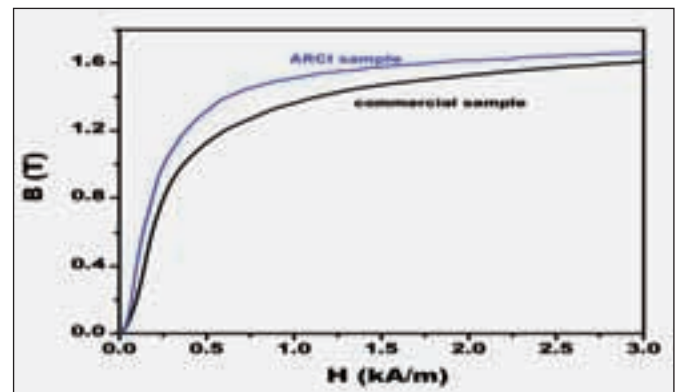


Fig. 3 The initial magnetization curve showing enhanced induction & permeability

Currently large quantity of alloy is melted for fabrication of a prototype alternator with ARCI sample for the purpose of testing the material in actual application. Figure 4 shows the 15 kg ingot melted for this purpose. Claw poles shall be fabricated by direct hot forging process from the discs cut of the melted ingot.



Fig. 4 The ingot of the ARCI sample which would be used for fabricating the prototype alternator

*Acknowledgement: The authors would like to acknowledge Director, DMRL, Hyderabad for providing melting and rolling facilities.*

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# Electrolyte Filling for Lithium Ion Batteries

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Electrolyte is one of the major components of lithium ion batteries. It is the medium of conduction for lithium ions between cathode and anode during charge or discharge. Cell properties like specific capacity, reversibility and calendar life are dependent on the composition of the electrolyte. Since, the lithiated carbon ( $\text{Li}_x\text{C}_6$ ) is extremely unstable in water, only non-aqueous electrolytes have to be used. Based on electrode materials and applications, appropriate volume ratio of different organic solvents [ethylene carbonate (EC), dimethyl carbonate (DMC), diethyl carbonate (DEC), ethyl methyl carbonate (EMC) and propylene carbonate (PC)] can be mixed together with lithium hexafluorophosphate ( $\text{LiPF}_6$ ) or lithium perchlorate ( $\text{LiClO}_4$ ) to get the desired electrolyte. The most commonly used salt is  $\text{LiPF}_6$ . The drawback of  $\text{LiPF}_6$  electrolyte is its decomposition at high temperature according to eq. 1, which leads to polymerization of cyclic carbonates and degrade the electrolyte and solid electrolyte interface layer. Thus, we have to select the solvent accordingly, either having a wide temperature/voltage window or additives/stabilizers. For example, we can use lithium bis-oxalato borate (LiBOB) as additives which has good thermal and chemical stability. For EV applications we have chosen  $\text{LiPF}_6$  as salt and EC:DMC:EMC with or without LiBOB additive based electrolytes.



Cathode and anode fabricated using pilot plant facility are calendered and slit according to the required dimensions for the fabrication of prototype cells. After winding of the electrodes and separator into jelly rolls, it is placed inside a suitable container (cylindrical/prismatic). The lid with GTM is welded hermetically to the can and the container and it is

placed inside the nest (cylindrical or prismatic). Electrolyte is filled into the cell by using a filling machine. As the electrolyte is extremely sensitive to both oxygen and moisture, the filling process is carried out inside 0.5% RH dry room. The moisture content of electrolyte should be less than 20 ppm for better performance of cell. Container containing electrolyte is connected to the hose of the filling machine. The filling machine operates under pneumatic pressure. The maximum fill volume is 260 mL. Required volume can be set manually by adjusting the micrometer attached to the dispense pump. Before filling, the cell is evacuated completely. After that, the electrolyte is pumped into the cell by the help of dispense pump. Besides, it has a suck back pump, which is used to suck back the over-filled electrolyte. Electrolyte can be filled either by direct fill or pre-fill mode. In the direct fill mode, the machine directly pumps the electrolyte into the dispensed pump and push it into the cell. In prefill mode, the dispense pump pushes the electrolyte into the prefill chamber, and then into the cell. Filling mode can be selected based on the application. The filling machine can be operated in fully automatic mode or manual mode.

Fresh dry air from the air inlet of 0.5 % RH room was connected to the electrolyte filling chamber (Figure 1a) to avoid any moisture absorption in the electrolyte. Accuracy of the dispensed volume was checked through repeated trials (10-260 ml) using pre-fill and direct fill modes. The observed error limit was <2% for both the modes. The appropriate volume of electrolyte was filled into the prototype cells using pre fill method. The cells were sealed and kept for equilibration. A typical calibration data for electrolyte filling in pre-fill mode is shown in Fig. 1b. This information will be useful while filling the predetermined volume of electrolyte for assorted type of cells.

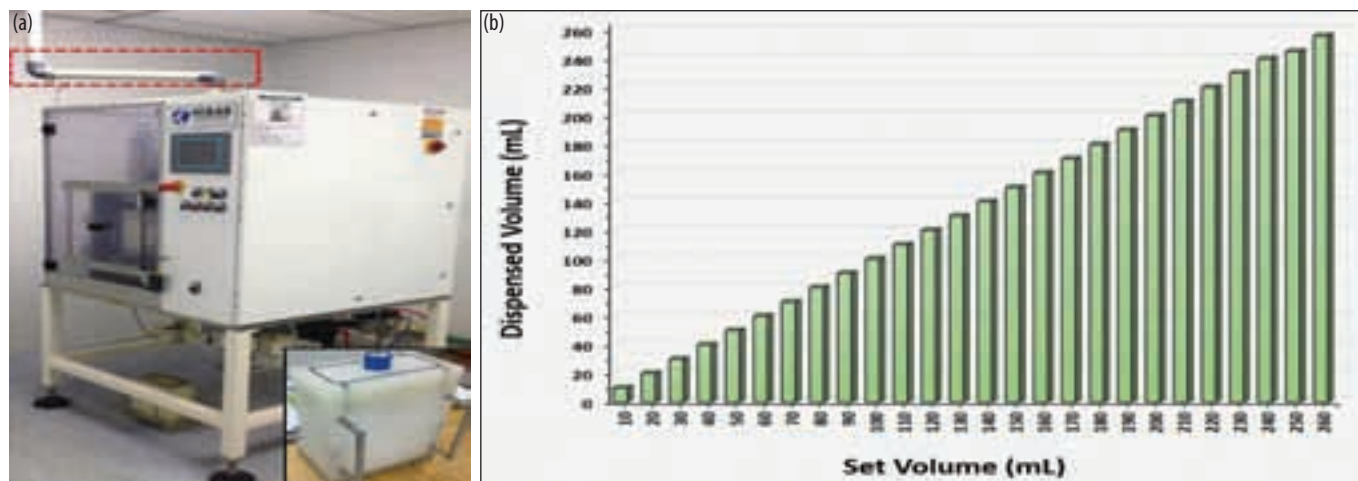


Fig 1. (a) Fresh dry air connection; nest with cylindrical cell (inset) (b) Calibration data for pre-fill mode

Contributors: K Shanmugam, R Prakash and R Gopalan



# Thermoelectric Properties of $\text{CoSb}_3$ based Skutterudite Materials for Automotive Applications

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Thermoelectric (TE) device converts heat directly into electricity when a temperature gradient is created across the device. The TE materials required for the device must have figure of merit (ZT)  $\sim 2$  and above at the operating temperature. Substitution and doping in the binary  $\text{CoSb}_3$  skutterudites draw enormous attention for automotive TE device due to their stable thermoelectric properties in the temperature range 573 K to 873 K. Moreover, skutterudites have advantages over other TE materials due to their flexibility of fabrication and cost effectiveness.

We are synthesizing polycrystalline  $\text{Sn}_x\text{Ba}_{0.4}\text{Co}_4\text{Sb}_{12-x}$  ( $x = 0$  and  $0.4$ ) skutterudite samples by ball milling and spark plasma sintering routes. The constituent elements of the alloys ( $\text{Ba}_{0.4}\text{Co}_4\text{Sb}_{12}$  and  $\text{Sn}_{0.4}\text{Ba}_{0.4}\text{Co}_4\text{Sb}_{11.6}$ ) are taken in the form of elemental powders and dendrites. The mixed powders are mechanically alloyed in Fritch Ball Mill for 12hrs under Ar atmosphere using WC-Co grinding medium. The milled powders are hot pressed using spark plasma sintering technique at 873 K to get samples having dimension of 20 mm diameter and 6 mm thickness with density  $\sim 99\%$ .  $\text{Cu}_2\text{O}$  nanoparticles are dispersed in  $\text{Sn}_x\text{Ba}_{0.4}\text{Co}_4\text{Sb}_{12-x}$  matrix to make the composite TE materials. Typical XRD pattern of the spark plasma sintered samples is shown in Figure 1. It shows that the sample is single phase and is stable upto 773 K, which is the typical operation temperature of skutterudite materials for automotive thermoelectric generator. Figure 2 shows the selected area electron diffraction (SAED) and dark field TEM images of the  $\text{Cu}_2\text{O}$  dispersed  $\text{Ba}_{0.4}\text{Co}_4\text{Sb}_{12}$  ball milled powders.  $\text{Cu}_2\text{O}$  is present as secondary phase and the milled powders are of nanocrystalline nature.

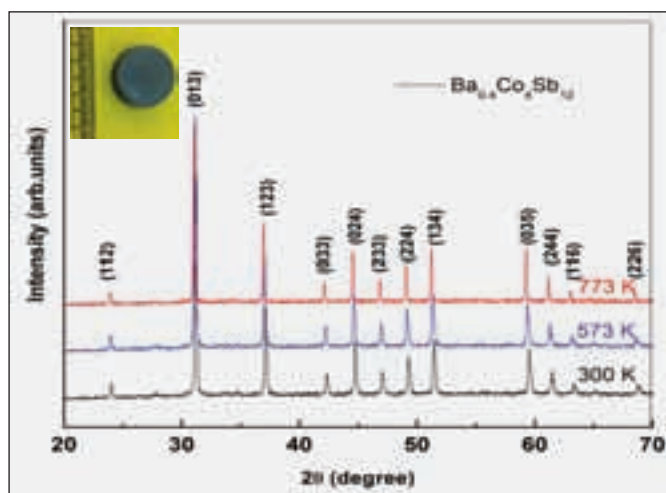


Fig.1 XRD of  $\text{Ba}_{0.4}\text{Co}_4\text{Sb}_{12}$  performed at 300 K, 573 K and 773 K. Peaks are indexed according to  $\text{CoSb}_3$  phase. Inset shows the spark plasma sintered  $\text{Ba}_{0.4}\text{Co}_4\text{Sb}_{12}$  sample

Electrical resistivity and thermopower measurements using Seebys system confirm that the electronic properties of  $\text{Sn}_x\text{Ba}_{0.4}\text{Co}_4\text{Sb}_{12-x}$  ( $x = 0$  and  $0.4$ ) are enhanced by the dispersion of  $\text{Cu}_2\text{O}$  nano particles. On the other hand, thermal conductivity is drastically reduced in  $\text{Cu}_2\text{O}$  dispersed  $\text{Sn}_x\text{Ba}_{0.4}\text{Co}_4\text{Sb}_{12-x}$  ( $x = 0$  and  $0.4$ ) samples. Highest ZT= 0.93 is achieved at 573 K. Further investigation is under progress to enhance the ZT of the samples for automotive TE generators.

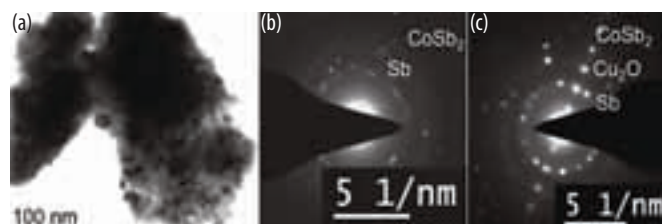


Fig.2 TEM images (a) bright field image of  $\text{Ba}_{0.4}\text{Co}_4\text{Sb}_{12}$  milled powders, (b) SAED pattern of  $\text{Ba}_{0.4}\text{Co}_4\text{Sb}_{12}$  milled powders, (c) SAED pattern of  $\text{Cu}_2\text{O}+\text{Ba}_{0.4}\text{Co}_4\text{Sb}_{12}$  milled powders

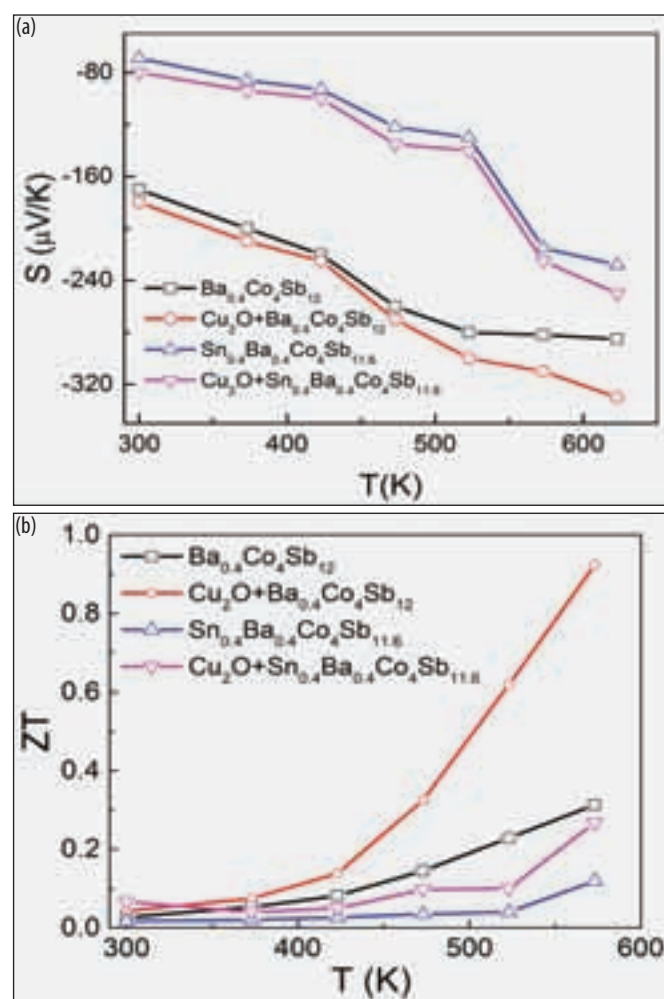


Fig.3 Temperature variation of thermopower (S) (a) and figure of merit (ZT) of the studied samples (b)

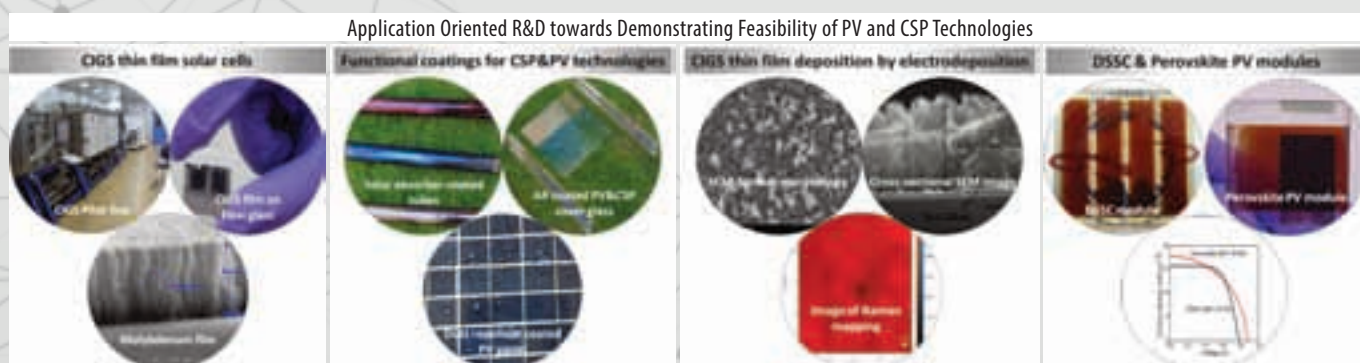
Contributors: B Priyadarshini, D Sivaprahasam and R Gopalan

## Centre for Solar Energy Materials

The Centre for Solar Energy Materials (CSEM) was established at ARCI with the aim of developing solar energy conversion technologies which are expected to create niche markets in the rapidly growing Indian renewable energy sector. The prominent R&D activities at CSEM have encompassed a pilot scale R&D of CIGS modules, development of cost effective CIGS and CZTS thin film solar cells by electrodeposition and ink-based routes and development of DSSC & perovskite-based solar cells. Development of solar receiver tubes comprising indigenous technology of selective absorber and antireflective coatings for low, medium and high temperature concentrated solar power (CSP) applications and development of antireflective and Dust repellent coatings for PV and CSP technologies constitute other major areas of activity. State-of-the-art characterization facilities are also now available at CSEM to assist in accurate measurement of performance and durability of various photovoltaic and solar thermal devices.

CIGS based solar cells possess several commercial advantages over the conventional Si-based solar cells including, low production cost, minimum material requirement, better performance in low-light or diffused light conditions and possibility of making on flexible substrates. Based on the various advantages of the thin film solar panels, many companies are now accelerating their production and installation of this technology. For certain features and given its low cost, thin film solar panels will join crystalline Si panels as a leading solar technology. Thus, these cost-saving alternatives also offer another important advantage as compared to wafer-based modules in that they can be used in a wide range of applications.

Organometal halide (i.e.  $\text{CH}_3\text{NH}_3\text{PbI}_3$ ) perovskites swiftly emerged as optimum light absorbing materials for next-generation sensitized solar cells. High light absorption coefficient and low temperature processing of perovskites offer the potential to realize ultra light and flexible solar panels for a range of practical applications. A maximum energy conversion efficiency of 8.1 % has been demonstrated in ambient atmospheric processed laboratory scale perovskite solar cells. Current efforts are being focused on Pb-free perovskite material, prototype module fabrication and outdoor durability studies. The major challenge in CSP technology is reducing the cost of solar collectors by employing an economical non evacuated solar receiver design. This demands development of low-cost solar functional coatings having the desired optical properties as well as very robust weather resistant properties. Considering the vast commercial potential, the centre has also been focusing on developing solar absorber, anti-reflection and self-cleaning coatings using cost effective processes (i.e., chemical, sol-gel, nanoparticle-incorporated precursor sols, nanoparticle suspensions) with demonstration of manufacturing feasibility on a commercially relevant scale.





# Cost Efficient Solar Absorber Coatings for Concentrated Solar Thermal Application

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Solar collectors are important devices for increasing energy efficiency in areas such as hot water, heating systems of buildings and steam generation for various industrial applications and power production. For such applications, the role of solar receiver tubes is important to convert sunlight to thermal energy and subsequently to electrical energy. An effective selective coating is defined as having a high solar absorptance ( $\alpha$ ) in the solar radiation range of  $0.3 \mu\text{m} < \lambda < 2.0 \mu\text{m}$  and low emittance ( $\epsilon$ ) in the heat irradiation range of  $2.0 \mu\text{m} < \lambda < 50 \mu\text{m}$  at the operating temperature of a receiver tube. A solar selective coating, in addition to having high absorptance and low thermal emittance, must be stable at high operating temperatures and resistant to atmospheric corrosion. However most of the current coatings do not have stability against weather and air, corrosion and oxidation are the two main problems when operating the receiver in an open atmosphere. This is mainly due to the inward diffusion of oxygen and subsequent oxidation of metal particles present in the absorber coating.

Conventionally, most of the solar selective coatings reported in earlier studies have been deposited using Physical Vapor Deposition (PVD) routes. Among the many available PVD routes, use of DC and RF magnetron sputter depositions for solar selective coatings have been widely reported. Although sputtering process has been preferred by virtue of its better productivity, it is known to be a very expensive process.

In contrast to the above expensive techniques, coating by an economic process (electrodeposition or chemical oxidation or sol-gel route) with a combination of

high selective optical properties together with other functional properties like good mechanical, thermal, weather and corrosion stabilities would be a desirable choice for economic power generation by a concentrated solar power (CSP) system.

After an extensive study and research of selective coatings, made under such situation, we found a novel approach involving the combination of chemical and sol-gel methods which can be used for developing highly efficient selective absorber coatings and having high solar absorptance, low thermal emissivity and high stability to all sort of environments.

In the present study, a tandem stack of two layers of Fe-Cr-Mn oxides and  $\text{SiO}_2\text{-ZrO}_2$  has been developed by a combination of chemical and sol-gel process over an economical variety of SS tube (SS-J4). Initially, a thin nanoporous composite oxide absorbing layer was developed on smooth highly specular reflecting SS tube by controlled chemical oxidation process. Following the development of nanoporous absorber layer, an optical enhancing layer (antireflective layer) comprising of  $\text{SiO}_2\text{-ZrO}_2$  nanocomposite having a high uniformity was deposited over on the absorber layer by sol-gel dip coating process and cured at  $300^\circ\text{C}$  for 1h. After curing, the tandem stack turned to dark blue colour (Figure 1). It showed efficient solar selective absorption with low thermal emissivity and high corrosion resistance in an open air atmosphere which is required very importantly for a solar energy collector suitable to use in concentrated solar thermal application.

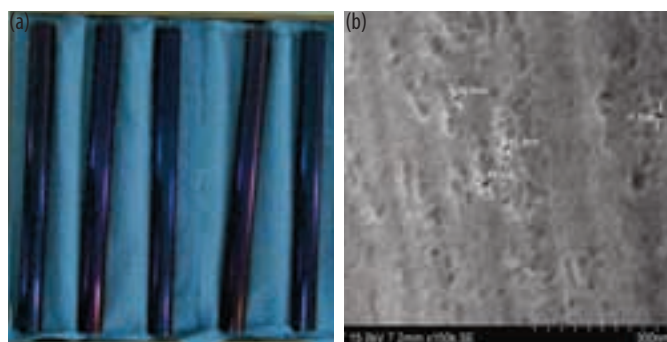


Fig.1 Photograph and FE-SEM image of the absorber tube developed by a combination of controlled chemical oxidation and sol-gel dip coating process revealing presence of nanoporous surface morphology comprising of Fe-Cr-Mn composite oxides, and covered by  $\text{ZrO}_2\text{-SiO}_2$  optical enhancing layer

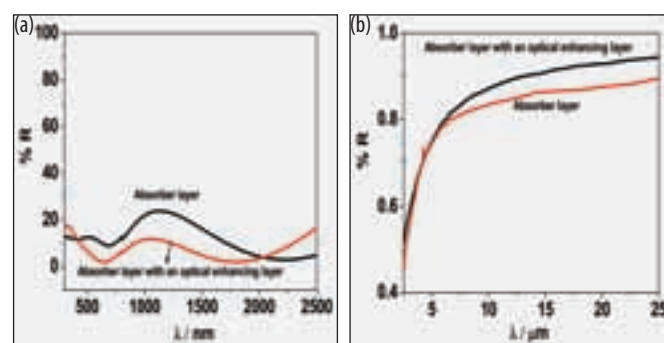


Fig. 2 (a) Reflectance spectra and (b) spectral emittance characteristic of the absorber layer with and without optical enhancing layer

Contributors: V Premkumar, T Vijayaraghavan and S Viswanathan



# Effect of Tri-sodium Citrate (TSC) on the Morphology and Stoichiometry of Pulse Electrodeposited CIGS Thin-Films

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Chalcopyrite  $\text{Cu}(\text{In,Ga})\text{Se}_2$  (CIGS) thin-film technology has already witnessed high conversion efficiencies due to its suitable bandgap ( $\approx 1.20$  eV) and large optical absorption coefficient ( $\approx 105 \text{ cm}^{-1}$ ). One of the major concerns to realize the commercialization of high efficiency CIGS technology is to fabricate the CIGS absorber layer by low-cost methods. Electrodeposition has been demonstrated to produce CIGS devices with high efficiency and it is easily amenable for achieving large area films of high quality with efficient material utilization and high rate deposition. In this context, our group has explored the solution based electrodeposition technique and successfully demonstrated simplified novel pulse electrodeposition process involving a two electrode system, for the fabrication of high quality CIGS thin-films by avoiding the approach of multi-step deposition, which also includes the conventional selenization step. However, it is well-known that In and Se are least abundant elements among Cu, In, Ga and Se and the frequent usage of In in the growing electronic and optoelectronic industries in the form of materials such as indium doped tin oxide (ITO), CIS, CIGS, InP, InN, InGaAs, InAlAs, etc. makes it one of the most scarce elements in the near future. Hence, it would be meaningful to use minimum In precursor and explore ways to minimize it. One of the convenient ways to minimize In precursor is the use of complexing agents such as tri-sodium citrate (TSC).

In the present work, TSC is used as the complexing agent during the pulse electrodeposition of CIGS films. In order to achieve the desired composition of CIGS, the concentration of TSC needs to be optimized. The concentration of TSC is varied from 40 to 120 mM in the present study and stoichiometric CIGS films are obtained for a TSC concentration of 100 mM. Figure 1 shows the SEM micrograph, XRD pattern and Tauc's plot of annealed CIGS film deposited with 100 mM of TSC. The SEM micrograph of CIGS film shows a compact morphology with coarse particles. XRD pattern reveals the formation of chalcopyrite CIGS without the presence of undesired secondary phases. Raman spectral studies further support the XRD results with the detection of  $A_1$  and  $B_2/E$  modes of chalcopyrite CIGS at 176 and 234  $\text{cm}^{-1}$ , respectively. The phase analysis reveals the formation of phase-pure CIGS without any secondary phases. In addition, the film exhibits a desired bandgap of  $\approx 1.28$  eV from optical absorption studies. Ultimately, high quality CIGS films are fabricated by the minimal use of In precursor by optimizing the concentration of tri-sodium citrate during the pulse electrodeposition of CIGS films.

The citrate ions form a complex with the copper ions in the solution, which narrows down the range of reduction potentials of Cu, In and Ga which in turn facilitates the minimization of In precursor.

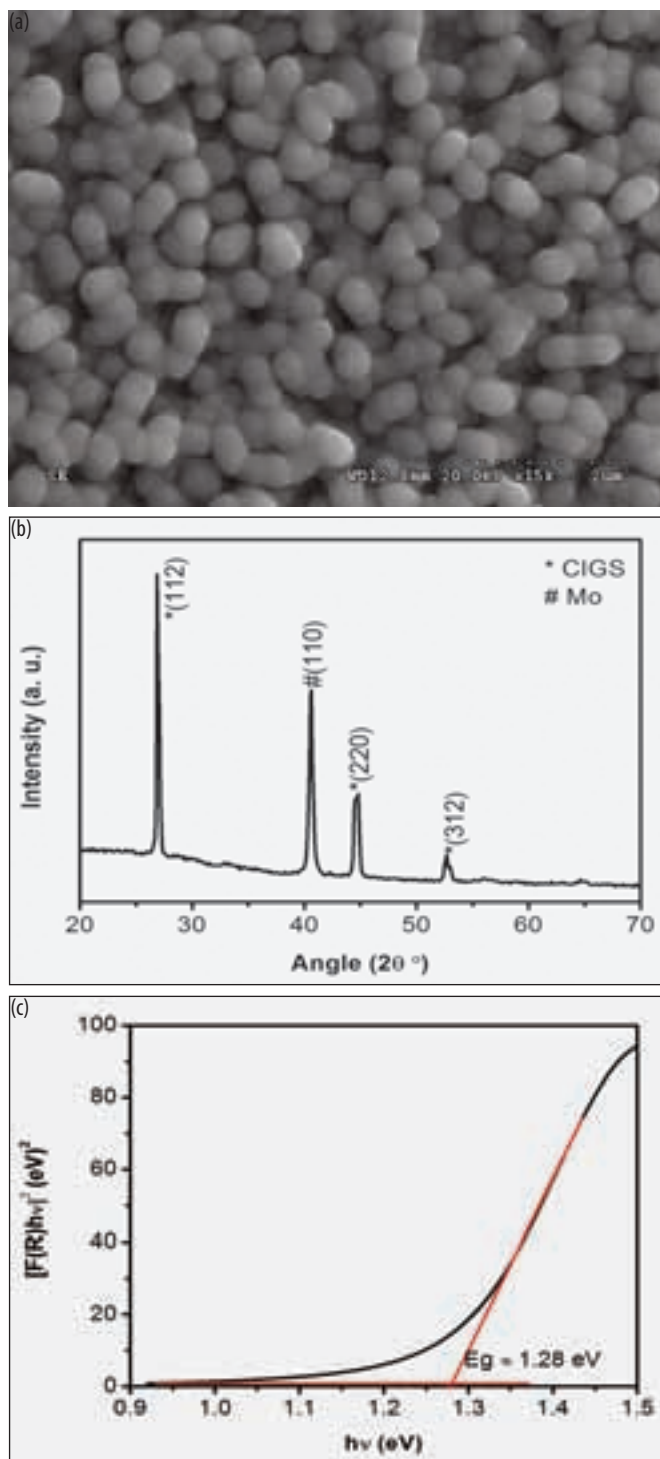


Fig. 1 (a) SEM micrograph (b) XRD pattern and (c) Tauc's plot of Pulse-electrodeposited and annealed CIGS thin-film deposited using 100 mM of tri-sodium citrate

Contributor: Sreekanth Mandati

# CIGS Thin Film Absorber on Willow Flex Glass by Non-Vacuum Ink Based Route

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Cu(In,Ga)Se<sub>2</sub> or CIGS is a promising candidate among thin film solar cell technology as a tunable direct band gap material with high absorption coefficient and high thermal chemical stability. CIGS solar cells has exceeded efficiency of 20% with absorber layer deposited by expensive vacuum based deposition methods on rigid Soda lime glass substrates. CIGS absorber layer deposition has been explored by solution based approaches like spin coating, doctor blade and ink jet printing consisting of two important steps of ink preparation and annealing/sintering post-treatment to make it commercially competitive. ARCI has successfully attempted CIGS thin film absorber deposition on willow flex glass substrates from Corning (Figure 1) by non-vacuum ink based route followed by intense pulsed light post-treatment.

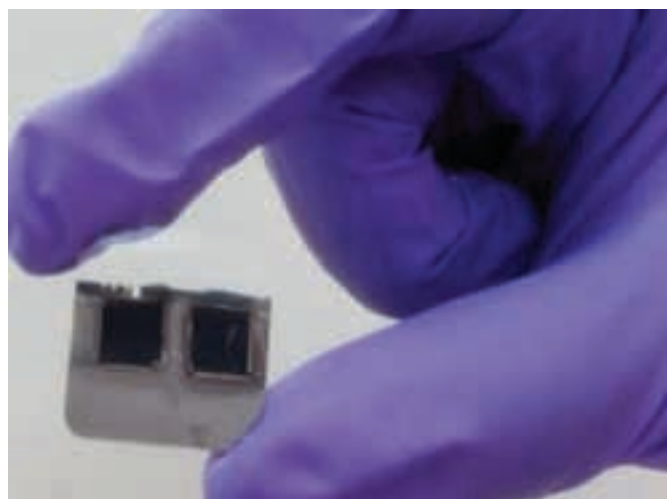


Fig. 1 CIGS thin film absorber on willow flex glass substrates from Corning obtained by IPL treatment

Ink has been prepared from appropriate blend of CIG metallic alloy nanoparticles (50 nm) and Se nanoparticle (80 nm) with Polyethylene glycol binder to adjust its rheology and make it suitable for doctor blade deposition on 8 mm x 8 mm Mo coated Flex glass substrate area. The doctor bladed ink containing CIG and Se was post treated by intense pulsed light (IPL). IPL treatment parameters such as power, pulse durations, delay and number of pulses are optimized to obtain uniform treatment on full sample area. The CIGS thin films obtained were characterized for thickness, composition, phase and surface characteristics.

CIGS thin film thickness uniformity and constituent elemental ratio were confirmed by XRF analysis over entire

coated area. The XRD pattern of CIGS thin films obtained by IPL post treatment using different filters is shown in Figure 2. Intense peaks corresponding to CIGS phase are in good agreement with reference pattern (JCPDS No 35-1102) for CuIn<sub>0.7</sub>Ga<sub>0.3</sub>Se<sub>2</sub>. The other phases present are unreacted CIG and Se of which phase fraction is reduced when fluence is increased to 225 J/cm<sup>2</sup>. Single set of IPL treatment was optimized to convert 90% of CIG into chalcopyrite CIGS phase using various light filters. The phase fraction and other properties of the obtained post IPL treated CIGS thin films are listed in Table 1. During the IPL post treatment formation of MoSe<sub>2</sub> at CIGS-Mo interface, which forms back surface field and is responsible for adhesion of CIGS thin film, is confirmed from XRD pattern. Surface morphology image of all sample revealed crystallization of constituent ink precursors to form dense CIGS thin film absorber. Fig. 3 shows a representative FESEM image of the obtained CIGS thin film after IPL post treatment.

Structural and morphological characterization of obtained films has confirmed the device quality of thin film absorber. Complete device fabrication (AZO/ZnO/CdS/CIGS/Mo/Willow flex Glass) using CdS deposition by chemical bath and ZnO-Al:ZnO front contact by sputtering is underway.

Table 1 Properties and phase fraction of IPL post treated CIGS thin film obtained using different filters

| IPL Filter | Fluence J/cm <sup>2</sup> | Thickness of CIGS (μm) | Crystallite Size of CIGS (nm) | Phase fraction |        |
|------------|---------------------------|------------------------|-------------------------------|----------------|--------|
|            |                           |                        |                               | CIG %          | CIGS % |
| Red        | 184                       | 4.48                   | 24.14                         | 39.74          | 60.26  |
| Yellow     | 205                       | 4.25                   | 32.66                         | 11             | 89     |
| Green      | 225                       | 3.68                   | 33.33                         | 8.42           | 91.58  |
| Blue       | 246                       | 3.82                   | 27.12                         | 8.58           | 91.42  |

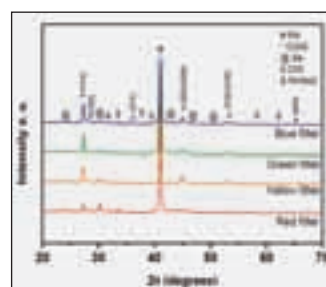


Fig. 2 XRD patterns of CIGS thin film absorber obtained by IPL post treatment using various filters



Fig. 3 Representative surface morphology FESEM image of CIGS obtained by IPL post treatment using green filter

Contributors: Amol C Badgujar and K Madhuri



# Design and Prototype of Sensitized Solar Cell Module

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Solar cells based on panchromatic dye-sensitized metal oxide photoanode and redox electrolyte are emerging as a cost-efficient way of solar energy to electric energy conversion. State of the art laboratory scale (active area: <math>< 1 \text{ cm}^2</math>) dye-sensitized solar cells (DSSCs) have been reported to exhibit around 13% power conversion efficiency under standard test condition (Air Mass 1.5G,  $100 \text{ mW}\cdot\text{cm}^{-2}$ ). The discovery of direct bandgap, high molar extinction coefficient perovskite-type light absorber (i.e.,  $\text{CH}_3\text{NH}_3\text{PbI}_3$ ) further enhanced the efficiency of sensitized solar cells to 19%, a value comparable to that of conventional silicon photovoltaics. Hence, the latest research is focusing on scale up of sensitized solar cell technology for practical applications. Figure 1 shows the design and prototyping of mesoporous  $\text{TiO}_2$  photoanode by high throughput screen printing technique. Ag metal grids were embedded on 50 mm x 50 mm FTO glass substrate to minimize the resistive losses and improve the charge carrier collection. Crack-free  $\text{TiO}_2$  layers with uniform thickness were printed on Ag grid/FTO glass substrate by using a paste made up of in-house synthesized anatase  $\text{TiO}_2$  nanopowders.

A prototype DSSC module was prepared by assembling a dye-sensitized  $\text{TiO}_2$  photoanode with a platinum counter electrode. The inter electrode space was filled with iodine/tri-iodide redox couple electrolyte. The module produced a peak power ( $P_{\text{max}}$ ) of 72 mW under standard test condition and was able to power the 50 mW electric fan under natural sunlight illumination (Fig.2). Comprehensive photovoltaic parameters of the module are shown and compared with a typical lab-scale device in Table 1. Prototype module exhibits analogous open-circuit voltage ( $V_{\text{oc}}$ ) and fill factor (FF) to those of lab-scale device. However, the photocurrent density decreased by a factor of 1.5 and leads to similar decrease in power conversion efficiency. Efforts are now underway to improve the efficiency and operational stability of module through optimum grid/ $\text{TiO}_2$  design, utilization of perovskite absorber and solid state hole transporting material.

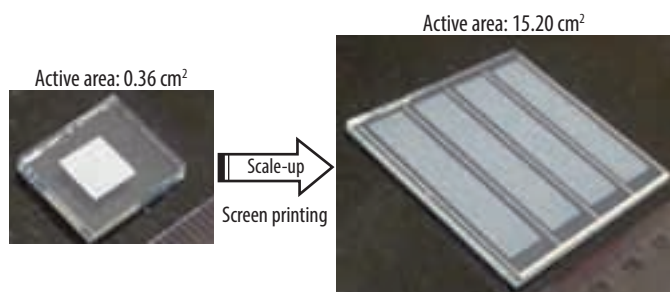


Fig.1 Scale-up of mesoporous  $\text{TiO}_2$  photoanode by screen printing method

Table 1 Photovoltaic parameters of DSSCs measured under standard test condition

| Parameters \ Device           | Lab-scale DSSC | Prototype module |
|-------------------------------|----------------|------------------|
| Active area ( $\text{cm}^2$ ) | 0.36           | 15.20            |
| $V_{\text{oc}}$ (V)           | 0.840          | 0.850            |
| $I_{\text{sc}}$ (mA)          | 4.59           | 124.47           |
| Fill factor                   | 0.70           | 0.68             |
| $P_{\text{max}}$ (mW)         | 2.70           | 71.90            |
| Efficiency (%)                | 7.50           | 4.73             |

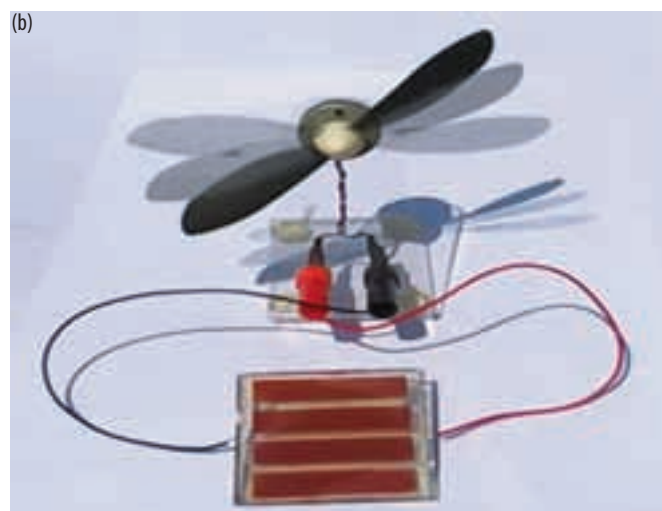
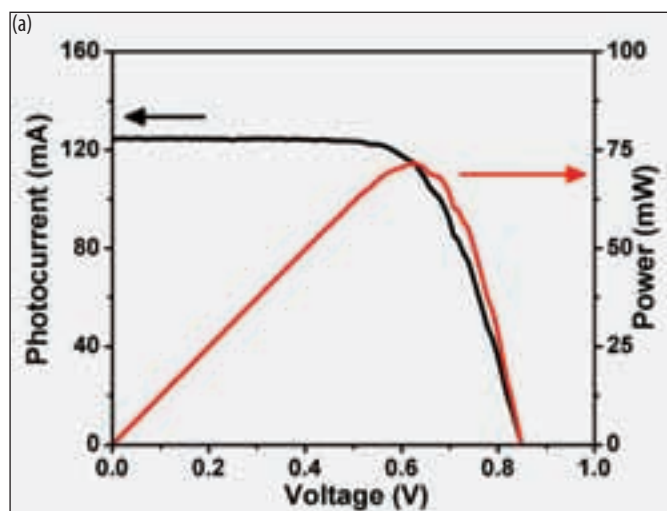


Fig. 2 Photocurrent-voltage power characteristics curve (a) and load test demonstration (b) of prototype DSSC module

Contributors: S Sakhivel and Nanaji Islavath

# CdS Thin Films on 300 mm x 300 mm Glass Substrate by Chemical Bath Deposition

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Chemical bath deposited (CBD) CdS buffer layer which is widely being used as a successful heterojunction partner to the p-type absorber layer in CIGS solar cells. At ARCI, a pilot line R&D for making CIGS thin film solar cell has been initiated. CdS films on substrate size of 300 mm x 300 mm can be realised by semi-automated CBD system established in the CIGS pilot line facility at ARCI. Thickness uniformity and transmittance are very important parameters to realise good quality device of large size. Series of experiments were carried out to ensure the uniformity throughout the substrate area.

Cd-salt, thiourea, ammonia and DI water were used as starting solution precursors. Three representative samples were taken diagonally from 300 mm x 300 mm substrate for characterization study. Thickness, structural, morphological and optical characterization of films was carried out on these films. X-ray photoelectron spectroscopy was carried out to check the presence of OH or any other oxide related impurities in CdS film. An experiment was carried out to study the effect of air annealing on film properties.

CdS film on 300 mm x 300 mm substrate found to be uniform and yellow in colour. The XRF average thickness is in the range 55 to 60 nm. Table 1 shows the characterization results of three representative samples. FESEM measurements reveal that surface is totally covered by the grains of sizes ranging from 25 to 50 nm.

Table 1 Characterization results of representative samples of 300 mm x 300 mm CdS film

| S.No.  | Thickness (nm) |              | Structure (XRD) | Transmittance (T%) | Band gap (eV) |
|--------|----------------|--------------|-----------------|--------------------|---------------|
|        | XRF            | profilometer |                 |                    |               |
| CdS_1a | 55             | 58           | Cubic/Hexagonal | 68                 | 2.50          |
| CdS_2b | 60             | 52           | Cubic/Hexagonal | 69                 | 2.52          |
| CdS_3c | 58             | 53           | Cubic/Hexagonal | 58                 | 2.46          |

Figure 1 & 2 shows the X-ray photoelectron spectrum of CdS film. An extra peak at 168.29 eV is observed in sulphur narrow scan which is attributed to sulphate ion. Along with carbon peak, an additional peak at 288.32 eV is seen which is due to the formation of carbonate ion. Efforts are underway to eliminate the formation of contaminants.

After annealing there is a slight improvement in crystallinity, and GIXRD (Figure 3) exhibits that films contain peaks related to cubic and hexagonal. The optical band gap value is found to have decreased from 2.46 to 2.2 eV, which might be due to the transformation of Cubic phase to hexagonal phase upon

reorganization and densification of crystallinity and annealing temperature at 300°C.

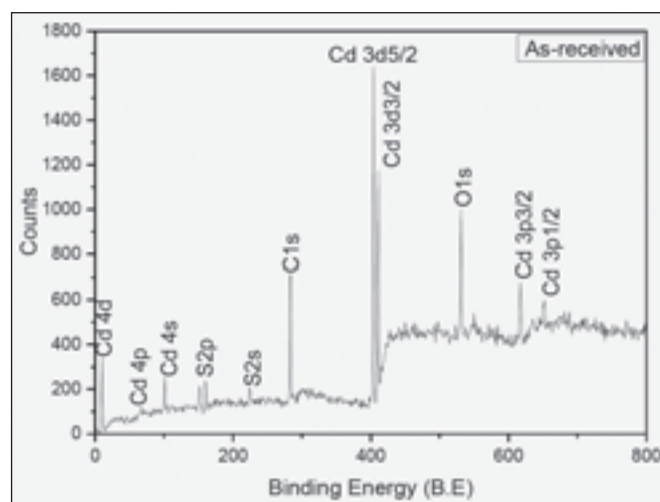


Fig.1 XPS As-received spectrum of CdS film

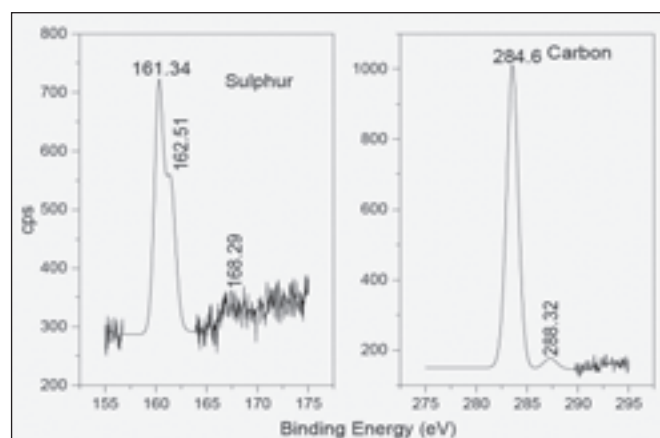


Fig.2 Narrow scan of sulphur and carbon peaks

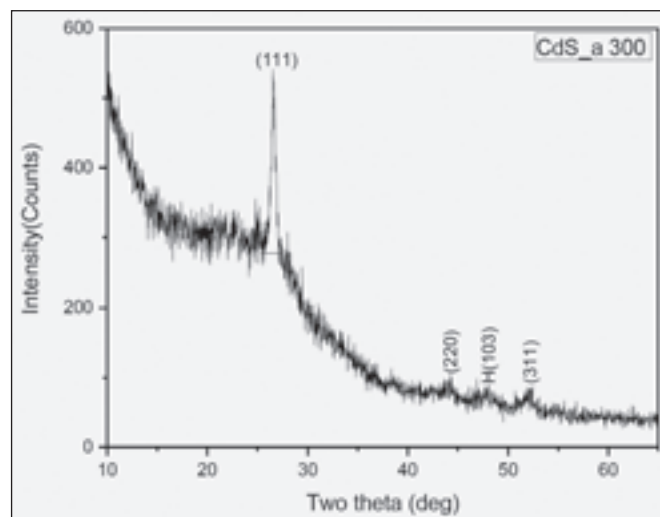


Fig.3 GIXRD pattern of annealed CdS film

Contributor: Sanjay R Dhage



# Bilayer Molybdenum Back Contact on 300 mm x 300 mm Area for CIGS Thin Film Solar Cell Application

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Cu(In,Ga)Se<sub>2</sub> or CIGS absorber material is popular among thin film solar cell technology as tuneable direct band material with high absorption coefficient, thermal and chemical stability, leading with photo-conversion efficiency exceeding more than 20% for laboratory scale area. Molybdenum (Mo) is preferred as back contact for CIGS thin film solar cells over other materials such as Pt, Al, Ni, Ag, Cu as it has excellent properties like high conductivity, chemical stability with CIGS precursor materials at elevated temperature conditions, matching thermal coefficient with Soda lime glass substrate and CIGS. Mo also makes strong ohmic contact with CIGS in the form of MoSe<sub>2</sub> at CIGS –Mo interface. One of the challenge in up scaling of CIGS solar cell technology is uniform Mo back contact since conductive, crystalline with preferred orientation (110), stress free, well adhered Mo thin film is desired for highly efficient CIGS solar cell over large area.

Rotating Cylindrical DC magnetron Sputter coater is being used to sputter bilayer Mo thin film comprised of Seed layer (50 nm) and bulk layer (400 nm) on pre-cleaned soda lime glass substrate over the area of 300 mm x 300 mm. Seed layer has been sputtered at high sputtering pressure and low power to obtain well adhered, dense Mo thin film. Sputtering parameters such as power and Ar gas flow rate are optimized to coat conductive, stress free, crystalline bulk layer on seed layer.

Thickness mapping of the bilayer Mo thin film as seen from Figure 1 carried out with XRF has revealed high degree thickness uniformity with maximum standard deviation of 3.16% which is also verified with optical profilometry. X-ray diffraction studies revealed highly crystalline Mo thin films with preferred orientation of (110). Average sheet resistance of 0.3 Ω/□ measured by four probe technique over entire area with variation of less than 5%. Variation of sheet resistance with sputtering conditions is shown in Figure 2. Sputtering parameters were also optimized for minimum mechanical stress on Mo thin film measured by Stress Analyser. Since all films comprised of seed layers, the films showed excellent adhesion to soda lime glass. Morphological characteristics of Mo thin film is shown in Figure 3 which reveals dense dead fish like grains structure on surface and desired columnar growth from cross section.

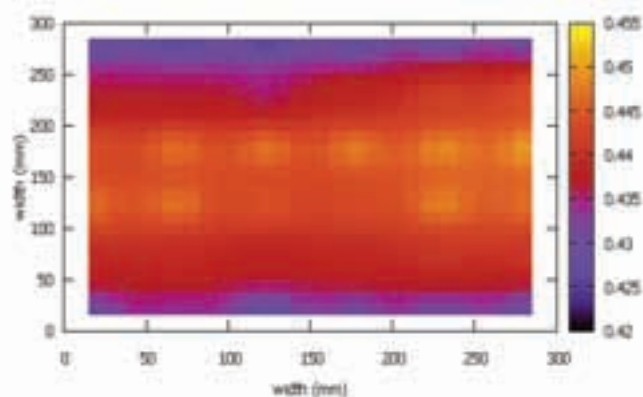


Fig. 1 Representative thickness map of a Mo bilayer thin film on 300 mm x 300 mm glass substrate

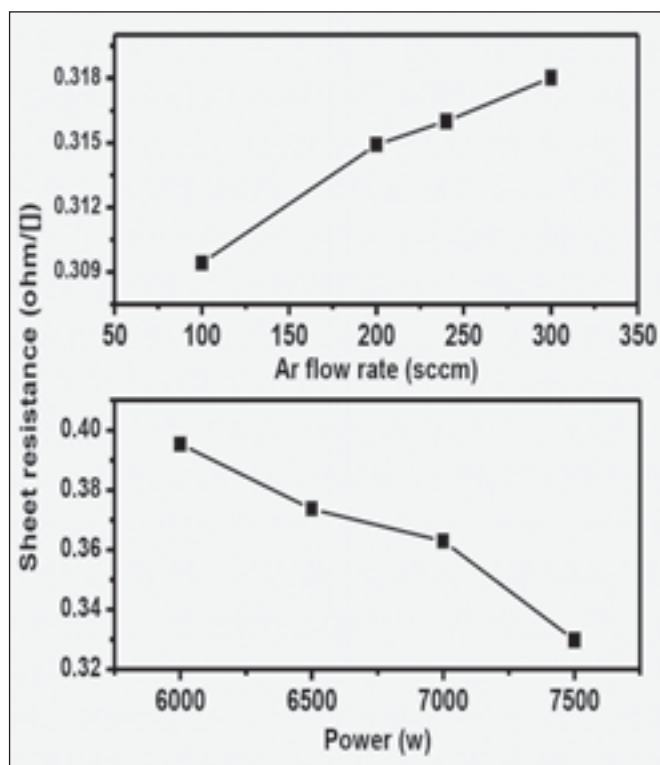


Fig. 2 Variation in sheet resistance with sputtering power and Ar gas flow rate for Mo bilayer thin films

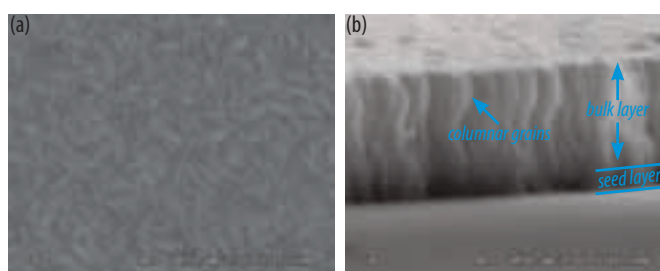


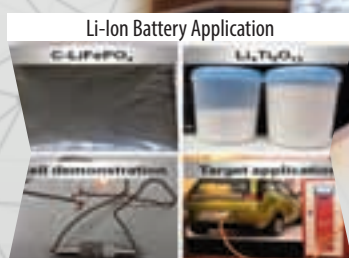
Fig. 3 Representative FESEM (a) surface and (b) cross-sectional image of a Mo bilayer thin film

Contributor: Sanjay R Dhage

# Centre for Nanomaterials

The Centre for Nanomaterials, established at ARCI a decade ago has quickly become visible in the country due to its proactive role in developing nanomaterials based technologies up to prototype or pilot level and transferring them to industries. There are in-house as well as sponsored application oriented projects which are efficiently handled, and a few technologies are already transferred to industries. Noteworthy is the recent technology transfer to a textile related chemical company, which received the technology related to photocatalytic self-cleaning  $\text{TiO}_2$  textiles. The company is planning to launch its first product into market in near future. Apart from water and textile related technologies, the Centre gives high priority to energy and automotive related technologies. These research areas include Li-ion batteries for electric vehicle (EV) applications, ODS steels for high temperature strength, aerogels for thermal insulation, high temperature lubricants, solar hydrogen materials and lead free bimetal bearings for automotive industry.

The Centre for Nanomaterials consists of a team of scientists from various science and engineering backgrounds, which is crucial for technology development. A strong technical team supports these activities. The real workforce to achieve the targets of the projects is the group of students who work on these topics as a part of their graduation projects. While the fundamental work is done by the students, the application research and the scale-up synthesis, prototype demonstration are done by the scientists with the help of technical team. The cost of production, scalability, worker and user friendliness, and the environmental impact are the key factors that are taken into consideration during the technology development. Some of the topics like water purification are taken up as in-house projects as they are more of societal relevance, and if the technology is successfully demonstrated, it is usually transferred to a suitable company or an NGO at free or at a nominal cost.



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# High Performance Lithium-Sulfur Batteries with Novel Cell Configurations

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The rechargeable lithium-ion battery technology has been proven to be one of the potent energy storage system in advance electronic devices and upcoming electric vehicle technology. But, the large scale commercialization of lithium-ion batteries is greatly hampered due to the use of highly expensive electrode materials. Moreover, the specific capacity and energy density delivered by present day lithium-ion battery are not high enough, which is why the battery driven vehicles are still not fully commercialized. One of the alternative energy storage systems to lithium-ion battery is lithium-sulfur battery, which works based on conversion chemistry ( $16\text{Li} + \text{S}_8 \leftrightarrow 8\text{Li}_2\text{S}$ ). The sulfur cathode has high theoretical capacity of 1672 mAh/g and high energy density of 2600 Wh/Kg, which is about 5 times higher than the conventional intercalation systems. Additionally, sulfur is inexpensive, non-toxic and naturally abundant, a common by-product of petroleum industry.

Despite its promising benefits, the realization of lithium-sulfur battery technology is greatly hindered due to reasons, such as polysulfide dissolution in the electrolyte leading to loss of active material, insulating nature of sulfur and its discharge products ( $\text{Li}_2\text{S}_2$ ,  $\text{Li}_2\text{S}$ ). This finally leads to severe capacity fading and low cycle performance of the cell. To mitigate these issues, many researchers have implemented various designs electrode designs on sulfur cathode side and demonstrated improved capacity and stability. The use of lithium metal as high capacity reference electrode in the lithium-sulfur cells is another limitation due to aggressive lithium chemistry. High capacity materials such as silicon and tin are best suitable anode for this high capacity sulfur cathode to form full cell from the practical point of view.

At ARCI, we are in the process of developing a simple and cost effective method to fabricate sandwich-type electrodes for lithium-sulfur battery application. This involves the use of protective carbon layer on the sulfur electrode that allows Li-ion diffusion through it, which prevents direct dissolution of lithium polysulfides into electrolyte. We have prepared sandwich-type sulfur cathode that has been combined with high capacity silicon anode to form full lithium-sulfur battery (Figure 1). The sulfur cathode has been sandwiched between two highly conductive carbon membranes. The use of sandwich-type electrode has an advantage that traps the dissolved lithium polysulfides in it and also increases the electrical

conductivity during charge-discharge process. For full cell fabrication a lithium-silicon-carbon composite anode was used, which acts as source of lithium ions.

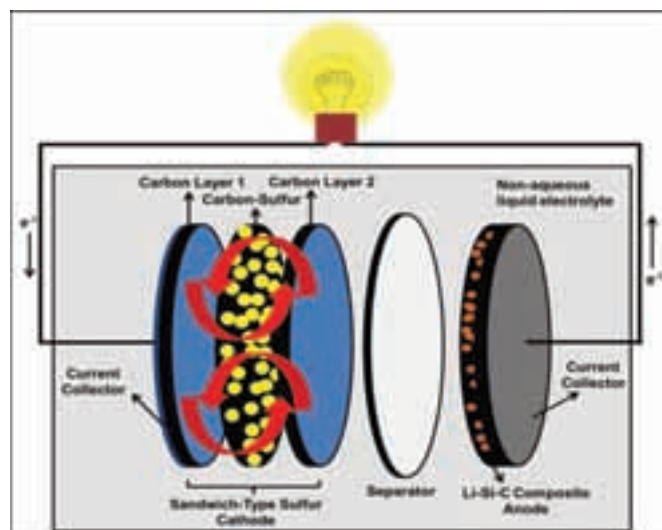


Fig. 1 Schematic representation for the fabrication full lithium-sulfur battery

The fabricated lithium-sulfur cell delivers high specific capacity up to 70 charge-discharge cycles (Figure 2), which is 3-4 times higher than conventionally used lithium-ion battery. We have been further focusing on improving the specific capacity and long term cycle performance of the cells by optimising both electrode designs and also studying the effect of electrolyte composition and their additives.

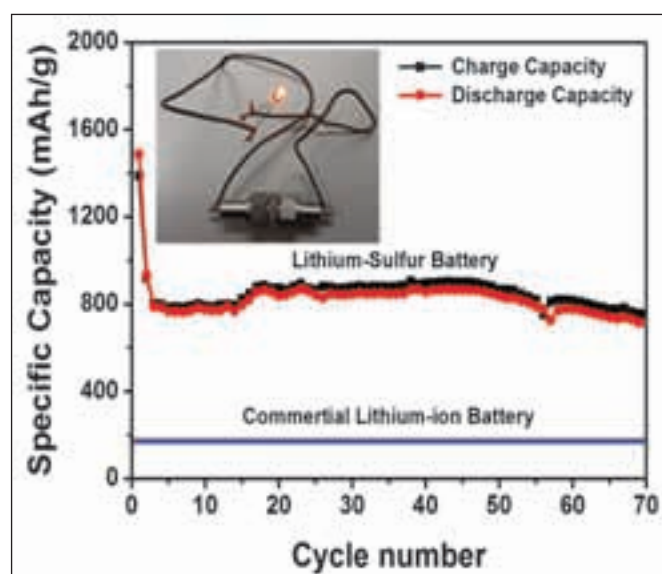


Fig. 2 Cycle performance of full lithium-sulfur battery

Contributor: E Hari Mohan

# Development of Lead free Copper Alloys for Bimetal Bearings

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The properties required for half bearings, piston pin bushes and cam bushes of internal combustion engines are moderate strength (110 MPa), wear and seizure resistance. Copper-tin alloys (bronze) containing lead are being widely used for the above applications. The presence of tin in copper alloy improves the strength, and lead improves wear and seizure resistance. The new environmental regulations demand that lead should not be used in these materials and hence there is a need for development of "Lead free copper alloys" for the above applications.

The following factors were considered for the design of new material. Sn should be in the range of 4-12%. Below 4% Sn, the alloy has poor strength and durability. Above 12% Sn, the alloy becomes brittle due to the formation of intermetallics. Bi is a soft phase and remains predominantly in elemental form. Bi is known to promote Sn migration from matrix to the surface thereby enhancing anti-seizure characteristics. Below 3% Bi, the alloy has poor seizure resistance. Above 5% Bi, the islands of Bi become coarse and effect hardness and durability. Nickel is a solid solution strengthener usually present in the range of 1-10%. The strength and corrosion resistance increase at about 1%. Other wear modification agents like graphite are to be added if nickel is more than 1%. Phosphorous is added in the range of 0.03 to 0.35%. P is an excellent deoxidiser and also strengthens matrix. It enhances sintering characteristics and reduces swelling tendency due to pore formation during sintering. The draw back of P is that it reduces the bond strength of copper alloy with steel.

Based on the above mentioned factors, two compositions i.e. Cu-8Sn-4Bi-0.05P (BMC840) and Cu-8Sn-4Bi-1Ni (BMC841) were selected for the study. The process for making bimetallic bearings involves loose powder sintering on steel plate, cold rolling, annealing and finish rolling. All the process conditions like sintering and annealing temperatures, initial and final cold work were optimised. Bimetal strips were produced using the optimised parameters, and microstructural and mechanical properties of these bimetal strips were evaluated. The microstructure of bimetal strips made from both the compositions indicates good bonding between the copper alloy and steel interface, good density (99%) and uniformly distributed fine lead islands. A typical microstructure of BMC841 bimetal strip is shown in Figure 1.

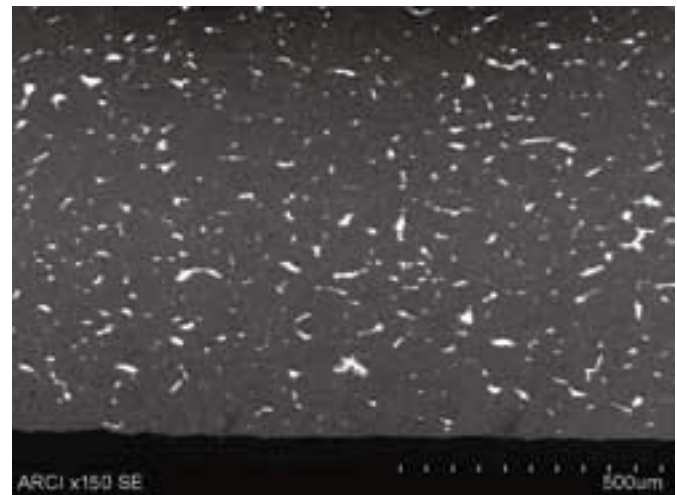


Fig. 1 Microstructure of BMC841 bimetal strip

Both hardness and tensile properties of BMC841 strip are higher when compared to BMC840 (Table 1).

Table 1 Mechanical properties of bimetal strips

| S.No. | Property                        | BMC840 | BMC841 |
|-------|---------------------------------|--------|--------|
| 1.    | Hardness [HVN]                  | 98     | 127    |
| 2.    | Yield Strength [Mpa]            | 451    | 470    |
| 3.    | Ultimate Tensile Strength [MPa] | 469    | 517    |

Both compositions exhibited moderate wear resistance, and both wear rate and coefficient of friction (COF) of BMC841 are lower when compared to BMC840 (Figure 2). The higher mechanical properties and wear resistance exhibited by BMC841 is due to the presence of nickel.

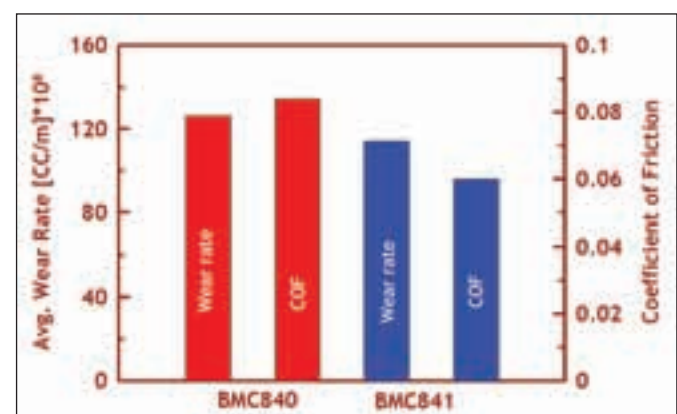


Fig. 2 Wear properties of bimetal strips

Field trials are being carried out by M/s. Bimetal Bearings, Chennai.

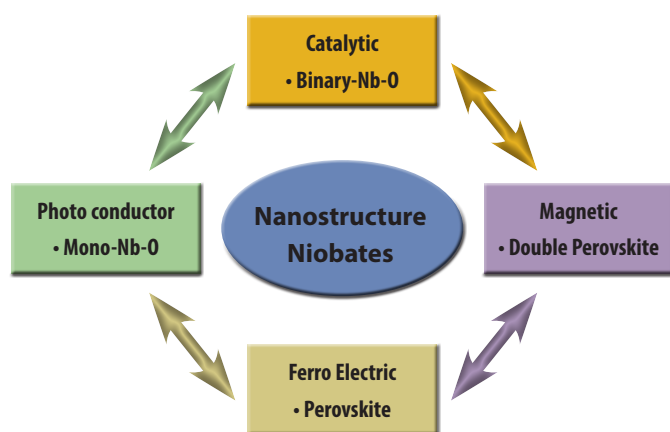
Contributors: P Suresh Babu, D Sen and A Venugopal Reddy

# Nano-Pyramid Niobate Films for Opto-/Electronic Energy Applications

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Nanostructured films of niobates ( $M_1M_2O_x$ ) find potential applications in energy generation and other related industries. Any efforts in depositing such films, which show improved performance and easy processability, would be desirable for their commercial acceptability. In one such effort, ARCI has demonstrated an economic spraying method to deposit the niobate films under moderate temperatures. The method deploys a low-cost precursor and has capability to deposit large area 50-300x50-300mm<sup>2</sup> nanostructured film on even glass substrate. Especially, the most complex configuration of double-perovskite ferroelectric material system viz.  $Sr_2FeNbO_6$  (SFNO), which is a potential visible light photocatalyst and solid oxide fuel cell material, has also been deposited.



It was shown that a nanostructure double-perovskite niobate film deposited by a simple, economic hybrid method has been used for hydrogen generating photo-electro chemical (PEC) cell. It is needless to say that hydrogen ( $H_2$ ) energy generated from the renewable sources has stimulated enormous R&D efforts worldwide.  $H_2$ -technology has been acknowledged for its potential applicability in energy & automotive sector. For solar assisted PEC  $H_2$  generation, there is need to identify simple process ability for an eco-friendly material. Present efforts are utmost important to realize a commercially viable solar  $H_2$ -energy technology.

In Figure 1, doped SFNO films deposited by hybrid technique for different dopant concentration are presented (a)  $x=0.1$  (b)  $x=0.2$  and (c)  $x=0.3$  (d)  $x=0.4$  deposited films. Titanium doping improves the conductivity that facilitates the enhanced transport properties of the films. The pyramidal faceted nan-grains assist in rendering the catalytic sites during the PEC water-splitting reaction.

Figure 2 shows the x-ray diffraction pattern of the various doped SFNO films. The doping does not induce the structural distortion of original double perovskite structure of the film that is deposited in transparent conducting glass. The substrate peak can be seen in the background.

We deposited various niobate films (>100-300mm area). This technique is well suited to deposit various types of  $M_1M_2O_x$  films for several applications in ferroelectric memories, super-capacitors, photoconductors, holographic-detectors and batteries etc. The economic nature of this methodology indicates its potential commercial viability in various industries.

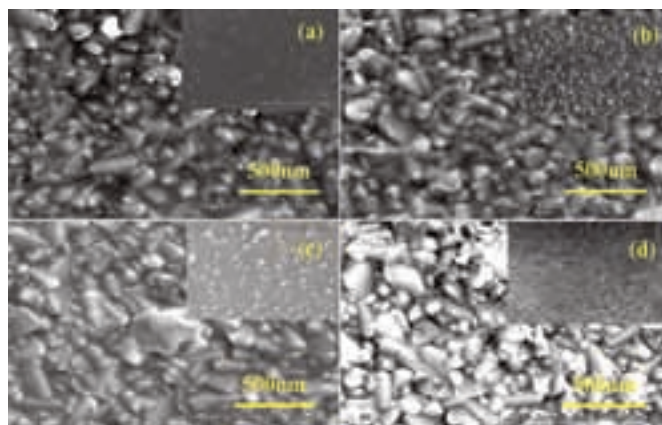


Fig. 1 FESEM image of doped SFNO films deposited for x (a)  $x=0.1$  (b)  $x=0.2$  and (c)  $x=0.3$  (d)  $x=0.4$

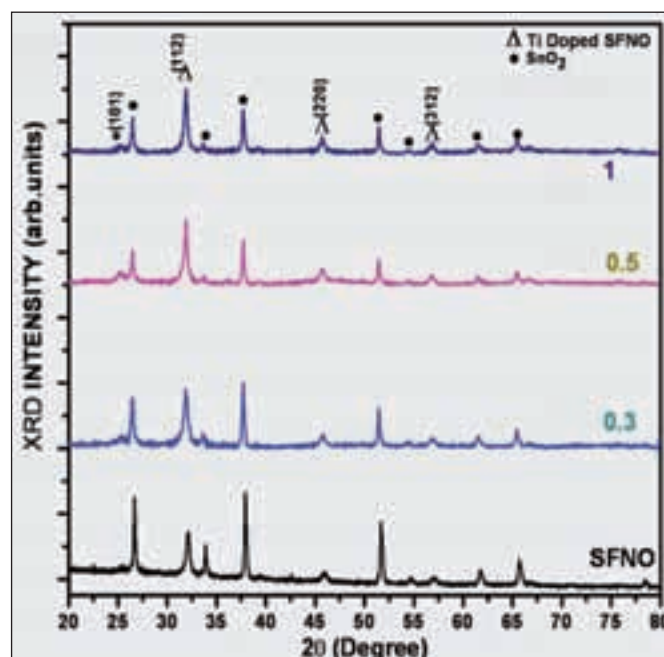


Fig. 2 XRD patterns of doped SFNO films deposited for x (a)  $x=0.1$  (b)  $x=0.2$  and (c)  $x=0.3$  (d)  $x=0.4$



# Silica Coated Fe-based Soft Magnetic Materials for AC Applications through Powder Metallurgy

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Soft magnetic alloys play a key role in both power generation and conversion of electrical energy. With the new paradigm emphasizing greater reliance on energy efficient renewable sources, the research is increasingly directed towards energy-related technologies. Thus, efforts are on to introduce soft magnetic materials (SMMs) in niche areas like electric and hybrid vehicles and improve the performance of the existing soft magnetic materials in electrical machines. Further, for minimizing electrical losses in generation, transmission and distribution of electricity from smart-grids operating under high voltage direct current (HVDC), advanced SMMs are being used for both DC and AC transmission. SMMs range from pure Fe for powder core inductor appliances to Fe-Si laminates, amorphous Fe or Fe alloys for transformer applications and soft ferrites for electromagnetic shielding interference (EMI) filters. Thus, advanced SMMs with higher values of saturation magnetization ( $B_s$ ), resistivity ( $\rho$ ) and permeability ( $\mu$ ) combined with lower coercivity ( $H_c$ ) and core loss are generally in demand. Based on specific application, minimum core-loss with high saturation induction ' $B_s$ ' as a function of frequency and applied field induction ' $B$ ' is the most sought after property.

PM processed soft magnetic composites (SMCs) are components consolidated through compaction & curing/sintering of electrically insulated coated magnetic powders. The core powder needs to be magnetically soft and retain its softness in the consolidated compact, normally achieved through optimized heat treatment below the Curie temperature ' $T_c$ '. PM processing reduces the size and weight of the devices by about 50% when operated at increasingly higher frequencies for the same power output. The thin electrical insulation between particles reduces eddy current losses while retaining the ferromagnetic coupling. This attribute provides high saturation magnetization with low loss.

In ARCI, PM technology is being pursued to produce Fe and Fe-P based SMCs for automotive applications. The process

adopted is (i) to coat  $\text{SiO}_2$  on 99.95 % pure Fe powder through solution technique (ii) to use spark plasma sintering to obtain compacts of high densities and (iii) subsequent annealing. Thus, samples of Fe- $\text{SiO}_2$  composites having silica from 0.1 to 2 wt% were obtained. Sinter-densities ranged from 91-98% TD. DC property data of 0.5 wt% silica coated powder composite is presented in this report. SEM with EDAX was used to observe morphology and silica coatings as seen from Figures 1 & 2. Hysteresis loops of the annealed and un-annealed samples SMCs are shown in Figure 3. Further work is under progress to improve density to about 98% and obtain the requisite heat treatment schedule for magnetic annealing of Fe-0.5wt%  $\text{SiO}_2$  soft magnetic composites for getting optimized AC properties.

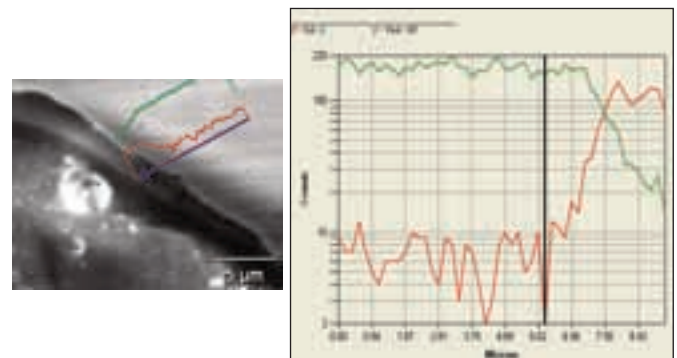


Fig.2 Elemental mapping showing the silica coating on the edges of sintered Fe-0.5wt%  $\text{SiO}_2$

Table 1 DC Properties of Fe-silica composites

| Sample | $\text{SiO}_2$ (wt%) | $B_s$ (kG) | P $\mu\Omega\text{-cm}$ | Br (kG) | $\mu_{\text{max}}$ | $H_c$ (Oe) |
|--------|----------------------|------------|-------------------------|---------|--------------------|------------|
| SMC5   | 0.5                  | 14.64      | 29.8                    | 12.60   | 1197               | 2.74       |

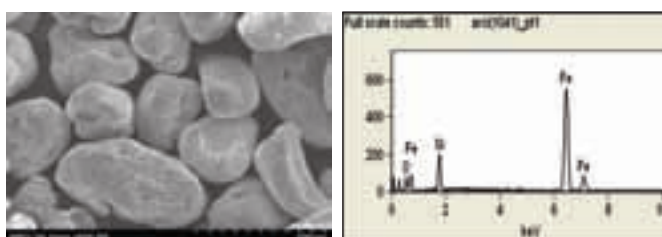


Fig. 1 Morphology of Fe-0.5wt%  $\text{SiO}_2$  and EDAX showing the presence of silica

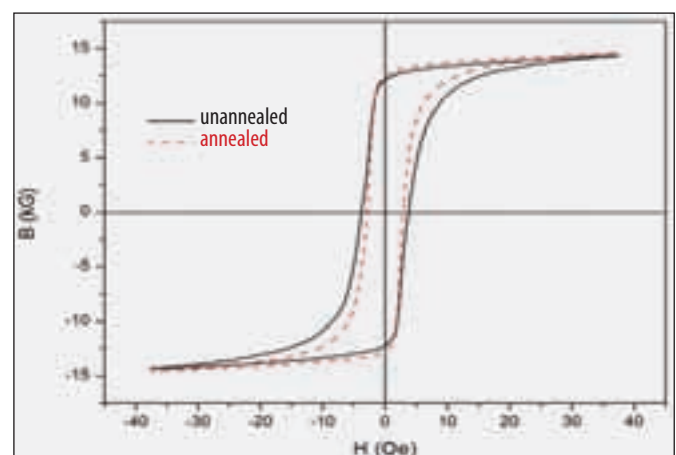


Fig. 3 B-H loop of Fe-0.5wt% silica

Contributors: Aviral Bisht, D Prabhu, Neha Y Hebalkar, V Chandrasekaran and R Gopalan

# Synthesis of Nanostructured Transition Metal Chalcogenides

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Nanostructured transition metal chalcogenides are known to have unique catalytic/electrocatalytic, opto-electronic as well as lubricating properties, which makes them a highly sought after material for wide range of applications in the field of aerospace, automotive, electronic, energy and petroleum/chemical industries. Various forms of nanostructured transition metal chalcogenides have been reported viz. inorganic fullerene (IF), inorganic graphene (IG) and nanosheets (NS). Each of these forms have unique role in imparting specific properties to these chalcogenides, which contributes to their properties.

Synthesis of these nanostructured form of metal chalcogenides, in bulk quantity and reproducible quality, is a challenging task. A novel synthesis route based on a mechanically activated solid-gas reaction under controlled temperature and pressure has been developed at ARCI for the generation of IF, IG and NS form of tungsten ( $WS_2$ ). Nanostructured pure iron ( $FeS_x$ ) and chromium ( $Cr_2S_3$ ,  $Cr_3S_4$ ) sulfides as well as Fe-Cr nanocomposite sulfides have also been successfully synthesized by a similar route.

A custom designed reactor has been designed (Patent pending) to perform such synthesis under controlled temperature and pressure in order to obtain tailor-made

microstructure of the final product. The unique design of the reactor also makes it suitable for processing of any other corrosive materials at elevated temperatures up to  $1000^\circ C$ .

Figure 1(a,b) shows the XRD patterns of pure  $WS_2$  nanosheets and  $FeS-Cr_2S_3-Cr_2FeS_4$  nanocomposite powders as synthesized by the ARCI method. Figure 2 (a, c and d) shows NS, IF and IG structured phases obtained for  $WS_2$  as synthesized by the ARCI method. Conventional commercial  $2H-WS_2$  is shown in Fig. 2b for comparison.

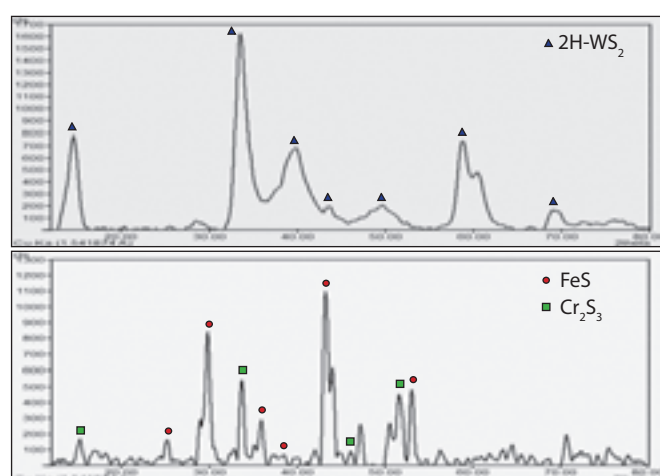


Fig. 1 (a) NS- $WS_2$  and (b) nano- $FeS-Cr_2S_3$  composite powder produced by ARCI method

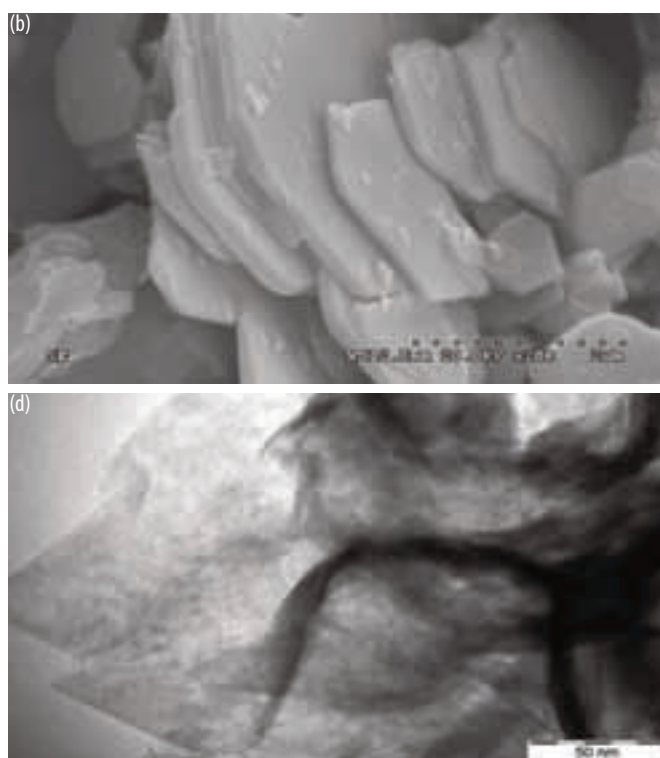
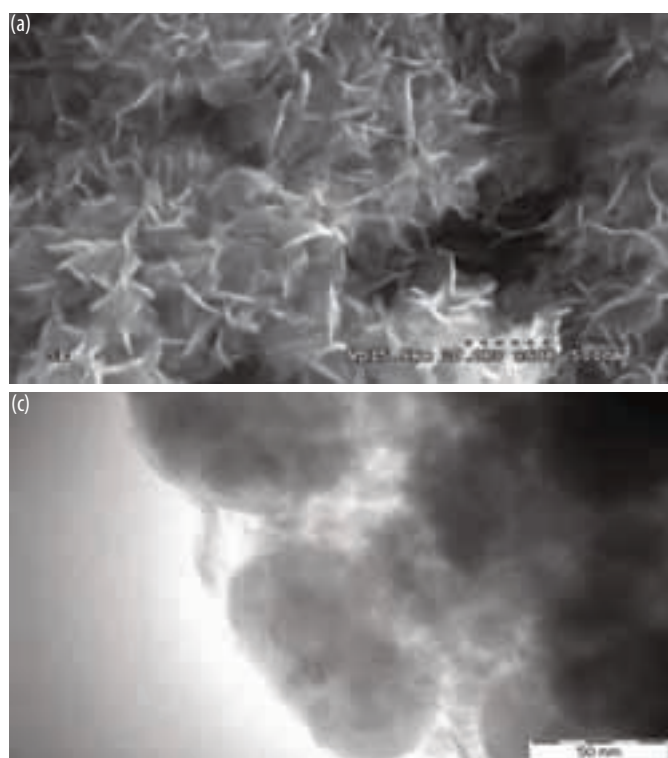


Fig. 2 (a)  $WS_2$  nanosheets produced by ARCI method (b) Conventional commercial grade  $2H-WS_2$  plates (c) IF- $WS_2$  particles produced at ARCI (d) IG- $WS_2$  as synthesized

Contributor: P V V Srinivas

# Development of Copper-Chromium-CNT Composites

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Copper is one of the most important materials for thermal and electrical applications due to its high thermal and electrical conductivity. The use of carbon nanotubes (CNTs) as reinforcements in copper is considered very attractive to meet the increasing demands for high performance materials. However, most of investigations focus on the application of CNT in polymer composites because of the flexibility and compatibility of CNTs with polymers. In recent years, CNT reinforced metal–matrix composites (MMCs) are attracting an increasing interest. But, CNT/MMCs have shown inferior mechanical properties than expected compared to CNT/polymer or CNT/ceramic composites. To prepare the high performance CNT/MMCs, two main issues have to be solved. The first one being the proper dispersion of individual CNTs in the metallic matrix and the second one is the effective interfacial bonding between CNTs and the matrix. To solve the former problem i.e. for dispersing of CNTs in the metallic matrix, use of ball milling has been proven to be a promising technique. To solve the interface problem between Cu and CNTs, matrix-alloying approach by the addition of matrix-alloying elements to ensure certain reaction (i.e. carbide formation) at the interface is being practiced. In the present study, the effect of chromium as a matrix-alloying element is evaluated on the improvement of interfacial bonding and enhancement in mechanical and electrical properties through the formation of a thin transition layer of  $\text{Cr}_3\text{C}_2$  at the interface between CNTs and Cu–Cr matrix.

The raw materials of copper and chromium powders, and CNTs were milled in a high energy planetary ball mill and subsequently reduced under hydrogen at 600°C for 1 hr. The CNT/Cu composite powders were sintered using spark plasma sintering at 650°C for 5 min under a vacuum of 6 Pa with an applied pressure of 75 MPa at a heating rate of 100°C/min. Microhardness and electrical conductivity measurements were made on the sintered samples and compared with Cu-CNT and reference electrolytic tough pitch (ETP) copper as shown in Table 1. Though the electrical conductivity of Cu-Cr/CNT composites decreases compared to ETP Cu, the increase in hardness is an added advantage for the engineering applications.

From Table 1, it is clear that Cu-Cr-CNT composite exhibits superior properties when compared to Cu-CNT composites.

Table 1 Electrical and mechanical properties of various CNT/MMCs

| Material  | Property                        |                  |
|-----------|---------------------------------|------------------|
|           | Electrical conductivity, % IACS | Hardness, HV0.05 |
| ETP Cu    | 95                              | 60               |
| Cu-CNT    | 44                              | 87               |
| Cu-Cr-CNT | 67                              | 110              |

The microstructure of the consolidated Cu-Cr-CNT composite was investigated by scanning electron microscope as shown in Figure 1. Homogenous distribution of CNTs in the copper matrix of 2  $\mu\text{m}$  is seen from the microstructure.

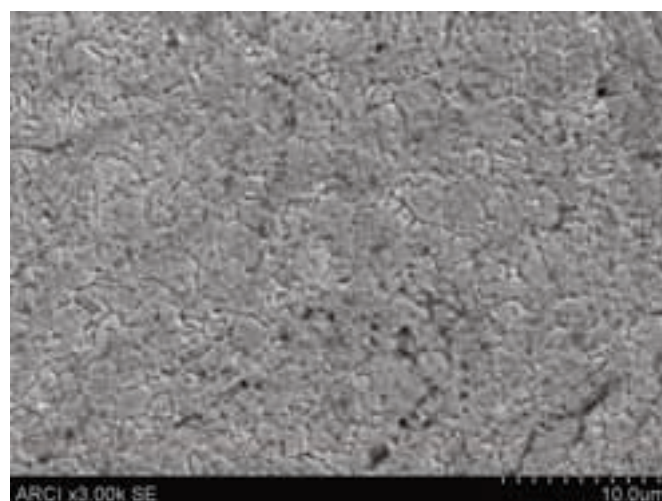


Fig. 1 Microstructure of Cu-Cr/CNT composite processed using MA and SPS, indicating the homogenous distribution of CNTs in the copper matrix

Therefore, it can be inferred that the enhancement of hardness and electrical conductivity by CNT-reinforcement is originated from the high interfacial strength at CNT-Cu-Cr interface and homogeneous distribution of CNTs within Cu-Cr matrix. In summary, powder metallurgical processing of Cu-Cr-CNT composites could show some major technological advantages due to their observed higher hardness and electrical conductivity. Further characterization of the consolidated samples for their tensile properties and in-depth TEM characterization are under progress.

Contributors: G Sivakumar, P V V Srinivas, D Chakravarthy, G V R Reddy and Tata Narasinga Rao



# Zirconia Nanoparticle Reinforced, Morphology Engineered Graphene Based Foams

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There have been several recent reports on the design and synthesis of ultra-low density, porous, three-dimensional foams based on graphene for applications ranging from mechanical to oil adsorption. Here we show that such graphene based foams can be engineered in morphology and density by reinforcing the graphene-like units with nanocrystalline zirconia. These reduced graphene oxide (rGO) foams with zirconia were prepared via solution methods where low zirconia fractions yield a flaky microstructure in which the zirconia nanoparticles prevent the flakes from tearing off by arresting the propagating cracks. A marked change in morphology of the foams is observed at higher zirconia concentration which possesses a complete mesh-like inter-connected structure of flakes with different morphologies varying from fully coiled at zirconia-rich regions to partially coiled at zirconia-depleted regions and completely uncoiled in regions devoid of zirconia. The change in morphology observed experimentally as a function of zirconia content is explained theoretically by molecular dynamic simulations and DFT calculations, leading to possible engineering of graphene based foams via second phase reinforcement. The higher zirconia content in the hybrid foams improves the oil adsorption capacity as well as mechanical reinforcement compared to pristine foams.



Fig. 1 Digital micrograph of the composite foam

The rGO used in this study was prepared by a modified Hummer's process using graphite powder as the starting material. Foams of three different compositions were

investigated a) pure rGO, b) rGO-25% ZrO<sub>2</sub> and c) rGO-50% ZrO<sub>2</sub>. A typical digital image of the low-density pristine foam ( $\rho = 4.5\text{-}5.0 \text{ mgcc}^{-1}$ ) is shown in Figure 1 and the coiled microstructure of the rGO-50% ZrO<sub>2</sub> composite is shown in the TEM micrograph, Figure 2.

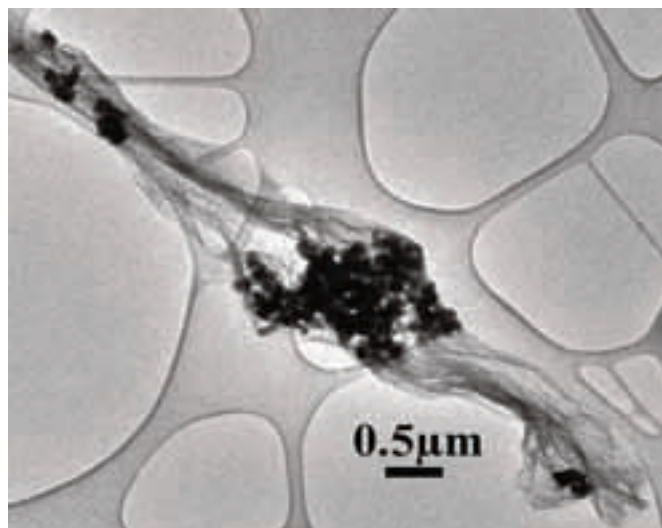


Fig. 2 rGO-50ZrO<sub>2</sub> composite with coiled rGO flakes along zirconia nanoparticles

The extremely high porosities in the foams give them the ability to adsorb different oils and organic solvents. Typically, 1 mg of the foams was put into various oil-water mixtures for experimentation. The oil adsorption per unit mass was obtained using the equation  $Q = (W_f - W_i) / W_i$  where  $W_i$  and  $W_f$  are the initial and final weights of foams. The composite foams yield an increment of adsorption capacities by ~ 30-40% over pure rGO sheet-like foams for various oils and organic solvents as tabulated in Table I, clearly validating the effect of morphology on adsorption due to second phase zirconia addition.

Table 1 Comparison of oil/solvent adsorption capacities (wt/wt) of different C-based materials

|             | CNT | Gr-CNT | Gr-sponge | rGO + coating | Current work |                         |                         |
|-------------|-----|--------|-----------|---------------|--------------|-------------------------|-------------------------|
|             |     |        |           |               | rGO          | rGO-25 ZrO <sub>2</sub> | rGO-50 ZrO <sub>2</sub> |
| Gasolene    | -   | -      | -         | 90            | 95           | 110                     | 125                     |
| Motor Oil   | 80* | 85     | 70        | 100           | 75           | 90                      | 110                     |
| Chloroform  | 120 | 105    | 85        | 100           | 105          | 110                     | 130                     |
| Toluene     | 60  | 120    | 55        | 95            | 80           | 90                      | 110                     |
| Cooking Oil | -   | 105    | 75        | 120           | 60           | 70                      | 90                      |

# Manufacturing of Multifunctional Titania Spheres for Self-Cleaning Textile Application

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A novel titanium dioxide based material has been developed at ARCI for self cleaning textile application. Titania is commonly used as self cleaning agent due to its good photocatalytic activity. In its nano form, the photocatalytic activity enhances as the reactive surface area increases. The titania in combination of anatase and rutile in about 70:30 ratio shows enhanced photocatalytic activity. However, being the most temperature stable phase, the rutile structured particles that possess the best photocatalytic activity, will be preferred for many applications. When modified with metal, it acts against micro organisms day and night. It is always preferred to have particle size larger than the 100 nm preferably in micron or submicron size and still possess all the properties as in the nano form. Such particles are easy to handle for safety reasons. Such materials have an advantage of possessing quantum size properties and do not release nanoparticles in environment freely which may cause serious toxic effects.

Taking account of all the available facts and preferences of the applications, the nanostructured titania material has been developed to have micron / submicron size spheres of radially self assembled rutile nanorods optionally having small concentration of anatase phased particles bonded to the rutile rods of titania and modified with silver metal. These particles contain major component of rutile phase of titania having high temperature stability. These possess excellent properties of self cleaning, anti-malodorant, air cleaning, UV protection, surface brightening etc. due to the nanostructured properties of all its individual components even though they exist in the micron or submicron size regime.

Figure 1(a) depicts the sphere formation of the size range from 0.5  $\mu\text{m}$  to 1.5  $\mu\text{m}$  in the scanning electron micrograph (SEM). Figure 1(b) shows the SEM image of the fractured particle which clearly indicates the radial self assembly of the nanorods in the sphere.

Property of maldeodorant is extremely important for self cleaning textile application. The organic pollutants are adsorbed and the body releases sweat, sebum in turn gives rise to odour. The deodorant property was tested through the gaseous pollutant degradation using gas chromatography. A cotton fabric piece of 1 inch square was dipped in the titania suspension of 0.2 wt% concentration and stirred for 15 minutes. Then it was removed and squeezed in the filter

paper to remove the excess liquid. This piece was then ironed to dry and then used for the testing purpose. This fabric was kept in the test reactor and exposed to solar simulator. The reactor was connected to the gas chromatography unit, and was injected with 500 ppm of acetaldehyde as a test gas pollutant as per the existing test standards. The conversion of acetaldehyde into carbon dioxide was monitored with time. The aliquots of gas in the reactor were drawn by gas chromatography unit periodically to measure the concentration of acetaldehyde and carbon dioxide. As a control sample, the fabric without any titania loading was tested. Figure 2 shows the results.

As depicted in the Figure acetaldehyde was completely decomposed due to Ag-TiO<sub>2</sub> spheres loaded on fabric. Within one hour of irradiation, 233 ppm of CO<sub>2</sub> was formed in the presence of Ag-TiO<sub>2</sub>, indicating the complete oxidation of acetaldehyde and CO<sub>2</sub>. The increase in CO<sub>2</sub> concentration at longer irradiation time may be due to the decomposition of binder, which was used to bind Ag-TiO<sub>2</sub> in fabric.

The patent for the manufacturing process was applied and the process technology was transferred to one of the leading Indian textile finish manufacturing company.

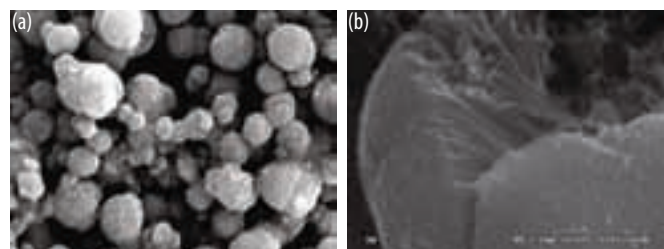


Fig. 1 SEM images of the (a) titania spheres (b) broken sphere showing radial assembly of rods

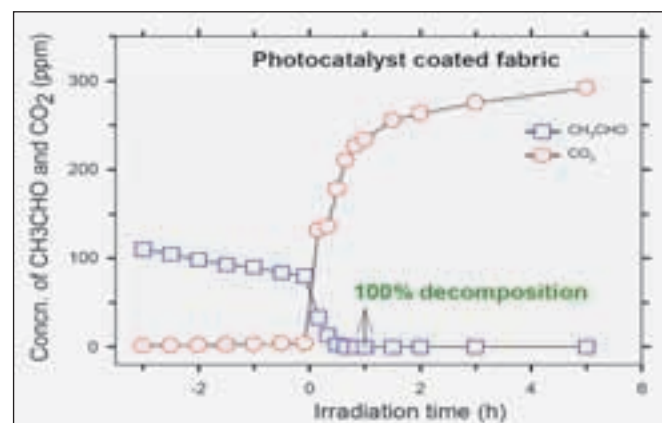


Fig. 2 Gas phase photocatalytic activity of titania spheres by acetaldehyde decomposition method

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# Grain Growth Kinetics Studies of Flame Spray Pyrolysis Synthesized Doped ZnO Nanopowders

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Doped ZnO nanopowders (varistor composition) of size ~ 25 nm was synthesized by Flame Spray Pyrolysis and sintered at different temperatures and times to understand the grain growth kinetic of the powders. The grain growth kinetics was studied using the phenomenological kinetics equation. The grain growth exponent can be estimated at isothermal conditions by the following equation (1):

$$\text{Log } G = 1/n \log t + 1/n(\log K_0 - 0.434Q/RT) \dots\dots\dots(1)$$

where, G is the average grain size at the time t, "n" is the grain growth exponent, K<sub>0</sub> is a constant, Q is the sintering activation energy, R is the universal gas constant and T is the sintering temperature in Kelvin. Under isothermal condition, the "n" value could be estimated from the slope of the log grain size verses log time plot, as shown in Figure1(a). The slope was equal to "1/n" which was obtained from linear fit of the data. The "n" values of 4.01, 3.92 and 3.54 for the sintering temperatures of 900, 1000 and 1100°C were obtained, respectively. An average "n" value of 3.82 was taken to be the for activation energy calculation for the process.

Activation energy of the grain growth was estimated by using equation (2):

$$\text{Log } (G^n/t) = \log K_0 - 0.434Q/RT \dots\dots\dots(2)$$

The activation energy (Q) of grain growth could be calculated from the Arrhenius plot of log (G<sub>n</sub>/t) verses 1000/T (K<sup>-1</sup>) as shown in fig.1(b). The activation energy of grain growth was found to be 306 ± 10 kJmol<sup>-1</sup>.

A grain growth exponent "n" of 3 is representation of uniform coarsening of the particles in three dimension (3D). In general the "n" value is found to be 3 for pure ZnO, due to solid-state diffusion of Zn<sup>2+</sup> ions. The reported values for the activation energy for grain growth for ZnO are in the range of 224-400 kJ.mol<sup>-1</sup>, depending on the initial characteristics of powders. On addition of liquid phase sintering additive (Bi<sub>2</sub>O<sub>3</sub>), the "n" value is reported to increase to 4-5 and the activation energy varies in the range of 150-270 kJ.mol<sup>-1</sup>. It is believed that with Bi<sub>2</sub>O<sub>3</sub> addition, a ZnO grain growth gradually slows down due to the diffusion of the solute atoms through the increasingly thick liquid phase layer coming from Bi<sub>2</sub>O<sub>3</sub> by solution-precipitation phase boundary reaction mechanism.

When the powder of ZnO-Bi<sub>2</sub>O<sub>3</sub> composition with particle size of ~ 20 nm was used, the grain growth exponent "n" of 3.5 and activation energy of grain growth of 148 kJ.mol<sup>-1</sup> were obtained. For varistor composition with particle size of 20 nm made by Sol-Gel method was used, the "n" value of 5 and activation energy for grain growth of 364 ± 24 kJ.mol<sup>-1</sup> were reported. The activation energy of grain growth is lower in nanopowder compared to the micron sized powder samples because of presence of higher surface energy in nanopowder compared to micron powder. In this study, nanopowder of size less than 25 nm was used, the "n" value in range of 3-4 and activation energy for grain growth of 306 ± 10 kJ.mol<sup>-1</sup> was obtained observations are in reasonable agreement with reported values.

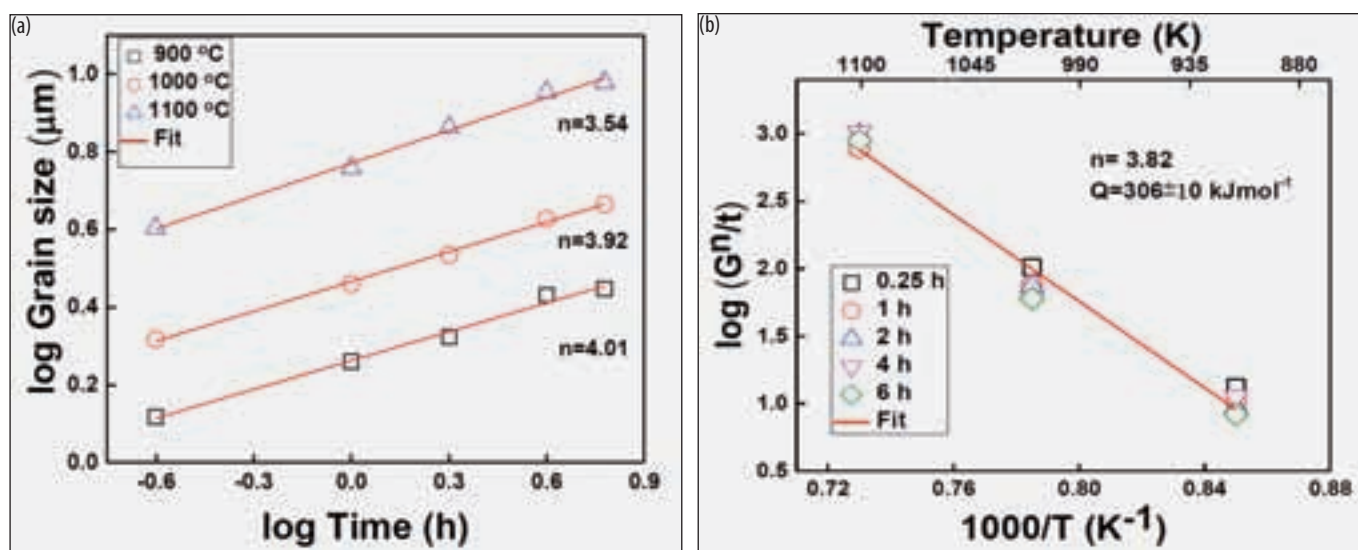


Fig. 1(a) Isothermal grain growth plot for samples (b) Arrhenius plot of grain growth

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# Uniform Carbon Coated Mesoporous SnO<sub>2</sub> Nanoparticles for Improved Electrochemical Performance in Li-ion Batteries

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As part of anode material development for Li-ion Batteries (LIBs), previously, ARCI succeeded to prepare an efficient Fe<sub>3</sub>O<sub>4</sub>/reduced graphene oxide as a high performance anode material for LIBs. Carbon coating on mesoporous SnO<sub>2</sub> nanoparticles has been carried out recently by adopting ARCI developed carbon coating technology, in which high quality thin carbon coating layer on SnO<sub>2</sub> is achieved with good uniformity and high electrical conductivity. Since large volume expansion and low intrinsic electrical conductivity hamper its electro-chemical performance, in the present study, we developed a novel hybrid approach (formation of mesoporous nanostructures SnO<sub>2</sub> and uniform carbon coating) to prepare an efficient SnO<sub>2</sub>-based electrode material with improved electro-chemical performance. This unique strategy may greatly enhance the electrochemical performance of SnO<sub>2</sub> by minimizing the volume expansion and increasing the electrical conductivity. Initially, mesoporous SnO<sub>2</sub> nanoparticles were prepared by surfactant assisted hydrothermal method. Then, post carbon coating on mesoporous SnO<sub>2</sub> was carried out by ARCI using carbon coating process. The resulting polysaccharide coated SnO<sub>2</sub> was dried, and later carbonized to obtain uniform carbon coated SnO<sub>2</sub> nanoparticles. As shown in TEM image (Figure 1a), the SnO<sub>2</sub> nanoparticles with sizes of ~ 8-15 nm are completely embedded in carbon matrix with thickness of  $4.2 \pm 1.9$  nm, which reveals that ARCI's process is found to be effective for homogeneous coating of thin layers of carbon. The selected-area electron diffraction (SAED) pattern (inset of Fig. 1a) indexes (110), (101), (200), and (211) planes of SnO<sub>2</sub>, confirming the presence of tetragonal phase SnO<sub>2</sub> with polycrystalline structure. Raman spectrum shows two Gaussian peaks namely G-band at 1585 cm<sup>-1</sup> and D-band at 1355 cm<sup>-1</sup> with low I<sub>D</sub>/I<sub>G</sub> ratio, in which G and D band are assigned to ordered sp<sup>2</sup> graphitic carbon and disordered sp<sup>3</sup> carbon respectively. The electrochemical performance of C-SnO<sub>2</sub> anode electrodes was investigated in a swage lock half cells in the potential ranging from 0.02 to 2V. Electrochemical results demonstrated that carbon coated SnO<sub>2</sub> nanoparticles exhibited stable capacity of ~ 755 mAh/g after 25 cycles at 1C rate compared to the capacity of pure SnO<sub>2</sub> (113 mAh/g) and conventional carbon coated SnO<sub>2</sub> particles (230 mAh/g), which is consistent with impedance results (Figure 1b and 1c). Further, it exhibited a stable capacity of ~ 396, 272 and 129 mAh/g at 5C, 8C and 10C current rate respectively over 250 cycles. The excellent electro-chemical performance of carbon coated

mesoporous SnO<sub>2</sub> is attributed to uniform carbon coating, high content of graphitic carbon, high surface area, short Li<sup>+</sup>-ion diffusion length, and a porous interior structure.

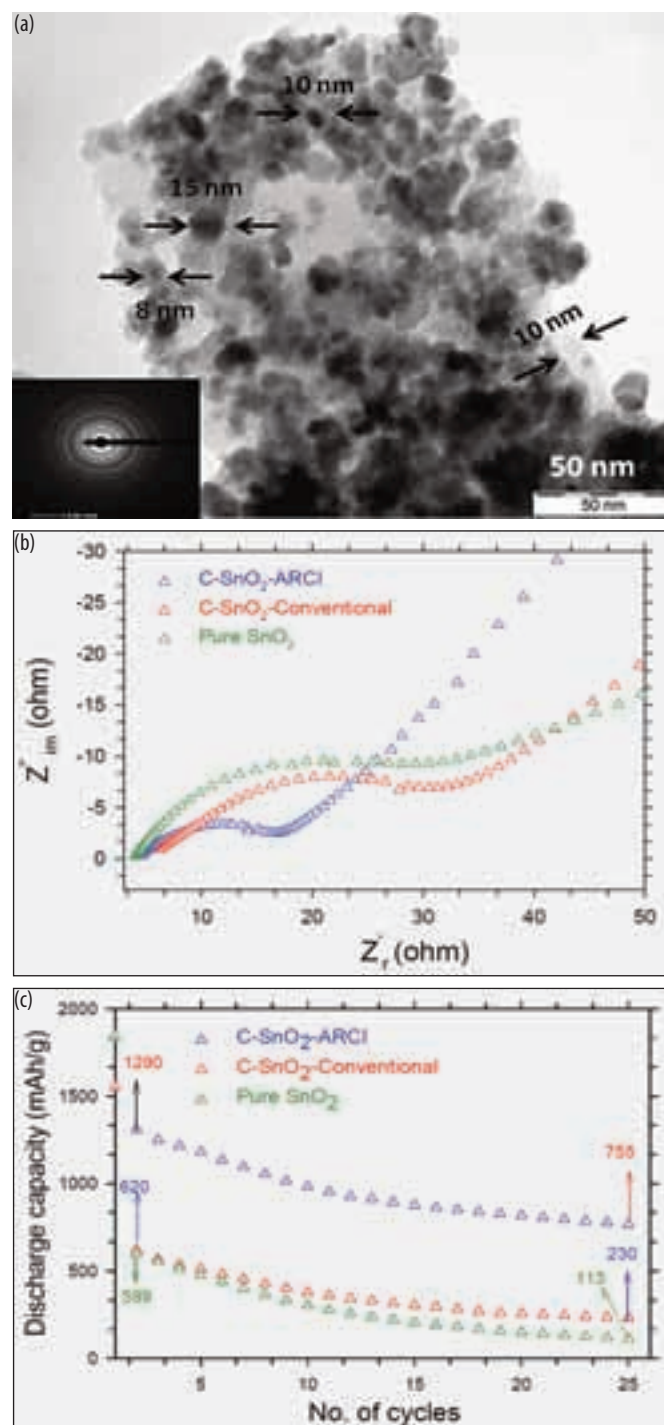


Fig. 1 Transmission electron microscope image (a) electro-chemical performance (b) and impedance spectra of carbon coated mesoporous SnO<sub>2</sub> nanoparticles (c). For comparison, the data of pure SnO<sub>2</sub> and conventional carbon coated SnO<sub>2</sub> are also included in Fig.1(b) and 1(c).

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# Processing of Fe Powder by Cryo Milling

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Potential use of nanostructured materials has led to invention of many processing techniques for synthesizing/producing the nanostructured materials. Cryo milling is one such technique that can be used for producing the large scale nanostructured metallic materials. Cryo milling is attritor type of mechanical milling but differs from the attritor milling since metallic powders are milled at cryogenic temperature. Schematic diagram of cryomill is shown in Figure 1. In this approach, powders are mechanically milled in liquid nitrogen slurry, followed by consolidation with established techniques such as hot extrusion.

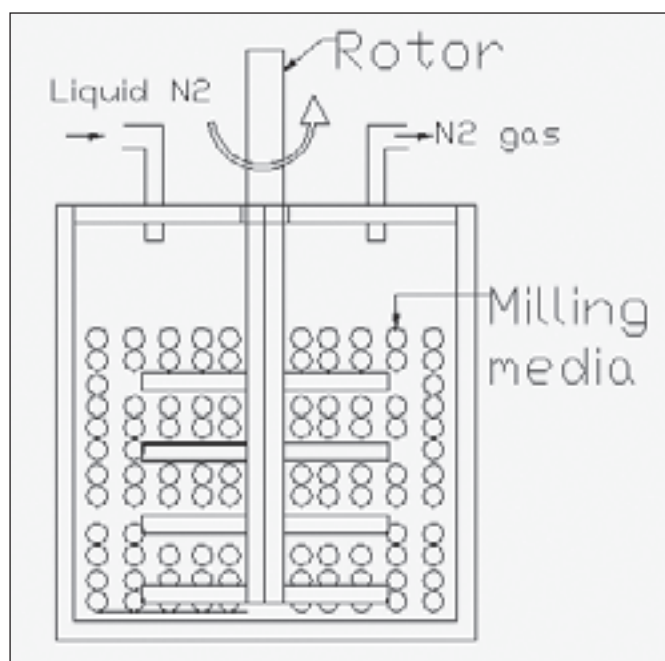


Fig.1 Schematic diagram of cryo mill

Cryo milling gives the advantage of milling at cryogenic temperature in addition to the advantages which will be achieved by attrition milling. Low temperature milling accelerates the fracture process and attains the steady state conditions quickly. This low temperature during the milling suppresses the recovery and recrystallization of the material which results in fine grained structures and more rapid grain refinement. There are several advantages with cryomilling compared to conventional milling. They are (1) sticking of the powder to the media and container can be eliminated (2) powder agglomeration can be reduced (3) takes less milling time to get the nanostructured powder (4) oxidation of powder during the milling can be avoided as the milling is done in liquid nitrogen (5) depending on the material,

there may be improved solubility (6) powder can be milled with high ball to powder ratio (32:1) (7) handling the milled powders is easier than other high energy milling processes. Considering all the above advantages, cryomilling can be used in material development to have Hall Petch, dispersoid, composite and solution strengthening. Limitations are (1) high process cost due to use of liquid nitrogen (2) Nitrogen may be picked up in the material (3) Effectiveness of the milling depends on the DBT (ductile to brittle transition) behavior of material.

ARCI has a cryo mill of 1.5 gallon tank capacity. Course Iron powder was milled in the cryo mill for 7 hrs. Micro structure and crystallite size of the milled iron powder were studied. Figure 2A and 2B shows the SEM microstructure of the unmilled and cryo milled Fe powder. With more milling time particle size and crystallite size can be reduced. Crystallite size of the cryo milled powder is 30 nm as observed by XRD after 7 hour milling. Fig (3) shows variation of crystallite size with milling time. As the milling time increases, crystallite size decreases. Milled powders can be used to develop high strength materials for structural applications.

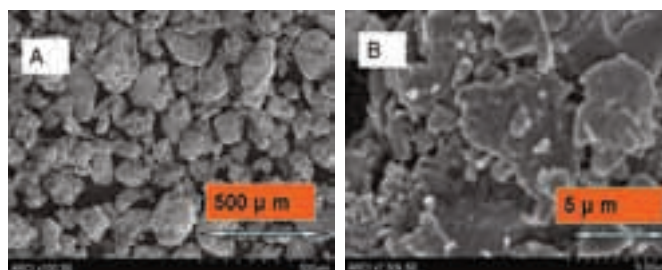


Fig. 2 Micro structure of a) Unmilled powder b) Cryo milled powder

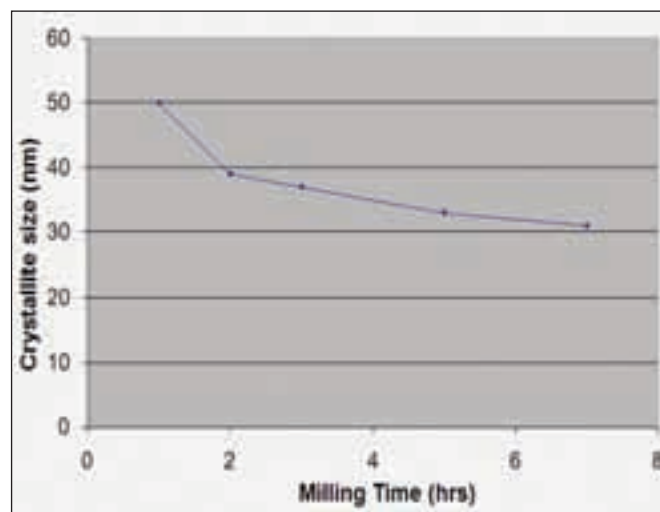


Fig. 3 Variation of crystallite size of cryo milled Iron powder with milling time

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# Wood Derived Carbon/SnO<sub>2</sub> Composite Anode for Lithium-ion Batteries

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Wood is a natural and abundant material with hierarchical architecture that extends from nanoscale to macroscopic level. A simplistic description of wood is that, it is composite of cellulose and other components such as lignin and hemicelluloses. Removal of lignin and hemicellulose by leaching with acid (maceration) leads to hierarchically porous structures. Such hierarchically porous structures can be very useful for rapid diffusion, transport of ions and small molecules, as well as for storage applications. Additionally, the hierarchical structure can impart greater structural stability. Carbonization of such porous wood structure can act as a suitable electrode material in Li-ion batteries.

High capacity active anode materials (Sn and SnO<sub>x</sub>) suffer from large volume expansion during alloying (lithiation), and dealloying (delithiation) of lithium. Eventually the material undergoes mechanical disintegration and the electrode fails due to capacity fading after few cycles. Porous carbon substrates such as above structures act as ideal substrates for high capacity anodes to compensate the volume expansion during lithiation and improve the cycling stability. Nanocasting of macerated wood with tin oxide followed by carbonization yields composites with hierarchical porous carbon matrix as seen in figure 1.

In such composites, the hierarchical porous carbon matrix improves the electronic conduction and acts as a

mechanical buffer to accommodate the volume change during charge/discharge. Raman spectra for carbon – SnO<sub>x</sub> samples in Figure 2 revealed that the structural hierarchy in wood template facilitates graphitization at low temperatures of carbonization. Synergistic effect of hierarchy in carbon–SnO<sub>x</sub> composite results in improved cycling stability and rate capability in carbon-tin oxide composite material with very low amount of tin oxide, as shown in Figure 3.

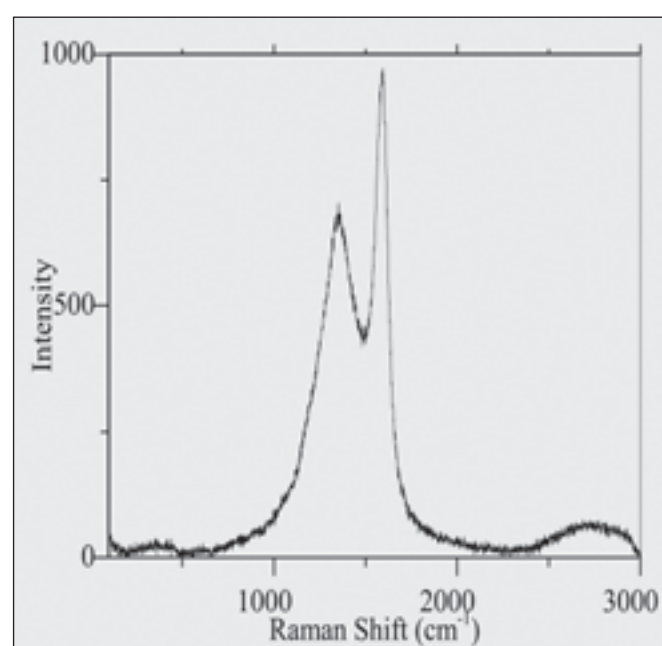


Fig. 2 Raman spectra for carbonized wood fiber with tin oxide

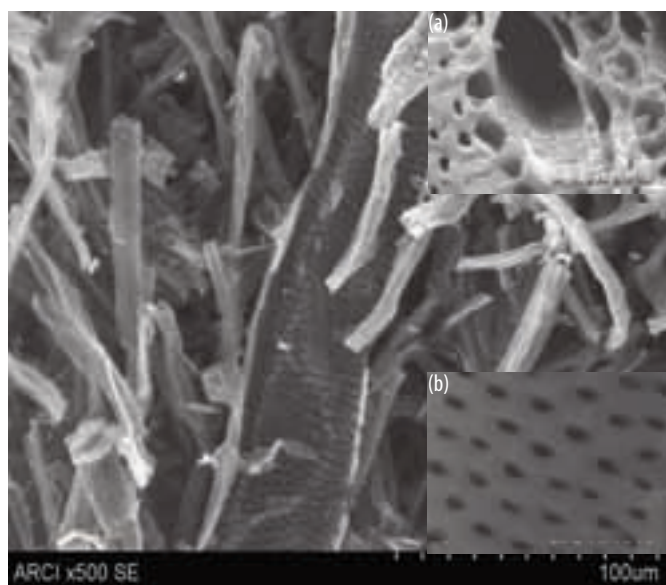


Fig. 1 SEM image of carbonized wood fiber with tin oxide. Inset (a) and (b) FESEM image of Hierarchical interconnected pores

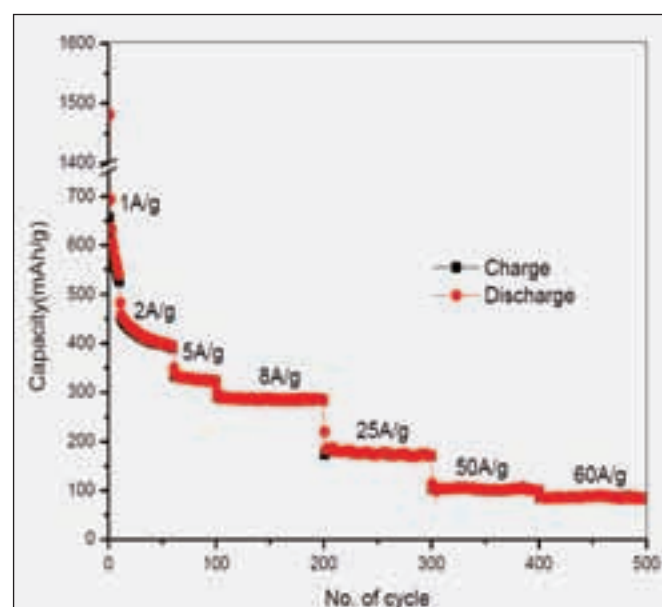


Fig. 3 Rate capability of carbonized wood fiber with tin oxide

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# Development of Soft Magnetic Fe-P Alloy through Powder Extrusion

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In search of an alternative for electrical steel in automotive applications, ARCI has identified Fe-P alloy as a potential soft-magnetic material with attractive magnetic properties. The material is being developed through wrought as well as powder metallurgy (PM) routes.

Fe-3.5wt%P master alloy was vacuum induction melted with required quantity of ARCO iron and the molten alloy was atomized in Ar atmosphere. The chemical analysis of the powder revealed the P content to be 1.8 wt% which is well within the solubility limit in iron. The spherical morphology and the dendritic structure of the particles are presented in Figure 1 (a) & 1(b). The sieve analysis of the atomized powder is given in Table 1 from which it can be seen that the average particle size is in the range of 65µm-75 µm. 500 g of Powder was taken in an MS can (Figure 2(a)) which was thoroughly degassed at 450°C and vacuum sealed at 0.02 Pa. The sealed can was subsequently upset-forged at 1050°C into a billet to 95% of the theoretical density (TD) (Fig. 2(b)). The billet was extruded into a Ø 16 mm rod (extrusion ratio of 9) of 30 cm-35 cm length at 1050°C and the density of the rod was found to be 99% of TD (Figure 2(c)).

The extruded rod sample was solutionized at 1050°C for 1 hour followed by quenching in water. Presented in Table 2 are the saturation magnetization values of the atomized powder, extruded sample & solutionized sample and typical vibrating sample magnetometer (VSM) curves of extruded and solutionized samples are given in Figure 3, from which it can be inferred that the samples exhibit high magnetic induction (Ms) and the process contamination is almost negligible.

The SEM images of extruded sample as shown in Figure 4(a) revealed segregation of Fe<sub>3</sub>P in Fe-P matrix while the a major removal of segregation upon solutionization can be seen in Figure 4(b).

Further experiments in the range of 950°C-1100°C have been planned to select the appropriate solution treatment.

Later, annealing schedules will be studied to optimize the hysteresis loop characteristics & development of device level components.



Fig. 2 (a) Vacuum sealed MS can (b) forged billet and (c) extruded rods

Table 1 Sieve analysis of atomized powder

| Size Fraction(µm) | Weight% |
|-------------------|---------|
| +150              | 5.6     |
| -150 +105         | 10.0    |
| -105 +75          | 15.2    |
| -75 +44           | 30.3    |
| -44 +37           | 10.3    |
| -37               | 28.6    |

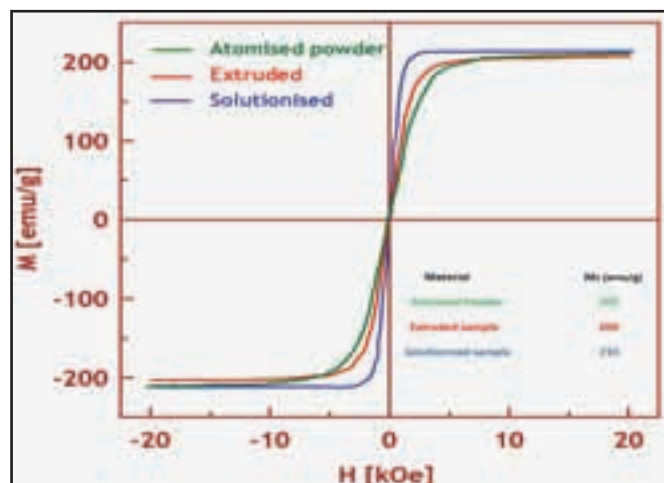


Fig. 3 VSM data of extruded & solutionized samples

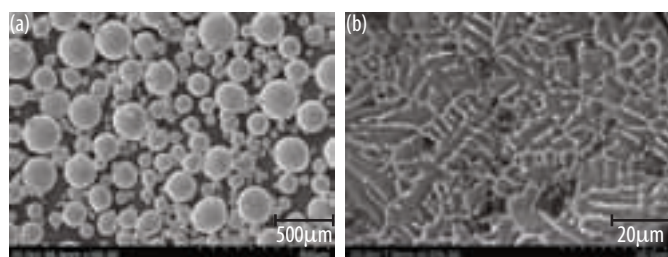


Fig. 1 (a) Atomized powder (b) Dendritic structure

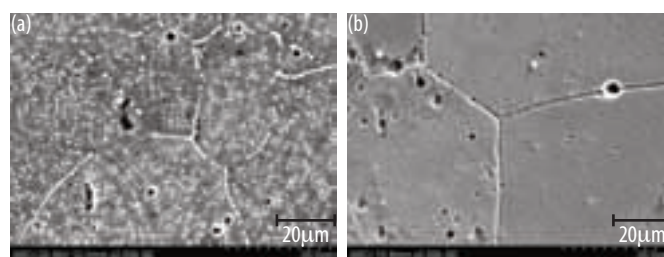


Fig. 4 SEM images of (a) extruded and (b) solutionized samples

Contributors: Ravi Gautam, V Uma, D Prabhu, R Vijay, V Chandrasekharan and R Gopalan

# RF Induction Plasma Synthesized Nano-aluminium as Binder to Improve the Hardness of Spark Plasma Sintered Al Disks

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Aluminum and its alloys have a great diversity of industrial applications because of their high specific stiffness and good workability. But the use of these alloys is limited due to their relatively low yield stress. Numerous investigations have shown that nanocrystalline (nc,  $d < 100$  nm) and ultrafine-grained (ufg,  $100$  nm  $< d < 1$   $\mu$ m) materials have very high strength or hardness. Though nc/ufg metals and alloys possess excellent mechanical strength, their path to commercial applications has been hampered by poor ductility, as necking usually occurs at low plastic strains. This has become one of barriers for advanced structural applications. Because of these concerns, current research has been focused on different strategies to increase the uniform tensile elongation of nc/ufg materials for practical structural applications. Some of these strategies include establishing bi-modal microstructures, deforming at high strain rates and/or low temperatures, utilizing the effect of high strain-rate sensitivity, etc. Here, we have adopted the former methodology to get the high strength and ductile aluminum suitable for automotive applications. The process of fabrication consists of blending the nano aluminium (n-Al) powder with micron sized aluminium (m-Al) powder in various proportions followed by spark plasma sintering.

n-Al powder (of size ranging from 50 to 200 nm) was produced using RF induction plasma system and m-Al as precursor powder. Figure 1 (a) and (b) shows the typical SEM micrographs of the m-Al and n-Al powders respectively. n-Al powder was degassed at 300°C for 12 hours in a vacuum furnace and blended with m-Al powder at various proportions ranging from 5-30 wt.%. The blended powders were spark plasma sintered at a temperature of 450°C under vacuum of 6 Pa at a heating rate of 100°C/min and at a pressure of 75 MPa.

Initially, the optimization of the sintered parameters was done in a 10 mm diameter die. The sintered sample densities were found to be varying very closely in the range of 97-98% of the theoretical density.

Hardness of the sintered samples was measured and, was found to increase with increase with the addition of n-Al to m-Al, as illustrated in Table 1.

The samples were polished metallographically and etched with 0.5% HF. Figures 2 (a) and (b) shows the sintered

microstructures of m-Al and n-Al, respectively. Refined microstructure may be seen in Figure 2(b). Metallographic examination revealed that grains in the range of 10-15  $\mu$ m for m-Al samples, whereas n-Al sample had a very fine microstructure not to be revealed by an optical microscope. Further characterization of the samples for their microstructural features is under progress. Samples with various fractions of m-Al and n-Al powders as given in Table 1 were sintered in a 30 mm ID graphite die and tensile specimens were cut from the sintered samples through wire cut electric discharge machining. The machined samples were polished mechanically to a mirror finish and the evaluation of their tensile properties is under progress.

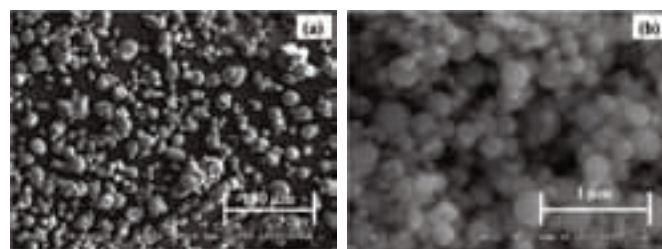


Fig. 1 (a) SEM micrograph of micron aluminium precursor powder (b) nano aluminium produced using RF induction plasma

Table 1 Variation of hardness of blended aluminium with n-Al. The increase in hardness with increase in n-Al may be noted

| Material Description | Hardness, HV <sub>0.05</sub> |
|----------------------|------------------------------|
| m-Al                 | 28                           |
| 5% n-Al + m-Al       | 37                           |
| 10% n-Al + m-Al      | 45                           |
| 20% n-Al + m-Al      | 52                           |
| 30% n-Al + m-Al      | 55                           |
| n-Al                 | 84                           |

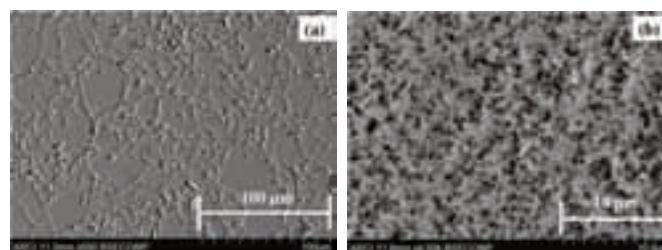


Fig. 2 SEM micrograph of spark plasma sintered (a) micron aluminium precursor powder (b) nano aluminium produced using RF induction plasma.

Contributors: S B Chandrasekhar, Dibyendu Chakravarthy, P Sai Karthik, G Siva Kumar and Tata Narasinga Rao





## Centre for Engineered Coatings

*The vast ability of Centre for Engineered Coatings (CEC) to provide few nanometer scale to couple of millimeter thick coatings through diverse technologies and corresponding achievements at ARCI have shaped the institution into a globally prominent player in the area of surface engineering. With an enormous impetus created by the transfer of several technologies as mentioned above to the multiple industries and different-locations in the country, the centre has gone ahead and has focused on many more surface engineering technologies that are expected to deliver a large deal of promise in the industrial arena. An excellent track record of the centre as well as Government of India's recent initiative under "Make in India" program for encouraging and strengthening of the manufacturing sector, noteworthy steps in this direction have been initiated an year ago and most critical tasks related to indigenous development of advanced Detonation Spray Coating (DSC) system has been completed. In the coming financial year, the centre is aiming to accomplish the system development in all aspects and transfer the newly fabricated DSC system to existing and new private entrepreneurs. Consistent with its main objectives, the CEC has successfully carried out R&D studies and extended important support to Navy by completing the prototype coatings on pilot batch of LPC blades and seals with erosion resistant and abradable coatings using DSC and Atmospheric Plasma Spraying (APS) techniques. Similarly, thermal barrier coatings as deposited through Electron Beam Physical Vapor Deposition (EBPVD) technology have been demonstrated on blades and vanes of aero-engine, similar efforts are likely to be continued for extending on more variety of blades and vanes of different fighter aircrafts. Considerable progress is also accomplished in development of the Cold Spray Coating (CSC) technology and launch of portable CSC system for transfer to Indian industries on commercial basis is planned shortly.*

*Yet another notable contribution of CEC is the development of fatigue resistant Al-alloys through Micro Arc Oxidation (MAO) coatings useful for various industrial applications including landing gear parts of aircraft. A patent application in this regard has already been filed in India. Also, with a view that the academic institutions in the country should also concurrently be benefited by the technologies that are developed at ARCI, the MAO technology is being suitably scaled and tailored in accordance with the interest of academic institutions. While one such academic unit has already been transferred and installed, it is expected that 3-4 more systems to be fabricated and transferred to various central, regional and private universities in the country. Another very recent development at Centre is the development of iron-tungsten (Fe-W) alloy utilizing the expertise gained during the development of nickel-tungsten (Ni-W) alloy. The Centre is also actively engaged in development of various nanocomposite based wear resistant coatings using a Cathodic Arc Physical Vapour Deposition (CAPVD) for developing tools for high speed dry machining applications and solar selective coatings for industrial and strategic sectors. Further, the Solution Precursor Plasma Spray (SPPS) technique has been successfully demonstrated to deposit a wide array of hybrid coatings suitable for conventional thermal barrier and non-conventional wear resistant applications for strategic power generation sector and gas turbine industry. In addition, the Centre has been actively engaged in development of a wide array of materials and related applications under numerous sponsored projects. Over the past two decades, the Centre has emerged to house unique combination of surface engineering technologies. The need for a centralized building to house all of them under a single roof for better capability demonstration has been realized and the same has been initiated. A brief technical report of the important activities at the Centre in the past year is provided subsequently.*



# Influence of Phase Gradient on the Mechanical and Tribological Behavior of Micro Arc Oxidation Coatings

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It is well known that the mechanism of coating formation in the Micro Arc Oxidation (MAO) process is quite complex. The complexity mainly arises out of multi-dimensional interactions between the substrate alloying elements-electrolyte constituents -plasma discharges driving such a coating formation. Although several attempts were made globally, no concrete evidences were demonstrated so far relating the mechanical and tribological properties with the phase gradient across the coating thickness. Towards achieving the above objective, a 6061 T6 Al alloy in which the total alloying content is  $\leq 2$  wt.% and a relatively lean electrolyte composition ( $\leq 4.5$  g/liter of de-ionized / distilled water) were chosen. A constant current density of  $0.3$  A/cm<sup>2</sup> was applied such that the plasma discharge behavior is identical in all the experiments.

The dense coating with  $100 \pm 5$   $\mu\text{m}$  was deposited, subjected to depth-wise XRD studies by employing intermediate grinding in between every X-ray scan. The relative peak intensities and area under the curves corresponding to (113) and (400) planes of  $\alpha$ - and  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phases were calculated and normalized based on highest intensity and integral area. The hardness and modulus gradients were evaluated across the coating thickness, the volume loss of MAO coated pin under sliding wear mode slid against the WC-Co sintered counter disc has been experimentally measured. The experimental data obtained has been modeled utilizing the linear rule of mixtures and inverse rule of mixtures.

The proportion of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phase is highest at the coating surface owing to the rapid solidification of molten reaction products as shown in Figure1; while the inner regions undergo  $\gamma \rightarrow \alpha$  phase transformation due to thermal energy as supplied by the erupting discharge events through the bulk of the coating. The coating hardness and modulus are strongly influenced by such a typical phase distribution and mainly depend upon how the relatively harder  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> phase varies across the coating thickness as shown in Figure 2. By applying linear rule of mixture, the individual hardness and modulus of  $\alpha$ - and  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phases were obtained as 2183 HV, 806 HV, 326.7 GPa and 68.7 GPa respectively. Further, as shown in Fig.3, the wear rate decreases with increasing fraction of harder  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> phase. Utilizing the inverse rule of mixture, the individual wear rates of  $\alpha$ - and  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phases were calculated to be  $6.47 \times 10^{-7}$  mm<sup>3</sup>/N.m and  $6.75 \times 10^{-6}$  mm<sup>3</sup>/N.m respectively. The present study not only establishes the typical relationships between the

phase gradient, mechanical and tribological properties; but also highlights the vital importance of having a harder and load bearing  $\alpha$ -alumina phase in the coating such that the overall wear resistance under high stress conditions can be significantly improved.

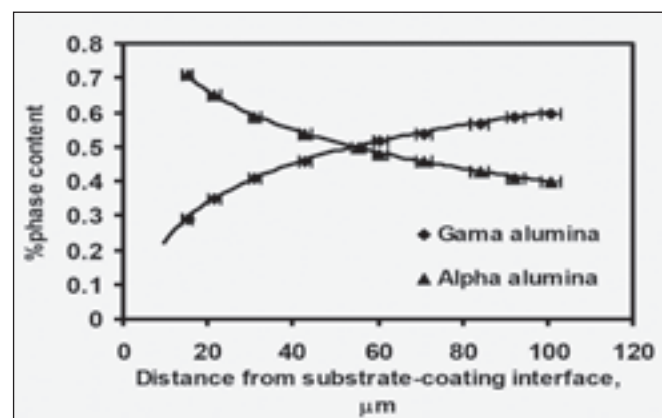


Fig. 1 Typical phase distribution across the MAO coating thickness

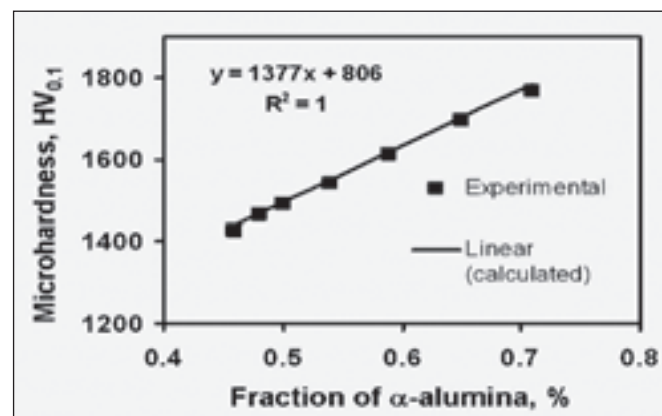


Fig.2 Experimental variation in localized coating hardness as a function of  $\alpha$ -alumina content as it changes across the coating thickness; calculated utilizing linear rule of mixtures

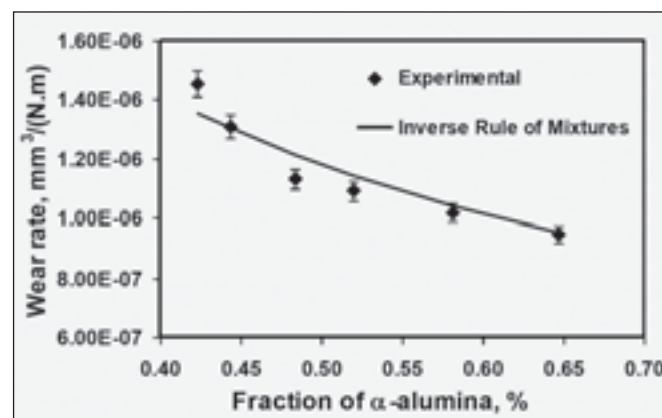


Fig.3 Experimental and inverse rule of mixture calculated wear rates as a function of  $\alpha$ -alumina content.

Contributors: P S V N B Gupta and G Sundararajan

# Cyclic Nanoimpact Behaviour of nc-TiAlN/a-Si<sub>3</sub>N<sub>4</sub> Nanocomposite Coatings Deposited by CAPVD Process

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Superhard nanocomposite coatings of the type nc-TiAlN/a-Si<sub>3</sub>N<sub>4</sub> are the recently developed thin hard coatings that find potential application in machining tools, dies, moulds etc., for operating temperatures of up to 1000°C. Being harder with the composite structure, these coatings are considered to perform better in interrupted high-speed machining operations such as end or face milling than any other conventional coatings, such as, TiN, TiAlN etc.

A cyclic nanoindentation impact testing machine used for simulating the milling or interrupted cutting operations of the coatings and bulk materials on laboratory scale to evaluate their behaviour during repetitive impact-induced damage with time has been installed recently at ARCI. The nanocomposite coatings with 2 µm thickness, deposited on highly polished ( $R_a=0.01\ \mu\text{m}$ ) high speed steel specimens by Cathodic Arc Physical Vapour Deposition (CAPVD) process, have been subjected to cyclic impact tests at a predetermined frequency of 0.25 Hz for a set time of 300 seconds using Vickers indenter. The load applied ranges in the steps of 30, 50, 70, 100, and 150 mN. A small clearance between the specimen and the indenter is maintained in order for the indenter to travel for the purpose of making the sudden impact on the surface of the specimen whose performance is to be evaluated. TiN coatings of identical thickness (2 µm) have also been evaluated by such tests for the purpose of comparison with nanocomposite coatings. The hardness values of nanocomposite and TiN coatings respectively are 40 GPa and 24 GPa while their toughness values are 5.4 and 7.8 MPa.m<sup>0.5</sup>.

Figure 1 shows the schematic of the cyclic indentation test while Figure 2 and Figure 3 show the time-depth curves at different applied loads for TiN and nanocomposite coatings respectively. While the initial increase in the curve shows plastic deformation of the coating material, the smooth progression of the curve indicates fatigue resistance of the coatings to the cyclic indentation into the surface of the specimen with minor or no change in depth of penetration. An abrupt change in the penetration depth as signalled by a sudden jump in the curve (such as shown in enlarged view in Figure 3b for nanocomposite coating at 150 mN) indicates occurrence of a fracture event in the coating. From the Figures, it is clear that the nanocomposite coatings, being harder and brittle, have failed at 150 mN whereas the softer and

tougher TiN coatings do not show any significant failure at any of the applied loads. These results indicate that the nanocomposite coatings despite their superhardness are to be modified to improve their toughness in order that they perform better than TiN coatings in machining operations such as milling, especially, at higher operating load environment.

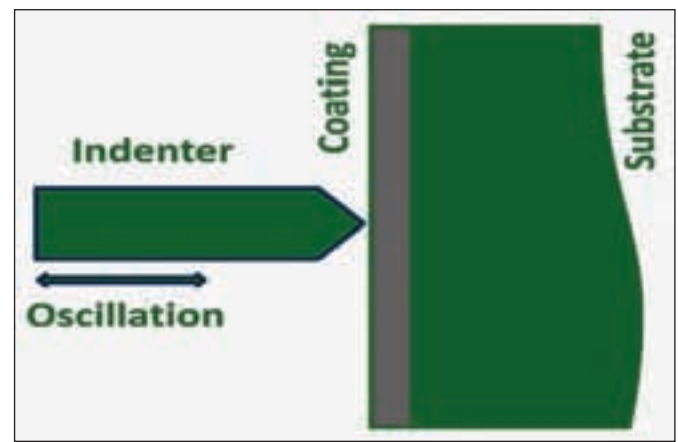


Fig. 1 Schematic diagram of cyclic nanoimpact test

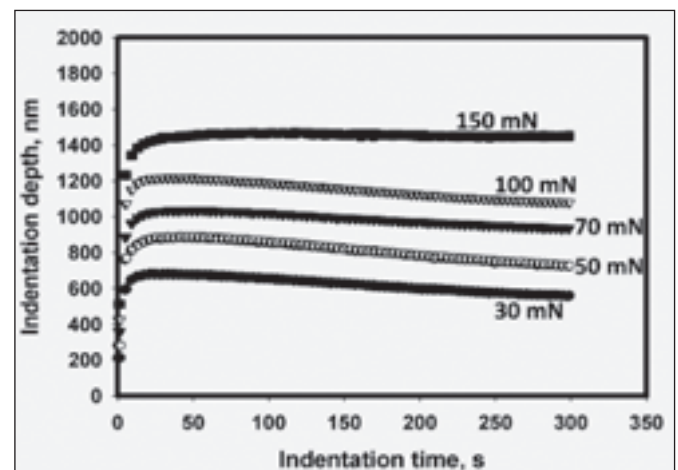


Fig. 2 Time-Depth curve for TiN coating at different loads

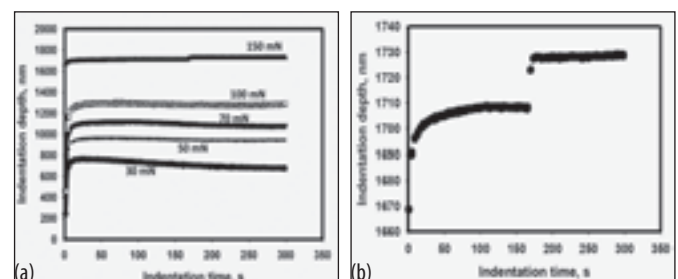


Fig. 3 (a) Time-depth curve for nanocomposite coating at different loads and (b) enlarged view of the curve at 150 mN

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# Hydrogen Production using a Single Channel with Cavities Plate Type Coated

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There is a greater interest in developing portable power sources capable of delivering power in 0.1-100 W range for potential uses in small electronic devices through the use of fuel cells which typically possess high-energy efficiency and are eco-friendly systems. For continuous operation, these fuel cells need a continuous supply of hydrogen gas which is considered to be the fuel for future. Hydrogen based Proton Exchange Membrane Fuel Cells (PEMFCs) are known for its higher energy density and require simple reformer systems for continuous hydrogen supply. It is therefore, preferable to have lightweight reformers as hydrogen feeder for the PEMFCs. Two types of reformers based on the catalyst layout are available including the conventional packed-bed type and reformers with wall coated or suspended catalyst layer. The wall coated reformers are reported to have an advantage of low pumping power without compromising on reforming performance, faster chemical reaction rate and also higher surface to volume ratio, compared to the traditional reformers. A reformer utilizing a composite CuO/ZnO/Al<sub>2</sub>O<sub>3</sub> catalyst was found to enable hydrogen production at relatively lower temperature than that of commercial catalysts. For a novel designed micro-reformer, Solution Precursor Plasma Spray (SPPS) technique was used for depositing the required catalyst coating for its reformation process.

In order to deposit catalyst films by the SPPS technique, the aqueous based precursor solution was prepared by mixing respective metal nitrate salts in distilled water. Zinc nitrate, cupric nitrate and aluminium nitrate were mixed at appropriate stoichiometry at room temperature, which was maintained at a concentration of 500 mM. The contents were stirred continuously for about 1 hour to ensure better homogeneity of metal salts within the precursor solution. A plasma power of about 42 kW was used for deposition on the pre-heated, grooved substrates, after subjecting the substrate to grit blasting, and thorough cleaning in ultrasonic cleaner. The injected precursor droplets undergo various stages of transition from a liquid into a solid deposit within few milliseconds and the thickness built up takes place through accumulation of fine-sized splats.

The top surface morphology of the as-deposited SPPS CuO-ZnO-Al<sub>2</sub>O<sub>3</sub> layer at varied magnifications is shown in Figure 1. It is clear that the surface of the deposits comprises of finely sized grains with uniformly distributed pores. The combination of pores and fine-sized features provides higher surface area and is essential to provide enhanced reactivity during

reformation, which can be realized through SPPS deposition. The phase analysis was carried out to assess the phases formed in the coating, as illustrated in Figure 2. It clearly revealed the presence of the individual CuO, ZnO and Al<sub>2</sub>O<sub>3</sub> peaks. It is also pertinent to note that the coating was nanocrystalline in nature, which can be understood from the broadening of peaks as shown in Fig. 2. Though, the compositional quantification was not possible, the elemental analysis carried out through EDS also confirmed the presence of above elements.

Methanol reforming refers to the chemical reaction between methanol (CH<sub>3</sub>OH) and water vapour (H<sub>2</sub>O) for the production of hydrogen (H<sub>2</sub>) gas. This process is typically carried out in the presence of metal oxide catalyst at temperature ranging from 195°C to 250°C. The performance of the microreformer unit in terms of methanol conversion rate and hydrogen production rate was measured at different inlet feed flow rates. The maximum hydrogen production rate of 0.9 mole/hr was obtained at 240°C for an inlet feed rate of 50 ml/hr and the methanol conversion for this operating condition was 64%. Assuming fuel cell efficiency of 70% and hydrogen utilization of 80%, the estimated electric power produced from this microreformer unit corresponds to 29W. A new deposition technology through SPPS was attempted for the first-time and could successfully be implemented for catalyst coating on SS substrate. The SPPS can be seen as a potential technique for catalyst film deposition meant for methanol steam reforming.

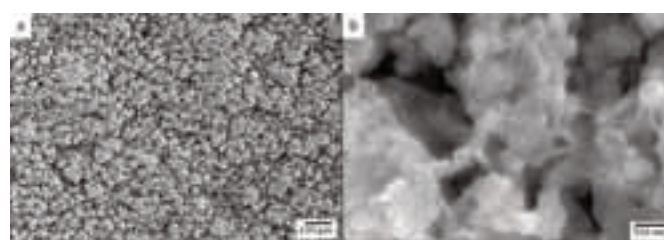


Fig. 1 Top surface morphology of SPPS deposited CuO-ZnO-Al<sub>2</sub>O<sub>3</sub> catalyst coating

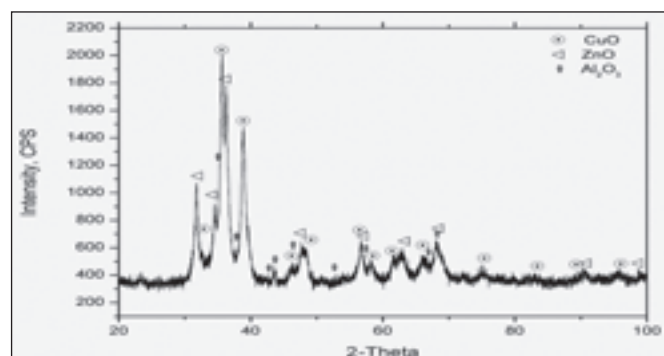


Fig. 2 Phase analysis of SPPS deposited CuO-ZnO-Al<sub>2</sub>O<sub>3</sub> catalyst coating

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# Comparative Dry Sliding Wear Performance of Electrodeposited Ni-W Alloy and Hard Chrome Coatings

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Electrodeposition has, over recent decades, evolved from an exact science to an economically viable technology for developing variety of coatings. Electrodeposition is economically important because of its low cost and technical flexibility. The ability to deposit a wide array of metals, alloys and composite materials by electrodeposition has graduated from a laboratory scale phenomenon to a practical industrial technology.

Pulsed Electrodeposition (PED), in which the current is imposed in a periodic manner with a rectangular waveform, is a powerful means of controlling the electrocrystallization process to produce nanocrystalline deposits with unique structure-property combination. In recent years therefore, many nanocrystalline metals, alloys and composites have been produced by PED successfully. With the ongoing research on finding suitable alternative for hard chrome replacement, ARCI has developed nanocrystalline Ni-W alloy coatings using PED. These coatings developed at ARCI have been found to exhibit superior corrosion resistance utilizing polarization and salt spray tests (ASTM B117). The present investigation deals with the feasibility studies on pulsed electrodeposited nanocrystalline Ni-W alloy coatings for hard chrome replacement in terms of dry sliding wear resistance.

Nanocrystalline Ni-W alloy coatings were pulsed electrodeposited using citrate bath on mild steel substrate. While, the hard chrome (HCr) coatings were obtained from a commercial source and used for relative comparison. As illustrated in Figure 1, Ni-W alloy coatings are found to be dense and devoid of micro cracks in contrast to hard chrome. The tungsten content of Ni-W alloy coatings was 10.5 (at) % with the hardness of 750 HV while the hardness of HCr is about 850 HV. The sliding wear was carried out using pin (Ni-W and HCr coated samples) on disc (WC-Co, 1750HV) configuration as per ASTM standard G99 at three different loads viz 10, 30 and 50 N.

The steady state wear rate is presented in Figure 2. Ni-W alloy coating is found to have less wear rate compared to HCr coatings at all loads. The improved wear resistance of Ni-W was attributed to its dense structure thereby leading to ductile wear. Whereas HCr coatings with numerous cracks lead to brittle fracture and higher wear rates.

Therefore, the present work clearly demonstrates the superior wear resistance of PED Ni-W alloy coatings in comparison to

hard chrome coatings. In conclusion, pulsed electrodeposited Ni-W coating can be considered as a potential candidate for replacing HCr coatings.

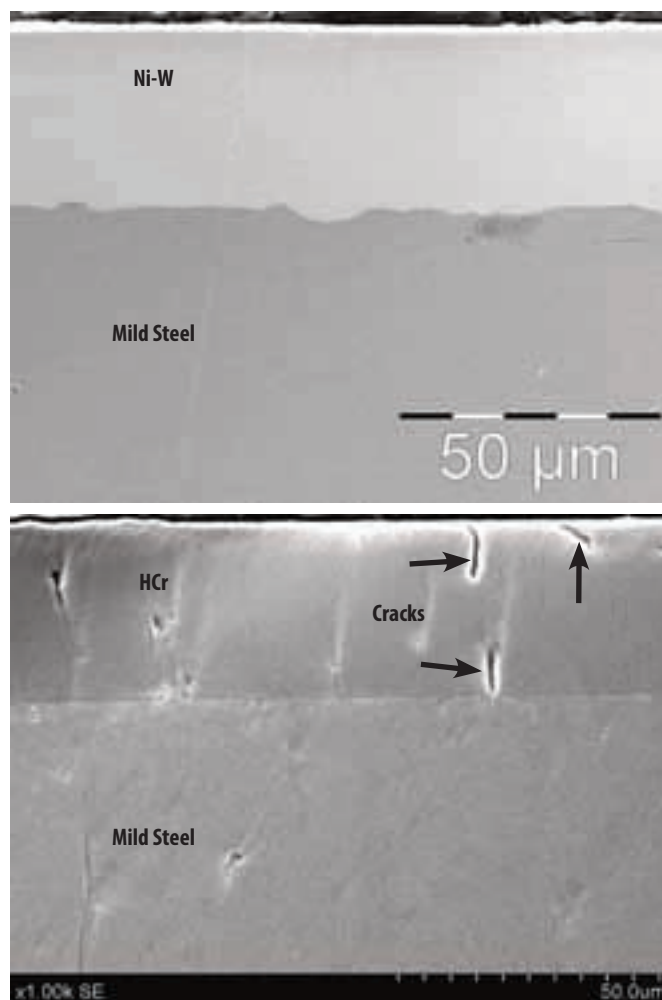


Fig. 1 SEM micrographs comparing Ni-W alloy and hard chrome coating cross section

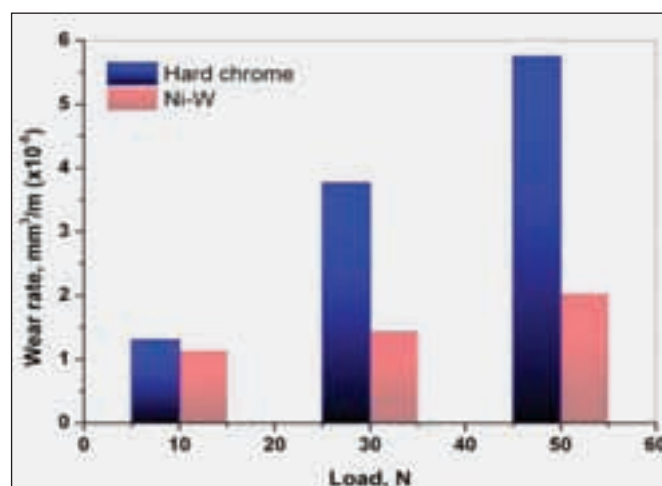


Fig. 2 Wear rate as a function of applied load

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# Effect of Pretreatment Processes on the Performance of Cathodic Arc Physical Vapour Deposition Coated HSS Twist Drills

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The development of hard coatings on cutting tools for providing a viable solution to the constantly growing need of high speed, MQL (minimum quantity lubrication) machining has been gaining interest in the recent years. Cathodic Arc based Physical Vapour Deposition (CAPVD) coatings have superior properties viz. excellent adhesion (due to ionized incoming flux) and high deposition rate over the other PVD techniques like magnetron sputter deposition, vacuum evaporation and ion plating. Therefore, the tools processed by this technique have garnered widespread interest and industrial viability. Since decades, owing to excellent wear and oxidation resistance, the cathodic arc deposited hard metal nitride coatings like Titanium Nitride (TiN) and Titanium Aluminum Nitride (TiAlN) have been most popular coatings employed for cutting tools. In order to improve tool life, surface treatment/cutting edge preparation is very essential prior to deposition of any coatings. Among the various pre-treatment methods the most popular ones are: Grinding, micro-blasting (wet/dry), drag finishing, magnetic polishing, abrasive water jet blasting, electro-polishing and laser texturing. In the present study, the effect of micro-blasting (with different particle size and shape) and edge rounding (for different rounding values) on TiN coated High speed steel (HSS) twist drill performance on EN24 work piece was studied and the conditions leading to the best tool life were obtained.

Figure 1 demonstrates the real time performance of TiN coated tools which are subjected to different blasting

durations. From the results it is observed that, the tools micro-blasted with  $15\ \mu\text{m}$   $\text{Al}_2\text{O}_3$  particles for 30 seconds have shown significantly enhanced tool life. With the increase in particle size of  $\text{Al}_2\text{O}_3$ , there is a considerable increase in surface roughness with reduced performance. The poor performance of the TiN coated tools may be attributed to the change in surface roughness.

The effect of edge rounding obtained by drag finishing in  $\text{SiO}_2$  media on cutting tools is shown in Figure 2. From the Figure it is observed that, there is a critical edge rounding (cutting edge radius of curvature) below or above which tool life is hampered notably. In the present study, the critical edge rounding seems to be  $\sim 15\ \mu\text{m}$  which corresponds to 10 min of drag finishing.

Further, to study the effect of Micro-blasting (MB) and Drag Finishing (DF) together on tool life, the best parameters concluding the study of micro-blasting and drag finishing were taken and drill bits were processed. The real life performance results shown in Fig. 3 indicate a considerable increase in the tool life with MB+DF.

In conclusion, employing MB and DF together have showed an excellent increase in tool life i.e.  $\sim 20$  times that of uncoated tool life. Therefore, it is envisaged that pretreatment is very essential for cutting tools prior to any protective coating deposition.

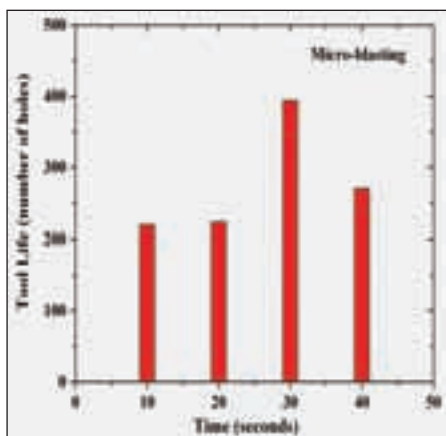


Fig. 1 6 mm TiN coated HSS twist drill performance (18 mm blind holes) with different blasting times using  $15\ \mu\text{m}$   $\text{Al}_2\text{O}_3$  powder

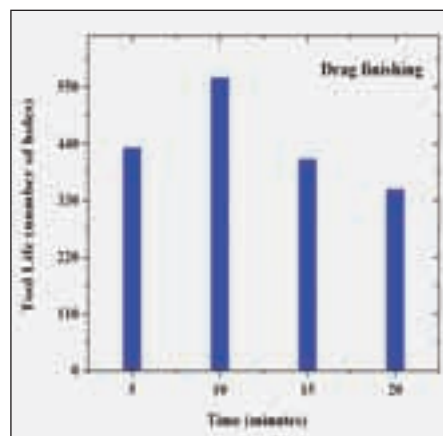


Fig. 2 6 mm TiN coated HSS twist drill performance (18 mm blind holes) with different drag finishing times using  $50\ \mu\text{m}$  particle size  $\text{SiO}_2$  powder

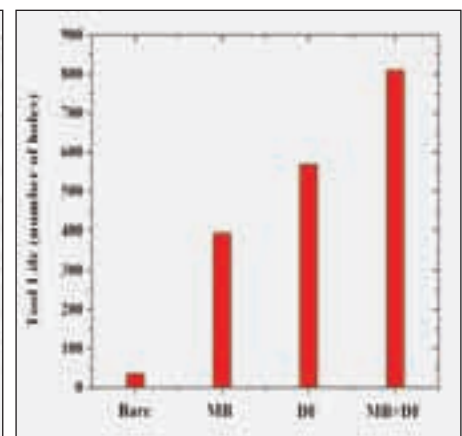


Fig. 3 6 mm TiN coated HSS twist drill performance (18 mm blind holes) with different pretreatment methods: Bare (without any pretreatment or coating), Micro-blasting (MB), Drag finishing (DF) and MB+DF

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# Deposition Features of Cold Sprayed Metal Matrix Composite Coatings

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Since the bonding / deposition mechanism is influenced by severe plastic deformation induced adiabatic heating of impacting bodies at interfaces, cold spray cannot be used to deposit materials prone to fracture such as cermets and ceramics. In cold spray, when a hard body impacts with softer body, the degree of plastic deformation is more in the softer body which leads to interface melting. Also, it was reported that degree of plastic deformation is more when the particle is deformed under successive impacts. Co-depositing ceramics / hard particles along with soft metals using cold spray technique has been demonstrated by many authors. The splats from soft feedstock can be acted as a binder in the coating. One needs to understand the physical properties such as density, size etc. of the feedstock properly for obtaining homogeneous distribution of particulates through cold spray. Prior to coating, a careful tailoring of feedstock is important to get desired coatings. It is also reported that presence of ceramic particle along with metal feedstock triggers the densification due to the activation of surfaces by tamping effect. The major challenge in depositing metal matrix composite using cold spray is to retain the amount of ceramic particles in the deposits and maintain the homogeneous distribution of ceramic particles in the composite coating.

Al-SiC system has been considered for carrying out scientific investigations keeping the possible applications in mind. The selection of initial feedstock ensures that Al and SiC attain the same velocity range upon impact on to the substrate while spraying, which leads to uniform distribution of SiC in the coating. Three powder mixtures were prepared having different SiC Wt% (Al25SiC, Al50SiC and Al75SiC) in the feedstock. Figure 1a shows the SEM cross section image of the composite coatings (50:50 wt %) which reveals the uniform distribution of SiC particles in aluminum matrix. High magnification images revealed that the particle size SiC in the coating is finer as compared to that of initial feedstock. Using image analyzer, the average size and the area percentage of SiC particulates in the deposits were estimated and shown in Figure 1b. SiC particulates have been retained in the cold sprayed aluminum metal matrix composite coatings with reduced size due to collision upon impact with each other (SiC particles) which can be explained from the plots. The results reported clearly demonstrate that by using cold spray, it is possible to achieve homogeneously distributed

metal matrix composite coating with fine ceramic particulates. Also it is proved that the quantity of ceramic can be retained in the coating. The wear performance of the coatings at different loads is shown in Figure 1c.

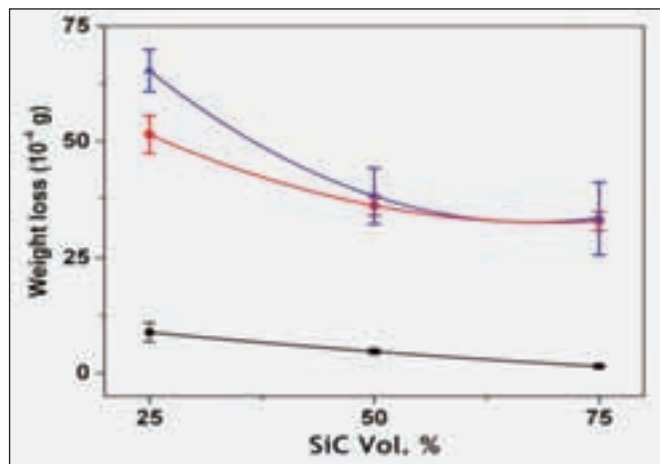
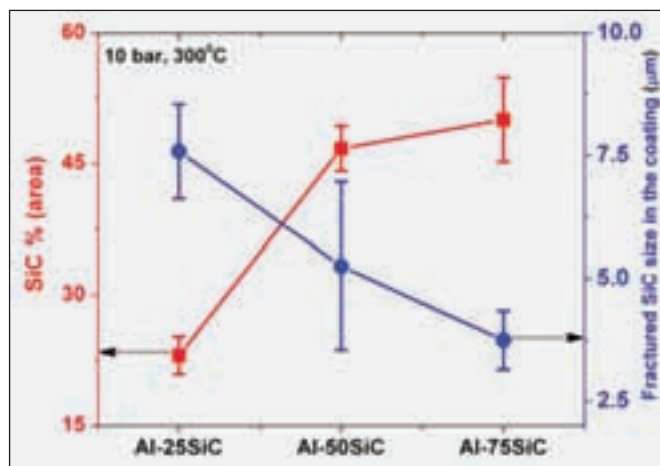
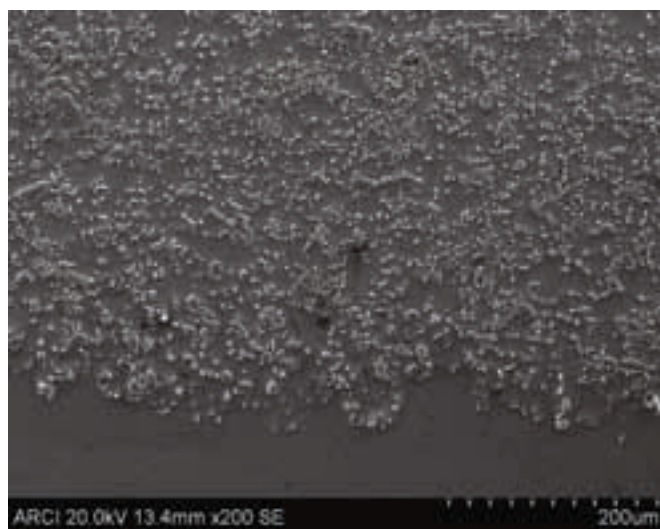


Fig.1(a) Cross-section microstructure of the coating (b) Fraction and particulate size in the coating (c) Wear performance at 3 different loads

Contributor: Sai Kiran Reddy



# Infrared Irradiation of Cold Sprayed Coatings: A Study on Microstructural Evolution and Resultant Properties

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A thick cold spray deposit consists of numerous splats (erstwhile particles) bonded to each other either mechanically or metallurgically. The coating properties depend on the soundness of the bonding between these individual splats. The coating property also depends on the deformation degree (degree of cold work) inside the individual splats. In this regard, a cold sprayed coating requires a post treatment in order to replenish the material properties and perform close to the bulk counterparts. In an industrial setup, it is cumbersome to heat treat each and every coated component in a furnace especially when the dimensions of the coated sample are long. Hence, there is a need for a mobile heating device, wherein coatings can be post treated (for stress relief, intersplat bonding etc.) in tandem with the coating process or at least immediately after coating process. In the present report, a mobile "Infrared (IR)" heating tool for post treating cold sprayed coatings is described. All materials have a tendency to absorb radiation. The absorption of IR radiation by metals and alloys is dependent on the wavelength of the IR radiation. It is well known that metals and alloys (eg. Cu, Ni, Steel etc.) exhibit maximum absorptivity to short wave IR radiations (0.7-2  $\mu\text{m}$ ). In this article, the microstructural evolution along with property changes in IR treated Cu, Ni, Ni-Cr, IN625 coatings is investigated.

Apparently, all the cold sprayed coatings have shown better absorptivity of IR radiation than theoretically reported values especially because of the surface roughness imparted by the deposition process. This is clearly shown by the increase in electrical conductivity of the IR treated copper coatings as presented in Figure 1(a). Polished copper coating surfaces showed minimum absorptivity and almost no change in electrical conductivity. Hardness of the coatings also dropped drastically as a function of IR treatment as shown in Figure 1(b) when compared to as coated (AC) and IR treated samples. The observed changes in the coating properties was consistent with the microstructural changes. Figure 1c and 1d show the etched microstructure of the crosssection of cold sprayed copper coating both in as coated and IR treated condition (9 kW and 5 minutes), it can be observed that the splat boundaries have diffused and the interior of the splat has been annealed with the emergence of coarser and equiaxed grains.

In addition to copper, recent investigation on the IR treatment of other cold sprayed coatings has shown that there was significant absorption and the same was evident from the change in hardness of the coatings as shown in Figure 2(a). Figure 2(b) shows the porosity data as well for the coatings other than copper that were IR treated. The porosity also exhibits a specific trend of reduction upon post treatment. The microstructure of all coatings showed healed intersplat boundary network to varying degrees.

It can be concluded from this study that cold sprayed coatings can be post treated (especially stress relief annealed) to replenish certain properties of interest which can be a value addition. Further detailed studies are required to understand the healing process between splat to splat interfaces.

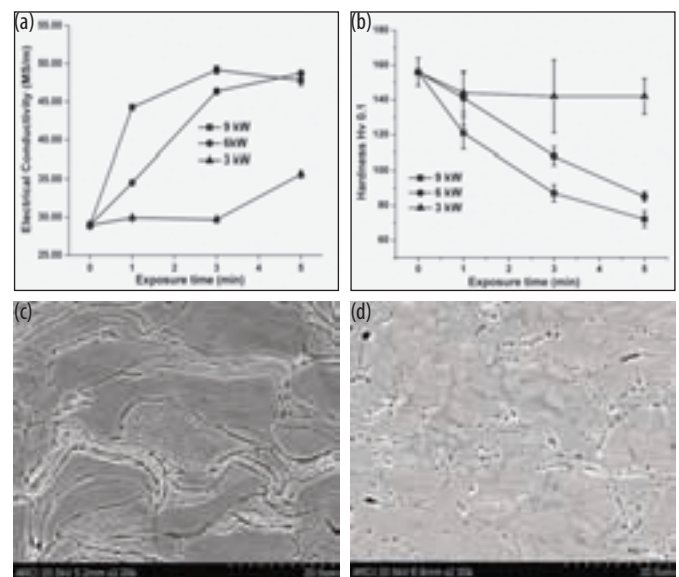


Fig.1 (a,b) Variation of electrical conductivity and hardness with IR power and exposure time (c,d) etched sectioned micrographs of As coated and IR treated (at 9 kW for 5 minutes) copper coatings

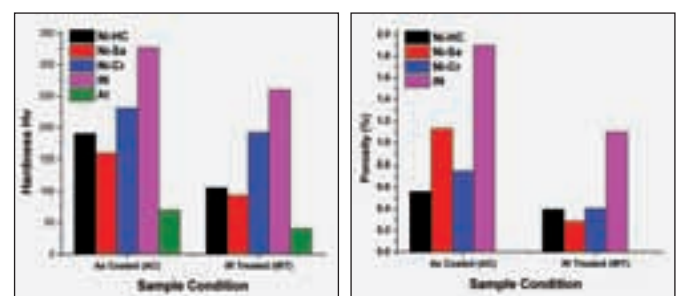


Fig. 2 (a) Microhardness (b) Porosity in as coated (AC) and IR treated (HT) conditions for different cold sprayed coatings

Contributor: M Pooja



## Centre for Ceramic Processing

*Unique combination of material properties has made advanced ceramics as one of the most efficient and interesting domain. However, the performance of these multifunctional materials is a strong function of the processing techniques employed. Centre for Ceramic Processing, with its vast experience and understanding in designing and developing ceramic components from simple to complex shapes and large sizes, has extended its expertise to meet user demand from time to time. In addition to the continued efforts to fulfill the commitments on the development and supply of deliverables of the sponsored programmes, the centre has initiated activities in few new areas such as thermal management, solid electrolytes and ceramic porous burners. Defect free processing through innovative ceramic forming processes being the core competence area, the centre has continued to pursue the activities in this direction through Chemical Vapour Deposition (CVD), Hot Isostatic Pressing (HIP) and Pressure Slip Casting. Centre has also demonstrated its defect free processing capability through successful fabrication of transparent ceramic components.*

*Centre has executed several job orders and fabricated prototypes demonstrating the technologies developed. As a part of international collaboration, the Centre has successfully completed a project on the development of thermal shock resistant cordierite-mullite formulations for the fabrication of crucibles under Indo-French Centre for the Promotion of Advanced Research. Centre was a part of organizing the ceramic session on “Ceramics for Health, Energy and Environment” under Indo-German Frontiers of Engineering (INDGFOE).*

*Centre has also initiated collaborative activities with Institute of Nuclear Medicine & Allied Sciences (INMAS) in the area of bio-ceramics and reported spinel as the biocompatible material as well as prospective transparent invisible orthodontic brackets. Centre is in the process of creating the facility for powder encapsulation through vacuum canning followed by HIPing for single step near net shaping of ceramics.*

# In-vivo Biocompatibility Studies of Spinel Ceramics

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Biocompatibility refers to the interaction of a living system with a material or a device. A biocompatible material is expected to be non-toxic, non injurious or not causing immunological rejection in a living system. Biocompatibility of a material is usually studied through in-vitro (experiments outside the living organism) in a controlled environment and in-vivo (experiments within the living organism).

Magnesium Aluminate Spinel ( $MgAl_2O_4$ ) is scientifically and technologically important ceramic due to wide range of applications in thermostructural implementations.  $MgAl_2O_4$  has also recently generated considerable interest as a transparent armour due to its inherent optical isotropy resulting from the cubic crystallography in combination with superior mechanical properties. The above attributes of spinel along with ease in machining without affecting the transparency, offers favourable opportunities in developing invisible orthodontic brackets. However, no data is available on the biocompatibility features of the spinel and the study is to evaluate the physico-chemical, optical and biocompatibility properties for assessing its feasibility for using as an orthodontic bracket material

Sintered magnesium aluminate ( $MgAl_2O_4$ ) spinel ceramic produced at ARCI has recently been confirmed for its biocompatibility based on the in-vitro studies carried out at the Institute of Nuclear Medicine and Allied Sciences, (INMAS), Defence Research and Development Organization (DRDO), Delhi. These studies evaluated the cytotoxicity of the material on human embryonic kidney cell lines in addition to its propensity towards inducing apoptosis and genotoxicity. Spinel exhibited excellent biocompatibility at par with known bioceramics. Currently, in-vivo studies on two animal models: Sprague Dawley (SD) rats and New Zealand White rabbits are in progress at INMAS to generate conclusive insight into the behavior of spinel on living systems.

For the in-vivo studies, the spinel samples were prepared at ARCI from commercially available 99.96% pure spinel powder. A slurry of spinel powder was made in the aqueous medium and cast into specimens of 50 x 50 x 5 mm thick samples. The green specimens were then fired at a peak temperature of 1650°C in an air sintering furnace. The physical properties of the samples were determined prior to the bio-compatibility studies (Table 1). Figure 1 shows the placement of samples in the rabbits.



Fig. 1 Placement of samples on Rabbit

Table 1 Physical properties of sintered spinel

| Property                         | Values/ Observations |
|----------------------------------|----------------------|
| Density                          | 98%                  |
| XRD Phases                       | $MgAl_2O_4$          |
| Hardness (GPa)                   | 12                   |
| Fracture Toughness $MPa.m^{1/2}$ | 1.90 + 0.1           |
| Flexural strength MPa            | 200 + 10             |

Spinel samples were cut and inserted into two sites of the rats: (i) parietal bone of skull and (ii) intra orally near mental foramen in the buccal side of mandible (Jaw bone). Spinel samples inserted at both the sites are shown in Figure 2(a) and (b). Post operatively, the animals are presently kept up to 12 weeks. Sequentially, they are being sacrificed at 2, 4, 6 and 12 weeks to evaluate the host tissue response and bone forming events adjacent to spinel using bone formation indices and histopathology.

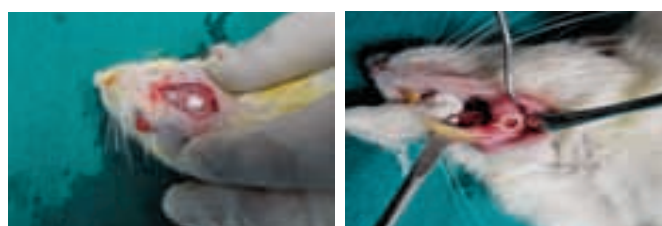


Fig. 2 Placement of samples on Sprague Dawley Rat (a) Parietal and (b) Mandible

Prior to sacrificing, animals are also injected with radiographic tracers like 18-Fludeoxy glucose (18-FDG) and Technetium 99 (99mTc). These tracers show bone formation or osteoblastic activity in Positron Emission Tomography (PET) and Single Photon Emission Computed Tomography (SPECT) exposures, respectively.

Preliminary results of histopathology and radiographic imaging reinforce the in-vitro biocompatibility of spinel and have opened up exciting options for this material as a new bio-ceramic for multiple medical applications.

Contributor: Manu Krishnan, Institute of Nuclear Medicine & Allied Sciences, DRDO



# High Coercive Nano Sr-Hexa Ferrite Powders by Pyrolysis for Hard Magnet Applications

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Strontium hexaferrite (SF), an important compound for permanent magnet based applications, is normally prepared by the conventional solid state synthesis route. This involves the calcination of the stoichiometric mixture, consisting of precursor oxides or salts, at temperatures  $\geq 1100^\circ\text{C}$ . In addition, soft chemistry routes such as co-precipitation, mechano-chemical and combustion synthesis also have been exploited. However, the important parameter from application point of view, i.e, the Coercivity ( $H_c$ ) has been low, about  $\leq 5$  kOe limiting the application fields. Taking into consideration the advantages of SF with high coercivity close to theoretical values ( $> 5$  or approaching 6.8 kOe) for permanent magnetic applications, series of stoichiometric compositions with combustion synthesis have been explored.

Present investigation reports the synthesis of SF with coercivity close to theoretical values through low combustion synthesis from the corresponding precursor salts. These salts in aqueous solutions according to stoichiometry were mixed with a fuel and the combustion synthesis was carried out at optimum temperature established through thermal analysis. In this process nano powders of average size  $< 50\text{nm}$  with established Strontium hexa ferrite phase (within the limits of detection by XRD) were produced in different batches of 100-250 gm consistently and repeatedly.

Figure 1 presents the XRD patterns recorded for the nano SF powders produced from the above cited combustion experiments calcined at different temperatures and that of compacted discs fired at higher temperatures. It can be seen that within the limits of detection, the XRD pattern clearly indicates the formation of SF phase. The purity at elemental level has been confirmed with EDS. The morphology of the nano SF powders produced in a representative scan of FESEM study is shown in Figure 2. From Figure 2, it is clear that the average particle size of SF powders is  $\sim 50$  nm; however, the particles are highly agglomerated. To ascertain the suitability of the nano SF powders produced for the permanent magnet applications, critical magnetic properties were also determined and a typical hysteresis curve is shown in Figure 3. The coercivity of 5.8kOe, 37 & 19 emu/g of  $M_{\text{Sat}}$  &  $B_r$  are encouraging to pursue further work on this material.

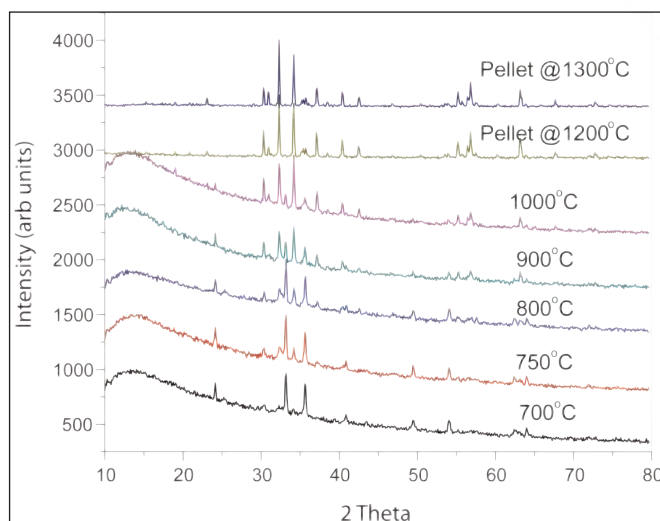


Fig. 1 Phase establishment of nano Sr-hexa ferrite as seen by the XRD profiles of powders and compacts

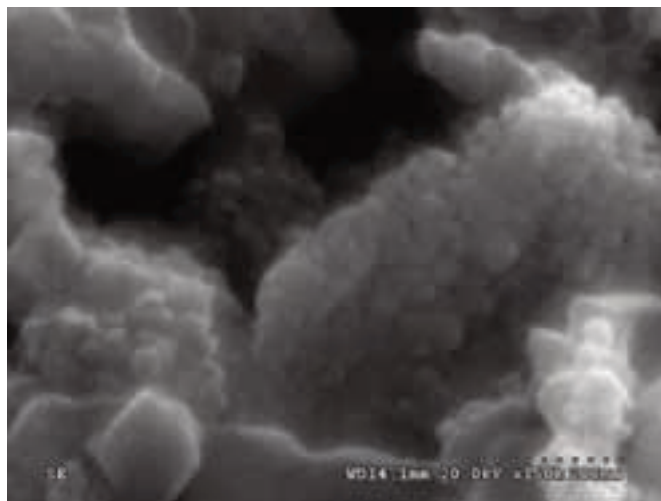


Fig. 2 FE SEM picture clearly bring out the nano size of the SF powders

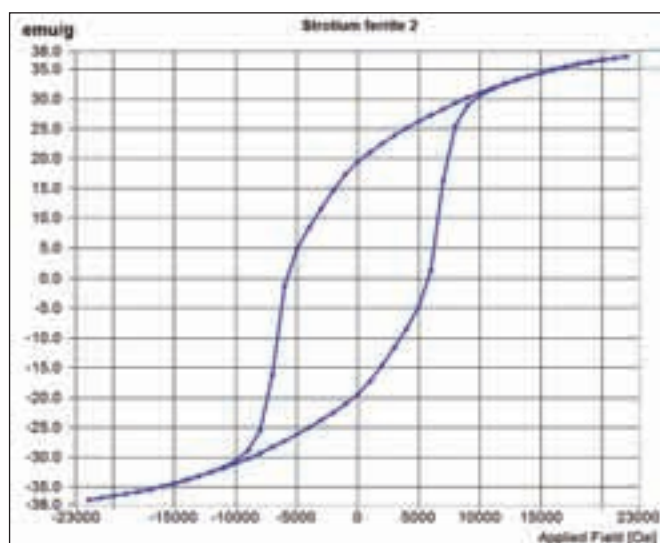


Fig.3 A typical hysteresis curve for Nano SF sample at CAEM, Chennai

Contributor: Roy Johnson

# Extrusion Processing of Sodium Beta Alumina Tubes and Conductivity Measurements

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Among the energy storage technologies, sodium-sulfur (Na-S) batteries are identified as a high efficiency and large duration devices. Sodium beta alumina (NBA) solid electrolyte is a key material that separates anode and cathode and acts as fast ion conductor.

In the present study precursor mix of alumina and sodium oxalate along with the sintering additives such as magnesium hydroxide were mixed according to the NBA stoichiometry (NBA-1S). A part of the precursor raw mix was calcined at 1650°C in order to ensure formation of NBA phase (NBA-2S) as confirmed by XRD pattern depicted in Figure 1. Moreover, the raw mix and the NBA powder were mixed with binder and extruded into tubes.

Typical load versus displacement curves are shown in Figure 2(a). The extruded tubes are also shown in Figure 2(b). Figure 2(a) indicates that calcination enhances the load required for extrusion considerably. However, the extrusion process is almost consistent in both cases showing an identical behavior after the ejection of extrudate. This can be attributed to the surface morphology of the calcined powder where there is an increase in the inter-particle friction and formation of occasional agglomerates. Additionally, the preformed NBA powder was also compacted into pellets and sintered for impedance measurements. The conductivity of sintered samples of NBA-1S and NBA-2S as a function of frequency is shown in Figure 3. AC conductivity increased marginally with frequency in the low frequency region and increased significantly in the high frequency region. In comparison to NBA-1S, NBA-2S sample has shown higher value of AC conductivity at RT ( $1.77 \times 10^{-4}$  S/cm at 10MHz) at all the frequencies. This may be due to enhanced NBA phase purity and crystallinity in combination with relatively higher density. Samples were also tested for high temperature conductivity with varying frequency. The samples exhibited an increase in the conductivity as a function of temperature. The Cole-Cole plot exhibited the contribution of grain and grain boundary conduction for the total conductivity. The prominent mechanism of conduction in the present study is found to be due to grains/grain boundary.

Sodium beta alumina synthesized in the present study is being subjected to shaping in order to explore the suitability of application as a solid electrolyte for high energy Na-S batteries.

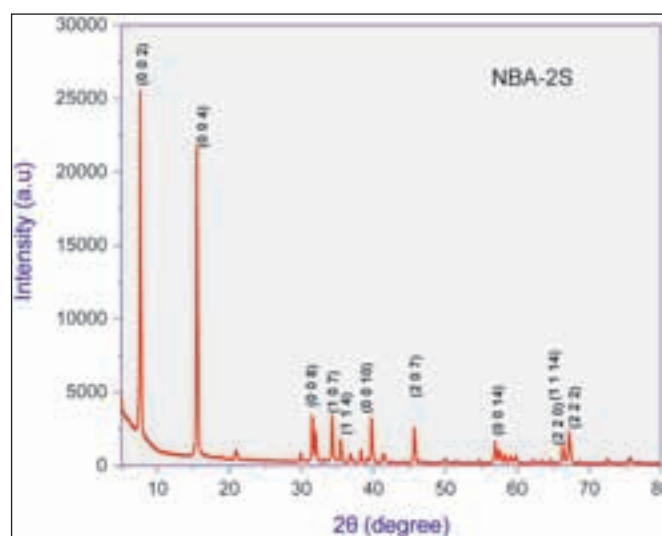


Fig. 1 XRD Pattern of NBA-2S sample

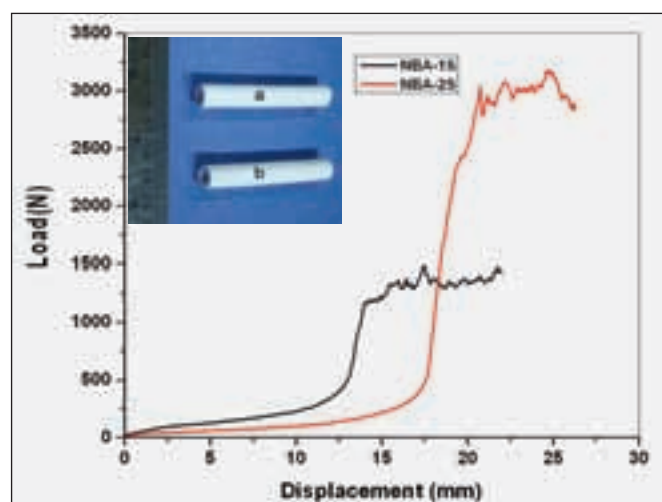


Fig. 2 Load versus displacement curves with in-situ extruded Tubes of NBA-1S and NBA-2S

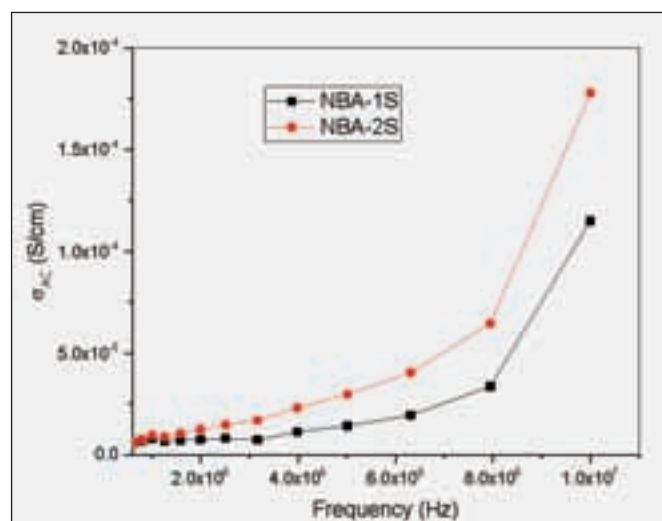


Fig. 3 AC conductivity variation with frequency of NBA-1S and NBA-2S

Contributors: K Avinash and Roy Johnson

# Effect of Additives on the Densification and Microstructural Control of Nano Structured Sol-gel Processed Alumina

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Dense alumina with nano structure exhibits superior mechanical properties over micron sized alumina in properties such as hardness, flexural strength and hence is ideal for various applications such as wear resistant components, orthodontic parts, cutting tools etc. Selection of starting powders, addition of grain growth inhibitors and optimum sintering parameters are the key functions to control the sintered grains in nano ranges. In the present investigation nano alumina powder has been produced through sol-gel method by dispersing boehmite powder in water at the weight ratio of 1:9. This dispersion was further peptized with dilute nitric acid for producing stable boehmite sol. Defined quantity of sintering additives or microstructure pinning agents such as MgO, TiO<sub>2</sub>, MgO+ZrO<sub>2</sub>, La<sub>2</sub>O<sub>3</sub> are added at this stage. In addition, about 10% of alpha phase alumina seeds with an average particle size of 200nm was added in the sol to obtain  $\alpha$  phase formation of sol-gel alumina at lower temperatures and to improve the densification tendency. The sol was stirred vigorously for 30 minutes followed by addition of ammonia for gelation and the gel was calcined at 1000°C to produce alpha phase alumina powder. The powder was milled with suitable binder such as poly vinyl pyrrolidone and shaped through uniaxial compaction followed by cold isostatic compaction at 200 MPa. The samples were sintered at temperatures between 1200°C and 1400°C for 1 hour in air atmosphere to achieve densification.

It is evident from the Figure 1 that the peaks corresponds to boehmite and also peaks of  $\alpha$ -phase alumina seeds are present. An intermittent  $\gamma$ -phase alumina is formed at 600°C and transforms to  $\alpha$ -phase. In order to retain the traces of  $\gamma$ -alumina, the calcination temperature was limited to 1000°C. This will enhance the final densification and to avoid the formation of hard granules. Figure 2 shows the comparison of densities, which increase from 90% T.D to 98% T.D as the temperature increased from 1200°C to 1400°C. Sintering additives have shown no significant difference in final densities at 1400°C; however exhibited significant effect on microstructure control. Addition of TiO<sub>2</sub> resulted in grain size around 1  $\mu$ m. However, MgO and La<sub>2</sub>O<sub>3</sub> resulted in grain sizes of 700nm and 640nm respectively. Addition of nano ZrO<sub>2</sub> with MgO showed substantial grain pinning reaching the mean size of 600nm.

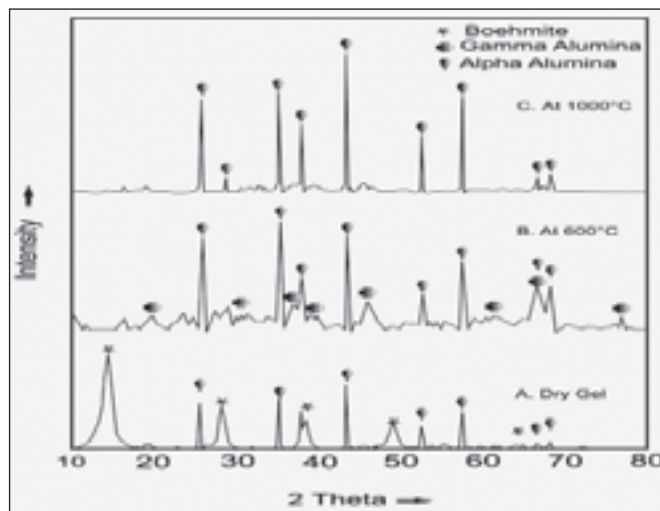


Fig.1 XRD pattern for the phase formation of alumina from boehmite

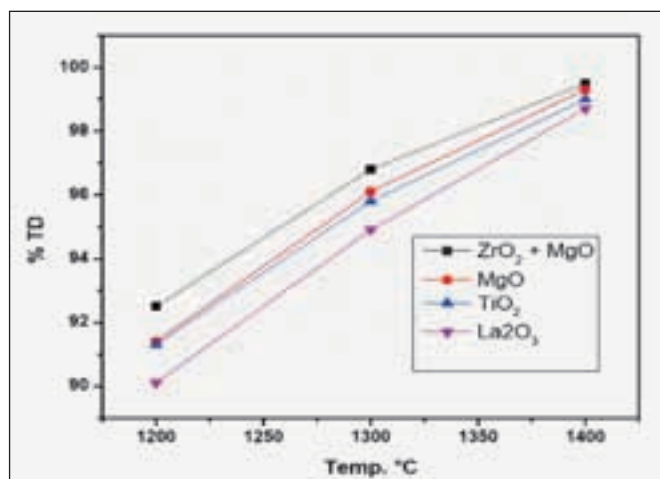


Fig.2 Sintering temperature Vs % theoretical densities for various sintering additives such as La<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, MgO and ZrO<sub>2</sub>+MgO

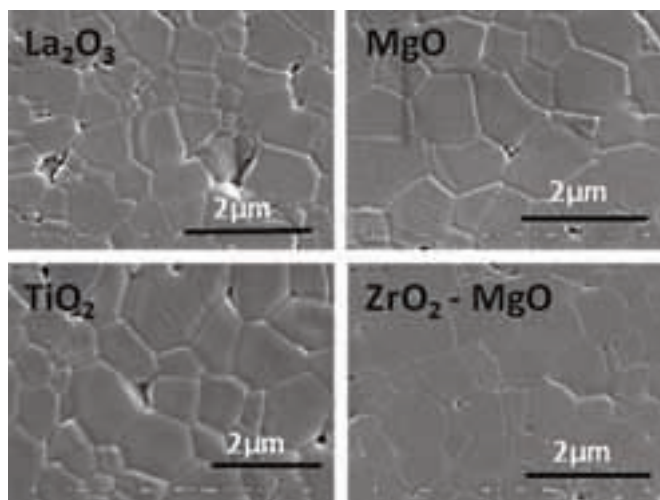


Fig.3 Microstructures of alumina sintered at 1400°C for 1 hour using various sintering additives such as La<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, MgO and ZrO<sub>2</sub>+MgO

Contributor: Roy Johnson



# Sonochemical Synthesis of Hydroxyapatite and Fabrication of Reticulated Cellular Foams

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Hydroxyapatite (HAp) on account of the similarity in mineral constituents with natural bone and teeth is widely used for biomedical applications. Various property requirements of HAp powder for applications and adaptability for shaping techniques depends on precise compositional control, particle size, morphologies, etc., which can be tailored using innovative synthesis techniques. Sonochemical synthesis employing acoustic cavitations to engineer the reactions are gaining interest due to increase in reaction kinetics by activation of reagents leading to efficient energy usage. Sonochemistry also permits to control the morphology of nanoparticles. In the present study, stoichiometric proportions of analytical grade Calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) and orthophosphoric acid ( $\text{H}_3\text{PO}_4$ ) were dissolved in water (according to the equation-1) and was irradiated using an ultrasonicator with a probe (VCX750, Sonics & Materials Inc., USA) under parameters optimized in our laboratory based on several experiments.



X-ray diffraction study confirmed HAp phase and average particle size was found to be 182 nm using photon correlation spectroscopy. Aqueous slurry of HAp powder with 55 wt% of solid loading was made with 1wt% dispersant (Darvan 821A) by ball milling for 2 hrs in polypropylene bottle in a pot jar mill. Polyurethane foam (PUF) with a pore density of 10 pore per linear inch (PPI) is coated with the homogeneous slurry. The foam with the coating of slurry on the struts was dried in an oven at 60°C followed by binder burnout and sintering at 1200°C. Typical burn out cycle for polymeric sponge and the dilatometric sintering schedule is shown in Figure 1 and 2, respectively. Typical foam produced by polymeric replication process is shown in Figure 3. HAp foam has a cell density of 13 PPI and relative density of 0.25. Foams are being characterized for mechanical properties such as compressive deformation behaviour in order to establish the suitability for scaffold applications. In addition, experiments are also in progress to simultaneously synthesize and coat HAp on bio-inert spinel foams through sonochemical process. HAp being one of the ideal bioceramic material which is processed in to reticulated foam structure is expected to be a prospective scaffold material for biomedical applications.

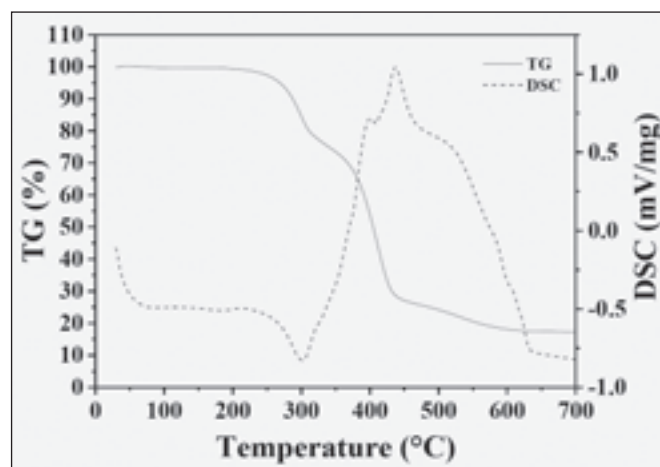


Fig. 1 TG/DSC plots of polymeric foam

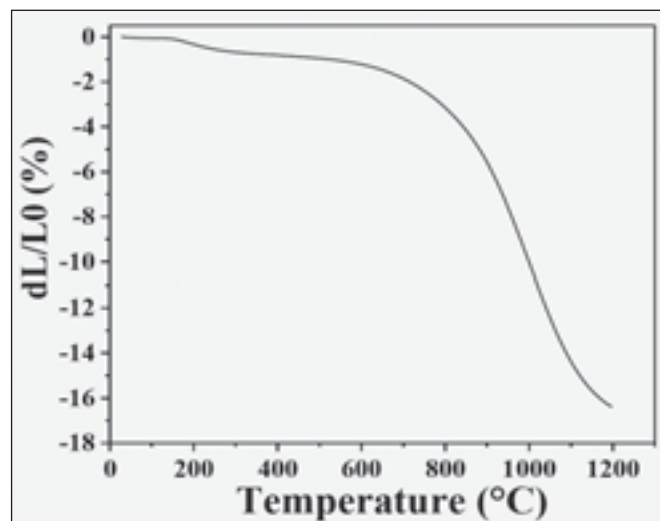


Fig. 2 Dilatometric sintering schedule of HAp sample



Fig. 3 Sintered HAp foam

Contributors: L Laxmi Sindura and Roy Johnson

# Microstructural and Transmission Properties of Hot Isostatically Pressed ZnS Powder and CVD Free Standing ZnS Ceramics

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Zinc sulphide (ZnS) is regarded as one of the potential material for electro-optic applications by virtue of its transmission properties in the visible (0.4-0.8  $\mu\text{m}$ ) through mid wave (3-5  $\mu\text{m}$ ) and long wave IR region (8 to 14  $\mu\text{m}$ ). The Chemical Vapour Deposition (CVD) is recognized as the most ideal route for the fabrication of ZnS articles. However, it requires Hot Isostatic Pressing (HIP) to make it multispectral. An alternative route to produce the transparent ZnS ceramics is through direct HIPing of ZnS powder encapsulated under vacuum in the specially designed metallic capsules. In the present study, the microstructures and transmission properties of Hot Isostatically Pressed ZnS Powder along with CVD Zinc Sulphide HIPed into free standing ceramics are compared.

CVD ZnS was fabricated using the reaction between Zinc and Hydrogen sulphide under thermodynamically favorable conditions of 650°C temperature and 50 mbar pressure. CVD ZnS samples (CVD+HIP) and Vacuum encapsulated ZnS powder cans (PRHIP) subjected to the HIP cycle employed in the present study is shown in Figure 1. Figure 2(a) and (b) shows CVDHIP and PRHIP samples after HIPing along with their microstructure.

It is evident that grain sizes were similar as is evident from the microstructures of CVDHIP and PRHIP ZnS specimens. However, the samples exhibited clear difference in morphology. CVDHIP exhibited substantial twinning, whereas such twinning was absent for PRHIP samples. Twinning of the samples can be attributed to the densification mechanisms involved. Predensified CVD samples undergo final densification by eliminating the porosity through recrystallisation whereas powder HIPing proceeds through plastic deformation.

FTIR transmission patterns in the visible, MWIR and LWIR region for CVDHIP and PRHIP samples are shown in Figure 3. PRHIP samples show lower transmission at < 70 % in the 3 to 5  $\mu\text{m}$  region. Presence of residual porosity evident from the marginally low density of 4.079 g/cc for PRHIP compared to 4.086 g/cc exhibited by CVDHIP samples are mainly responsible for the low transmission values. Figure 3 also shows 45-65% transmission for the CVDHIP sample in the visible range demonstrating multispectral capability.

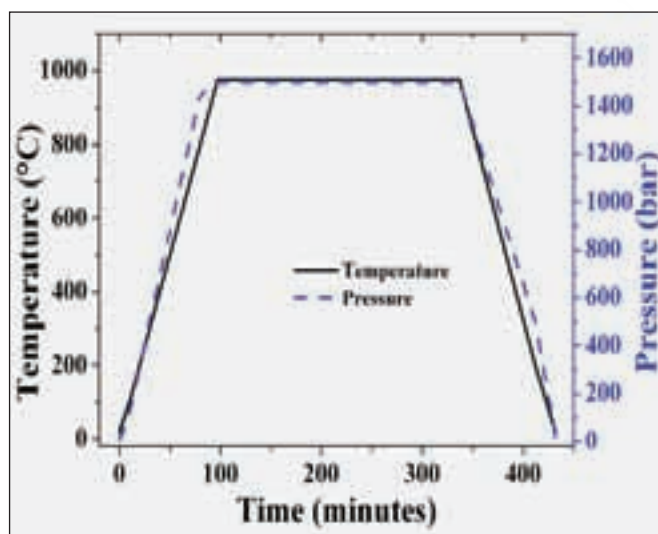


Fig. 1 HIP cycle

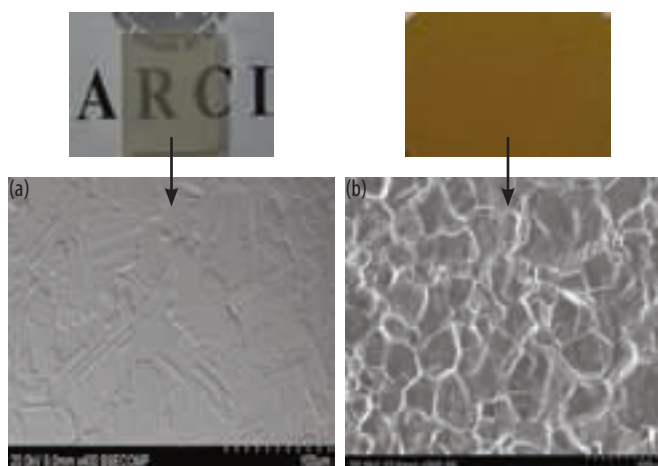


Fig. 2 (a) CVDHIP and (b) PRHIP ZnS Specimens along with the corresponding microstructure

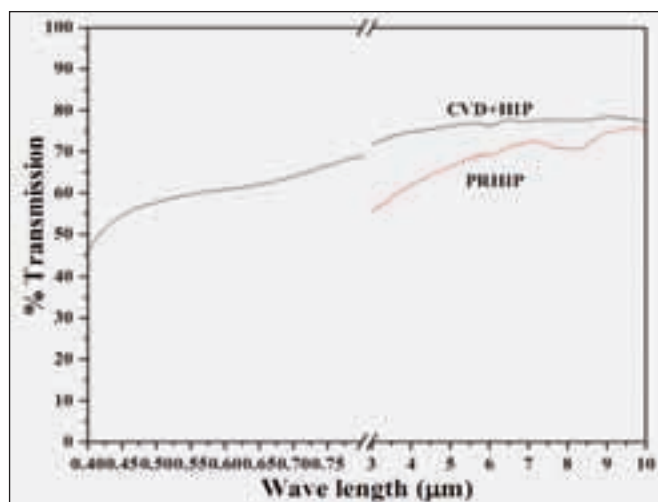


Fig. 3 Transmission plots of ZnS by CVD+HIP and PRHIP

Contributor: Roy Johnson

# Effect of Cone Angle and Land Parameters on the Properties of Extruded Alumina Ceramics

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Function of an extrusion die is to shape the plastic deformable dough. For optimum extrusion process, the die should have a balance in order to provide a uniform exit velocity to the extrudate, across the entire die exit in combination with minimal pressure drop. Hence, defect free products during extrusion processing not only depends on the dough rheology, but also on the die design especially the cone angle of the die and the die land parameters. In the present study extrusion dies are designed and fabricated with three different cone angles of 30°, 45° and 60° in order to study the properties of green extrudates and a typical design is shown in Figure 1 and the fabricated die is mounted on press as shown in Figure 2.

Extruded green samples are characterized for their green density by dimensional method and are also characterized for the bend strength. The results are presented along with cone angle in Table 1. In order to evaluate the strength, the dough was extruded into standard specimens and dried. Dried samples are further subjected to the 3-point bend test to estimate the modulus of rupture (MOR) using a universal testing machine (Instron 5584). Samples are also sintered in order to assess the effect of cone angle on the finished product.

It is evident from the table that a cone angle of 45° has resulted in the maximum theoretical density of 54 % and a maximum strength of 15 MPa. This can be attributed to the uniform pressure distribution throughout the dough that leads to maximum particle packing while extrusion processing.

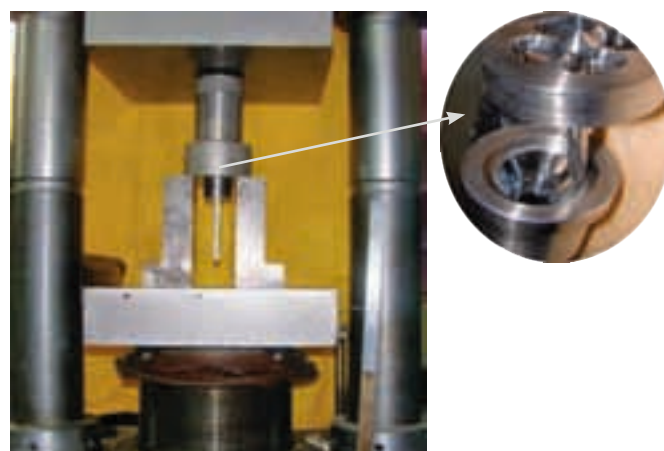


Fig. 2 Extrusion process

Table 1 Density and Flexural strength of green samples extruded using dies with various cone angles

| Cone angle of the die | % Theoretical density (g/cc) | Flexural strength (MPa) |
|-----------------------|------------------------------|-------------------------|
| 30°                   | 49                           | 11                      |
| 45°                   | 54                           | 15                      |
| 50°                   | 51                           | 13                      |

Sample with the maximum green density of 54 % of TD was subjected to dilatometry and the curve is shown in Figure 3. Shrinkage starts at around 1200°C and exhibits a slope change from 1600°C indicating the final densification regime. The samples are sintered in a laboratory furnace as per standard sintering schedule and a theoretical density of 99% was achieved at 1650°C.

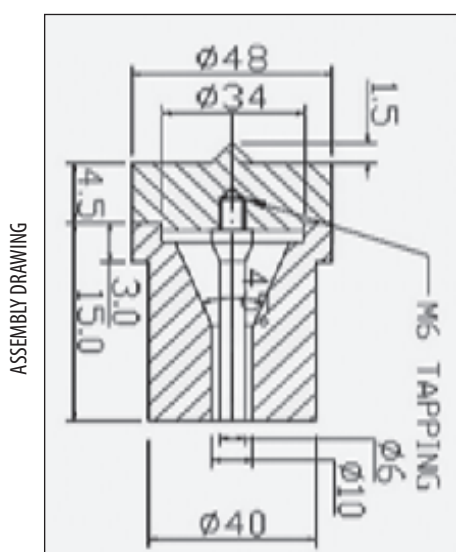


Fig. 1 Typical die design

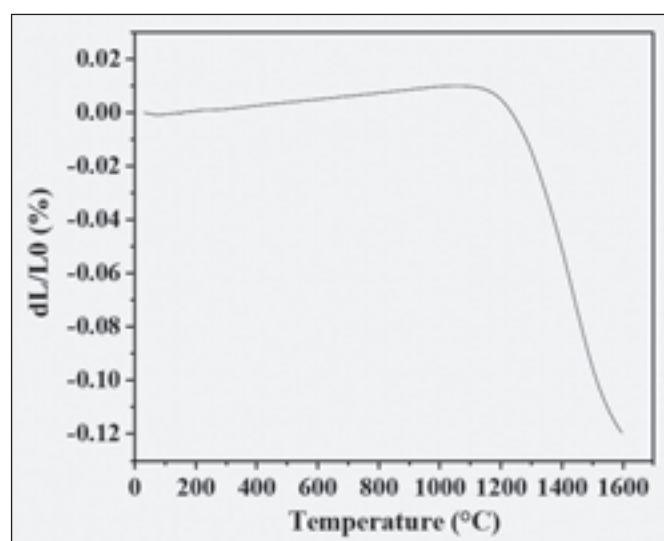


Fig. 3 Densification behavior of Alumina

Contributors: A Rajashekar Reddy, M Buchi Suresh and Roy Johnson





## Centre for Laser Processing of Materials

**L**aser as a high intensity, precise, flexible and clean heat source is used as a manufacturing tool in several industrial sectors such as automotive, aerospace, energy, electronics and nuclear sectors. With the advent of more robust, energy efficient, cheaper and low foot print lasers such as fiber lasers, laser based manufacturing is increasing rapidly. Laser based processing can be classified as macro processes (cutting, welding, cladding, alloying, drilling brazing) and micro processes (micro texturing, scribing, micro drilling etc) depending on the scale of effects induced during the process.

The Centre for Laser Processing of Materials (CLPM) aims at promotion of laser based manufacturing in the industry. Various laser based processes being pursued include:

- Surface engineering processes such as hardening, cladding, texturing;
- Joining processes such as welding, hybrid welding and brazing;
- Machining processes such as drilling, cutting, scribing;
- Repair and refurbishment through laser material deposition; and
- Metal additive manufacturing

The activities span from R&D work towards in-depth understanding of various processes, development of applications & manufacturing solutions followed by know-how transfer to the user industries.

The centre has been active in macro processing applications using high power lasers available viz., a 6kW fiber coupled diode laser, a 3.5 kW slab CO<sub>2</sub> laser and a 400W Average Power pulsed Nd:YAG laser. However, during the current year, the centre established a state-of-the-art femtosecond T-Sapphire laser based micromachining system and carried out several micro processing investigations. Some of them are micro surface texturing of automotive engine component materials, fabrication of micro heaters, micro cutting of co-fired ceramic PCBs etc.

A very innovative method of laser hardening under water of very low carbon and thin automotive steel sheets was demonstrated. Laser hardening method was investigated on bearing steels and substantial improvement in wear behaviour was observed.

In the material joining area, the centre actively pursued the project "MultiJoin" along with consortium partners in India and Germany towards demonstration of joining technologies for Aluminium, Steel and Plastic in different dissimilar combinations. These are mainly meant for lightweight construction of automotive bodies. Laser brazing and cold metal transfer brazing processes were successfully developed at the centre for joining of aluminium alloys to steels and fracture loads as high as 280 N/mm were achieved, which is a usable strength in actual applications. On the thick section welding side, the CO<sub>2</sub> laser in combination with MIG welding was successfully used to welding thick sections of maraging steels and Ni based alloys are prelude to applications in aerospace and power plants.

Based on laser metal deposition methods, a major direction pursued was repair and refurbishment of expensive pressure die casting dies and used diesel engine blocks. At the same time, first attempts of layer by laser deposition of hot work die steel to build up complicated profiles using the diode laser cladding system were made and such additively manufactured shapes were metallurgically evaluated. The centre in collaboration with the Engineering Staff College of India organised the International Conference on Additive Manufacturing ICAM-3D by organising a special session on repair and refurbishment.

In summary, laser based manufacturing processes were developed for application in various industrial sectors such as automotive, thermal power, electronics and tools & dies. In the current year the centre made successful entry into laser micro processing as well.

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# Autogenous Laser Welding of Thick Section of Ni-based Alloy IN617

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Inconel 617 (Ni-Cr-Co-Mo) alloy is primarily a solid solution and carbide strengthened nickel-based super alloy with superior engineering properties. This alloy has been widely employed in power plants, aerospace, chemical and nuclear industries because of its exceptional properties of high temperature strength and creep resistance. The primary welding issue in nickel based super alloy is the sluggish nature of the molten pool to flow, liquation cracking at fusion boundary and microfissuring in reheat zones of multi-pass welds. Laser welding being a low heat input process is expected to obviate many of these problems. Accordingly, investigations were carried out to study laser weldability of 6.3 mm thick sections of Inconel 617 alloy.

Autogenous laser welding of 350 mm x 150 mm x 6.3 mm Inconel IN617 plates have been carried out using high beam quality (Gaussian,  $K > 0.9$ ) DC035 slab CO<sub>2</sub> laser using a focal spot size of 180 microns. Parametric optimization has been done by conducting exhaustive bead on plate (BoP) welding studies. BoP welds have been analysed extensively by microhardness measurements, macro & microstructural analysis.

The butt welds were successfully made in straight butt configuration with a set of optimised parameters selected from BoP studies and full penetration was achieved at three heat inputs of 175J/mm, 210J/mm and 300J/mm. The weld bead geometry of all weld joints is Y-type. The effect of heat input on the weld bead geometry is shown in Figure 1. Macrostructural analysis showed that the welds were free from defects such as cracks, porosity etc. Occurrence of under cut is significant with increase in heat input. The weld bead subsidence is prominent with highest heat input and arises from the downward melt flowing along the front of keyhole which is caused by the intense recoil pressure of evaporation.

Laser weld microstructure was found to be fully austenitic with dendritic solidification, finer at bottom and coarser at top. Heat affected zone (HAZ) is almost absent with very narrow zone containing coarsened precipitates and thickened grain boundaries. Coarsening of precipitates and thickening of grain boundaries near fusion boundary appear to be confined near neck region, probably due to more heat stagnation and accumulation of heat in this zone. Microhardness survey was conducted both along and across the welds at various locations which revealed that there is a mild variation in the hardness of the fusion zone as compared to the base material. The butt welds were subjected to bend, tensile and impact testing according to ASTM standards. The tensile testing has shown that low heat input tensile specimens fractured at the base metal while high heat input tensile specimens fractured through weld metal. Impact energy of weld with low heat input is higher than the base material impact strength, while higher heat input weld impact strength is lower than base material impact strength. Mode of fracture in both tensile and impact test were found to be ductile. Energy dispersive spectra of tensile fractured specimens revealed that some dimples are enriched with titanium and less in molybdenum, whereas certain dimples found to be rich in Cr and Mo, but less in cobalt and nickel. Further, the butt welds have sufficient bend ductility. Inconel IN617 super alloy found to be laser weldable and laser welding may be considered as a potential joining technique as the process did not adversely influence the strength and resulted in very narrow HAZ etc., resulting in acceptable mechanical properties. Further studies are being carried out to understand the microsegregation of the elements such as Mo, Cr, and Ti into the interdendritic regions leading to the formation of a second phase at the end of solidification.

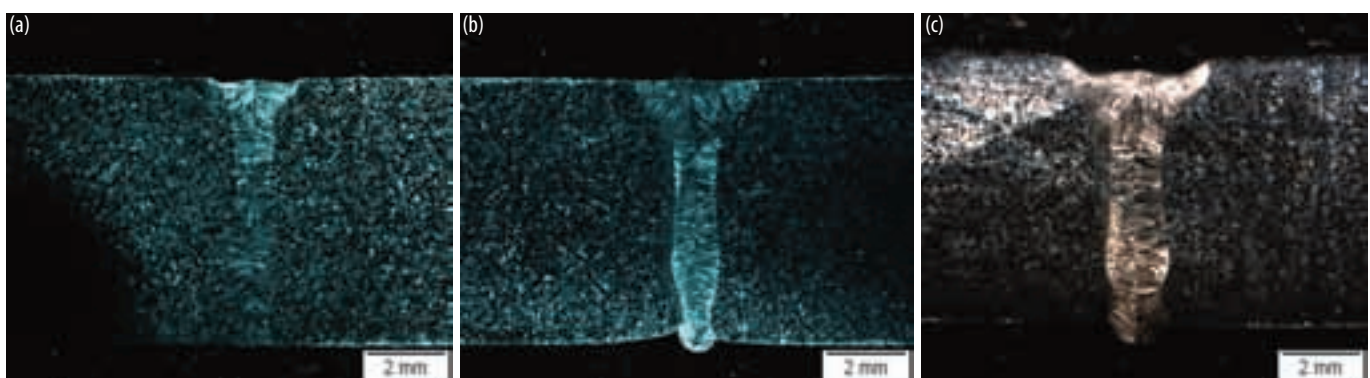


Fig. 1 Transverse cross-sectional macrostructures of butt welds in ascending order of heat inputs (a) 175 J/mm (b) 210 J/mm (c) 300 J/mm

Contributor: E Anburasu

# Laser Micro-fabrication of Heating Element for MEMS Device

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A new generation of ultrafast lasers with high pulse energy and pulse repetition rate has emerged as a new technique for micro/nano structure fabrication and precision manufacturing of all kinds of materials. ARCI's ultrafast laser micromachining system is equipped with some of the most cutting edge optics configurations, wide range of pulse duration (ns, ps, fs), broad spectrum of wavelength (uv, visible, NIR), multiple beams at workstation, precision motion system and fast speed galvo scanner. Apart from improving the laser systems, it is therefore crucial to realize that material removal can be influenced by other process parameters. The absorption of laser pulse in material is an energy transfer from the laser pulse to the electrons of the materials. For femtosecond pulse the duration is so short that there is not enough time for temperature equalization of electron and lattice. Then the heat diffusion from the "hot electrons" to the surrounding lattice after a characteristic time; this electron-phonon-relaxation-time is a material property and has a typical value on the order of 1-10 ps; an abrupt energy transfer between the hot electrons and the lattice takes place, resulting in a phase explosion, i.e., evaporation of the excited volume. This process is often called "cold" or athermal laser ablation. This salient characteristic of ultrafast laser processing produces remarkable clean micro-scale machined features free of burrs, melting, re-cast layer and heat-affected zone. The cutting of microheaters from electric heating foil material is important in the development of micro-electro-mechanical system (MEMS), where the machined feature sizes approach micron scales, resulting in increased integration capabilities and reduce power consumption.

An amplified Ti:Sapphire femtosecond laser (central wavelength  $\lambda=800\text{nm}$ , 10 kHz repetition rate, 100 fs measured at full width half maximum by an autocorrelator) was used for micro cutting of heaters from nickel foil of 20  $\mu\text{m}$  thickness. The precision of ultrafast laser micro-cutting depends on several femtosecond laser and material relationship parameters and the ablation threshold was one of the important parameter. In case of a Gaussian intensity distribution of the laser focus, the diameter of the ablated structures (D) can be given by following equation:

$$D^2 = 2\omega_0^2 \ln\left(\frac{F}{F_{th}}\right)$$

where F is the pulse energy,  $F_{th}$  is the threshold pulse energy and  $\omega_0$  is the waist radius of the laser focus. If the fluence

is just slightly above the threshold it is seen that structures smaller than the minimum spot size can be generated; in this way structures down to a submicrometer are possible to be machined. Figure 1 shows the ablated structure diameter squared as a function of the pulse energy for nickel. The waist radius  $\omega_0$  can be determined from the slope of the linear curve and a value of  $\omega_0 = 6.8 \mu\text{m}$  and the ablation threshold  $F_{th}$  is 0.29 J/cm<sup>2</sup> by extrapolating the linear fit to  $D^2 = 0$  in a plot of the diameter square vs logarithm of the pulse energy. Figure 2 shows the optical microscope images of the microheater cutting of nickel sheet using femtosecond laser. High speed and multipass processing with low thermal effect was used to cut the microheaters. No melting, microcracks and heat induced distortion were absorbed. This leads to cutting edges with no burr and high strength, which is essential for the MEMS membrane microheaters to heat uniformly without any hot spot.

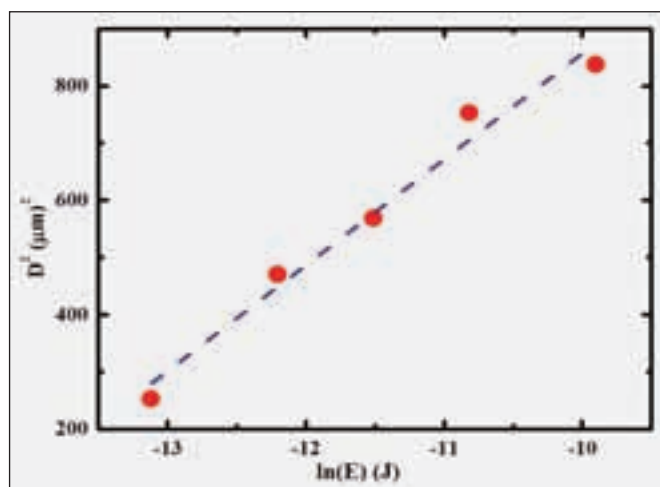


Fig. 1 Ablated structure diameters squared versus logarithm of the laser pulse energy

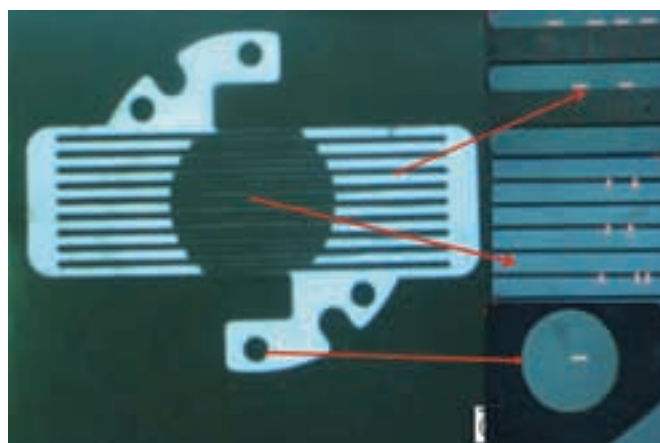


Fig. 2 Micro cutting of heaters from nickel foil with sharp edges, no burr or melt and low surface roughness

Contributor: G Padmanabham



# Performance Evaluation of Laser Deposition Refurbished PDC Die Under Simulated and Production Conditions

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Die components are subjected to premature failure due to thermo mechanical fatigue assisted cracking, high temperature softening and erosion. Thermo-mechanical fatigue resistance can be improved by increasing strength, hardness and resistance to softening at higher temperature. In the present study, detailed studies of softening resistance and thermal fatigue resistance of laser surface clad H13 tool steel has been evaluated in a specially designed test rig (shown in Figure 1) containing molten aluminium alloy (A380) melt maintained at 660-680°C, which simulates the casting conditions. Test samples were alternatively dipped in Al-alloy (A380) melt and commercially available water based die coat solution maintained at 30 - 40°C. Test was conducted for 3000 cycles with each cycle of 28s. Subsequently, field trials were conducted in an industry and performance evaluated in actual production conditions, in terms of number of castings produced. A damaged component (H13 hot work tool steel) of running die assembly, shown in Figure 2a, was chosen for this study. Figure 2a shows chipped off at inside corner of the die. Chipped off location of the die component was machined to the repairable dimension and laser clad deposition was carried out without any preheating step.

Microhardness data (Figure 3a) shows that the rate of decrease in hardness at later stage of soaking is much lower in laser clad zone. The decreased drop rate of microhardness in clad layer with increase in soaking time is possibly attributed to the retention of the original microstructure after soaking. A close comparison of the soaking behavior of laser surface engineered sample with that of hardened and tempered H13 tool steel shows that softening resistance increases in laser surface engineered surface as compared to as-received substrate.

Conventionally hardened and tempered H13 tool steel (46 to 48 HRC) and laser surface cladding were subjected to simulated thermal cyclic test. There is substantial drop in hardness due to thermal cycling (Figure 3b). However, the clad surface showed higher hardness compared to H13 substrate. This shows that the laser clad samples showed improved cyclic softening resistance. This is attributed to the initial refined and supersaturated microstructure with high dislocation densities, residual compressive stress and high hardness and strength of the surface developed during laser processing.

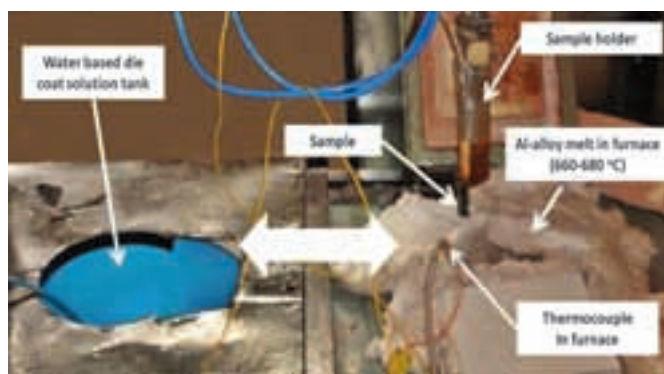


Fig. 1 Photograph of simulated thermal fatigue test rig

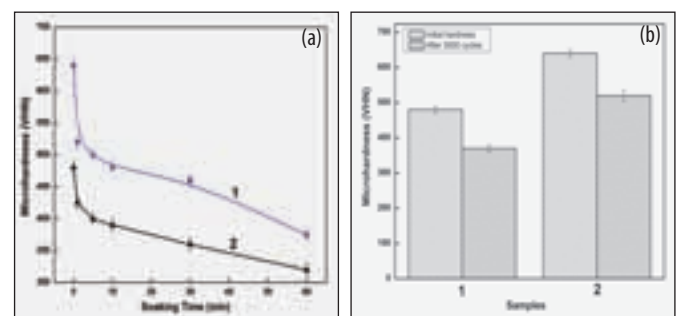


Fig. 3 Surface hardness observed after different test (a) Softening resistance test (b) thermal cyclic test for 3000 cycles for (1) H13 tool steel substrate and (2) laser clad H13 tool steel

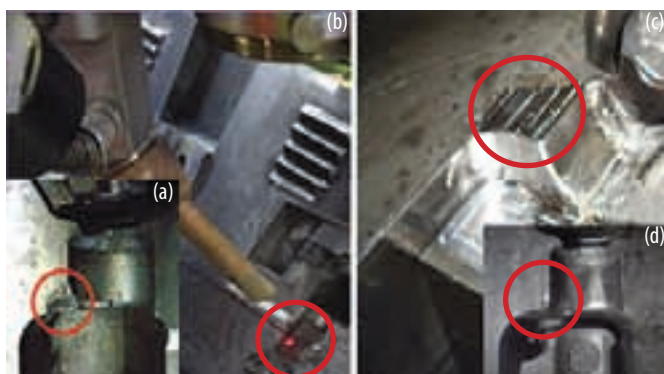


Fig. 2 Photographs of (a) chipped-off die supplied by the industry (b) deposition by laser cladding (c) post machining and (d) Repaired die component after 30,000 shots

The laser clad repaired actual die component shown in Figure 2b, is machined to match the actual dimensions of original die. Subsequently, repaired die component was subjected to stress relieving at 550°C for 2 h, before loading the die for casting process. The performance of the repaired location was closely observed and successfully completed 30,000 shots so far without any problems. The latest condition of the die is shown in Figure 2d.

Contributor: G Padmanabham

# A Novel Method to Harden the Thin Component and Low Carbon Steel

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The present invention is to harden the conventionally unhardenable low carbon structural steels or thin steel components which require extremely high cooling rates to attain the required phase transformation.

Laser surface hardening has advantage over the conventional hardening techniques as it gives very high cooling rates due to localized heating and subsequent self-quenching caused by heat-sink effect of the remaining unaffected cooler bulk part of the steel. Although laser surface treatment by hardening induces higher cooling rates than that of conventional treating methods, it is still not sufficient enough to achieve hardness improvement on surface and subsurface in low carbon structural steel materials. If the same steel is a thin one, it then further becomes impossible to harden the surface and subsurface as heat accumulation in the remaining bulk reduces the cooling rates resulting in no surface hardening effect. Additionally, significant rise in surface temperature results in adverse effects such as melting and/or heavy distortion. Distortion is another vital barrier in surface hardening of thin steel sheets or components.

The underwater laser hardening process is a novel technique, invented to address the issues related to surface modification of low carbon structural steel or thin steel sheets or components. The under water laser surface hardening technique increases the cooling rate sufficiently high enough to achieve the phase transformation not only on the surface but also in sub-surface to a depth of few hundreds of microns. The underwater laser surface hardening process also facilitates in reducing distortion substantially associated with induced uneven stress distribution on the steel and thus making the process most suitable for achieving surface hardening on thin sheets or components.

In the underwater laser hardening technique, a high power laser beam is scanned over the substrate which is immersed in water (Figure 1). The laser beam penetrates through the thin layer of water, which is transparent to the laser beam, and raises the temperature of the substrate above the austenising temperature. The surrounding immersed water facilitates fast dissipation of heat from the laser-treated surface and thereby result in producing hardened surface up to a depth of few hundreds of microns. Experiments were conducted on the 1mm thick CRCA grade steel sheets with 0.05% carbon content. A 6kW high power fiber coupled

diode laser system was used for the experimentation. Figure 2 shows the cross-sectional macrograph of the hardened substrate with martensite phase in the hardened zone. Figure 3 shows the cross-sectional hardness profile with significant improvement in the hardness value. This technique can be used to enhance the surface hardness of ultra low carbon steel components or to process any thin sheets and small components with very less heat sink, without any distortion.



Fig. 1 Process setup



Fig. 2 Cross-sectional macrograph

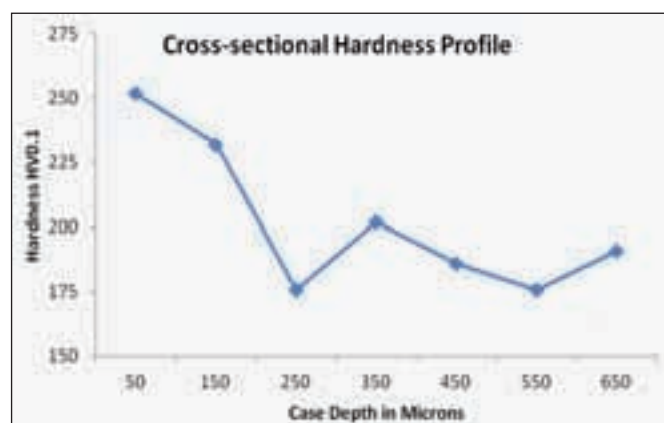


Fig. 3 Cross-sectional micro hardness profile

Contributors: S M Shariff and G Padmanabham



# A Novel Cobalt Oxide Based Thin Film Electrocatalyst for Water Oxidation Catalysis

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Development of effective catalysts for performing water oxidation reaction is of great importance since they play a vital role in artificial photosynthesis development. In 21<sup>st</sup> century, development of artificial photosynthesis, i.e., conversion of waste-stream greenhouse carbon dioxide into value added chemicals using water and sunlight or electricity derived from sunlight, has been considered to be one of the top most research priorities as it deals with the problems related to i) carbon dioxide associated global warming, ii) energy storage, and iii) depletion of fossil fuels. It is a well-known fact that the artificial photosynthesis like natural photosynthesis consists of water oxidation (i.e., light) reaction and carbon dioxide reduction (i.e., dark) reaction. It is also known that water oxidation reaction needs both thermodynamic energy input as it is an endothermic reaction and an effective catalytic system to perform four consecutive one-electron transfer reactions ( $2\text{H}_2\text{O} \rightarrow \text{O}_2\uparrow + 4\text{H}^+ + 4\text{e}^-$ ;  $E_0 = 1.23 \text{ V} - 0.059 (\text{pH}) \text{ vs. NHE}$ ). However, inexpensive catalysts that perform water oxidation reaction at low over potentials with required efficiency and stability are yet to be developed fully. As a part of this objective, we at ARCI have undertaken a systematic study to develop inexpensive cobalt (abundantly available base metal) oxide based thin film electrocatalyst, which showed water oxidation ability at an overpotential of only about 400 mV and was found to be quite stable (Figure 1 (a) & (b) in comparison to cobalt phosphate (Nocera catalyst) water oxidation (Figure 2) catalyst. The ARCI water oxidation catalyst was prepared following a sol-gel dip-coating method, whereas, the cobalt phosphate catalyst was formed on the surface of Fluorine doped Tin Oxide (FTO) conductive glass substrate *in situ* from a sodium phosphate buffer solution containing desired amounts of cobalt nitrate during cyclic voltammetry. The high activity and stability of

ARCI thin film electrocatalyst could be attributed to its unique chemical composition, surface morphology (Figure 3) and high surface area (>1500 m<sup>2</sup>/g) of the thin film.

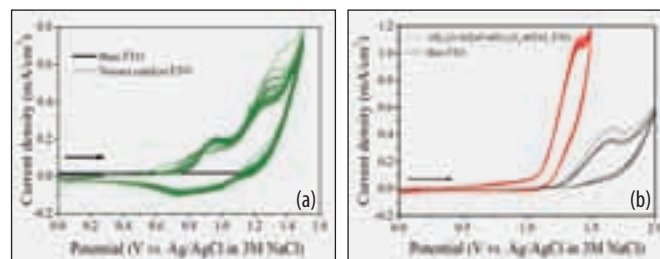


Fig. 1 Cyclic voltammetry profiles of (a) Nocera catalyst on FTO (for 30 cycles), and (b) ARCI thin film electrocatalyst deposited on FTO (for 30 cycles) recorded in 0.2 M NaPi (pH = 7.4) (saturated and blanketed with Ar) at a sweeping rate of 100 mV/s. The CVs recorded for bare FTO under identical experimental conditions for different potential ranges can also be seen.

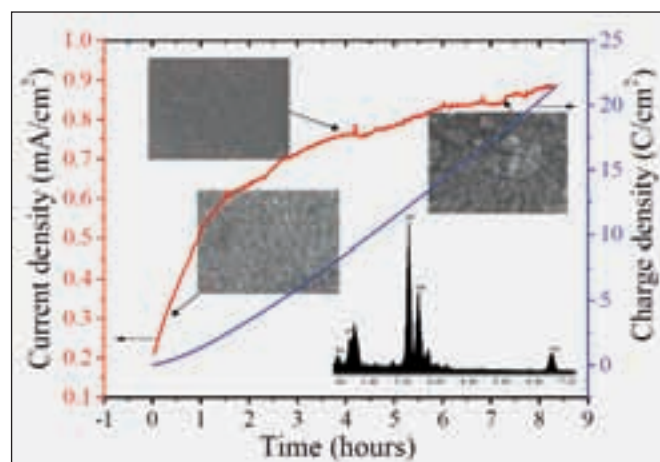


Fig. 2 The profiles of current density and charge density vs. time recorded during the *in situ* formation of Nocera catalyst on the surface of FTO from a quiescent 0.2 M NaPi (pH = 7.4) containing 0.5 mM  $\text{Co}^{2+}$  ions at 1.30 V (vs. Ag/AgCl) in a two-compartment electrochemical cell. An EDAX spectrum recorded after about 8 hours, and the SEM micrographs of film formed for different time intervals can also be seen.

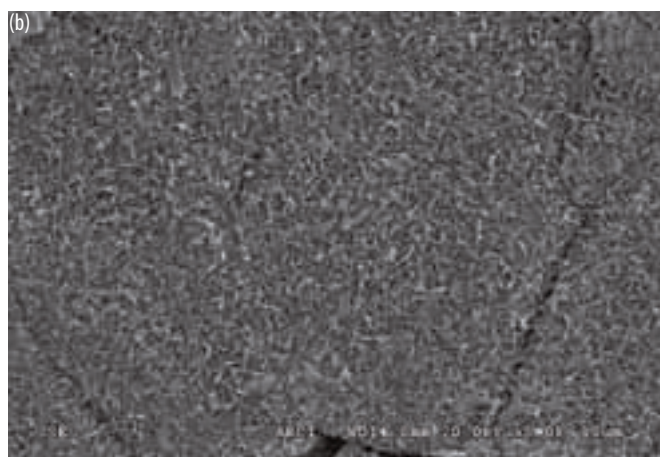
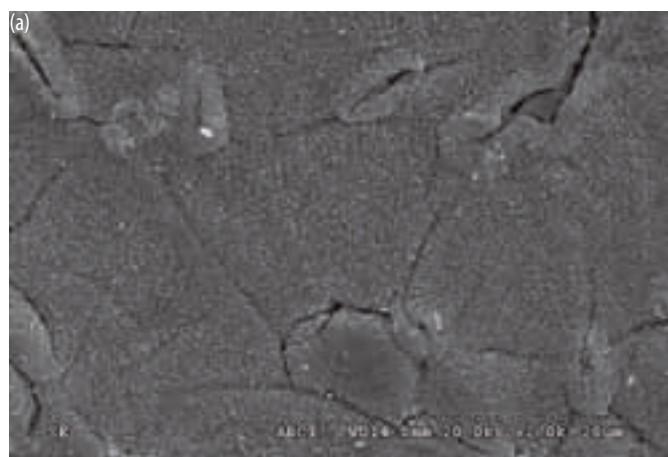


Fig. 3 SEM micrographs of ARCI catalyst thin film captured at two different magnifications (a) & (b)

Contributor: G Padmanabham





## Centre for Fuel Cell Technology

*The R&D efforts at the Centre for Fuel Cell Technology (CFCT), under the general theme of Energy Conversion Devices, include work on improvements in LT-PEMFC fuel cell systems, demonstration of HT-PEM fuel cell stacks, development of alternative materials for use in hydrogen generation (electrochemical reformation & photo electrochemical route), hydrogen storage and batteries.*

*Under the fuel cell research programme, the “5 KW – 48VDC Lab “powered by LT-PEMFC has been operated intermittently for 200 hrs without any degradation of the cells. A 1 kW HT-PEMFC built using exfoliated graphite bipolar plates has been demonstrated. A hydrothermal route has been developed to make N-doped Graphene for use as catalyst support. Investigations on the effects of impurities on fuel cell performance and mitigation strategies is another area of research under the DST-UKRC programme. Impedance and thermal imaging are used as diagnostic tools for identifying malfunctioning cells in a stack.*

*In the area of supercapacitors development, carbon materials with well-defined micro and meso pores have been made which show a high surface area (~2700 sq.m/g) and high specific capacitance. For the first time, a high temperature super capacitor has been developed which exhibited a maximum capacitance of 128 Fg<sup>-1</sup> at 140°C. Use of anion exchange membrane in super capacitor development has also been demonstrated. The maximum specific capacitance, power density and energy density of the supercapacitor were 45 F/g, 23 kW/kg and 6.25 Wh/kg, respectively.*

*Under MNRE project, new activated carbon materials have been derived from agricultural wastes and some of them have shown a hydrogen storage capacity of ~ 4.3 wt% at 40 atm and 30°C and reproducibility has been tested for 30 cycles.*

*Under the program on Hydrogen generation, a novel polymer composite has been made from sulphonated TiO<sub>2</sub> with sPEEK to reduce the cost of the electrochemical methanol reformer (ECMR) disclosed in the previous years. A mesoporous palladium catalyst also shows performance similar to platinum based catalyst, thereby offering cost reduction. Hydrogen generator using anion exchange membrane is also being investigated. Under PEC route to make hydrogen, carbon and CdS quantum dots capped TiO<sub>2</sub> nanotubes and ZnO nanorods have been prepared which show a high photo current density of 6.5 mA/cm<sup>2</sup> which corresponds to a hydrogen generation rate of 0.03 mMol/sec.*

*In the area of battery research, under the rechargeable metal – air batteries with aqueous electrolytes, high activities for ORR and OER has been achieved with electro deposited MnO<sub>2</sub>, and more significantly stability upon cycling (40 cycles). In the field of REDOX flow battery, a novel method has been developed to increase the concentration of the vanadium species in the electrolyte, to increase the energy density. A Metal-Hydrogen Peroxide based electrochemical cell has also been developed.*

*During the year, the Centre completed the project “Centre for Fuel Cell Technology – Phase-2” and carried out a contract project sponsored by M/s. NATCO Pharma. During the course of the year, 11 papers have been published in peer reviewed International Journals.*

# Investigations on LT-PEMFC with Impure Fuel and Air

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PEM fuel cells are being demonstrated increasingly for various applications due to their advantages like easy start up, high efficiency, less pollution, modular nature etc., Since these applications are in different locations with variable air quality and hydrogen from different sources, it is important that the fuel cells are tested for under several simulated conditions which include impurities of various kinds in different concentration. The impurities commonly encountered in the oxidant and fuel streams are CO, H<sub>2</sub>S, and NH<sub>3</sub>, organic sulfur-carbon, and carbon-hydrogen compounds. These impurities can cause reversible or irreversible damage to the cell either by attacking the catalyst sites or the electrolyte. Concentration of the impurities, some times, decide whether it is irreversible or reversible process. In this connection the Centre has carried out extensive work during the last year, bulk of the work under the DST-UKRC project.

The studies include standardisation of methods to study the poisoning effect, extent of poisoning and develop mitigation strategies. The impurities investigated were CO and chloride ion in the anode gas feed and SO<sub>2</sub>, NH<sub>3</sub> and chloride ion in the cathode gas feed. The mitigation strategies for different impurities include development and testing of electro catalysts with mesoporous structure [Pt and Pt-Ru for the anode; Pt-Co, Pt-Ni for the cathode prepared by hard template method], non-noble metal catalyst such as TiC (from New Castle university, UK), Platinum supported on graphene & nitrogen doped graphene, voltage cycling, treatment with oxidising agents like ozone, increasing the concentration of oxygen in air and water washing of the electrodes. The studies were extended from half cells to single cells and then to multiple cells. With respect to chloride ions contamination, it has been observed that chloride ions adversely affects both Hydrogen Oxidation Reaction (HOR) as well as Oxygen Reduction Reaction (ORR) reactions. However, the poisoning effect is reversible and the rate of recovery is dependent on the concentration of the chloride ions. Even a small amount of SO<sub>2</sub> contamination reduces the fuel cell performance considerably and this effect is only partially reversible. We find that mesoporous Pt catalyst are more tolerant to SO<sub>2</sub> poisoning compared to microporous catalyst. Pt supported on nitrogen doped graphene also shows higher SO<sub>2</sub> tolerance as well as quicker recovery. It is also observed that ozone treatment can recover the SO<sub>2</sub> poisoned electrodes. The use of O<sub>3</sub> for recovery can be attributed to both electrochemical and chemical processes. However, this method has to be carried out cautiously. These studies are being continued for all the impurities.

In a multi cell study it was observed that the graphene based catalysts got recovered in 4 hours compared to 9 hours required for the conventional catalysts at the same impurity level.

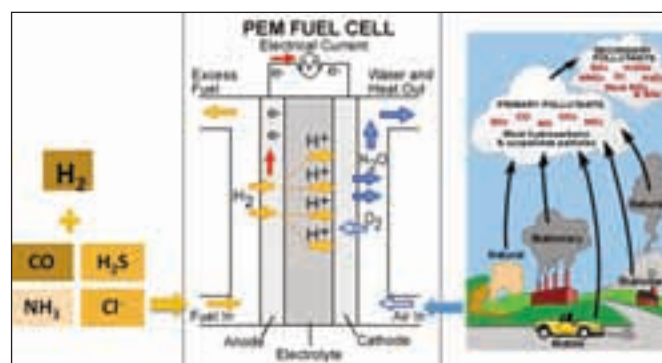


Fig.1 Impurity sources and effect of impurity on the fuel cell performance

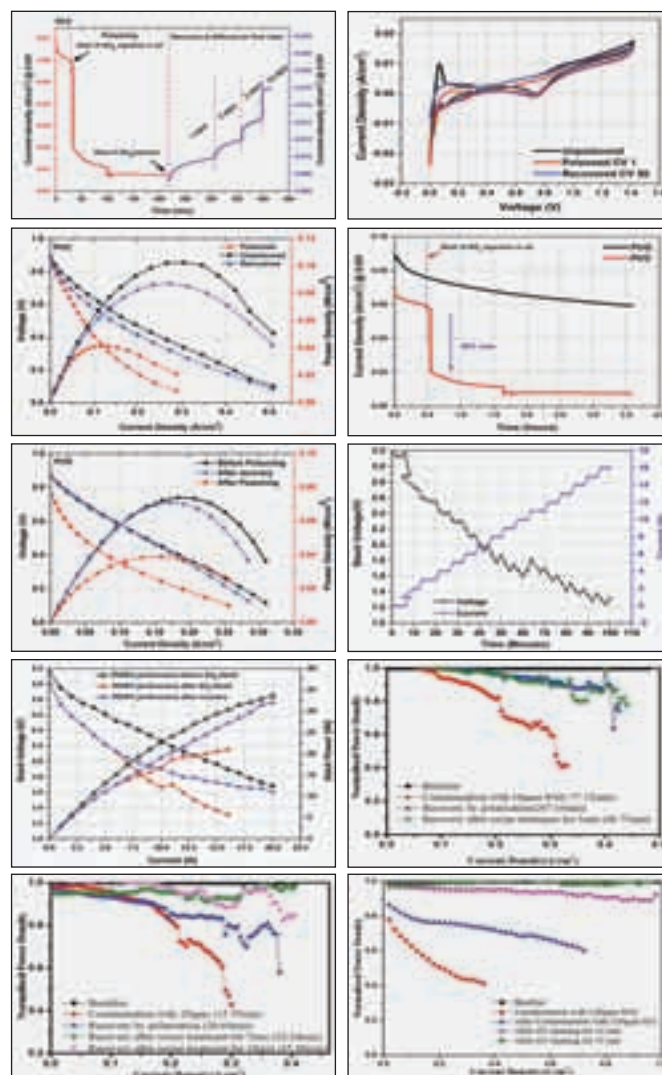


Fig. 2 Recovery of contaminated fuel cells by various methods

Contributors: Prithi Jayaraj, Anusree Unnikrishnan, Karthika and Imran Jafri



# High Temperature PEMFC: Stack Development, *in situ* Characterization and Waste Heat Recovery

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High temperature PEMFC systems operate at temperatures above 150°C and offers the advantages of better thermal management and impurity tolerance compared the low temperature PEMFC systems. As a  $\mu$ CHP system, HTPEMFC stack's ability to provide both power and heat (as hot water) was demonstrated earlier with a 600W HTPEMFC stack. Further developments has resulted in the fabrication of a 1kW HTPEMFC stack consisting of 56 cells operating at 180°C with dry hydrogen and air as the reactant gases with exfoliated graphite plates for gas distribution and current collection.

To optimise the fabrication ,operation parameters and for cell diagnostics, electrochemical impedance spectroscopy (EIS) has been extensively used. A multi (08) cell HT-PEMFC stack under operation was used in these studies which can provide an insight into electrochemical reaction kinetics, ohmic process and the mass transport processes in the system. In addition, EIS allows modelling of the system with an equivalent circuit to distinguish the individual contributions of membrane, electrodes, interfacial charge transfer, mass transport etc. EIS studies showed that there is not much variation in resistances when the stack was operated at constant temperature and at high frequency. However, at low frequency there was no clear trend. It was also observed that with increasing currents ohmic resistance decreases and at high currents mass transfer limitation is the main reason for voltage and power decrease. Further, cells with higher temperature show lower resistance and variation of ohmic resistance at low currents is larger for individual cells as hydration levels for various membranes may be different. Variation of ohmic resistance among individual cells is not high at high currents indicating uniformity in the cells of the stack as even a small increase in ohmic resistance of the individual cells at high currents indicates a large voltage loss in the cell. The charge transfer resistance was found to decrease with increasing current indicating increase in driving force of

the electrode process. Further studies are being carried out to calculate the parameters for a larger stack. Studies to identify the stack and cell parameters with current interrupt method and Impedance study at single frequency are being carried out.

In order to improve the efficiency of the fuel cell systems, incorporation of devices that can utilize the waste heat generated by HTPEMFC stack to produce electricity have been investigated. Towards this effort the centre has initiated studies on development of high temperature super capacitor and incorporation of thermoelectric power generators to utilize the high quality heat generated. Single cell studies of the supercapacitor developed using phosphoric acid incorporated Poly (2, 5 benzimidazole) based cells have shown that maximum capacitance of 128F/g can be achieved with commercial carbon black based electrodes. Similarly, a maximum power of 1.2W was obtained with a single thermoelectric cooler (bismuth telluride) with a hot junction at 180°C and the cold junction at 78°C. It is proposed to integrate these with HTPEMFC stacks to study system efficiency in the coming year.

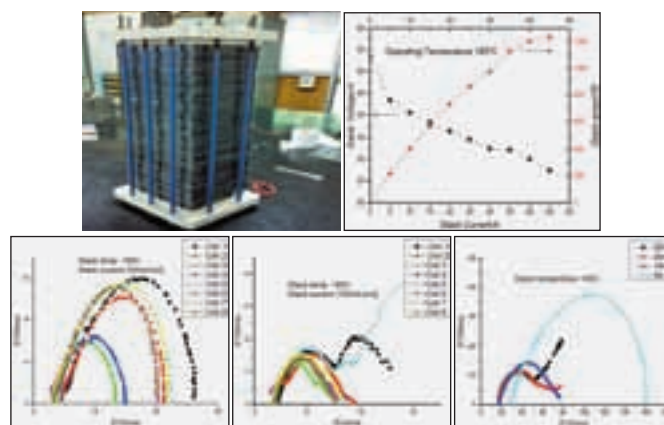


Fig. 1 One kW HT-PEMFC stack and Impedance characteristics of the individual cells and the stack (8 cells)

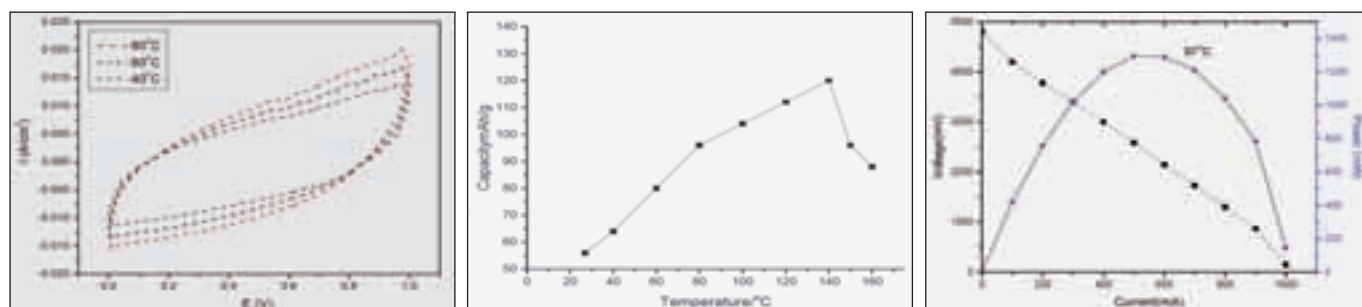


Fig. 2 Super capacitor and thermoelectric power generator performance curves

Contributor: G Vijaydev



# Activated Carbons with Tunable Properties Derived from Cotton as Supercapacitor Electrodes

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Supercapacitors which store energy in the form of double layer created between electrode and electrolyte interface has been widely explored as an alternative for high energy density batteries and high power density conventional capacitors. Nano structured and highly conductive carbons have been extensively used as electrodes for supercapacitors. However, the synthesis of these nano structured carbon materials (CNT, Graphene, Carbon nanorods etc.) is time consuming and require sophisticated equipments. The structure, porosity and electrical conductivity play a major role in determining the power density and energy density of the final supercapacitor device. Hence the present work is a multi pronged project in which many aspects are being addressed:

- (i) The complexities involved in the synthesis of carbon nanomaterials have been addressed by using simple agricultural product such as cotton and
- (ii) Structural properties (porosity, surface area, pore size distribution) which affects the power density of supercapacitors have been tuned by using simple activation procedures

Cotton has been chosen as a starting material which was first carbonised under argon (800°C, CC-800), followed by activation with KOH (Post-KCC-800). In another activation procedure carbonization and activation were performed simultaneously (Pre-KCC-800). The SEM pictures (Figure 1) show a highly porous structure for the activated samples a remarkable change in the BET surface area, pore volume and specific capacitance depending on activation process. The surface area and pore volume are important parameters which determine the cyclic stability and specific capacitance of supercapacitors.

With pre activation, the surface area increased from 667.9 m<sup>2</sup>/g to 1333.5 m<sup>2</sup>/g and with post activation, it increased to 2313.9 m<sup>2</sup>/g. The specific capacitances also increased from 30 F/g to 62 F/g and 92 F/g for Pre-KCC-800 and Post-KCC-800 respectively (Table 1). Figure 2 shows the cyclic voltammograms of CC-800, Pre-KCC-800 and Post-KCC-800, ideal rectangular shaped patterns have been obtained. It is interesting to note that the ideal rectangular patterns seen at lower scan rates (10 mV/s) gets distorted at higher scan rate (100 mV/s) for

CC-800 and Post-KCC-800, whereas in the case of Pre-KCC-800, the rectangular shape is still intact even at a scan rate of 200 mV/s. This implies that Pre-KCC-800 can be used for higher power density applications where huge energy delivery is required. While coming to the stability, the supercapacitor cells were tested till 10,000 cycles of charge discharge at 1 A/g. As shown in figure 3, Post-KCC-800 shows excellent stability (retaining the capacitance) even after 10K cycles of charge discharge. Whereas, CC-800 and Pre-KCC-800 show rapid decline in capacitance with increasing number of cycles. Hence the base cotton material can be modified by simple activation procedure to suit for the requirement.

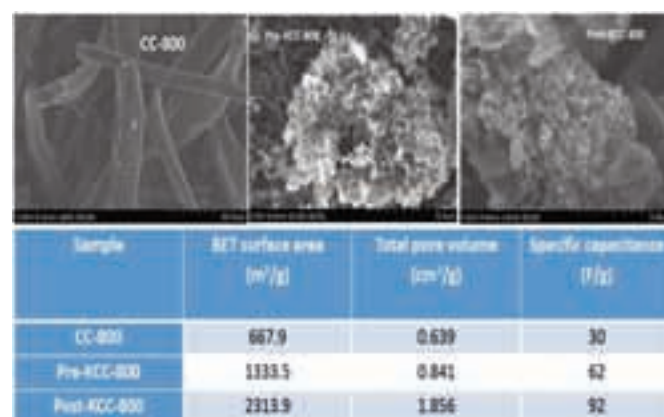


Fig. 1 SEM images of CC-800, Pre-KACC-800 and Post-KACC-800 and some characteristics

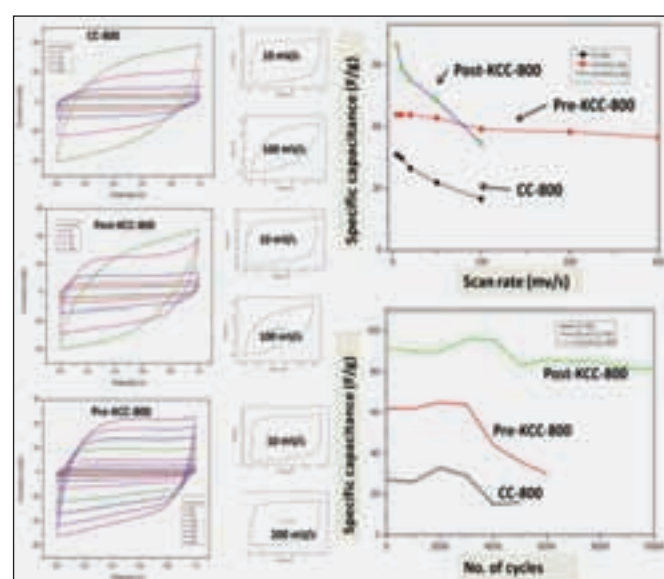


Fig. 2 Cyclic voltammograms recorded with CC-800, Pre-KACC-800 and Post-KACC-800 at different scan rates plots of specific capacitance vs increasing scan rates and specific capacitance vs. No. cycles (charge discharge cycles performed at a current density of 1 A/g)

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# Studies on Performance Improvement and Development of Alternative Membrane for PEM Based ECMR

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Cost reduction through performance improvement and development of alternative materials has been focus of development under the programme on hydrogen generation through electrochemical reformation of methanol which was demonstrated at 1000 litres/hr capacity in the previous years. The cost reduction has been addressed through evaluation of non noble metal catalysts, reduction in catalyst loading, use of non fluorinated composite membranes as electrolyte and operation at higher current density at the same voltage.

Performance improvements have been addressed through modification of electrode structure ( conductivity, porosity, hydrophobicity, hydrophilicity). Optimization of the various electrode preparation parameters evaluated in a single cell with 30 sq.cm electrode area resulted in doubling the current density to 200 mA/sq.cm. The performance was stable in an 100 hour operation. The electrode was scaled up to 150 sq. cm and a short stack of 2 cells was tested which also could be operated at the higher current density. This performance improvement indicates that the capital cost can be reduced by 50% from the earlier cost estimates.

One of the major cost component in Electrochemical Methanol Reformer (ECMR) is the perfluorinated membrane which is used as the electrolyte. Replacement of this membrane with indigenous low cost hydrocarbon membrane such as sulphonated poly-ether ether ketone (SPEEK) are being studied for their stability, methanol permeability and performance. A composite SPEEK membrane prepared by solvent casting technique with nano titania tubes was also tested for hydrogen generation. Among the series of composite membranes prepared with SPEEK and nano titania, the 5% titania incorporated membrane gave the highest performance. Membranes with 30-50 cm<sup>2</sup> area were tested for their hydrogen production capabilities.

In another approach cross linked SPEEK membranes were prepared using a polyhydric alcohol based cross-linker. The reason for this approach is that, although the proton conductivity of SPEEK increases with degree of sulphonation, above 70% DS, the membrane loses its physical strength especially above 50°C. This problem can be overcome by covalent crosslinking and ionic crosslinking using polyhydric alcohol. The crosslinking parameters were optimized and the resulted membranes were characterized in terms of water swelling, ion-exchange capacity, methanol permeability and proton conductivity. These membranes are nearly 20 times

cheaper than the conventional perfluorinated membrane . These cross linked polymeric membranes were tested in a standard cell with 30 sq. cm electrodes. Hydrogen generation was achieved at an energy consumption of ~ 1.7-1.8 kWh/Nm<sup>3</sup> compared to ~1.4kWh/nm<sup>3</sup> achieved with perfluorosulphonic acid based membranes. A trade-off between membrane cost and energy cost is indicated. Studies were also carried out with test solutions containing varying concentrations of methanol at various temperatures. Long term stability tests, performance tests are currently being carried. Solvent casting techniques to prepare larger area membranes (~ 300 cm<sup>2</sup>) using doctor blade techniques are underway.

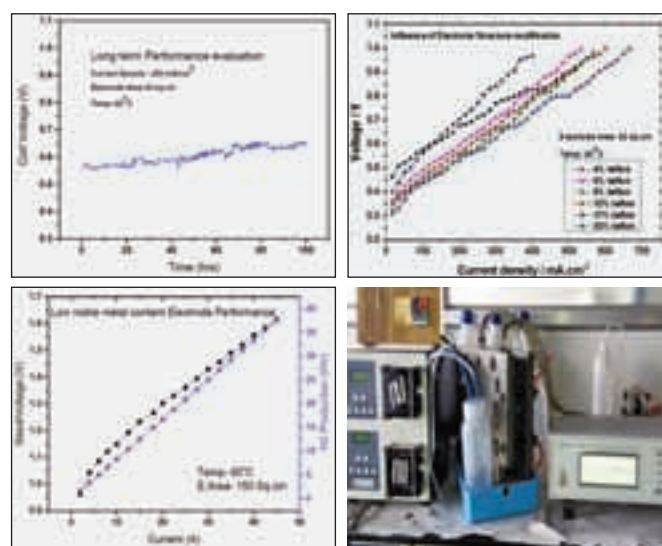


Fig.1 Performance and testing of ECMR

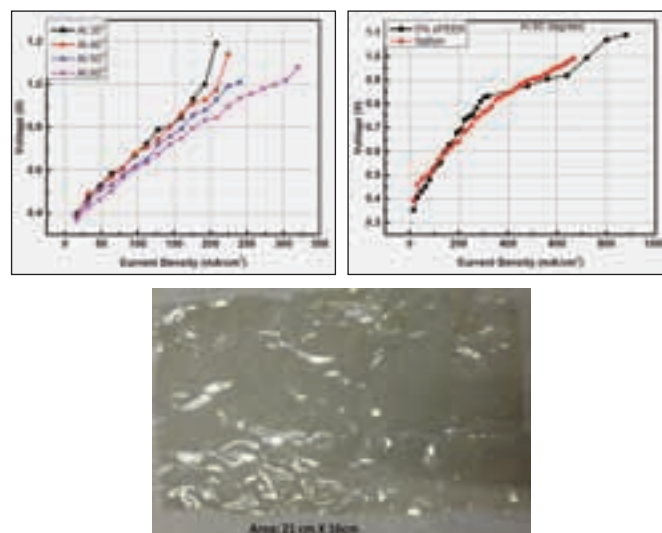


Fig.2 Performance of SPEEK membrane based ECMR cell

Contributors: K Ramya, Naga Mahesh, Manjula Reddy, Shanmuga Priya and K S Dhathathreyan



# Functionalised Metal-Oxide Nanostructures for Photo-Electrochemical Hydrogen Generation

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TiO<sub>2</sub> and ZnO are well known photo-catalysts that efficiently produce electron-hole pairs upon irradiation of light. However, due to the lower conduction band edge positions compared to the hydrogen reduction potential and also because of larger band-gap (3.2 – 3.3 eV), these materials are not suitable for hydrogen generation by photo-electrochemical water splitting under visible light irradiation. Functionalization of TiO<sub>2</sub>/ZnO with quantum dots having smaller band gap and a higher conduction band edge is one of the practical alternatives to overcome the problem. We have synthesised TiO<sub>2</sub> nanotubes and ZnO nanorods and functionalised them with carbon and CdS quantum dots. TiO<sub>2</sub> nanotubes were grown on titanium foil by electrochemical anodization in a two electrode configuration. The amorphous nanotubes are crystallised to anatase phase by annealing at 500°C for 2 hours. Carbon quantum dots were synthesised separately by electrochemical etching of graphite rods and then deposited on TiO<sub>2</sub> nanotubes arrays by dip coating technique. On the other hand, for the preparation of CdS quantum dots, CdSO<sub>4</sub> and Na<sub>2</sub>S solutions were prepared in two separate beakers. TiO<sub>2</sub> nanotube sample was successively dipped into two beakers to deposit layers of Cd and S ions and finally CdS quantum dots on TiO<sub>2</sub> nanotubes were obtained through drying. The SEM/TEM images of carbon/CdS functionalised TiO<sub>2</sub> nanotube arrays are shown in Figure 1. Further, ZnO nanorods were also grown on ITO coated glass substrates by chemical technique. These ZnO nanorods were functionalised with BiVO<sub>4</sub> nanoparticles by dip coating techniques. Figure 1(d) shows SEM image of ZnO nanorods. Finally, TiO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> composite were synthesized by hydrothermal technique and was pasted on conductive glass substrates. Formation of TiO<sub>2</sub>

and ZnO crystalline phases were confirmed by X-ray diffraction data. Change in the absorption edge resulting from quantum dots functionalisation of these oxides was examined by UV-Visible spectroscopy. Photocurrents for all TiO<sub>2</sub> and ZnO based samples were measured in three electrode system: TiO<sub>2</sub>/ZnO sample was used as anode, Pt wire as cathode and Ag/AgCl were used as reference electrode.

Photocurrent density for all the samples is mentioned in block diagram of Figure 2. Highest photocurrent density of 6.5 mA/cm<sup>2</sup> has been obtained from CdS functionalised TiO<sub>2</sub> nanotube that correspond to a hydrogen generation rate of 0.03 mmol/sec. This is attributed to efficient visible light absorption and charge separation caused by the CdS quantum dots.

To understand the mechanism of electron-hole pair creation, charge transfer and enhancement of photocurrent in quantum dots functionalised metal oxides, Mott-Schottky measurements and electrochemical impedance spectroscopy were carried out. Interfacial charge density and flat-band potential were estimated from the Mott-Schottky plots, whereas the charge transfer resistance was measured using the Nyquist plots obtain from impedance spectra. The interfacial charge density was found to increase and the charge transfer resistance across semiconductor-electrode decreased as a result of quantum dot functionalization. This can be attributed to better separation of the photo-generated charge carriers and their availability at the interface, caused by the presence of quantum dots. It is concluded that quantum dots help to obtain higher photocurrent from metal oxide nanostructures.

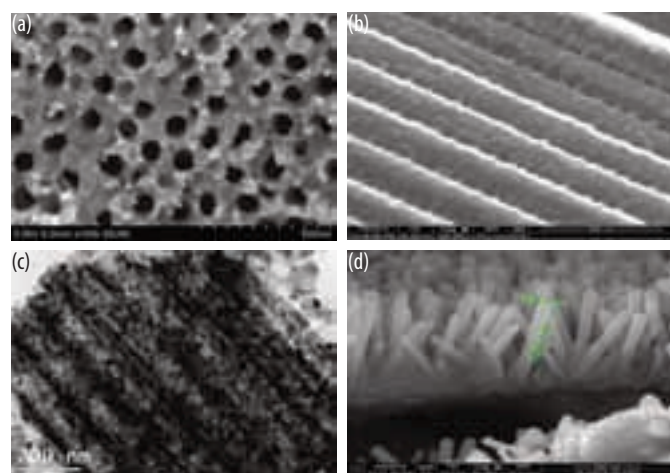


Fig. 1 (a) Top (b) Side view of CdS functionalised TiO<sub>2</sub> nanotube arrays (c) HRTEM images of CdS functionalised TiO<sub>2</sub> nanotube arrays (d) SEM of ZnO nanorods

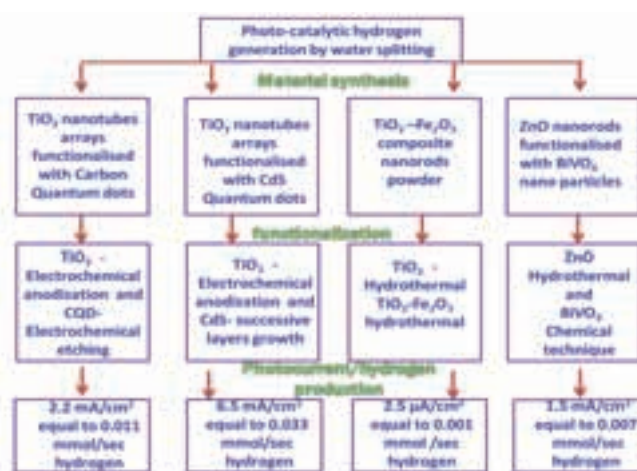


Fig. 2 Material synthesis, functionalization technique and photo current densities for different samples

Contributors: N Rajalakshmi and Shanmugapriya





## Centre for Non-Oxide Ceramics

**C**entre for Non-Oxide Ceramics (CNOC) at ARCI has been actively pursuing R&D activities in the area of various carbides, nitrides, and borides, their coatings and composites for wide range of applications. The centre has established the state of the art facilities including large size cold isostatic press (CIP), vacuum hot press, atmosphere controlled high temperature sintering furnace, chemical vapour deposition (CVD) system and CNC machining facilities for production of large size high performance ceramic components for critical applications.

The centre has already developed silicon carbide based light-weighted substrates up to the diameter of 730 mm for space optics applications under a sponsored program. The centre has also developed large area CVD coated SiC parts for many applications due to their relatively low co-efficient of thermal expansion and extremely smooth surface that can be achieved upon polishing. Recently, a high level of surface finish ( $< 1$  nm RMS roughness) has been achieved on the mirror blanks by adopting a specialized coating and subsequent polishing. The other ongoing major sponsored program in the centre include the development of non-oxide ceramics for ballistic applications. In view of this vital technology become indigenously available and to explore the export potentiality, ARCI has established most of the state of the art processing facilities for producing large size ceramic ballistic protective components.

The centre developed near-net shape ceramics including dense and porous SiC parts which find applications in hot gas and molten metal filtrations, heat exchangers, and volumetric solar radiation absorbers etc. SiC foams with wide range of porosity have been produced through optimization of various gelcasting parameters including surfactant concentration, slurry viscosity and solid loading. Ongoing R&D activities of the centre also include development of ready to press SiC powder through proper selection of additives and binders, nano composites either by using nano powder as primary phase or incorporating them in the matrix as a secondary phase. Recently, the centre has adopted a superior powder processing technique namely spray freeze drying to process powders of various sizes including nanopowders. The centre is also actively working on development of SiC based receiver tubes and joining the same for use in solar thermal power generation, nitride based ceramics with low dielectric constant and excellent mechanical properties to protect antenna systems for hypersonic space vehicles. In addition, the centre has successfully developed the technologies for range of products such as reaction bonded and pressureless sintered silicon carbide based mechanical seals, wear resistant parts etc.

# Effect of Porosity on Thermal Conductivity of SiC Foams by Aqueous Gelcasting

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Silicon carbide (SiC) based ceramic foams exhibit unique advantages of low density, high surface area, high permeability etc. in combination with the excellent properties of SiC including high temperature stability, high mechanical properties, high thermal conductivity, chemical inertness and long life in severe environments. Thermal conductivity of SiC foams plays an important role in many applications including in hot gas and molten metal filtrations, heat exchangers, thermal protection system and volumetric solar radiation absorbers. The study highlights the effect of porosity on thermal conductivity of solid-state sintered SiC foams processed through direct foaming followed by gelcasting and sintering technique.

SiC foams were prepared by foaming of SiC slurries in presence of cetyl tri-methyl ammonium bromide (CTMAB) as surfactant by tumbling in a roll mill using zirconia balls. Subsequently, foamed slurries were cast in aluminium moulds, gelled at normal temperature and dried in a humidity controlled drier followed by binder removal at 500°C and sintering at 2150°C. SiC foams with relative density ( $\rho/\rho_s$ ) ranging between 0.12 and 0.34 were prepared through optimized gelcasting parameters including surfactant concentration, slurry viscosity and solid loading. Fig. 1 shows the decrease in thermal conductivity (measured by laser flash technique) of SiC foams with increase in temperature. It is clear from Fig. 1 that thermal conductivity of dense SiC is much higher than SiC foams and the foams with lower relative density exhibited lower thermal conductivity. The decrease of thermal conductivity with increase in temperature is attributed to the increased phonon scattering from pores and grain boundaries which are typical for polycrystalline materials.

Several heat transfer models based on the thermal conductivity of constituent phases were considered to analyze the heat transfer mechanism in SiC foams. The decrease of thermal conductivity with relative density ( $0.12 < \rho/\rho_s < 0.34$ ) was explained by considering the heat conduction by gas and solid phases. The negligible contribution of convection and radiation effect to the effective thermal conductivity could be attributed to the lower cell size ( $< 800 \mu\text{m}$ ) in the foams prepared by direct foaming. The decrease of thermal conductivity at 500°C with relative density in Fig. 2 showed close agreement with the model as predicted by Ashby:

$$k = \frac{1}{3} k_s \frac{\rho}{\rho_s} + k_g \left(1 - \frac{\rho}{\rho_s}\right)$$

where  $\rho$  is the overall density of the foam,  $\rho_s$  is the density of solid phase in the cell wall,  $k_g$  is the conductivity of the gas phase and  $k_s$  is the conductivity of the solid phase. According to Ashby's model the structure of low density isotropic foams are consisted of cells with thin walls and edges where approximately one-third of struts are aligned towards the direction of the heat flow, resulting in effectively 1/3 of solid phase contribution to the heat transfer.

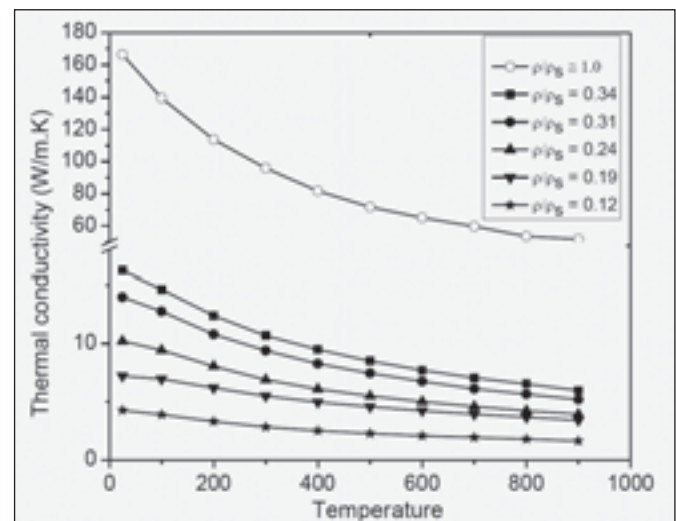


Fig. 1 Thermal conductivity versus temperature of SiC foams with different relative density

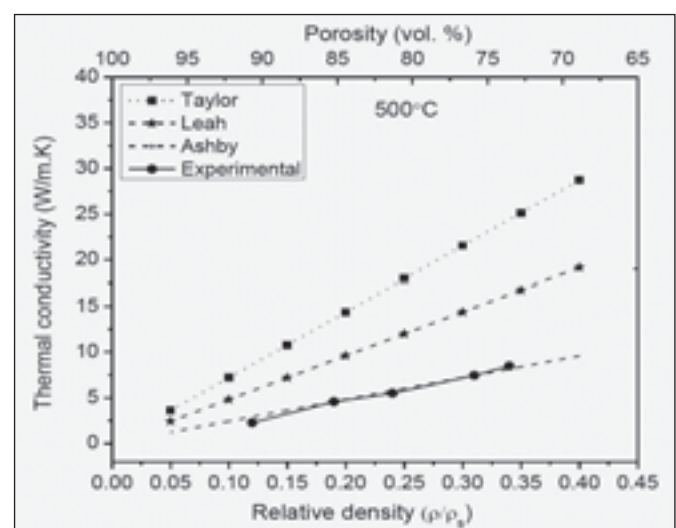


Fig. 2 Comparison of thermal conductivity versus porosity of SiC foams between experimental measurements and analytical predictions at 500°C

Contributor: G Sundararajan

# Development of Nano-Silicon Carbide Based Ready to Press Granules using Freeze Granulation Technique

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Silicon carbide (SiC) is a potential candidate material for numerous applications such as protective armors, aluminium electrolysis cell, as well as nuclear fuel coating for next generation fusion reactors because of its outstanding mechanical, thermal, and oxidation resistance properties. Nevertheless, the incessant demands for strategic and high performance applications require the properties of SiC to be continuously upgraded. In order to improve the properties of sintered SiC, basic granule properties of starting powder plays an important role.

The objective of the present study is to develop ready to press (RTP) SiC granules comprising primary nanoparticles, using freeze granulation (FG) technique. In FG techniques, the ceramic slurry is atomized into droplets and immediately fed into liquid nitrogen where instant freezing of the droplets taken place. The frozen granules are dried using freeze drier through the sublimation of ice into vapour. The superiority of FG as compared to spray drying technique is reported in literatures, as FG produces soft granules with homogeneous distribution of binders, additives across the granules. A higher density of green compacts is achieved through better pressing efficiency of soft granules during compaction.

In this present work, nano-SiC based spherical shape RTP granules (Figure 1) have been successfully produced adopting freeze granulation technique. Further, the investigation has been carried out on variation of green density of compacts with applied pressure. The results are shown in Figure 2 indicating that as the applied pressure increases from 200MPa, the relative green density also increases progressively upto 600MPa; however, there is no substantial increase in green density is observed beyond

600MPa. Therefore, 600MPa is considered as optimized compaction pressure.

As the applied pressure increases, the granules started rearrangement within themselves leading to improvement in green density. Further, with the increase in pressure, the intergranular and intragranular pores collapsed and primary particles come closer to each other. As the pressure increases more, the extent of rupturing of such pores is progressively higher, which eventually increases the density. As no increase in green density is observed beyond 600MPa indicating that pore closure is not possible anymore and particles are at shortest distance. Further, increase in applied pressure may generate laminations in the compacts. A high relative green density (~ 62%) was achieved in green compacts obtained from such granules, which is difficult to achieve using conventional processing route.

A suitable carbon (sintering aid) imparting agent was selected to process the nano-SiC powder for freeze granulation process. Sintering of the compacts was carried out at 2100°C with dwell of 1 h under argon atmosphere. Figure 3 depicts the carbon content vs. density plot, which shows that a maximum relative density ~ 97.4% is obtained using 3 wt% carbon and 1 wt % boron based compound as sintering activators. The present experimental results show the efficacy of FG process to produce nano-SiC based RTP granules in commercial scale.

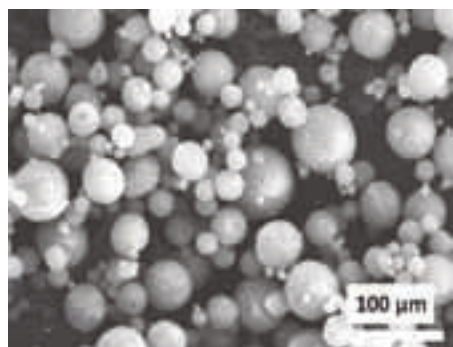


Fig.1 SiC Granules made adopting freeze granulation technique

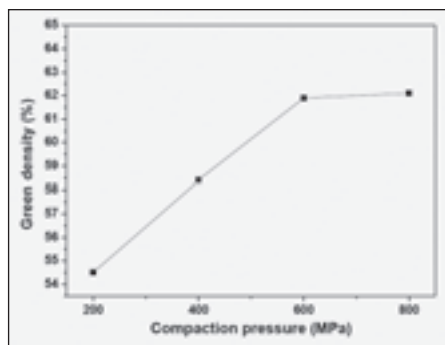


Fig. 2 Applied pressure vs. green density

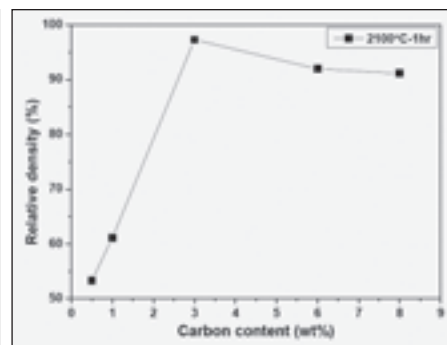


Fig. 3 Carbon content vs. sintered density

Contributors: Bhaskar Prasad Saha and B V Shalini





## Centre for Carbon Materials

Carbon has been a part of our life since ancient times. The benefits of carbon have never been far away from mankind and making our lives more comfortable. Carbon has long been known to exist in three forms: amorphous carbon, graphite, and diamond. However, the Nobel Prize-winning discovery in 1985 of buckminsterfullerene C<sub>60</sub>, which is a pure carbon molecule with a soccer ball-like structure consisting of 12 pentagons and 20 hexagons facing symmetrically, has created an entirely new branch of carbon chemistry. The subsequent discovery of carbon nanotubes in early nineties opened up a new era in materials science and nanotechnology. Carbon nanotubes (CNTs) have been under scientific investigation for more than fifteen years because of their unique properties that predestine them for many potential applications. The field of nanotechnology and nanoscience push their investigation forward to produce CNTs with suitable parameters for future applications. There are various techniques to synthesize carbon nanotubes such as arc discharge, chemical vapor deposition, laser ablation etc. However, each technique has its advantages and limitations. In the centre of carbon materials, carbon nanotubes are synthesized through electric arc discharge in which two graphite electrodes are used and carbon nanotubes were synthesized whereas in chemical vapor deposition technique vertically aligned carbon nanotubes were grown on a substrate such as silicon wafer, quartz, etc. To get consistent properties of carbon nanotubes is still a challenge and subject of research for many research groups. These vertically aligned carbon nanotubes find applications in the field emitter, as they possess very high aspect ratio, atomically sharp tip, very high thermal & electrical conductivities apart from their chemical inertness. These vertically aligned carbon nanotubes were patterned with the help of ultra fast laser technique in turn which enhances the emission properties due to more edge carbons.

Carbon nanomaterials have an extraordinarily high surface area to volume ratio and electrochemical redox properties. These properties would bring beneficial effects if they could be retained when the material is assembled into a structure capable of being used as a nano-structured electrode. Among all the nanomaterials, carbon nanotubes (CNTs), graphene and nano-structured carbon have been widely studied as anode materials for lithium batteries since their unique structure allows rapid insertion/deinsertion of lithium ions as compared to conventional graphite. Surface layer formation and safety concerns limit performance of pure CNTs. Composites or hybrid structures of CNT / graphene will provide improved charge cycling characteristics. In the centre, activities related to development of carbon nano-structured based nano-electrodes for super-capacitor and batteries which find applications in energy storage devices have been initiated.

The properties of CNTs, strongly depend on the extent of CNT dispersion and strength of the interfacial adhesion, because CNTs are generally considered to be non-uniformly dispersed in polymers because of van der Waals attraction between the carbon nanotubes, which can lead to the formation of strongly bound aggregates. The nanotubes agglomerates reduce the surface area and interrupt formation of the network structure which is essential to improve electrical and mechanical properties and to induce efficient transfer of their superior properties to the polymer matrix. Improved wetting and interfacial bonding between polymer matrix and carbon nanotubes are needed to produce superior carbon nanotube composites. In Centre for Carbon materials, we have been working for the synthesis of carbon nanomaterials via various techniques and their processing such as purification, functionalisation and their dispersion for various applications.

# Carbon Nanotube Based Nanofluid for Heat Transfer Applications

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Nanoparticles suspended or dispersed in a base fluid are termed as nanofluid. Depending on their thermal conductivity, various types of nanoparticles (ceramics, carbon, metals and metal oxides) are used in nanofluids. Carbon nanotubes possess remarkable thermal conductivity (~3000 W/M/K) due to which it may be an ideal particle for development of nanofluid. Production processes for carbon nanotubes often produce mixtures of solid morphologies that are mechanically entangled or that self-associate into aggregates. Entangled or aggregated nano particles often need to be dispersed into fluid suspensions in order to develop materials that have unique mechanical characteristics or transport properties. The extent of dispersion of CNTs in aqueous media is a method to qualify and compare the efficacy of functionalization procedures. Figure 1 shows the carbon nanotubes micrographs and the dispersed nanotubes solution.

Herein, the optimization of process parameters (surfactant concentration, type of surfactant, sonication duration) for the efficient dispersion of CNT in water was optimized. Two types of surfactants were used, cationic and anionic. The effect of concentration of cationic (CTAB) and anionic (SDS) surfactant and sonication duration on dispersion of CNT was illustrated in Figure 2 (a) & (b), respectively. Heat transfer was measured in double pipe counter flow heat exchanger system as shown in Figure 3. In this system cold water will flow in from one end i.e.  $C_1$  and flow out from  $C_2$  and simultaneously hot water as shown as  $H_1$  and  $H_0$  is flowing in the outer pipe. The overall heat transfer was enhanced by 1.51% and 0.83 % for ARCI CNTs and commercially available CNTs as shown in the Figure 4. When the CNTs percentage is very low i.e 0.1 & 0.5% due to fouling effect (CNTs get deposited in the side walls of the heat exchanger) over all heat transfer comes down.



Fig. 1 CNTs Micrograph and dispersed nanotubes solution

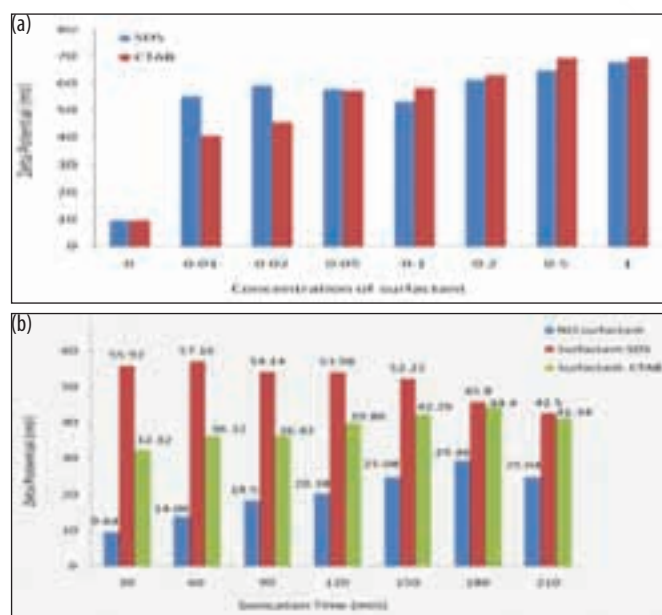


Fig. 2 (a) Effect of concentration of cationic (CTAB) and anionic (SDS) surfactant and (b) sonication duration on dispersion of CNT

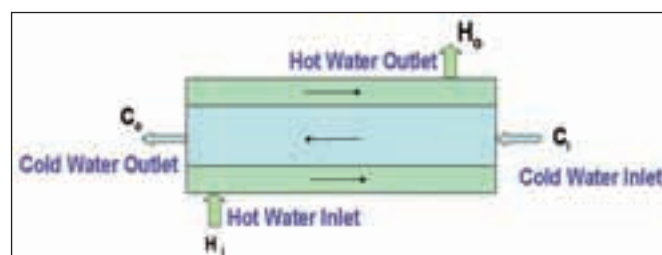


Fig. 3 Double pipe-counter flow heat exchanger

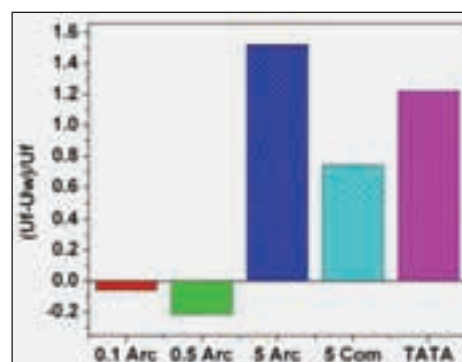


Fig. 4 Graph showing the overall heat transfer enhancement for ARCI CNTs and commercially available CNTs

# Preparation and Optoelectronic Properties of Few Layer Nanoscaled-Graphene Sheets/Polyaniline Nanocomposites

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Graphene, one-atom-thick 2-dimensional planar sheet of carbon atoms densely packed in a honeycomb crystal lattice, has grabbed appreciable attention due to its remarkable electronic, optoelectronic, thermal, mechanical and electrical properties.

Conducting polymers are well known for their high flexibility, electrical / thermal / optical properties, environmental stability and relatively high specific capacitance. Among these materials, polyaniline (PANI) has been considered as one of the most promising electrode materials because of its low cost, easy synthesis and relatively high conductivity. PANI exists in several oxidation states with electrical conductivity varying progressively from 1-100 S/cm.

Compared to carbon nanotubes, graphene nanosheets (GNS) are predicted as an excellent support material due to their high surface area, remarkable mechanical stiffness and excellent thermal and mechanical conductivity.

Herein, GNS-PANI (GNP) composite was prepared by in-situ chemical polymerization by varying the concentration of few layered graphene sheets. The oxygen related surface active sites on GNP surface were generated by functionalization to increase the interfacial bonding between GNS and PANi. As expected, properties of PANI-GNS composites are intermediate between pure PANI and GNS but vary depending on GNS content loading and the extent of its integration with PANI molecules. Composite material exhibits a three-step decomposition i.e., release of water molecules (<100°C), dopant anion (180–280°C), and decomposition of PANI (280–630°C). Thermal stability of composite was improved with increase in GNS loading.  $\pi$ - $\pi$  Interaction between PANI and GNS lead to conformal coating as well as firm bonding. Thus, the functional groups attached on GNP surfaces will act as anchoring sites for the PANi nucleation and firm interfacial strength.

The absorption coefficient  $(\alpha h\nu)^2$  is plotted against the photon energy ( $h\nu$ ) for GNP, PANI and composites and was illustrated in Figure 2. The intercept of this plot on the photon energy axis gives the bandgap ( $E_g$ ) of the PANi. Bandgap of the composite material was drastically decreased by GNS inclusion in composite.

The synergetic performance of GNS-PANI composites finds application in batteries, supercapacitors, dye-sensitized solar cells, biosensors and microbial fuel cell.

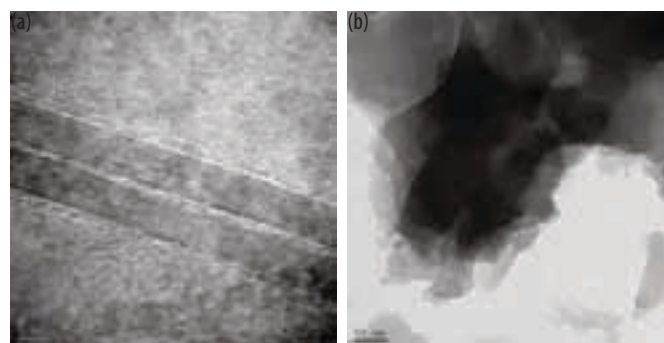


Fig.1 (a) TEM micrographs of GNS and (b) GNS-PANI composite

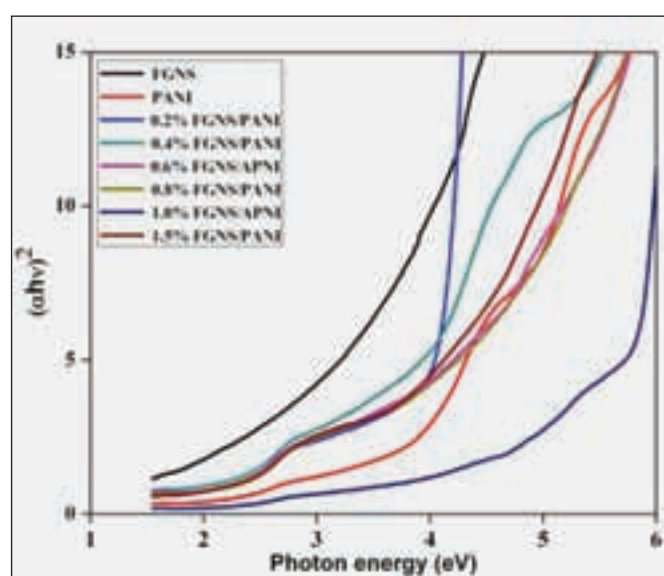


Fig. 2 Tauc plot for GNS-PANI composites

Table 1 Electrical conductivity and bandgap energy of PANI-GNS composites

| Sample        | Band gap (eV) | Electrical Conductivity (S/cm) |
|---------------|---------------|--------------------------------|
| GNS           | 2.40          | -                              |
| PANI          | 3.44          | 2.54                           |
| 0.2 FGNS/PANI | 3.34          | 1.41                           |
| 0.4 FGNS/PANI | 3.19          | 4.92                           |
| 0.6 FGNS/PANI | 3.18          | 2.78                           |
| 0.8 FGNS/PANI | 3.06          | 0.27                           |
| 1.0 FGNS/PANI | 3.03          | 0.26                           |
| 1.5 FGNS/PANI | 3.01          | 0.17                           |

Contributor: P K Jain





## Centre for Sol-gel Coatings

**O**rganic-inorganic hybrid nanocomposite coatings generated through sol-gel technique has been an active field of research since they are extremely promising for commercial exploitation. The Centre has been working with several industrial partners for development and demonstration of sol-based nanocomposite coatings for a wide variety of applications and has recently been focusing on the following areas:

1. *Decorative coatings on glass for architectural applications*
2. *Chrome-free, self-healing, corrosion protection coatings on aluminum and its alloys*
3. *Anti-tarnish coatings on noble metals*
4. *Fully dielectric solar control coatings on glass for automotive and architectural applications*
5. *Eco-friendly, halogen-free flame retardant coatings on textiles*

*New sol formulations for decorative coatings for architectural applications were developed to achieve the target properties specified by the user industry. Challenges in the coating deposition using spray technique were overcome and the coating technology has been successfully transferred to an entrepreneur.*

*The development on hexavalent chrome-free, self-healing coatings on aluminum and magnesium alloys using organic/inorganic encapsulation materials like polymeric microcapsules, clay nanotubes and layered double hydroxides for containing the corrosion inhibitor is ongoing. Investigations were also carried out to evaluate the efficiency of sol-gel layers for sealing of the porosity in anodized layers. Promising preliminary results have been obtained and further work is underway.*

*Development of anti-tarnish coatings is ongoing. Consistency in obtaining good and uniform coatings on silver and gold coupons/articles possessing anti-tarnish property and perspiration resistance has been achieved after persistent efforts.*

*Use of metal-dielectric stacks and transparent conducting oxides for achieving solar control property on glass are cost-intensive. Hence, development of cost-effective, fully dielectric solar control coatings on glass for automotive and architectural applications has been initiated.*

*A new halogen-free, flame retardant formulation has been developed, which can not only delay the burning of the fabric, once ignited, but can also extinguish the flame.*

# Solar Control Coatings on Glass for Automotive and Architectural Applications

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There are concerted efforts by researchers and technologists on making energy efficient windows for buildings and automobile application due to the growing awareness on global warming. A solar control glass, usually based on multilayer stacks of metal (mostly Ag or Au) in conjunction with dielectric layers either reflects or absorbs a high percentage of incident near-infrared (NIR) and UV radiation, while maintaining a high level of visible light transmittance. Inherent disadvantages with such coating stacks are poor visible light transmittance and sensitivity to environment, in addition to being cost-intensive. A cost-effective substitute would be to have a sol-gel derived, fully dielectric system consisting of alternating high and low refractive index material layers of appropriate thickness required by the optical design. An interference filter is thus generated that can be tuned to reflect radiation in the near-infrared region, while transmitting a high percentage of visible light. From a technology point of view, it would be beneficial to minimize the time taken for intermediate firing of the layers in the coating stack by employing a rapid firing rate using a conveyORIZED belt furnace. In this backdrop, the effect of heat treatment time on the optical properties of the solar control coating stack designed with the configuration Glass|T|S|T was studied. Here, T refers to a  $\text{TiO}_2$  layer and S refers to a  $\text{SiO}_2$  layer. The stack was fired simultaneously in a conventional muffle furnace (total cycle time per layer: 330 minutes) and in a conveyORIZED belt furnace (total cycle time per layer: 30 min). The optical properties obtained using different firing schedules have been analysed using spectroscopic ellipsometry.

The spectral variation of transmittance for the trilayer solar control coating stack after batch and belt furnace heat treatments along with that of a bare soda lime glass (SLG) substrate is shown in Figure 1. In order to explain the difference in the solar control properties of the coated glass substrates, the single layers of  $\text{TiO}_2$  and silica fired in batch and belt furnaces were studied using spectroscopic ellipsometry. The fitting of the experimental data obtained from ellipsometry analysis for single layered  $\text{TiO}_2$  and  $\text{SiO}_2$  coatings was carried out using the Cauchy model. The variation of refractive index of the layers with wavelength is shown in Figure 2. It could be independently discerned that the decrease in the overall transmittance in belt furnace treated trilayer stack is due to the effect from  $\text{TiO}_2$  layer only, because no variation was found in the optical properties of pure silica layers whether heat treated in a

belt or batch furnace. The reason for this could be that one of the additives used in the  $\text{TiO}_2$  sol synthesis is retained during the rapid heat treatment in the belt furnace and this material, could additionally block the UV, visible light and NIR radiation.

The present study showed that slow firing of the trilayer solar control coating stack could be employed for automobile windows and windscreens, and fast firing could be suitable for architectural applications.

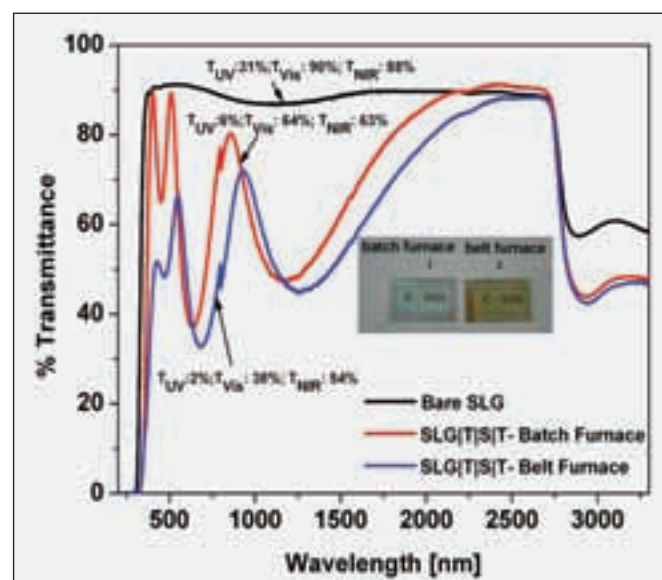


Fig. 1 Comparison of the spectral transmittance of trilayer coating stack fired in batch and belt furnace with that of bare soda lime glass substrate

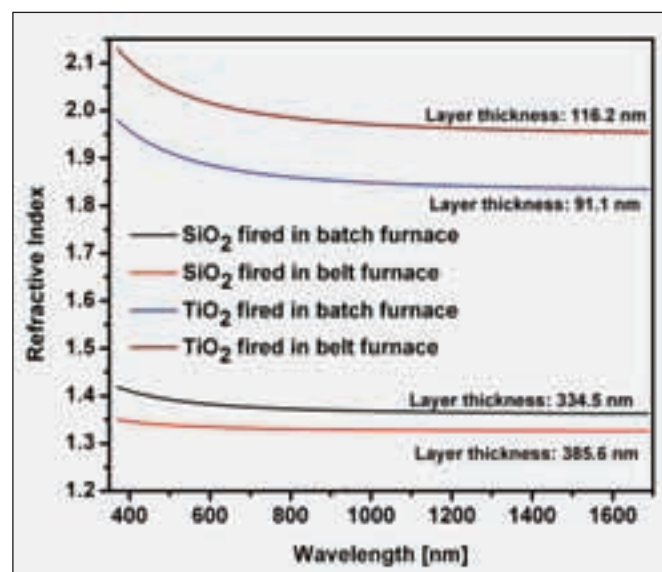


Fig. 2 Refractive index values of single layer  $\text{TiO}_2$  and  $\text{SiO}_2$  coatings fired in batch and belt furnace as a function of wavelength along with layer thicknesses

Contributor: S Manasa



# Improvement in Borosilicate Glass Transmittance by an Etching Process

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Optical elements based on glass have indices of refraction ( $n$ ) in the range of 1.45–1.7 and, as a result, reflect from 4% to more than 6.5% of normal incident light from each air–substrate interface. The additional energy loss of reflected radiation can be especially detrimental to a system's performance when there are several optical components involved. Reflection losses from optical components are notably disadvantageous in technologies such as solar cell and particularly in CSP collectors, which rely on efficiently transmitted energy. It is therefore necessary to reduce the amount of reflected light to improve the overall performance and efficiencies of such systems.

A reduction in surface reflection is typically accomplished by the application of an anti-reflection coating of a quarter-wavelength optical thickness. When the index of refraction of the coating ( $n_c$ ) is equivalent to the square root of the product of the indices of the surrounding medium ( $n_o$ ) and the substrate ( $n_s$ ), that is,  $n_c = (n_s n_o)^{0.5}$ , reflections are suppressed at wavelengths near the quarter-wavelength optical thickness. Consequently,  $n$  must be between 1.2 and 1.3 for efficient anti-reflection coatings to be suitable for glass optical elements. This is, in general, a difficult task because readily available low  $n$  materials are limited to values of  $\sim 1.35$ . However, the effective refractive index of 1.35 can be achieved by introducing porosity and graded index (or moth eye structure). A sol-gel film containing sacrificed porogen would also generate about 40% porosity after calcinations. However, for products intended at outdoor applications, such as solar panel and solar thermal cover glasses, the coatings need sufficient mechanical strength to withstand the weather conditions.

A novel alternative etching method has been realized to improve the borosilicate glass (BSG) transmittance significantly. Figure 1 shows the transmittance spectrum of bare and etched BSG substrate. Bare BSG shows average transmittance (400 to 1200 nm) of 91.6% whereas etched BSG shows 97.1%. The under and over etched BSG shows relatively lower transmittance in the required spectrum of wavelength. Figure 2 shows the surface morphology of etched and bare BSG substrate. It has been confirmed by X-ray photo electron spectroscopy (XPS) that the etching process selectively leaches sodium oxide, one of the constituents of BSG. As the graded index or moth eye structure introduced by this method is a part of the substrate, it can have long lasting service properties.

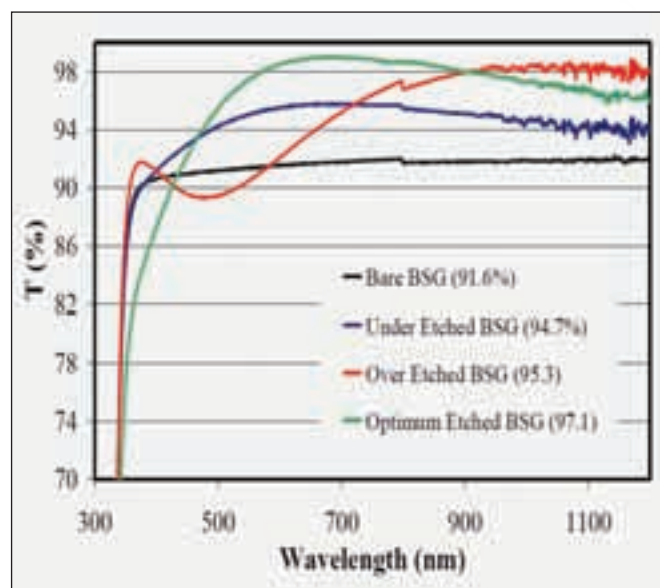


Fig. 1 Improvement in the borosilicate glass transmittance by etching process

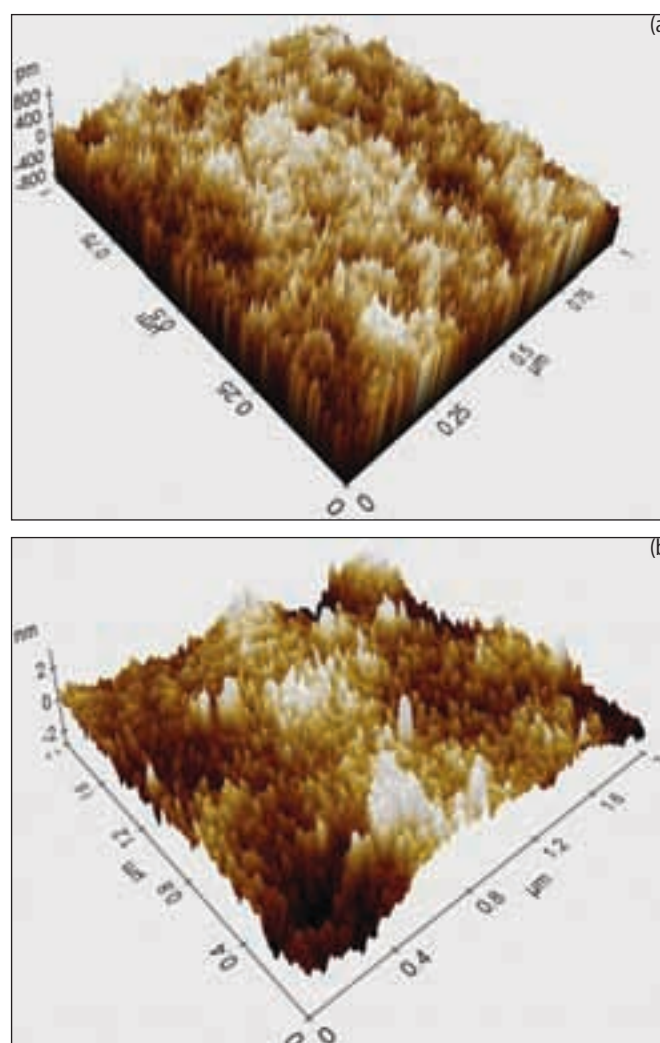


Fig. 2 AFM topographic image BSG substrate (a) bare (b) etched



# Assessment of Corrosion Resistance of Sol-Gel Coated Anodized Aluminium

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Aluminium and its alloys are being extensively used as part of house hold appliances, industrial machine components and aviation structural elements for many decades. Low density, high thermal conductivity, excellent machinability and formability are some of the advantages favoring the material for such applications. Nevertheless, they are susceptible to corrosion in acidic and saline environments, thereby limiting their service life. Many surface engineering techniques such as anodizing, electrochemical plating, conversion coatings such as chromating and organic films such as painting are being employed for improving the corrosion resistance of aluminium. Low temperature curable hybrid sol-gel coatings have been proven to be effective when compared to some of the above-mentioned surface engineering techniques in enhancing corrosion resistance. Sol-gel compositions can be tailor-made depending on the substrate compositions and end property requirements. Coatings can be cured using either thermal energy or UV/NIR irradiation depending on the temperature sensitivity of substrate.

Published literature shows that irrespective of the composition employed, all sol compositions such as silica, alumina, zirconia and titania have shown some amount of corrosion protection of aluminium substrates. But, none of the published literature so far has brought out the effect of a combination of sol-gel coatings and anodizing on corrosion protection. Hence, the present investigation was undertaken with a view to fill the gap in assessing the corrosion resistance of optimally thick sol-gel coating vis-à-vis equivalent coating thickness samples of anodized and a duplex sol-gel coating on anodized aluminium substrates.

A low temperature curable organic-inorganic hybrid silica sol was deposited on commercially pure aluminium substrates in uncoated and anodized condition and was thermally

cured in an air circulating drying oven at 130°C for 1 h. The coating thickness was varied over a range so as to identify a critical thickness that will yield the best corrosion resistance. Corrosion resistance of the only sol-gel coated aluminium with optimized thickness those of minimum coating thickness anodized along with anodized and sol-gel duplex coated substrates having an equivalent thickness was measured by potentiodynamic polarization measurements in 3.5% NaCl solution. The experimental results presented in Table 1 reveal that the maximum possible thick sol-gel coated aluminium exhibited similar corrosion resistance as that of minimum coating thickness of anodized aluminium when individual layers were compared. However, an enhanced corrosion resistance was observed when employed as a duplex coating system as compared to equivalent coating thickness of the anodized layer alone. Figure 1 depicts the surface morphology of sol-gel, anodized and duplex coating system. Poor performance of the anodized layer could be explained based on the open surface porosity of anodized coating when compared to a dense microstructure of hybrid sol-gel coatings.

Table 1. Results of potentiodynamic polarization tests of duplex and anodizing coated samples in 3.5 wt% NaCl solution

| Coating/coating thickness, $\mu\text{m}$ | $R_p$ (Ohms/cm <sup>2</sup> ) | $I_o$ (A/cm <sup>2</sup> ) | $E_o$ (Volts) |
|------------------------------------------|-------------------------------|----------------------------|---------------|
| Minimum sol-gel, 2.3 $\pm$ 0.32          | 86568                         | $3.013 \times 10^{-7}$     | -1.2182       |
| Minimum anodizing, 6 $\pm$ 0.34          | 82306                         | $3.170 \times 10^{-7}$     | -1.10721      |
| Duplex, 8.3 $\pm$ 0.3                    | $1.8122 \times 10^5$          | $1.4395 \times 10^{-7}$    | -1.2814       |
| Equivalent anodizing, 8.5 $\pm$ 0.47     | $0.821 \times 10^5$           | $3.1776 \times 10^{-7}$    | -1.7899       |
| Uncoated aluminium                       | 10225                         | $2.5513 \times 10^{-6}$    | -0.81844      |

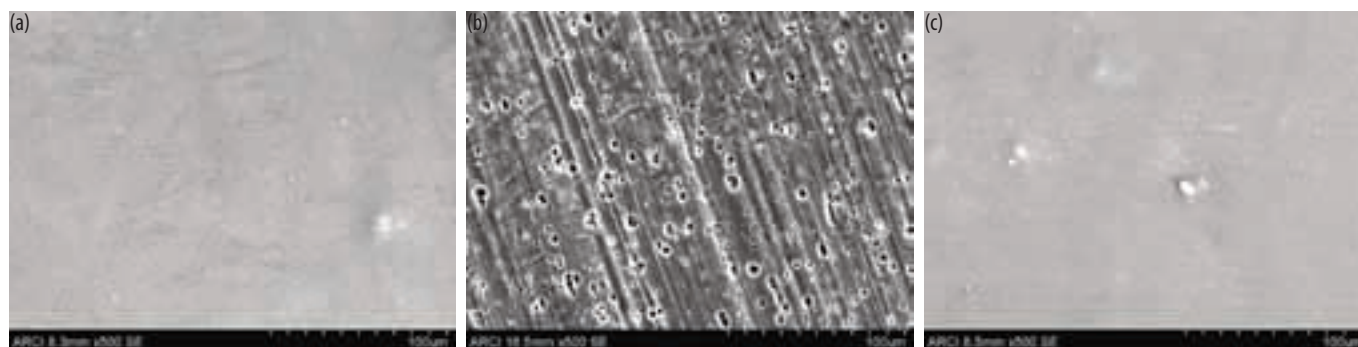


Fig. 2 SEM images of a) sol-gel coated, b) anodized and c) anodized & subsequently sol-gel coated aluminium samples

Contributor: A Jyothirmayi

# Automation of Flow Coating Process: Feasibility Studies

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Flow coating is one of the wet chemical sol-gel coating deposition techniques on the substrates. This coating technique is suited to coat large or oddly shaped parts that are difficult to coat with dip coating technique. Literature study describes that tailored made apparatus have been designed for specific application like for continuous type of flow coatings on magnetic tapes and batch type flow coating on electronic displays and circuit boards to name a few. They are also called as "curtain coatings". In general flow coating is achieved on the substrate by positioning the substrate in a suitable manner and the coating apparatus moves over the substrate allowing a laminar flow.

In the present application the apparatus is intended to have a uniform coating through the length of the substrate. The idea was to apply coat on a 4 meter long pipe. Automation is necessary to have the flow rate along the pipe to be constant to achieve uniform coating. This can be achieved by the pressure compensation method, best suited since the apparatus is fixed in a linear position for the gravity aided flow. The feasibility study was carried out for a length of one meter flat substrate.

The apparatus as shown in Figure 1, consists of a 1 meter long glass tube. The other end of the glass tube contains two valves. The first valve has fine control on the orifice of the outlet. The fine control approximately varies the opening of the valve from a minimum value of 0.4 mm to maximum of 6 mm. The second valve is an ON/OFF valve. This helps in quantifying the flow rate. The top of the glass tube can be closed with a bell shaped lid. The lid has internal provision to hold the one meter substrate and externally can be connected to a hose to feed compressed air pressure. The diameter of the apparatus is 30 mm with 120 cm length and the orifice is 6mm diameter, which can hold 850 cu.cm of liquid.

Four experiments were conducted using water and solution, with and without air pressure. The time was measured at equal intervals of height as the liquid was flowing out of the column. Graph 1 shows the volume out flow vs. time. It is observed that the positive pressure has an effect on the constant flow rate. The results may not be absolute since the operation was manual and the pressure compensation was not dynamic. Graph 2, shows second derivate of  $dV/dt$  Vs time. Pressure level compensation for the time duration adjustment can be seen clearly in the graph for uniform flow of liquid along the column.

Automation will be worked out subsequently after studying the uniformity, coating thickness and the thickness tolerances levels for the application.

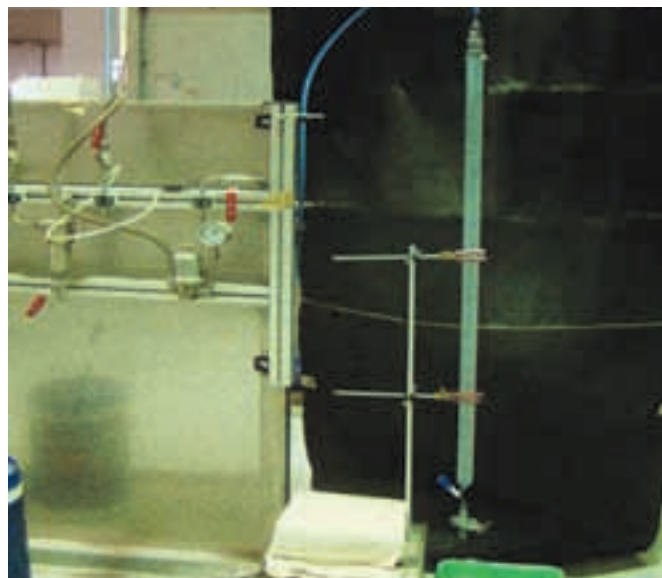


Fig. 1 Flow coat apparatus

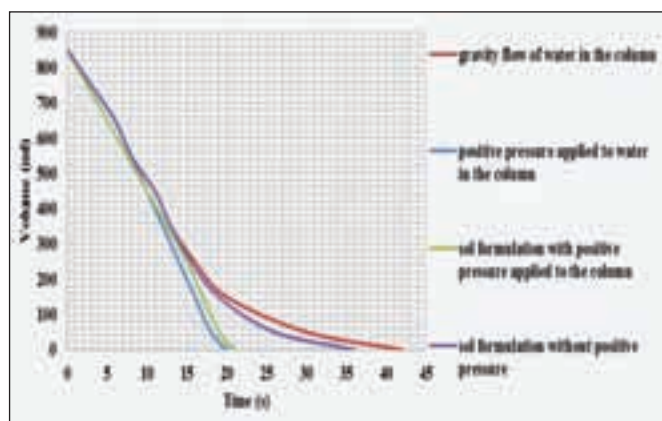


Fig. 2 Volume out flow vs time

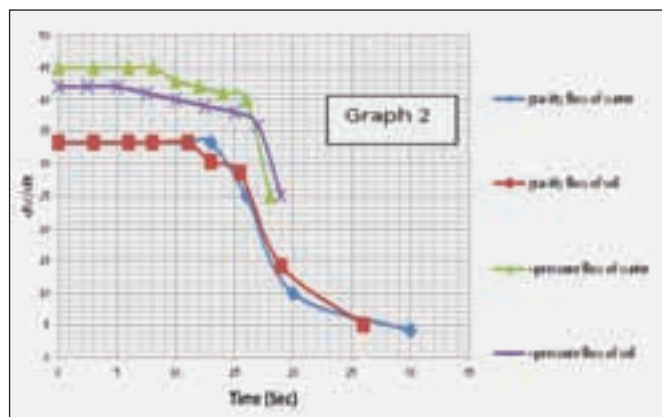


Fig. 3 Pressure compensation for the outflow

# Hybrid Sol-Gel Decorative Coatings on Glass and Plastics

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Colored coatings on glass are replacing colored glass due to difficulties in recycling colored glass as well as due to limitations in obtaining a wide variety of colors in case of the latter. Single layered hybrid sol-gel coatings on glass and plastics are being investigated as a substitute to other conventional polymer-based decorative coatings, which usually are easily susceptible to scratches and do not possess sufficient solvent, detergent and hot water resistance. The advantages of organic-inorganic hybrid coatings are that they can be obtained as thick and scratch-resistant coatings in addition to being amenable to densification at low temperatures or sometimes even at room temperatures. In the present study, different sol formulations were generated by varying the raw material contents in a sol composition comprising various organically modified silanes and metal alkoxide. Each organically modified silane and metal alkoxide bestows different properties in the multifunctional coating derived from the sol. The different raw materials used have been abbreviated as (A) Acrylic modified silane, (B) Aryl modified silane (C) Alkyl modified silane, (D) epoxy modified silane and (E) Aluminum/Titanium/Zirconium alkoxide. Four sol compositions with varying contents of A, B, C, D and E were synthesized and investigated to obtain an optimized composition, that would yield the maximum coating thickness and scratch resistance after deposition on glass and plastic substrates.

Table 1 shows the thickness of the coating deposited and shelf-life of the synthesized sol, obtained by varying the composition and ratio of the used precursors, respectively. The optimized sol composition yielding transparent and thick coatings was combined with suitable additives to generate different colored sols. The colored sols were deposited on glass and plastic substrates by spray coating followed by subsequent densification at room temperature and curing in an oven at temperatures lower than 150°C for 1 h. Table 2 presents the mechanical properties of the coatings generated with the optimized composition. Coating applied on glass resulted in coatings with excellent scratch hardness of 9H when heat treated up to 150°C, while the same composition when applied on plastic and heat treated up to 130°C resulted in a scratch hardness of 5H only. Coating cured at higher temperature also shows better adhesion indicating a minimum curing temperature of 150°C is essential for good mechanical properties.

The optimized sol composition could yield thick (12-15  $\mu\text{m}$ ), adherent, decorative, scratch, solvent and hot water resistant coatings. The results indicated that the hybrid coatings could be an eco-friendly substitute for normal paints. Decorative coatings as presented in Figure 1 were successfully demonstrated on glass idols and flat glass to assess commercial viability. The shelf life of the sol was also found to be one month, which is technologically desirable.

Table 1 Thickness of coatings obtained along with pot life of the sol for varying compositions

| Sl.No. | Composition               | Coating condition | Thickness           | Shelf life |
|--------|---------------------------|-------------------|---------------------|------------|
| 1      | BCDE / Equimolar          | cracked           | 6-7 $\mu\text{m}$   | 1 day      |
| 2      | ABCDE / High A            | cracked           | 6-7 $\mu\text{m}$   | 1 day      |
| 3      | ABCDE/ High A, Low D      | good              | 9-10 $\mu\text{m}$  | 3 days     |
| 4      | ABCDE/ High A, very Low D | good              | 12-15 $\mu\text{m}$ | 30 days    |

Table 2 Scratch hardness, densification condition and adhesion of coatings derived from the optimized sol composition (4)

| Sl.No. | substrate | Curing condition | Scratch hardness | Adhesion |
|--------|-----------|------------------|------------------|----------|
| 1      | glass     | Room temperature | 8H               | --       |
| 2      | glass     | 150°C/1 hour     | 9H               | 4B       |
| 3      | plastic   | Room temperature | 4H               | --       |
| 4      | Plastic   | 130°C/1 hour     | 5H               | --       |



Fig. 1 Decorative sol-gel coated (a) glass article and (b) flat glass sheet using optimized sol composition





## Centre for Materials Characterization and Testing

*The primary mandate of ARCI is develop and transfer technologies to the industry, and materials play a very important role in realizing this. Once a material is synthesized, either with a new composition or a novel microstructure, the immediate need is to characterize and understand it to the fullest extent possible. Thus, the need for accurate and timely characterization can never be overemphasized.*

*The Centre for Materials Characterization and Testing at ARCI has established facilities that are especially suited for in-depth study of microstructure. Electron microscopes (both scanning and transmission) and other equipment are specifically geared to this end. The ARCI website lists the facilities at the Centre and these are available to users within the Organization and to external users from the Industry and Academia.*

*To augment the existing facilities and to enhance the scope of the work, the following three systems have been procured and installed during the previous year:*

- Microfocus x-ray diffraction unit. This system is equipped with a rotating anode generator for high intensity x-rays and the spot size can be collimated to 50  $\mu\text{m}$ , making it particularly suitable for the study of secondary phases that occur as precipitates.*
- Nanoindenter system with impact testing facility. This unit is also capable of performing scratch and wear measurements and a built-in atomic force microscope provides the capability to carry out imaging in-situ.*
- Creep testing: Two units have been installed, one each with constant load and constant stress capability. Peak load is 53.4 kN and measurements can be performed up to a temperature of 1000°C.*

*Reports highlighting the R&D activities in the Centre are being presented.*

# Nano Mechanical Testing of Thin Hard Coatings

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The development of high performance nanostructured materials and thick/thin coatings requires suitable testing techniques to evaluate material properties at small length scales. ARCI is actively involved in the development of such materials and coatings for specific applications. It is very difficult to test these materials through conventional testing methods which require large sample volumes. In recent years, instruments have become available with various testing features for the study of the deformation behavior and material performance. In order to cover the testing of materials at various length scales and to carry out advanced tests such as nano-impact, nano-wear and profiling, in addition to conventional hardness and modulus measurements, the Nano-Test System from M/s. Micro Materials, U.K., has been successfully installed at the Centre. A photograph of the facility is shown in Figure 1. The system is equipped with the following five modules: low load head, high load head, optical microscope, nano-positioning stage and AFM.

The 'NanoTest' is a pendulum-based depth sensing system with electromagnetic loading. The indenter moves into the sample surface by movement of a frictionless pendulum by varying the current in the coil. The displacement is measured by capacitor plates up to the sub-nanometer range. The NanoTest System can also be used for impact testing of thin coatings. In this module, testing is possible either by oscillation of the sample (high cyclic fatigue) or by oscillation of the pendulum (low cyclic fatigue). In the case of pendulum impulse impact testing, it has been reported that results obtained from the nano-impact test are closely related to the cutting tool life as against statically measured hardness and scratch behavior obtained from instrumented indentation of thin coatings.

Nanoimpact testing is a rapid indentation technique involving repeated impact at fixed loads for preset time intervals. The high strain rates achievable in this technique can be used to closely simulate actual test conditions, especially in real life applications involving repeated contact such as machining where cutting tools experience fatigue due to constant on-off contact with the work piece. This technique has begun to be used to study thin film coatings, especially hard nitride coatings, and a close correlation is found between actual tool life and the impact resistance of these coatings. This proves to be an advantage since time-consuming industrial machining and testing can be replaced by the simple, lab-based impact test which closely simulates conditions seen in real life.

Shown below are selected results from initial experiments carried out with the Nanotest Vantage system at ARCI. Figure 2 (a) shows curves of impact depth vs. time on  $Ti_{1-x}Al_xN$  coatings while Figure 2 (b) shows the curves on TiAlN multilayers with bilayer periods ranging from 40 nm to 160 nm. From Figure 2 (a), it is evident that the impact resistance increases with increasing Al content. This is on account of the gradual change in the microstructure from coarse columnar grains at low Al content to finer, equiaxed grains and a nanocomposite grain structure at the highest Al content. From Figure 2 (b), an inverse linear dependence of the impact resistance with multilayer period is seen. It can be concluded that multilayers with low bilayer periods show increased impact resistance which can be attributed to the increased number of interfaces at lower bilayer periods which act as barriers to crack propagation. Thus, the nanoimpact test enables simulation of the small repetitive stresses that many modern materials undergo in real life over time and allows more accurate predictions of their behaviour and the design of better materials.

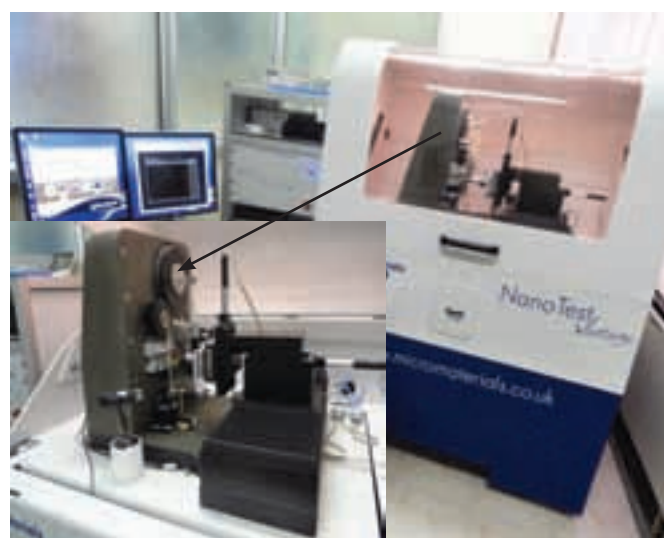


Fig. 1 Nano Test System

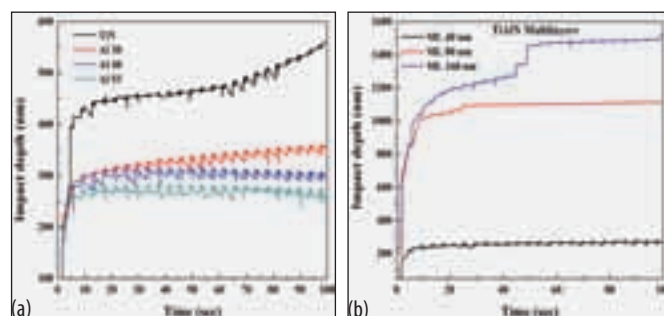


Fig. 2 Nano-impact test results on CAPVD (a)  $Ti_{1-x}Al_xN$  coatings monolithic and (b) TiAlN multilayers with bilayer periods ranging from 40 nm to 160 nm

# Transmission EBSD: A New Technique to Enhance Spatial Resolution

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A new technique called Transmission EBSD (t-EBSD) that improves spatial resolution of orientation imaging in FESEM which has gained impetus in recent times has been explored at ARCI. Conventional EBSD is carried out in reflection mode and the best possible spatial resolution is around 100 nm. Collecting kikuchi patterns in transmission from a sample in FESEM improves the resolution by an order of magnitude. The second major advantage is that the interaction volume in transmission mode is more symmetric when compared to that of in reflection. There are two reasons for this. First, the interaction volume is smaller in transmission mode and second, the angle that the sample makes w.r.t the beam is not so steep. For these reasons this technique can be applied well to materials having fine grain structure, i.e. < 100 nm, the size domain which is not resolvable using the conventional EBSD technique.

In order to generate and capture EBSD patterns in transmission, two conditions have to be met. First, the sample thickness should be decreased to around 100 nm. Second, positioning of the sample w.r.t both the electron beam and the phosphor screen should be such that the transmitted diffracted cones formed due to diffraction of backscattered electrons fall on the screen. To attain the first objective we can use the specimens prepared for TEM examination, as these contain a sufficiently thinned area. The Second objective can be met by tilting the sample to around  $40^\circ$  w.r.t the horizontal plane so that the exit plane of the EBSD patterns (the sample surface from which the patterns arise) faces the phosphor screen and also maintain a very low working distance (WD), so that sufficient number of Kikuchi patterns fall on the phosphor screen. In addition to these conditions the microscope imposes one more condition that the stage has to be tilted to  $70^\circ$  for the EBSD camera to be inserted into the specimen chamber. Finally, the recommended accelerating voltage for t-EBSD ranges from 25 to 30 kV. If the sample is too thin it might be necessary to decrease the accelerating voltage in order to increase the interaction volume for generating sufficient yield of backscattered electrons.

In order to carry out the experiment with the above mentioned conditions a sample holder has been designed and fabricated. Figure 1 shows the design of the holder and positioning of the sample during experiment. A steel sample that has been prepared for TEM using twin-

jet electro polishing has been employed to demonstrate t-EBSD. In order to make sure that the patterns captured are in transmission, the beam was moved from the edge of the hole to the periphery of the sample and image on the EBSD camera is observed. As we move away from the edge the pattern intensity decreases and dies down after some distance, which would not have been the case reflection geometry. A scan was run at the edge of the central hole and the results are shown in Figure 2.

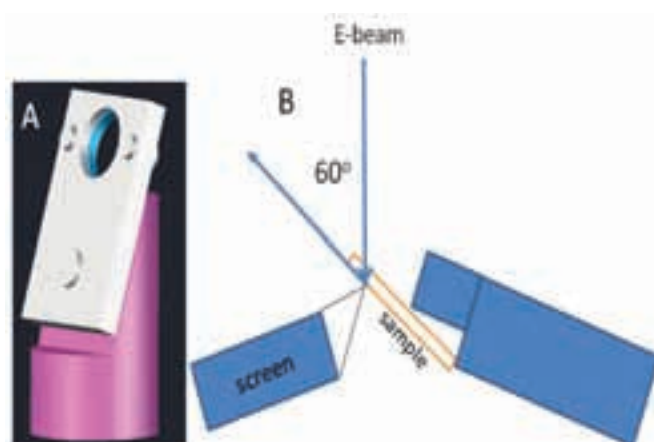


Fig. 1 (a) schematic of the t-EBSD sample holder, and (b) sample positioning in SEM chamber with reference to beam and phosphor screen

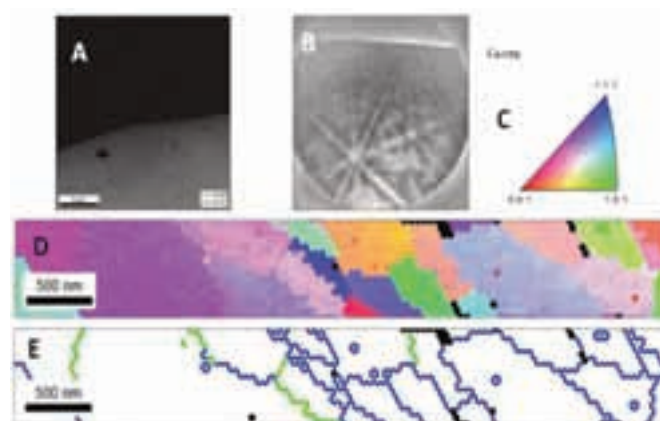


Fig. 2 a) SEM image of thin region close to the hole, b) t-EBSD pattern captured from same region, d) OIM image showing grains colored according to the inverse pole figure as shown in (c), and e) map showing distribution of high and low angle grain boundaries as blue and green lines, respectively.

This technique will help us to characterize microstructural features that were not possible to be accessed using conventional EBSD. Representative examples are the study of recrystallization behavior across splat boundaries in cold sprayed coatings and study of micro-texture in fine grained pulsed electrodeposited coatings.

Contributor: G Ravichandra



# Effect of Heat Treatment on Corrosion Performance of Cold Sprayed Tantalum Coatings

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Tantalum is a refractory metal with excellent machinability and a low ductile-to-brittle transition temperature. It has excellent resistance to corrosion by various acids (except HF), salt solutions and various organic chemicals even at elevated temperatures. A stable passive oxide film is formed on tantalum, which can provide protection against extreme corrosion. As cold gas dynamic spraying (CGDS) has emerged as a promising coating technique for deposition of high-density coatings, a portable cold spray system was fabricated in-house and the same has been used to deposit tantalum coatings of thickness up to 300  $\mu\text{m}$  on low carbon steel. The coatings were carefully removed from the substrate and heat treated at different temperatures in a vacuum furnace for two hours in order to see the effect of heat treatment on corrosion properties. Potentiodynamic polarization and impedance spectroscopy tests were carried out using SI 1260 Impedance/gain phase analyzer with SI 1287 electrochemical interface (Solatron, UK) for as-coated, heat-treated and bulk Ta in 1M KOH solution, (which is an aggressive environment) after exposure for 1 hour. The corrosion cell had a classic configuration of three electrodes, platinum as counter electrode, a saturated calomel electrode as reference electrode and the uncoated/coated sample as the working electrode.

The resulting potentiodynamic polarization plots are shown in Fig.1(a), and the analyzed data of corrosion potentials ( $E_{\text{corr}}$ ) and current densities ( $I_{\text{corr}}$ ) in Table 1. All the polarization plots show passive region showing the formation of protective passive film. However, there is some fluctuation in current density in the passive region of as coated sample which could be due to the presence of heavily deformed splats and strain hardened inter splat boundaries which are active sites for corrosion and prevent the formation of uniform passive film. The defects are decreased with heat treatment and uniform passive region is observed for sample heat treated at 1500°C. The current density and potential indicate that heat treatment enhances the corrosion resistance of the coatings. The corrosion currents of as-deposited coatings and those heat treated at 1500°C were found to be 7.184  $\mu\text{A}/\text{cm}^2$  and 0.703  $\mu\text{A}/\text{cm}^2$ , respectively, indicating a substantially superior performance of the heat-treated coatings approaching that of the bulk with a corrosion current of 0.528  $\mu\text{A}/\text{cm}^2$ . The corrosion potential for heat-treated coating at 1500°C was better than that of bulk Tantalum, even though it exhibited lower passivity.  $E_{\text{corr}}$  value is found to be less negative with increasing heat treatment temperature up to 1500°C. It is evident from the results that closing of pores and cracks, with

reduction in grain boundaries on account of heat treatment is responsible for increased corrosion resistance.

The impedance test results plotted for all the samples in the form of Bode plots are shown in Figure 1(b). The charge transfer resistance ( $R_{\text{ct}}$ ), pore resistance ( $R_{\text{pore}}$ ) and Warburg impedance ( $W_s$ ) of coatings obtained after fitting with suitable circuits are given in Table 1. The as-coated samples and those heat-treated at 750°C show Warburg impedance due to diffusion process but it is less in samples heat treated at 750°C, which indicates that the porosity is decreased with heat treatment. On the other hand, samples heat-treated at 1000°C and 1500°C do not show any diffusion, however they show some pore resistance, which is improved with heat treatment from 1000°C to 1500°C. The total resistance value increases with heat treatment and samples heat-treated at 1500°C show better results, which are near to that of bulk tantalum samples. Recrystallization during heat treatment at temperatures exceeding  $\sim 1000^\circ\text{C}$  enhances the functional properties to a considerable extent that they are comparable with those of bulk Tantalum. This is due to the fact that recrystallization process induces the diffusion of inter-splat boundaries and reduces the number of potential weak sites.

Table 1  $E_{\text{corr}}$  and  $I_{\text{corr}}$  from polarization results shown in Fig. 1(a);  $R_{\text{ct}}$ ,  $R_{\text{pore}}$  and  $W_s$  from circuit fit of Bode plots in Fig. 1(b)

| Sample ID    | $E_{\text{corr}}$ (mV) vs SCE | $I_{\text{corr}}$ [ $\mu\text{A}/\text{cm}^2$ ] | Data from impedance analysis                 |                                                |                                    |
|--------------|-------------------------------|-------------------------------------------------|----------------------------------------------|------------------------------------------------|------------------------------------|
|              |                               |                                                 | $R_{\text{ct}}$ [ $\Omega\cdot\text{cm}^2$ ] | $R_{\text{pore}}$ [ $\Omega\cdot\text{cm}^2$ ] | $W_s$ [ $\Omega\cdot\text{cm}^2$ ] |
| Ta- Bulk     | -631                          | 0.528                                           | 1.744E5                                      | -                                              | -                                  |
| As coated    | -632                          | 7.184                                           | 93.11                                        | -                                              | 12033                              |
| HT at 750°C  | -561                          | 6.591                                           | 296.1                                        | -                                              | 5422                               |
| HT at 1000°C | -706                          | 1.065                                           | 33686                                        | 81.28                                          | -                                  |
| HT at 1500°C | -516                          | 0.703                                           | 83886                                        | 104.5                                          | -                                  |

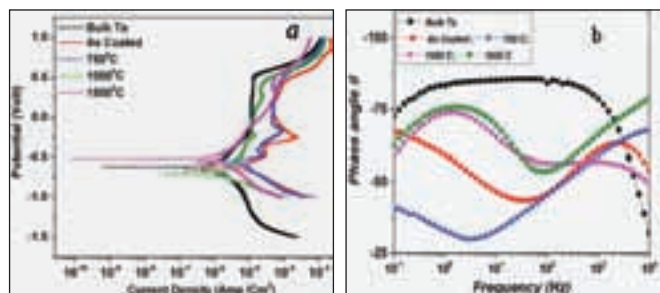


Fig.1 (a) Potentiodynamic polarization vs. SCE and (b) Bode plots of bulk, as coated and heat treated Ta coatings after 1hr exposure to 1M KOH solution

Contributors: S Kumar and V Vidyasagar

# Crystallographic Orientation Dependence of Properties

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It is well known that properties of materials are strongly dependent on crystallographic orientation. In this study, effect of orientation of the chromium carbide grains on the properties of chromium carbide-NiCrMoNb metal matrix composite has been studied. The samples were prepared by laser cladding chromium carbide-NiCrMoNb powder on a medium carbon steel substrate. The laser power was varied from 1600 W to 2400 W with 200 W increments. The powder feed rate and laser beam scanning speed were maintained constant at 14 g/min and 8 mm/s respectively. Hexagonal chromium carbide solidified dendritically and Nickel rich FCC phase solidified later between the interdendritic spaces.

It was found that at 1600 W, the chromium carbide had a strong preferred orientation with the basal plane (0001) parallel to the surface normal. However with increased laser power the randomness in the orientation of the carbides gradually increased. The cause for this is that at lower laser powers the heat extraction is highly directional with the substrate acting as an infinite heat sink, whereas at higher powers where the substrate is also heated significantly, and the heat flow may not be unidirectional.

Figure 1 shows the crystal orientation map of the  $Cr_7C_3$  phase in the laser clad layer processed at 2000 W. Microhardness and nano impact testing of the basal orientation and two prismatic orientations were carried out to understand the effect of orientation. Figure 2 shows the IPF with basal and prismatic orientations and the measured hardness values are mentioned. There is a significant reduction in the hardness of the composite as the orientation of the carbides changes from basal to prismatic. The impact measurement curves

shown in Figure 3 also show that the depth of penetration is higher in the prismatic orientation than the basal orientation. Close observation of the Vickers indentations shown in Figure 4 reveals that there were initiations of the cracks in the prismatic orientation. This study clearly shows that crystallographic orientation influences the properties significantly. It also shows that the preferred growth can be controlled by optimizing the process parameters. By choosing right process parameters, the microstructure can be tailored for better performance.

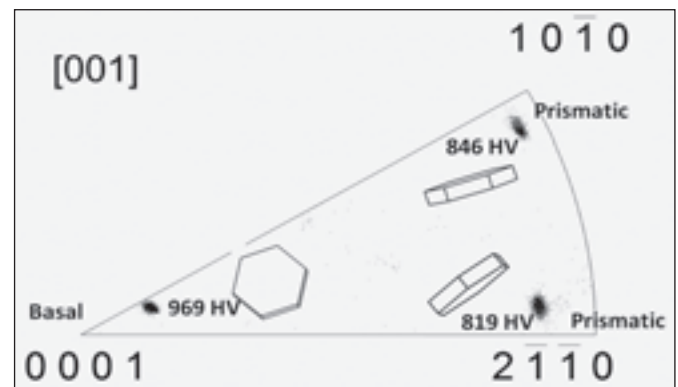


Fig. 2 Inverse pole figure of  $Cr_7C_3$  phase showing the basal and prismatic orientation with hardness values included

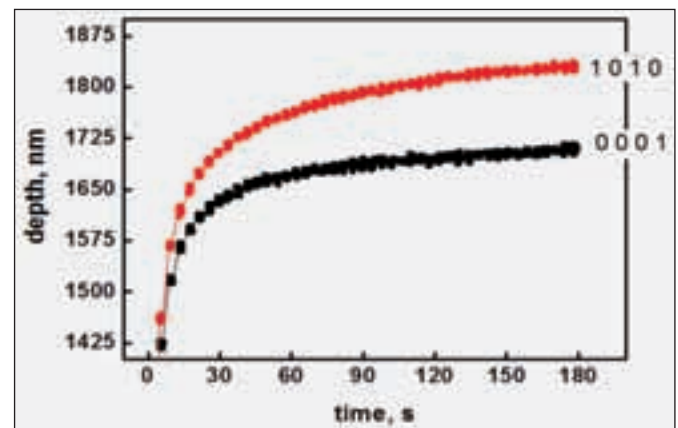


Fig.3 Impact test curve profiles for basal and prismatic orientations

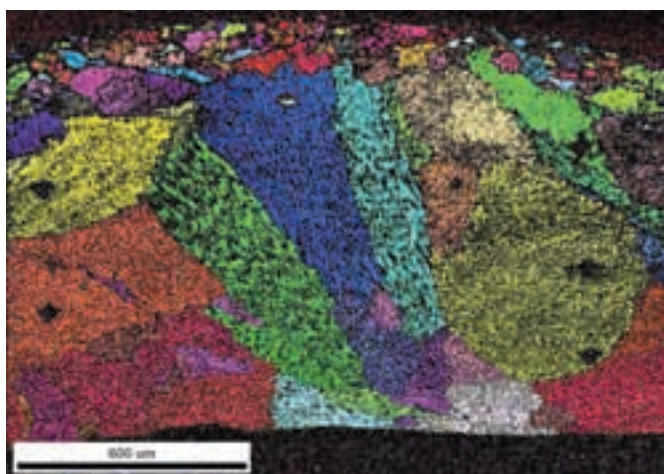


Fig.1 Crystal orientation map of  $Cr_7C_3$  phase in laser clad layer processed at 2000 W

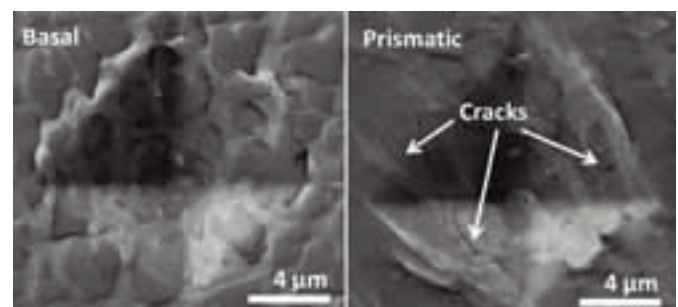


Fig.4 Microstructure of the Vickers indentation at basal and prismatic orientations

Contributor: P Suresh Babu



# Understanding the Microstructure of Mesoporous TiO<sub>2</sub> Beads by Small Angle X-ray Scattering

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Mesoporous metal oxide beads with high internal surface area and interconnected pore structure have received significant attention due to their potential for applications in various energy conversion and storage devices. For example, sub-micron size anatase TiO<sub>2</sub> beads with custom designed microstructures have recently achieved > 10% solar to electric energy conversion efficiency in dye-sensitized solar cells (DSSCs) with higher dye loading and improved electron diffusion coefficient. Such beads are usually made up of TiO<sub>2</sub> nanoparticles assembled in a random network. The surface area and porosity of the beads can be tuned to some extent by altering the constituent nanoparticles. However, quantifying the internal structure and porosity of the mesoporous beads for high efficiency DSSCs is a challenging task. Electron microscopy (SEM and TEM) techniques are routinely employed to probe the microstructure of mesoporous beads. SEM is an effective tool to study the size distribution and surface morphology of the mesoporous beads, whereas TEM is used to probe the internal assembly and features (size, shape) of constituent particles. As the particles are too thick for electron transmission, the observations are mostly confined to particles that lie at the surface of the TiO<sub>2</sub> micro-spheres. The size, shape and assembly of the primary particles may vary from the surface to the inside of the micro-sphere due to the influence of the processing conditions. Therefore, it is important to understand the influence of specific processing conditions on the size, shape and the assembly of the primary particles.

In order to study the primary TiO<sub>2</sub> particles, we have applied small angle x-ray scattering (SAXS), which is a very sensitive technique that can be used to probe the size and size distribution, shape, surface area and fractal structure of the primary TiO<sub>2</sub> particle from a large probe volume (3 to 4 orders higher than that of the volume investigated by TEM). Therefore, statistically averaged, high quality microstructural information is obtained, which is representative data that

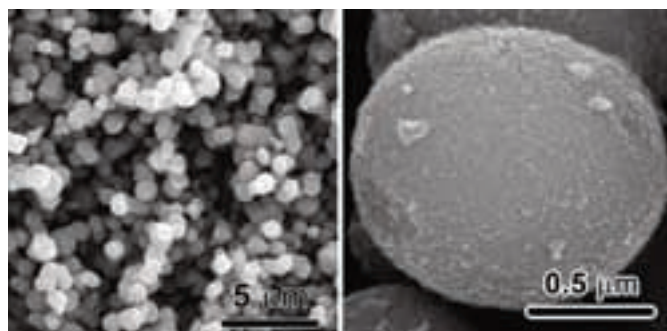


Fig.1 SEM-SE images of mesoporous TiO<sub>2</sub> beads (MA)

can directly be correlated with a property or functionality of the device. Mesoporous TiO<sub>2</sub> beads employed in this study were synthesized by the hydrothermal method. By changing the titanium precursor and process parameters, mesoporous TiO<sub>2</sub> beads with different microstructure and porosity (labeled as MR (rutile), MG (glycolate derived), MA (anatase) and MH (hollow anatase)) were obtained. SEM observation of these powders indicated that the sizes of the beads are in micron length scale. Within the beads, nano-pores are present which was confirmed by BET surface area analysis and N<sub>2</sub> adsorption-desorption isotherms.

SAXS measurements were performed using a high flux/ high transmission Mo laboratory source in transmission geometry. Figure 2(a) shows the SAXS intensity vs. modulus of the momentum transfer vector ( $q$ ) of mesoporous TiO<sub>2</sub> microbeads. A strong scattering intensity is observed between  $q = 0.2$  to  $0.8 \text{ nm}^{-1}$ , owing to the primary TiO<sub>2</sub> particles. In the Porod region, SAXS intensity decreases as  $q^{-4}$ , which indicates that the shape of the primary particles is spherical with a smooth surface. Further, the size and size distribution of the primary particles were extracted using least-square profile fitting with a spherical model and log-normal size distribution. The size distribution and mean size of the primary TiO<sub>2</sub> particles are depicted in Figures 2(b) and (c). A narrow size distribution with a mean size of  $11.0 \pm 0.5$ ,  $10.8 \pm 0.5$ ,  $10.33 \pm 0.3$ , and  $9.4 \pm 0.4 \text{ nm}$  were observed for the primary TiO<sub>2</sub> particles with -MR, -MG, -MA, -MH, respectively. Combining the SAXS and SEM studies, the morphology and internal structure of mesoporous TiO<sub>2</sub> microbeads could thus be investigated.

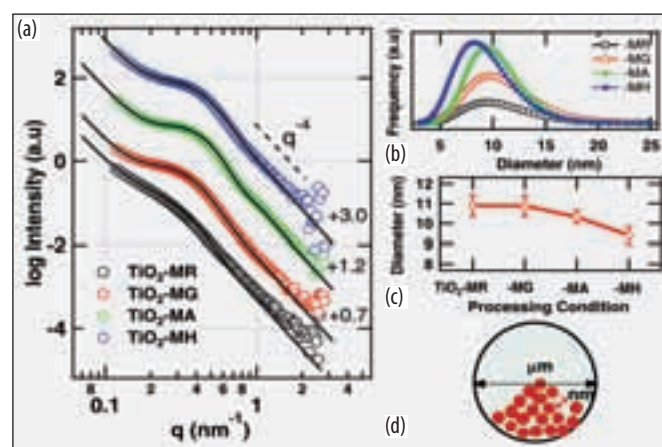


Fig. 2 (a) SAXS profiles of TiO<sub>2</sub> with different processing conditions. Solid lines show the fitting of the curves assuming the spherical model and broken line indicates that SAXS intensity decays with  $q^{-4}$ , (b) Size distribution of the TiO<sub>2</sub> particles, (c) variation of average particle diameter and (d) Schematic showing the hierarchical structure of TiO<sub>2</sub> nanoparticles which leads to micron size TiO<sub>2</sub> beads

Contributor: Easwaramoorthi Ramasamy



# Multi-functional High-Intensity 2D-X-ray Diffraction System

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A new advanced and multi-functional x-ray diffraction laboratory has been setup at the Centre for Materials Characterization and Testing with the installation of a new Rigaku Rapid-II D/MAX x-ray diffraction unit (Figure 1). This system is equipped with a very high intensity microfocus x-ray source viz. Rigaku MicroMax 007HF, which is a high-flux rotating anode system with excellent beam stability and brightness close to a second generation synchrotron (x-ray flux of  $10^{14}$  x-ray photon/mm<sup>2</sup>/s at the focal point). The system also has a highly sensitive and very large image plate-based two dimensional (2D) detector, which collects the diffracted Debye rings in 2D and ensures a full scan from 0 to 150° or 10 to 160° for 2θ in a single exposure of very small duration, making it a truly rapid system.

The system has several unique features, which makes it the most versatile laboratory scale x-ray diffraction system. The x-ray generating unit has a dual target (copper and chromium) option for the rotating anode and has a set of beam collimators with different aperture diameters in order to vary the x-ray beam spot size from a lowest possible 10 μm to 800 μm diameter. Coupled with the high intensity source, it is possible to obtain x-ray diffraction rings/peaks for features with dimensions even less than 1-10 microns. The system can be operated in different modes viz. reflectance, transmission and glancing angle configuration. It can perform various types of studies using these modes of operation like phase analysis, texture, stress analysis, failure analysis, trace phase detection (even for phase fraction of 0.1% or less) in micro/macro-area apart from thin film analysis in the glancing incidence mode. Figure 2(a) shows a typical 2D-XRD pattern obtained using the system for a sintered solid sample while the corresponding 1D-XRD pattern is shown in Figure 2(b) and Figure 2(c) shows a magnified section of the pattern revealing the well defined peaks of trace phases in the sample.

In addition to the above mentioned features, the XRD system has a provision to perform fully automated area mapping using auto stage. The wide range of studies that are possible with this system and the data quality has thrown open a new and efficient approach to material characterization of the most challenging samples.



Fig. 1 (a) Rapid II D/MAX XRD system (b) setup showing the rotating anode, optics and detector behind the sample stage installed on the goniometer

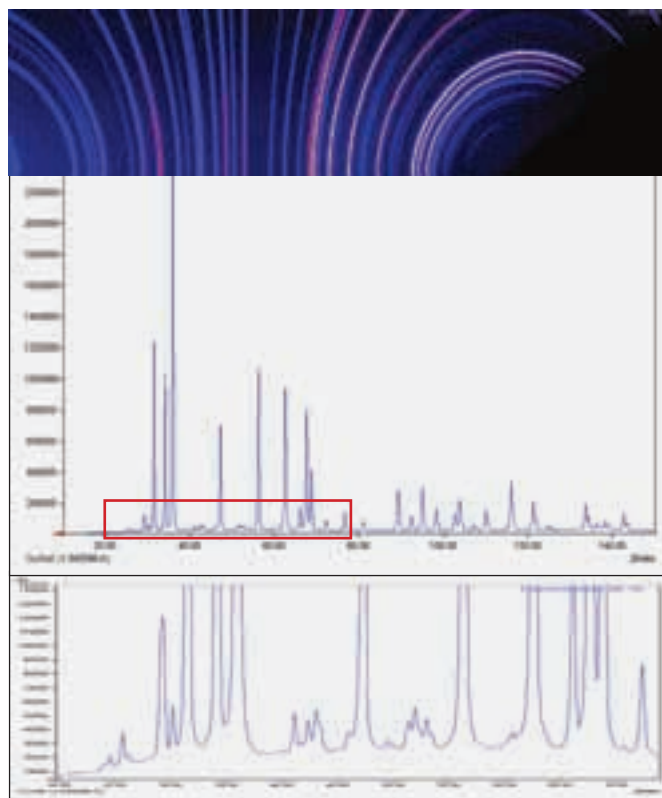


Fig. 2 (a) Typical 2D-XRD pattern (b) corresponding 1D-XRD pattern (c) magnified view of the highlighted section in (b) indicating trace phase peaks

*Contributor: Joydip Joardar*

# Centre for Knowledge Management of Nanoscience and Technology

Centre for Knowledge Management of Nanoscience and Technology (CKMNT) was set up in 2009 by ARCI with partial financial assistance from Nano Mission, Department of Science and Technology, Government of India. CKMNT uses unique strategies to search, monitor and analyze the information available in the patents, literature, and business databases. Insights drawn from analyses are being used by stakeholders for taking crucial decisions. The centre has gained requisite experience and expertise in the area, besides putting together a competent team. CKMNT has proven its utility for the end-users by providing the following: (1) on-demand / sponsored techno-commercial reports, (2) patent analyses reports, (3) inputs to Nano Mission, (4) multi-client techno-commercial reports and databases, (5) *Nanotech Insights*, a quarterly newsletter.

On-demand/sponsored techno-commercial reports, prepared at the request of interested organizations, are expected to be useful for making decisions pertaining to product, application and market development; technology commercialization; ongoing and proposed innovation programmes; patenting possibilities and promising business opportunities for Indian and international markets. During last year, an industry-solicited report was completed for a leading oil and gas company. Work on another industry-solicited report is ongoing.

Patent analyses reports in the form of Freedom to Operate (FTO) analysis and landscaping studies etc. have been prepared. FTO analysis for silica aerogels' granules in India, analysis of technology evolution and future opportunities for sol-gel coatings in automotive applications, patent landscape analyses on incorporation of nanomaterials in Li-ion batteries, nanotechnology applications in agriculture sector, and iron oxide nanoparticles for biomedical applications were completed during last year. Patent landscape report on heterocyclic compounds in polymers and materials: 2002-2012 is being prepared for a client.

As a part of inputs to Nano Mission, CKMNT has been providing yearly updates about the status of nanoscience and technology R&D in the country as well as the global trends. A detailed bibliometric analysis of global nanotechnology research publications during 2014 was submitted.

Multiclient techno-commercial reports and databases have been prepared by CKMNT on themes of relevance to all stakeholders in the field of nanoscience and technology. For instance, updating of techno-commercial report on nanofibers

for biomedical and healthcare applications prepared earlier is taken up. In addition, a directory containing academic institutions, R & D centres and industrial organizations operating in the area of nanoscience and technology has regularly been brought out by CKMNT. Directory is also being revised.

*Nanotech Insights* is a quarterly newsletter having information on topics that are relevant for the "nano" stakeholders including researchers, industries, policy makers, financial institutions and venture capitalists. The information presented in each issue is in the form of guest articles by identified experts, nanotech patent and literature analysis, emerging nanotechnologies, commercial and business issues, new products, green nanotechnologies, safety, healthcare and environmental issues, Indian nanoscience and nanotech scenario, Nano Mission activities etc. Special issues on "Defence, Aerospace and National Security" and "Nanotechnology for Remediation of Contaminated Water" were published during the year.

In near future, it is planned to continue the above work as well as to initiate following new activities:

- Due diligence, patentability, potential applications, market analysis etc. for projects that are under consideration for financial assistance by Nano Mission programme.
- Inputs to translate knowledge/Intellectual Property (IP) generated in the projects assisted through Nano Mission programme to useful products, processes and technologies.
- Organizing events that facilitate interaction between industry/entrepreneurs, academic and research institutes for initiating collaborative projects, forging academia-institute-industry partnerships, and technology licensing/transfer
- Broad-basing the CKMNT's capability utilization to sectors such as healthcare, energy, oil and gas, chemicals, plastics and composites, advanced materials, and electrical and electronics.



# Centre for Technology Acquisition, Transfer and International Cooperation

Centre for Technology Acquisition, Transfer and International Co-operation (CTATIC) works towards leveraging knowledge-base, intellectual property and technologies available with ARCI for organizations from public and private sectors, from India and abroad. CTATIC's role as an interface between R&D centres of excellence and external stakeholders is depicted in Figure 1.



Fig.1 Interfacing role of CTATIC

CTATIC performs the following activities:

- Identifying possible collaborators for ongoing and new R&D programmes
- Identifying start-ups / established companies to effect technology transfers through various direct and indirect channels such as participation in industrial exhibitions, business opportunity workshops, web and tele-marketing
- Understanding competing technologies and preparing techno-commercial feasibility analysis report for ARCI technologies available for transfer
- Formalizing contractual agreements to forge alliances at different points of ARCI's science and technology value chain
- Facilitating patent related services such as prior art searches for patent filing, research planning, market research etc., and coordinating patent drafting and filing activities

- Costing for technologies and projects
- Receivables management, monitoring of technology development programmes
- Performance reporting to different government agencies
- Coordinating visits of ARCI personnel deputed abroad for project related activities, equipment inspection and training, conferences, workshops, seminars etc.

CTATIC contributes at multiple points of ARCI's science and technology value chain, expressed in terms of Intellectual Property Development Indices (IPDIs), as shown in Figure 2. Recently, efforts have been made to assess the ongoing R&D programmes and, to identify appropriate partners for possible collaborations and technology transfers by using IPDIs. Appropriate contractual agreement is selected based on the IPDI of a particular R&D programme; the type, number and role of the partner organizations; intellectual property ownership and licensing methodology; expected deliverables; commercialization route and the financial arrangement including the manner of sharing the proceeds of intellectual property licensing/technology transfer etc. This approach is expected to help in enhanced utilization of the knowledge assets and R&D capability.

In all, 12 confidentiality Agreements to explore the possibility of entering into partnerships and 9 Agreements to utilize ARCI resources/capabilities were signed during last year. In addition to other efforts to identify possible partners and technology receivers for ARCI's research & technology development programmes, CTATIC participated in Advances in Welding and Surface Engineering (AWSE) Workshop cum Exhibition on October 17, 2014 and in a Buyer-Seller Meet cum exhibition organized by FICCI on November 24, 2014. Costing of over 30 projects and technologies was undertaken during last year. Patent related inputs were provided for 10 R&D projects.

Fig. 2 Schematic showing IPDIs and applicable contractual agreements

| IPDI                               | 1                                                                                                                                                            | 2                                | 3                                                                | 4                                            | 5                               | 6                                                                                                                        | 7                               | 8                                                                        | 9                                                                                         | 10                                |
|------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------|------------------------------------------------------------------|----------------------------------------------|---------------------------------|--------------------------------------------------------------------------------------------------------------------------|---------------------------------|--------------------------------------------------------------------------|-------------------------------------------------------------------------------------------|-----------------------------------|
| Activities                         | Basic concepts and understanding of underlying scientific principles                                                                                         | Possible applications forecasted | Research to prove technical feasibility for targeted application | Coupon level testing in simulated conditions | Check repeatability/consistency | Prototype testing in real-life conditions                                                                                | Check repeatability/consistency | Re-assessing of feasibility (competitive technologies and cost analysis) | Initiate technology transfer                                                              | Support in stabilizing production |
| Technology chain milestone(s)      | Exploratory studies                                                                                                                                          |                                  | Laboratory demonstration                                         |                                              |                                 | Field trials                                                                                                             |                                 |                                                                          | Technology transfer                                                                       |                                   |
| Possible contractual agreement (s) | <ul style="list-style-type: none"> <li>• Co-operative R&amp;D</li> <li>• R&amp;D Consortium</li> <li>• Inter-institutional</li> <li>• Sponsorship</li> </ul> |                                  |                                                                  |                                              |                                 | <ul style="list-style-type: none"> <li>• Joint demonstration</li> <li>• Technology demonstration and transfer</li> </ul> |                                 |                                                                          | <ul style="list-style-type: none"> <li>• Option</li> <li>• Technology transfer</li> </ul> |                                   |



# Portfolio of ARCI Technologies

## Technologies Transfers Undertaken

Based on the perceived market size of products/services based on ARCI technologies, ARCI has adopted exclusive and non-exclusive modes of technology transfer to facilitate healthy competition in the market. So far, ARCI has successfully transferred 15 technologies to 27 receivers and few technologies are under transfer. The following table depicts the technologies transferred:

| Sr. No | Technology                                                                            | Technology Recipient                                                      | Status                                                |
|--------|---------------------------------------------------------------------------------------|---------------------------------------------------------------------------|-------------------------------------------------------|
| 1-8.   | Electro Spark Coating (ESC) equipment                                                 | Hard, wear resistant coatings                                             | Transferred to 8 companies on non-exclusive basis     |
| 9.     | Magnesia Aluminate Spinel (MAS)                                                       | Steel, cement and power plants                                            | Transferred on exclusive basis                        |
| 10.    | Ceramic crucibles                                                                     | Carbon and Sulphur analysis                                               | Transferred on exclusive basis                        |
| 11.    | Energy efficient air heaters from ceramic honeycombs                                  | Industrial heating                                                        | Transferred on exclusive basis                        |
| 12-15  | Detonation Spray Coating (DSC)                                                        | Wear and corrosion resistant coating for various components               | Transferred to 4 companies on region-exclusive basis  |
| 16.    | Reinforced graphite sheets and seals                                                  | Automotive sector                                                         | Transferred on exclusive basis                        |
| 17.    | Heat pipes heat sinks                                                                 | Waste heat recovery systems, solar energy applications, power electronics | Transferred on exclusive basis                        |
| 18.    | Evaporation boats                                                                     | Metallization                                                             | Transferred on exclusive basis                        |
| 19.    | Ceramic honeycomb molten metal filters                                                | Molten metal filtration                                                   | Transferred on exclusive basis                        |
| 20.    | Calcium aluminate cements and furnace sealants                                        | Refractory castables                                                      | Transferred on exclusive basis                        |
| 21-23. | Micro Arc Oxidation (MAO)                                                             | Hard (1800 VHN) wear resistant coatings on aluminum and titanium alloys   | Transferred to 3 companies on region-exclusive basis. |
| 24.    | ESC equipment manufacturing                                                           | Diverse segments                                                          | Transferred on non-exclusive basis                    |
| 25.    | Nanosilver impregnated ceramic water filter candles to impart anti-bacterial function | Water purification                                                        | Transferred on non-exclusive basis                    |
| 26.    | Nano silver based textile finishes for anti-bacterial applications                    | Anti-bacterial applications                                               | Transferred on exclusive basis                        |
| 27.    | Nanotitaniumdioxide based textile finishes for self cleaning applications             | Self-cleaning applications                                                | Transferred on exclusive basis                        |
| 28.    | Decorative coatings on glass substrates                                               | Aesthetic applications                                                    | Ongoing                                               |

## Technologies Available for Adaptation/Transfer

| S. No | Technology and Related Issues                                                                                                                                                                                                                             | Key Features and Applications                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |                                                                                                                                                                                                                                                                                         |
|-------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1.    | <p><b>Decorative, Corrosion Resistant, Easy-To-Clean (ETC) Coatings on Metals</b><br/>(Indian Patent Application Number 620/DEL/2010 filed on 17/03/2010)</p> <p>Level of Maturity: In-house testing completed</p>                                        | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Water contact angle <math>95^{\circ} \pm 5^{\circ}</math></li> <li>- Can be directly applied on Aluminium/Stainless steel/mild steel substrates without need for primer</li> <li>- Can be transparent or decorative</li> <li>- High scratch hardness and abrasion resistance</li> <li>- Good corrosion resistance &gt; 720 hrs Salt Spray Test (for Aluminium)</li> <li>- Good adhesion</li> <li>- Can be made anti-bacterial</li> </ul> | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- On Aluminum for use of chromate-free, decorative multi-functional coatings for blades of ceiling fans</li> <li>- On SS sheets as decorative, abrasion resistant coatings for modular kitchens</li> </ul>  |
| 2.    | <p><b>Hard Coatings on Plastics like Polycarbonate, PMMA, Carbon Epoxy Composites etc.</b><br/>(Indian Patent Application Numbers 2427/DEL/2010 dtd. 12/10/2010 and 1278/DEL/2011 dtd. 02/05/11)</p> <p>Level of Maturity: In-house testing completed</p> | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- High scratch hardness and abrasion resistance</li> <li>- Long life</li> <li>- Good adhesion</li> <li>- Coloured coatings possible</li> <li>- Can be made easy-to-clean with low surface free energy</li> </ul>                                                                                                                                                                                                                           | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- Helicopter and automobiles windshields and windows</li> <li>- Aircraft canopies</li> <li>- Helmet visors</li> <li>- Road markers</li> <li>- Bi-aspheric lenses used in indirect ophthalmoscopy</li> </ul> |

| S. No | Technology and Related Issues                                                                                                                                                                                                                                                  | Key Features and Applications                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                                                                                                                                                                                                                                                                                                                                                          |
|-------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 3.    | <p><b>Decorative Coatings on Glass and Ceramics</b><br/>(Indian Patent Application Number 2427/DEL/2010 filed on 12/10/2010)</p> <p>Level of Maturity: In-house testing completed</p>                                                                                          | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Adjustable transmission and refractive index of the coatings</li> <li>- Colour of the coating can be controlled by suitable choice of dopants</li> <li>- UV temperature stable and weather proof</li> <li>- Recyclability of glass due to complete degradation of organic constituents at high temperatures</li> <li>- Opaque coatings possible with high temperature durability</li> </ul>               | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- Coloured glasses for aesthetics or decoration</li> <li>- Scratch resistant coloured coatings for glass bottles used in various industries such as perfume and fashion fields</li> <li>- Architectural Applications</li> </ul>                                              |
| 4.    | <p><b>Durable Single Layer Anti-Reflective Coating on Glass</b><br/>(Indian Patent Application No.: 2330/DEL/2013 dated: 05/08/2013)</p> <p>Level of Maturity: In-house testing completed</p>                                                                                  | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Economical possibility of large area applications</li> <li>- Low temperature curability</li> <li>- Developed and demonstrated sol-gel based antireflective coatings on 1 m long, borosilicate glass cover tube of 120-130 mm diameter to maximize visible light transmission to 97%</li> </ul>                                                                                                            | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- Showroom display glass</li> <li>- Ophthalmic lenses</li> <li>- Solar thermal plants</li> <li>- Automobile</li> </ul>                                                                                                                                                       |
| 5.    | <p><b>Nanocrystalline Zinc Oxide (ZnO) based Varistors</b><br/>(Indian Patent Application Number 1669/DEL/2006 dtd. 20/07/2006)</p> <p>Level of Maturity: In-house testing completed</p>                                                                                       | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Higher Breakdown voltage (5 times); Higher Coefficient of non-linearity (3 to 4 times); Lower leakage current compared to that of commercial varistors</li> </ul>                                                                                                                                                                                                                                         | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- Surge voltage protection in electrical and electronics industry</li> </ul>                                                                                                                                                                                                 |
| 6.    | <p><b>Nano Silver Impregnated Ceramic Candle Filter</b><br/>(Indian Patent Application Number 2786/DEL/2005 dtd. 19/10/2005)</p> <p>Level of Maturity: Small scale production (Technology transferred to one company and is available for transfer on non-exclusive basis)</p> | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Successfully field tested at various villages in Andhra Pradesh with a Non-Governmental Organization</li> <li>- No electrical power and pressurized water required:</li> <li>- Ease in maintenance</li> <li>- Commercially attractive {very low amount of silver used (0.2 wt %), Cost increase : candle (30-50%) and filter assembly (3-5%)}</li> <li>- Replacement needed once in six months</li> </ul> | <p><b>Application:</b></p> <p>Ceramic candles for drinking water purification</p>                                                                                                                                                                                                                                                                        |
| 7.    | <p><b>Silica Aerogels</b><br/>(Indian Patent Application Number 2406/DEL/2010 dtd. 08/10/2010)</p> <p>Level of Maturity: In-house testing completed</p>                                                                                                                        | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Stable from cryo (-50°C) to 1000°C</li> <li>- Thermal conductivity (0.03 W/mK)</li> <li>- Fire resistant</li> <li>- Chemically inert</li> <li>- Easily cut</li> <li>- Hydrophobic</li> <li>- Thickness range from 5-25 mm can be produced</li> </ul>                                                                                                                                                      | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- Thermal insulation in automotives</li> <li>- Heating/cold storage</li> <li>- Thermal clothing</li> <li>- Aerospace etc.</li> </ul>                                                                                                                                         |
| 8.    | <p><b>Laser Welding and Laser-MIG Hybrid Welding</b></p> <p>Level of Maturity: Testing on some actual components as per users' requirements done successfully</p>                                                                                                              | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- High power density</li> <li>- Single pass welding of thick sections</li> <li>- Controlled heat input welding with precision</li> <li>- No vacuum requirement</li> </ul>                                                                                                                                                                                                                                   | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- Tailor welded blanks for automotive applications etc.</li> <li>- Can weld a wide variety of materials and thicknesses</li> <li>- Can weld magnetic materials unlike Electron Beam Welding</li> <li>- Steel plates, thick section welds, ship building etc.</li> </ul>      |
| 9.    | <p><b>Laser Surface Hardening Treatment</b></p> <p>Level of Maturity: Testing on some actual components as per users' requirements done successfully</p>                                                                                                                       | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Selective localized area hardening with minimal heat input</li> <li>- No quenchant requirement</li> <li>- No surface damage</li> <li>- Excellent reproducibility with ease of automation</li> <li>- Negligible post process machining requirement</li> <li>- Controlled case depth</li> <li>- Refined homogenous microstructures</li> <li>- Minimal distortion</li> <li>- Chemical cleanliness</li> </ul> | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- Suited to wide range of steels, cast irons and profiles</li> <li>- The process can be developed for hardening of a variety of components such as crankshafts, camshafts, piston rings, tooling and dies, bearing steels, steam turbine blades, sheet metal etc.</li> </ul> |

| S. No | Technology and Related Issues                                                                                                                                                                                                                                                                                                                  | Key Features and Applications                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                                                                                                                                                                                                                                                                                                                                                                        |
|-------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 10.   | <p><b>Laser Surface Coating (Alloying and Cladding)</b></p> <p>Level of Maturity: Testing on actual components done successfully</p>                                                                                                                                                                                                           | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Material to be coated is fused using a laser beam and deposited on a substrate with good metallurgical bonding but with minimal base metal dilution</li> <li>- Low heat input resulting in fine microstructures</li> <li>- Provides crack-free clad layers without porosity</li> </ul>                                                                                                                                                                                                             | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- Wear plates for different applications</li> <li>- Component repair and refurbishment</li> </ul>                                                                                                                                                                                          |
| 11.   | <p><b>Laser Drilling</b></p> <p>Level of Maturity: Testing on actual components done successfully</p>                                                                                                                                                                                                                                          | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Non-contact drilling method</li> <li>- Holes of large aspect ratio and very small diameter (0.3 mm) can be drilled</li> <li>- Precise control of heat input</li> <li>- Holes can be drilled at shallow angles to the surface</li> </ul>                                                                                                                                                                                                                                                            | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- A wide variety of materials such as metals, ceramics and composites etc., can be drilled</li> <li>- The process can be used for specific applications such as drilling of fine holes on high pressure nozzle guided vanes and combustion liners for aero-engine applications.</li> </ul> |
| 12.   | <p><b>Micro Arc Oxidation</b><br/>(Indian Patent Number 209817 granted on 06/09/2007; US Patent Number 6893551 granted on: 17/05/2005)</p> <p>Level of Maturity: Small scale production (Technology transferred to 3 entrepreneurs and is available for export and for states in India other than Andhra Pradesh, Tamilnadu and Karnataka)</p> | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Ability to coat Al, Ti, Mg and Zr metals and their alloys</li> <li>- Ease to coat complex shapes and difficult to access regions</li> <li>- Uniform, dense, hard and thick coatings</li> <li>- Superior coating properties and performance compared to other conventional acid based processes like anodizing and hard anodizing</li> <li>- Excellent tribological properties and corrosion resistance</li> <li>- Eco friendly</li> <li>- 5 to 40 times service life enhancement</li> </ul>        | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- For a wide array of applications in industries such as textile, automobile etc.</li> </ul>                                                                                                                                                                                               |
| 13.   | <p><b>Detonation Spray Coating (DSC) Technology</b></p> <p>Level of Maturity: Small scale production (Technology transferred to 4 entrepreneurs and is available for all Indian states and for export)</p>                                                                                                                                     | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Attractively priced compared to imported HVOF units</li> <li>- Extreme versatility</li> <li>- Capable of depositing a vast range of metals, alloys, cermet, ceramic and composite coatings for varied functional properties</li> </ul>                                                                                                                                                                                                                                                             | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- Coatings for applications such as wear and corrosion resistance etc., for various industries</li> </ul>                                                                                                                                                                                  |
| 14.   | <p><b>Electro Spark Coating (ESC) Equipment Manufacturing Technology</b><br/>(Indian Patent Application Number 1610/DEL/2005 dtd. 21/06/2005)</p> <p>Level of Maturity: Small scale production (Technology transferred to one company and is available for transfer to all Indian states on non-exclusive basis)</p>                           | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Simple and cost-effective</li> <li>- Metallurgical bonded coatings with low heat input to the substrate</li> <li>- Any electrically conductive material available in electrode form can be coated on any conductive substrate</li> <li>- Equipment is portable and lends itself easily to automation for ensuring reproducibility</li> <li>- Capable of providing coating thickness in the range of 10 to 130 <math>\mu\text{m}</math></li> </ul>                                                  | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- Component refurbishment and to combat severe conditions of wear</li> <li>- Can be used for enhancing life of cutting tools such as end mills, taps and lathe bits</li> </ul>                                                                                                             |
| 15.   | <p><b>Exfoliated Graphite and its Value Added Products</b><br/>(Indian Patent Number 187654 granted on 07/06/1995)</p> <p>Level of Maturity: Commercial Scale (Technology transferred to one company and is available for transfer to all Indian states on non-exclusive basis)</p>                                                            | <p><b>Key Features:</b></p> <ul style="list-style-type: none"> <li>- Impermeable to fluids</li> <li>- Leak proof sealing under low turning torque</li> <li>- Easily cut and punched</li> <li>- Can withstand temperature range from <math>-200^{\circ}</math> to <math>+500^{\circ}</math> C in oxidizing and up to <math>3000^{\circ}</math>C in inert atmosphere</li> <li>- Excellent thermal shock resistance</li> <li>- Does not age or creep</li> <li>- Cannot get wetted by molten glass, metal etc., self-lubricating, and resistant to all chemicals</li> </ul> | <p><b>Possible Applications:</b></p> <ul style="list-style-type: none"> <li>- Fuel Cells</li> <li>- Automotive</li> <li>- Oil refineries</li> <li>- Petrochemical industries etc.</li> </ul>                                                                                                                                                                           |





# Support Groups



# Design and Development of a Test Set-up for Comparison of Solar PV Panel Parameters

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A simple microcontroller based test facility was set up for comparing the power outputs of two solar PV panels each 12V, 40Wp under identical test conditions. The two devices under test are placed side by side so as to receive the same solar radiation. The outputs of both were connected to a single charge controller through rectifier diodes and current sensors. A battery and load were connected to the charge controller, as is done conventionally. In this way, the same load shares the solar energy generated by the two panels. If the voltage generated by any of the panels is more, the corresponding current drawn from that panel is more and vice versa. Basic scheme is shown in Figure 1.

The output voltages and currents drawn from each panel are monitored by an Arduino microcontroller. The controller is programmed to log this data i.e. Panel 1 voltage & current, Panel 2 voltage & current on a real time basis with required sampling rates in Excel compatible files for post analysis.

A sample of the logged data (Table 1) and a chart showing the plot of the recorded parameters (Figure 3) are shown below.

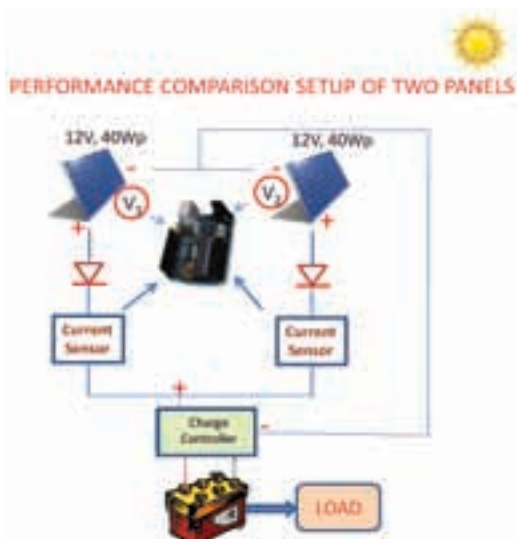


Fig. 1 Basic scheme of the test set-up

An LCD display has been also interfaced to display this data in real time. The following figures (Figure 2,) shows the connection scheme, the LCD display and the associated hardware.



Fig. 2 Physical arrangement of the test set-up

| Date and Time   | Panel 1 (V1) | Panel 1 (I1) | Panel 2 (V2) | Panel 2 (I2) | panel 1(W=V1*I1) | panel 2(W=V2*I2) |
|-----------------|--------------|--------------|--------------|--------------|------------------|------------------|
| 9/11/2014 13:39 | 14.64        | 0.43         | 14.51        | 0.52         | 6.2952           | 7.0432           |
| 9/11/2014 13:40 | 14.64        | 0.43         | 14.51        | 0.52         | 6.2952           | 7.0400           |
| 9/11/2014 13:40 | 14.64        | 0.43         | 14.51        | 0.52         | 6.2952           | 7.0400           |
| 9/11/2014 13:40 | 14.66        | 0.44         | 14.53        | 0.53         | 6.4004           | 7.2009           |
| 9/11/2014 13:40 | 14.66        | 0.44         | 14.56        | 0.56         | 7.1232           | 8.2096           |
| 9/11/2014 13:40 | 15.25        | 0.54         | 15.07        | 0.6          | 8.235            | 9.042            |
| 9/11/2014 13:40 | 15.33        | 0.63         | 15.13        | 0.66         | 9.6579           | 9.9858           |
| 9/11/2014 13:41 | 15.29        | 0.59         | 15.11        | 0.63         | 9.0211           | 9.5193           |
| 9/11/2014 13:41 | 15.17        | 0.49         | 15           | 0.56         | 7.4333           | 8.4              |
| 9/11/2014 13:41 | 15           | 0.46         | 14.94        | 0.54         | 6.9              | 8.0136           |
| 9/11/2014 13:41 | 14.74        | 0.44         | 14.58        | 0.52         | 6.4856           | 7.5816           |
| 9/11/2014 13:41 | 14.7         | 0.43         | 14.58        | 0.52         | 6.321            | 7.4338           |
| 9/11/2014 13:41 | 14.64        | 0.43         | 14.51        | 0.52         | 6.2952           | 7.0400           |
| 9/11/2014 13:42 | 14.62        | 0.43         | 14.51        | 0.52         | 6.2886           | 7.0400           |
| 9/11/2014 13:42 | 14.62        | 0.42         | 14.47        | 0.5          | 6.1404           | 7.235            |
| 9/11/2014 13:42 | 14.62        | 0.43         | 14.47        | 0.52         | 6.2886           | 7.5244           |
| 9/11/2014 13:42 | 14.6         | 0.42         | 14.45        | 0.5          | 6.132            | 7.225            |
| 9/11/2014 13:42 | 14.6         | 0.43         | 14.49        | 0.5          | 6.278            | 7.245            |
| 9/11/2014 13:42 | 14.58        | 0.43         | 14.45        | 0.5          | 6.2884           | 7.225            |
| 9/11/2014 13:43 | 14.6         | 0.43         | 14.47        | 0.5          | 6.278            | 7.225            |
| 9/11/2014 13:43 | 14.62        | 0.43         | 14.45        | 0.52         | 6.2886           | 7.3695           |
| 9/11/2014 13:43 | 14.6         | 0.43         | 14.45        | 0.5          | 6.278            | 7.225            |
| 9/11/2014 13:43 | 14.57        | 0.43         | 14.43        | 0.52         | 6.2051           | 7.3993           |
| 9/11/2014 13:43 | 14.57        | 0.42         | 14.41        | 0.5          | 6.1094           | 7.205            |
| 9/11/2014 13:43 | 14.58        | 0.42         | 14.37        | 0.5          | 6.1236           | 7.185            |
| 9/11/2014 13:44 | 14.57        | 0.42         | 14.41        | 0.5          | 6.1294           | 7.205            |
| 9/11/2014 13:44 | 14.55        | 0.42         | 14.41        | 0.5          | 6.131            | 7.205            |

Table 1 Sample of logged data

In this particular case, the two solar panels have shown only marginal difference in their behaviour.

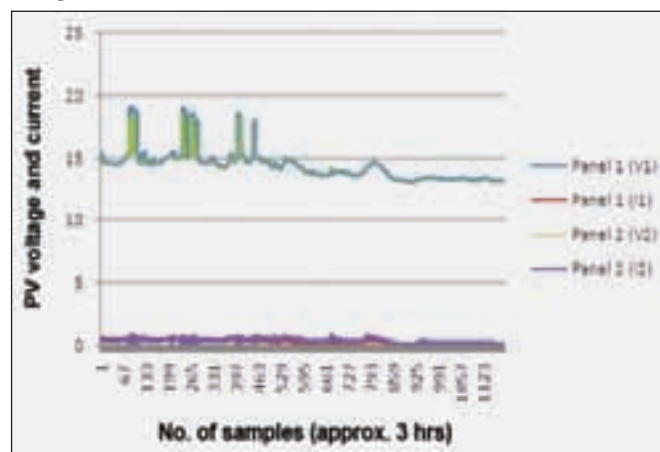


Fig. 3 Plot of recorded parameters

Though the above scheme is designed for two PV panels, it can be expanded to monitor and log the parameters of any number of PV panels with minor modifications in the hardware and software.

Contributors: A Gouthami and A S Joshi

# Development of an Arduino based Charge Controller for a Solar Panel

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Conventionally, any solar panel is connected to a charge controller, a battery and a load placed in series in that order. The main function of the charge controller is to protect the battery against overcharging and to disconnect the load from the panel if its voltage falls to a very low value. However, the charge controller does not have a provision to disconnect the battery from the load. We have developed a Charge Controller using an Arduino board to include this additional function. The status of the battery and solar panel are monitored continuously at all times. The battery is charged by the solar panel, whenever needed, provided the solar power is adequate. However, if the battery output or solar energy is low, the battery is isolated from all loads to prevent possible damage on account of under-voltage operation.

Our charge controller is built around the Arduino microcontroller kit to minimize the cost. It is shown in Figure 1. A program has been developed to measure the status of the Battery and the Solar panel independently. The battery and solar panel voltage levels are monitored by the Arduino board.

The battery voltage is measured and categorized in one of four preset levels, as shown in Table 1. The lowest battery level i.e. a battery with 25 % residual charge is deemed to be that which gives an open circuit voltage equal to its rated value, which in our case is 12 V. This classification that we have adopted may vary marginally with that used by other developers.

In this project, we have considered loads which can vary in discrete steps, which is generally the situation in a domestic environment where the lights or fans or TV sets or similar appliances are switched on or off periodically as per the demands of the situation. Such connected loads can be grouped into four priority segments P-I to P-IV, each segment covering 25 % of the total power rating. The most important load is included in P-I and the least required one being in P-IV, as shown in Table 2. An LCD indicator is used to display the status of charge of the battery and panel voltage as shown in Figure 2.

For testing the above concept, a 12 V Lead acid battery having a capacity of 7 AH was used to test the battery sensing feature of the charge controller. More elaborate tests with solar panel and connected loads are required to validate all

other aspects of the integrated system. This study, which is being initiated, requires a great deal of time to gather and analyze the data.

A customized microcontroller based charge controller has been designed, developed and tested in the laboratory under simulated conditions. The developed program is flexible and can be modified to add more features.

Table 1 Preset voltage levels corresponds to % state of charge

| % state of charge | 12V battery voltage level |
|-------------------|---------------------------|
| 100               | 12.6+                     |
| 75                | 12.4                      |
| 50                | 12.2                      |
| 25                | 12.0                      |

Table 2 Load grouping and Priority levels

| Load grouping (Battery level) | Load quality            | Priority level |
|-------------------------------|-------------------------|----------------|
| 76-100%                       | Least essential         | P-IV           |
| 51-75%                        | More important than PII | P-III          |
| 26-50%                        | More important than PI  | P-II           |
| ~25%                          | Most essential          | P-I            |

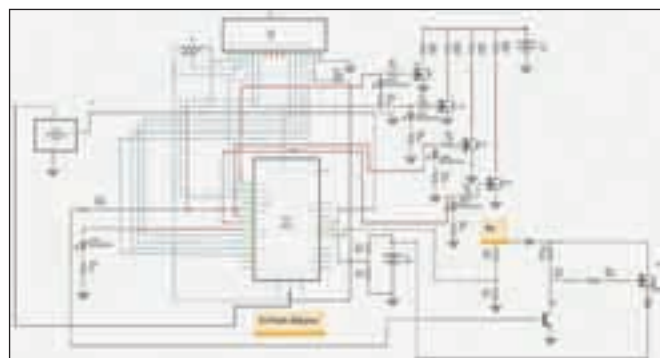


Fig.1 Schematic of Arduino Microcontroller with Battery, MOSFET etc.



Fig.2 LCD Indicator

Contributors: S Nirmala, N Aruna and A S Joshi



# Flexibility and the Challenges in Power Systems

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Power systems have been designed and operated so that the demand for electricity can be met at all times and under a variety of conditions. Depending on the season, the climate, and the weather, demand can fluctuate significantly over a single day, week, or month. In addition to meeting the variability requirements, there is always some inherent uncertainty about future demand and the future availability of generators. The power system must thus be able to manage both variability and uncertainty.

Sources of variable renewable generation such as wind, tidal, wave, solar, and run-of-river hydro have one common characteristic i.e. is having an output governed by atmospheric conditions. Wind and solar energy generation may consequently be difficult to predict over some time scales. Large penetrations of variable generation (VG) lead to increases in the variability and uncertainty in the system's generation output, driving a need for greater flexibility. This flexibility needs to come either from flexible generation technologies or from alternative sources of flexibility such as flexible demand and storage.

The flexibility of the system represents its ability to accommodate the variability and uncertainty in the load-generation balance while maintaining satisfactory levels of performance for any time scale. There is no uniform definition of flexibility. Ramp rate, minimum up/down time, and start-up time are used as indicators of flexibility, measured as megawatts available for ramping up and down over time.

At each stage of planning and operations, an understanding of variability is applied in different ways. Traditionally, long-term resource planning required little information about the variability of the net load in time scales of minutes to days, whereas characterizing the diurnal cycle is an important feature of day-ahead operational planning. The variability and uncertainty of VG production give rise to challenging ramping issues in the operational time frame. Characterizing

those issues in a planning contest is becoming increasingly necessary. Operational flexibility is related to the system's ability to deal with variability within system operation time scales (normally from a day ahead down to real time). The type of operational flexibility required will depend on the time scale: increased frequency response and reserves for seconds to minutes, increased ramping capability for minutes to hours, and scheduling flexibility for hours to a day ahead. The time scales of flexibility, from the system-planning perspective down to very short-term operation, and the impacts of variable generation on flexibility can be seen in Figure 1.

The need for additional flexibility will depend on the increase in the demand for flexibility related to the penetration of variable renewables and also on the flexibility that already exists in the system.

Figure 2 depicts how each part of the system affects the need for and supply of flexibility. The variability sources drive the need for flexibility to restore a system's energy balance, whereas the flexibility sources respond to restore that balance. In the middle, the system context oval contains facilitators that influence how much of the technically available flexibility may be deployed in real time.

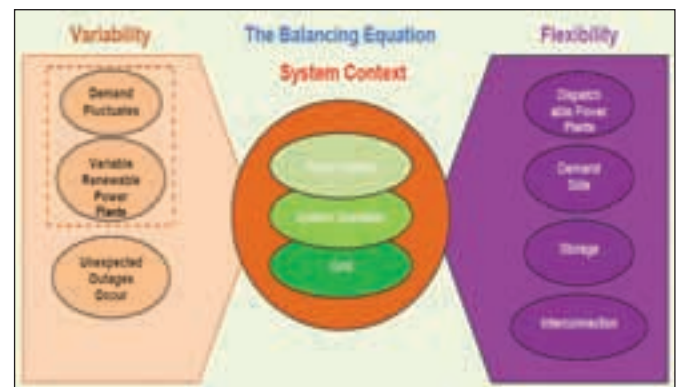


Fig. 2 Flexibility needs, sources and enablers



Fig. 1 The impacts of variable generation on the flexibility time line

Source: IEEE Power and Energy Magazine, November/December 2013, by Hannele Holttinen and Others

# Catering Science and Technology Information Requirements

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Technical Information Centre (TIC) aims at providing critical scientific information support to the scientific and technical staff of ARCI by way of providing access to both the print as well as online resources. It has made significant progress by supporting its R&D community by providing the required science & technology information by rendering timely services.

TIC has a collection of 1621 books and 2084 bound volumes of journals. It continues to subscribe to over 40 National and International journals relevant to the core areas of interest of ARCI. In addition to the print journals, it also subscribes to several e-journals apart from the abstracting database Scopus.

Being the member of the National Knowledge Resource Consortium (NKRC), the TIC has access to over 1700 electronic journals including major STM publishers like Elsevier, IEEE, NPG, RSC, Springer, Taylor & Francis and Wiley, in addition to several online databases which were well utilized by the Scientists of ARCI. Apart from the above resources, during the period of this report, TIC has provided access to Maney online journals. In addition to the existing Materials Science collection, the Engineering collection is also additionally being subscribed to on the ScienceDirect platform. Further, TIC has subscribed to the plagiarism check software iThenticate.

## Services Offered by TIC:

- **Lending Service:** Officers and Research Scholars are allowed to borrow three books and other members, two books at a time.
- **OPAC:** TIC offers Online Public Access Catalogue (OPAC) service, which helps users to browse the TIC collection by Title, Author, Keywords, etc. OPAC can be accessed on ARCI's Local Area Network (LAN).
- **e-resources:** TIC subscribes to several e-journals and A&I databases either directly or through NKRC. All these e-resources can be accessed throughout the IP ranges of ARCI, CKMNT and ARCI's project sites CFCT and CAEM, Chennai. As on date, all the e-resources are activated on the newly acquired IP ranges from National Knowledge Network (NKN) at both Hyderabad and Chennai.
- **Resource Sharing:** TIC maintains close relations with libraries of DST, CSIR and other National Laboratories.

During the past year, it had procured about 60 documents from other Institutions on Inter Library Loan (ILL) basis and fulfilled ILL requests from other DST and CSIR laboratories.

- **Scientometric Analysis:** TIC extended its support to the scientific community by way of scientometric analysis to understand the current global research trends and also to help them be aware of the changing interests of policy makers which has direct impact on the research fields.

Table 1 Electronic resources subscribed through NKRC

| #  | e-resource                      | #   | e-resource                                 |
|----|---------------------------------|-----|--------------------------------------------|
| 1. | American Chemical Society (ACS) | 8.  | Royal Society of Chemistry                 |
| 2. | AIP and APS                     | 9.  | SciFinder                                  |
| 3. | ASTM Standards Digital Library  | 10. | Springerlink                               |
| 4. | Science Direct                  | 11. | Taylor & Francis                           |
| 5. | IEEE Explore Digital Library    | 12. | Web of Science & Derwent Innovations Index |
| 6. | Maney Publishing                | 13. | Wiley Blackwell                            |
| 7. | Nature Publishing Group         |     |                                            |



Fig. 1 Wide range of books and journals at ARCI's Technical Information Centre

# Events, Data and Statistics





## Major Events

### Sports

ARCI constituted a sports committee to conduct sports and games for the year 2014-15. Sports and Games 2014-15 was inaugurated on April 11, 2014 by Dr. S. V. Joshi, Additional Director and Dr. G. Padmanabham, Associate Director. In all, more than 20 events were conducted and more than 100 employees actively participated in games such as Volleyball, Cricket, Badminton, Football, Carom, Athletics etc. Prizes were distributed to the winners and runners-up by Dr. S. V. Joshi, Additional Director, Dr. G. Padmanabham, Associate Director and Shri R. Prabhakara Rao, Chief Admin and Personnel Officer.



Rolling Shield being presented to the Winners Team for the year 2014-15 by Dr. S. V. Joshi and Dr. G. Padmanabham on the Sports Day celebration

### Jayanthi Celebrations

ARCI celebrated Dr. B. R. Ambedkar Jayanthi and Dr. Babu Jagjivan Ram Jayanthi on April 14, 2014.

### Independence Day

ARCI celebrated Independence Day on August 15, 2014. Dr. H. Purushotham, Scientist 'G' and Head-Centre for



Dr. H. Purushotham hoisted the National Flag as a part of the celebrations

Knowledge Management for Nanoscience and Technology (CKMNT) hoisted the National Flag and addressed the gathering on the occasion.

### Annual Medical Check-up and Health Talk

The Annual Medical Check-up programme for ARCI employees was organized during August 27-28, 2014. Employees were categorized into two age groups i.e. below and above 45 years of age and special medical tests such as TMT, 2D ECHO etc. were undertaken for employees above 45 years of age. Bone densitometry (for the spine) test was also undertaken for women employees.



Annual Medical check-up in progress

### Official Language (Hindi) Implementation at ARCI

The Official Language Implementation Committee (OLIC) under the chairmanship of Dr. G. Sundararajan-Director - ARCI has been successful in the implementation and progressive use of Hindi in ARCI. During the year 2014-15, ARCI issued more than 3300 letters etc. in bilingual form and surpassed the target set by the Dept. of Official Language (D.O.L), Ministry of Home Affairs, Govt. of India. The Department of Science and Technology (DST), in its review, has appreciated the achievement of ARCI in this regard. To propagate the use of Hindi during work, ARCI not only conducted Hindi workshops on a quarterly basis for its employees but also imparted training on 'Typing in Hindi', Unicode etc. to more than 100 employees. ARCI has also been imparting Training in Hindi to its Employees under the Hindi Teaching Scheme and has trained a number of employees in Prabodh and Praveen levels. Employees were also provided cash awards for their good performance.

ARCI conducts internal OLIC meetings on a quarterly basis to review the progressive use of Hindi ARCI. The minutes of the meeting are sent to DST as well as D. O. L. for review. ARCI celebrated Hindi Week during September 08 to 12,



Participants and OLIC Members during the Hindi week celebrations

2014. Various programmes and competitions like Quiz, Elocution, Noting and Drafting in Hindi, Essay Competition were conducted, and prizes were distributed to the winners. On this occasion, ARCI organized two lectures on 'Uses of Science in Official Language' and 'Invisible Pollution and Health' delivered by Dr. D. D. Ojha, member of Hindi advisory committee.

DRDO organized the "10<sup>th</sup> All India Official Language Scientific and Technical Seminar" held at RCI, Hyderabad on February 12 and 13, 2015. Dr. P. K. Jain, delivered a technical and non-technical lecture in Hindi during the Sammelan. ARCI is a member of Town Official Language Implementation Committee (TOLIC). Mr. R. Prabhakara Rao, Chief Admin and Personnel Officer and O. L. Officer has been nominated as member in the core-committee of TOLIC.



Dr. Sanjay Bhardwaj addressing the participants during Hindi week celebrations

### 'Swach Bharat Mission' Implementation at ARCI

As per the directives of Department of Science and Technology, Govt. of India, ARCI observed the 'Swach

Bharat Mission' from September 25 to October 02, 2014. As a part of this initiative, ARCI undertook cleanliness drive in and around all its buildings. As a part of this mission, Dr. G. Sundararajan, Director-ARCI and Dr. S. V. Joshi, Additional Director-ARCI administered the Swach Bharat pledge in the presence of ARCI personnel.

### Vigilance Awareness Week

ARCI observed Vigilance Awareness Week from October 27 to November 01, 2014. As a part of this occasion, Dr. R. Vijay, Vigilance Officer-ARCI administered the pledge in the presence of personnel from Administration, Stores, Finance & Accounts, Computer Centre, and Centre for Technology Acquisition, Transfer and International Coordination. The Team Leaders from other centres of excellence administered the pledge at their respective centres in presence of their team. During the week, ARCI also organized a lecture titled 'Combating Corruption', which was delivered by Shri Kumar Viswajeet, I. G., Director-Anti Corruption Bureau, Hyderabad, Government of Telangana. An exhibition displaying posters on different aspects of vigilance awareness was also organized.



Shri Kumar Viswajeet delivering the lecture on vigilance awareness



## Annual Day

ARCI celebrated its 18<sup>th</sup> Annual Day on December 26, 2014. On this occasion, Dr. G. Sundararajan, Director delivered a speech detailing various achievements during the year. Dr. S. V. Joshi, Additional Director, Dr. G. Padmanabham, Associate Director and Dr. (Ms.) R. Subasri, Convener-Annual Day Committee also addressed the gathering. On this occasion, Dr. G. Sundararajan, Dr. S. V. Joshi, and Dr. G. Padmanabham planted trees to encourage the concept of keeping the environment clean and green. As a part of these celebrations, various cultural events with participation from employees and their family were also organized enthraling the live audience. After the events, prizes were distributed to the participants.



Dr. S. V. Joshi, Additional Director-ARCI planting a tree on the occasion



Dr. G. Sundararajan, Director-ARCI addressing the gathering



Dr. G. Padmanabham enthraling the audience



Participation of Dr. Tata Narasinga Rao in the cultural events

## Republic Day

ARCI celebrated Republic Day on January 26, 2015. Dr. S. V. Joshi, Additional Director hoisted the National Flag and addressed the gathering on the occasion.



Dr. S. V. Joshi hoisted the National Flag as a part of the celebrations

## National Science Day

National Science Day (NSD) was celebrated on February 26, 2015 at ARCI. Dr. G. Padmanabham, Associate Director welcomed the audience comprising of Scientists, Officers, Research Fellows, Project Students, Trainees of ARCI, 60 IX<sup>th</sup> & X<sup>th</sup> class students and teachers from nearby Government schools and briefed about the importance of National Science Day Celebrations. The theme for National Science Day 2015 was "Science for Nation Building". Dr. T. Narasinga Rao delivered a talk on "Role of Science in Turning 'Make in India' into a Reality". Dr. R. Subasri delivered a talk on "Science for Society".

A science quiz was conducted by Dr. Y. Srinivasa Rao, Dr. Neha Y. Hebalkar and Dr. R. Easwaramoorthi for the school students. Dr. G. Sundararajan, Director ARCI handed over the prizes for winners of science quiz competition. Schools students were taken around for visit to various Centres of Excellence showing live demonstration of various equipments and R&D activities.





Dr. Y Srinivasa Rao, Dr. Neha Y Hebalkar and Dr. R Easwaramoorthi conducting the Science Quiz



Participants at the In-House Training Programme on Industrial Safety at ARCI



Dr. G Sundararajan, Director-ARCI addressing the students

## National Safety Week

ARCI observed National Safety Week during March 09 to 13, 2015. As a part of this, a first aid training programme was organized on March 09, 2015 through St. John's Ambulance. The programme was conducted by Dr. N. Nityanand who demonstrated the importance of first aid to the participants. The programme was attended by 28 participants including scientists, technical officers and security personnel. Participation certificate was also issued to all the participants. A two day in-plant training programme on "Industrial Safety" was organized on March 11 and 12, 2015 through National safety Council (NSC), Mumbai. Shri T. Nityanand and Shri P. M. Rao were designated by NSC as faculty members who conducted classes on industrial safety and its importance.

The programme was attended by 45 participants and participation certificates were issued to all of them. On March 13, 2015, Shri S Kalyanaraman, Security, Fire and Safety Officer arranged a demo on the usage of portable fire extinguishers kept handy at all major locations in ARCI. He explained in detail the function of various fire extinguishers in case of emergencies and the precautionary measures one has to adopt in case of emergencies.

## ARCI Internal Complaints Committee (AICC)

An awareness programme about the new rules on Sexual Harassment of Women at Workplace (SHWW) for Research Fellows/Trainees/Students was conducted on June 2, 2014 by AICC. ARCI celebrated International Women's Day on March 10, 2015. As a part of this occasion, AICC invited Smt. Swati Lakra, I.P.S., Additional Commissioner of Police (Crimes & SIT) and Head-SHE Teams, Telangana as Chief



Smt. Swati Lakra I.P.S., delivering the lecture as a part of Women's Day celebrations



IX<sup>th</sup> and X<sup>th</sup> class students and teachers from nearby schools during the National Science Day celebrations





Participants during the International Women's day celebrations

Guest. She delivered a lecture on 'Women Safety' on the occasion. Awareness about the new rules on SHWW for all contract staff of ARCI was conducted by Asmita Resource Centre for Women on the same day.

### Conference/Workshops/Symposia Organized by ARCI:

- **6<sup>th</sup> Asian Thermal Spray Conference (ATSC 2014)**

The 6<sup>th</sup> Asian Thermal Spray Conference (ATSC 2014), organized by ARCI in association with Asian Thermal Spray Society (ATSC) and SAHTSE, held at Hyderabad during November 24-26, 2014, for the first time in India, was a stupendous success. The conference witnessed huge participation cutting across various categories such as authors, exhibitors and conference delegates. Over 300 participants from 16 different countries attended this event which is the highest number in ATSC conference series. Quality research work was presented by Scientists and Engineers in thermal spray field along with a large number of plenary and invited lectures. Exhibitors and sponsors also had the opportunity to showcase their products and services to a wide section of audience. The conference was preceded by a two day Thermal Spray course attended by over 80 participants with a good number of foreign

participants as well. Clearly, ATSC 2014 not only realized the objective of providing an attractive forum for all stakeholders from across the region to network in order to foster a fruitful interaction during and after the conference but also served as an important step in putting India on the thermal spray map.



Participants at the ATSC 2014

- **International Conference on Additive Manufacturing, 3D Printing & 3D Scanning (ICAM 3D)**

International Conference on Additive Manufacturing, 3D Printing & 3D Scanning (ICAM 3D) was organized by Vel Tech, Chennai and ESCI-IE (I) in association with ARCI at Chennai during February 05-07, 2015.



Participants at the two-day Thermal Spray course

## Human Resource Development

### ARCI-IIT Fellowship Programme

ARCI continues to sponsor fellowship programmes at Indian Institute of Technology (IIT) – Bombay, IIT-Hyderabad and IIT-Madras. As a part of these ARCI-IIT Fellowships, ARCI supports the doctoral study of talented students selected as ARCI Fellows to work in areas of immediate interest to ARCI under the expert guidance of an identified Faculty member. The ARCI support includes stipend, procurement of consumables and essential equipment. After successful completion of the programme, the ARCI Fellow is awarded a Ph.D. degree by the respective academic institution.

The status of projects being undertaken is as follows:

| Project                                                                                        | Collaborating Institute | Name of the Fellow | Date of admission | Status                 |
|------------------------------------------------------------------------------------------------|-------------------------|--------------------|-------------------|------------------------|
| Study of Multiferroic Composite Thin Films                                                     | IIT - Bombay            | Tarun              | 16.07.2009        | Ongoing                |
| Molybdenum Oxide and Tin Oxide/Sulfide Nanostructured Materials for Anodes in Li-Ion Batteries | IIT- Hyderabad          | A. Bhaskar         | 01.08.2010        | Ph.D. thesis submitted |
| Stable and Highly Efficient Copper Zinc Tin Sulphide (CZTS) Thin Film Photovoltaics            | IIT - Madras            | Deepak Kumar       | 01.08.2012        | Ongoing                |

### Post Doctoral Fellows, Research Scholars, Senior/Junior Research Fellows, Post Graduate/Graduate Trainees and M.Tech/B.Tech./M.Sc. Project Students joined during the year at ARCI

|                                 |    |
|---------------------------------|----|
| DST-Inspire Faculty             | 01 |
| DST-WoS (DST - Women Scientist) | 02 |
| Post-Doctoral Fellows           | -  |
| Research Scholars (Ph.D.)       | 01 |
| Senior Research Fellows         | 07 |
| Junior Research Fellows         | 21 |
| Post Graduate Trainees          | 03 |
| Graduate Trainees               | 40 |
| M.Tech Project Students         | 25 |
| B.Tech./M.Sc. Project Students  | 25 |

### Recognition of ARCI as an External Centre for Carrying Out Ph.D. Research

The following academic institutes recognized ARCI as an External Centre for carrying out Ph.D. Research. Accordingly, interested ARCI employees, Project Scientists and Research Fellows can register for Ph.D. (as per university norms) at the University.

- |                                                 |                                                              |
|-------------------------------------------------|--------------------------------------------------------------|
| 01. Indian Institute of Technology – Bombay     | 07. National Institute of Technology – Tiruchirappalli       |
| 02. Indian Institute of Technology – Kharagpur  | 08. Visvesvaraya National Institute of Technology – Nagpur   |
| 03. Indian Institute of Technology – Kanpur     | 09. University of Hyderabad (Central University) – Hyderabad |
| 04. Indian Institute of Technology – Hyderabad  | 10. Andhra University – Visakhapatnam                        |
| 05. Indian Institute of Technology – Madras     |                                                              |
| 06. National Institute of Technology – Warangal |                                                              |

| Sl. No. | Name of the Student (Mr./Ms.) | Topic                                                                                                                                            | Registered at           | Status    |
|---------|-------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------|-----------|
| 01.     | R. Papitha                    | Investigations on pressure casting and extrusion processing parameters and thermo-mechanical properties of low expanding ceramics                | University of Hyderabad | Completed |
| 02.     | Y. Krishna Priya              | Joining of aluminum alloy and steel by thermal joining techniques                                                                                | University of Hyderabad | Ongoing   |
| 03.     | Ch. Leela Pydi Pavithra       | Structure - property correlations in nanostructured copper and copper Nanocomposite foils prepared by pulse and pulse-reverse electro deposition | University of Hyderabad | Ongoing   |
| 04.     | M. S. Archana                 | Development of TiCN - metal/intermetallic based nanocomposites for cutting tool applications                                                     | University of Hyderabad | Ongoing   |
| 05.     | P. Sai V Pramod Kumar         | Effect of individual layers and their microstructure on tribological behavior of cathodic arc deposition multilayer coatings                     | University of Hyderabad | Ongoing   |
| 06.     | M. Nagini                     | Structure - property correlation in ODS 18 Cr steels                                                                                             | University of Hyderabad | Ongoing   |
| 07.     | Alka Pareek                   | Stabilization of cadmium chalcogenide based photo anode for photo electro chemical hydrogen production using solar light                         | University of Hyderabad | Ongoing   |
| 08.     | N.S. Anas                     | Effect of dispersion of carbon nanotube/grapheme on aluminium alloys                                                                             | University of Hyderabad | Ongoing   |



| Sl. No. | Name of the Student (Mr./Ms.) | Topic                                                                                                                                                                                             | Registered at                                     | Status    |
|---------|-------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------|-----------|
| 09.     | L. Subashini                  | Investigation of laser hybrid weldability of special steels                                                                                                                                       | University of Hyderabad                           | Ongoing   |
| 10.     | Mandati Sreekanth             | Fabrication of $\text{CuInSe}_2$ and $\text{Cu (In, Ga) Se}_2$ absorber layers by pulse and pulse-reverse electrochemical techniques for solar photovoltaic applications                          | Indian Institute of Technology, Hyderabad         | Completed |
| 11.     | K. H. Anulekha                | Synthesis characterization and electrochemical performance of electrospun electrode materials for lithium ion batteries                                                                           | Indian Institute of Technology, Hyderabad         | Ongoing   |
| 12.     | Bolla Reddy                   | Spherical indentation behaviour of porous copper and cold sprayed copper coatings                                                                                                                 | Indian Institute of Technology, Hyderabad         | Ongoing   |
| 13.     | Anusree Unnikrishnan          | Polymer Electrolyte Membrane Fuel Cells : Impurity Studies – Experimental & Modelling Investigations                                                                                              | Indian Institute of Technology, Hyderabad         | Ongoing   |
| 14.     | J.A. Prithi                   | Experimental Investigation of PEMPC – ORR Catalysts _ Imp                                                                                                                                         | Indian Institute of Technology, Madras            | Ongoing   |
| 15.     | K. Nanaji                     | Development of Mesoporous Metal Oxides/Carbon electrode materials for Energy Storage Applications.                                                                                                | Indian Institute of Technology, Madras            | Ongoing   |
| 16.     | S. Bhuvanewari                | Structure, Morphology and Electrochemical Performance Correlation in Metal Doped Spinel ( $\text{Li M}_x \text{Mn}_{2-x} \text{O}_A$ ) (M=Transition Metals) as Li ion Battery Cathode Materials. | Indian Institute of Technology, Madras            | Ongoing   |
| 17.     | Ravi Gautam                   | Microstructure_ Magnetic Property Correlation of $\text{LiNiO}_2$ based Cathode Materials.                                                                                                        | Indian Institute of Technology, Madras            | Ongoing   |
| 18.     | Sumit Ranjan Sahu             | Carbon Nanohorns based Anode Material for Lithium Ion Battery                                                                                                                                     | Indian Institute of Technology, Madras            | Ongoing   |
| 19.     | N. Sasikala                   | Structure and Electrochemical Property Correlation of $\text{LiNiO}_2$ based Cathode Materials.                                                                                                   | Indian Institute of Technology, Madras            | Ongoing   |
| 20.     | S. Harish                     | Development and Performance Testing of Thermo-electric Devices for Automotive Waste Heat Recovery.                                                                                                | Indian Institute of Technology, Madras            | Ongoing   |
| 21.     | S. Vasu                       | Structure and Electrochemical Property Correlation of Lithium Rich Layered Oxide and Layered Oxide of Lithium Ion Batteries for Electric Vehicle Applications.                                    | Indian Institute of Technology, Madras            | Ongoing   |
| 22.     | Amol C. Badgujar              | Development of Copper Indium Gallium Selenide (CIGS) Solar Cells.                                                                                                                                 | Indian Institute of Technology, Bombay            | Ongoing   |
| 23.     | Vallabharao Rikka             | Investigation on Ageing Mechanism of Lithium Ion Cell ( $\text{LiFePO}_4/\text{Graphite}$ )                                                                                                       | Indian Institute of Technology, Bombay            | Ongoing   |
| 24.     | Kumari Konda                  | Importance of Coating, Mixing and Calendaring Lithium Ion Batteries.                                                                                                                              | Indian Institute of Technology, Bombay            | Ongoing   |
| 25.     | Puneet Chandran               | Development of coated tools for high speed machining of hard to cut materials under dry conditions                                                                                                | National Institute of Technology, Warangal        | Ongoing   |
| 26.     | P. Tejavvi                    | Electrospun inorganic materials for battery applications                                                                                                                                          | National Institute of Technology, Warangal        | Ongoing   |
| 27.     | E. Hari Mohan                 | Development of high capacity nanostructured anode and sulphur cathode for lithium sulphur battery applications                                                                                    | National Institute of Technology, Warangal        | Ongoing   |
| 28.     | V.V.N. Phani Kumar            | Synthesis, Characterization and Doping of Olivine/Spinel based Materials and its effective binding nature for Lithium Ion Batteries.                                                              | National Institute of Technology, Warangal        | Ongoing   |
| 29.     | N. Manjula                    | Studies on the Aspects of Depolarized Electrolysis for Hydrogen Generation.                                                                                                                       | National Institute of Technology, Warangal        | Ongoing   |
| 30.     | T. Ramesh                     | Activated Carbons for Energy Storage.                                                                                                                                                             | National Institute of Technology, Warangal        | Ongoing   |
| 31.     | P.M. Pratheeksha              | Development of Nanostructured Electrode Materials of High Energy lithium ion battery Applications.                                                                                                | National Institute of Technology, Warangal        | Ongoing   |
| 32.     | VV Ramakrishna                | Microstructural_ Magnetic Property Investigation of MnBi Alloy to Develop Heavy Rare Earth Free Permanent Magnets.                                                                                | National Institute of Technology, Tiruchirappalli | Ongoing   |

## Appointments

ARCI has added the following employees to its fold to take up varied responsibilities:

| Employee Name          | Designation                                     | Date of Joining |
|------------------------|-------------------------------------------------|-----------------|
| Dr. D. Prabhu          | Scientist "C"                                   | 04.06.2014      |
| Ms. Rambha Singh       | Hindi Translator (Contract)                     | 04.06.2014      |
| Dr. R. Prakash         | Scientist "E"                                   | 12.06.2014      |
| Dr. Srinivasan Anandan | Scientist "D"                                   | 12.06.2014      |
| Mr. S. Kalyanaraman    | Security, Fire & Safety Officer (on deputation) | 30.06.2014      |
| Dr. R. Balaji          | Scientist (Contract)                            | 02.12.2014      |
| Dr. Sathiya Mariappan  | Scientist (Contract)                            | 09.02.2015      |

## Promotions

ARCI has been following its existing assessment and promotion policy since the year 2000-01. As per the policy, assessments were carried out for all eligible employees and the following were promoted during the year 2014-15:

| Name of the Promotees     | Effective Date  | Promotion for the post  |                       |
|---------------------------|-----------------|-------------------------|-----------------------|
|                           |                 | From                    | To                    |
| Dr. T. Narasinga Rao      | October 1, 2014 | Scientist "F"           | Scientist "G"         |
| Dr. Pramod H Borse        | October 1, 2014 | Scientist "E"           | Scientist "F"         |
| Dr. Bhaskar Prasad Saha   | October 1, 2014 | Scientist "E"           | Scientist "F"         |
| Dr. R. Subasri            | October 1, 2014 | Scientist "E"           | Scientist "F"         |
| Mr. S.B. Chandrasekhar    | October 1, 2014 | Scientist "D"           | Scientist "E"         |
| Mr. S. Sudhakara Sarma    | October 1, 2014 | Scientist "C"           | Scientist "D"         |
| Mr. K. Srinivasa Rao      | October 1, 2014 | Technical Officer "B"   | Technical Officer "C" |
| Mr. J. Nagabhushana Chary | October 1, 2014 | Technical Officer "A"   | Technical Officer "B" |
| Mr. A. Raja Shekhar Reddy | October 1, 2014 | Technical Officer "A"   | Technical Officer "B" |
| Mr. R. Anba Rasu          | October 1, 2014 | Technical Assistant "A" | Technical Officer "A" |
| Ms. N. Aruna              | October 1, 2014 | Technical Assistant "A" | Technical Officer "A" |
| Mr. G. Venkata Rao        | October 1, 2014 | Technician "C"          | Technician "D"        |
| Mr. KVB Vasantha Rayudu   | October 1, 2014 | Technician "C"          | Technician "D"        |
| Mr. D. Krishna Sagar      | October 1, 2014 | Technician "C"          | Technician "D"        |
| Mr. M. Satyanand          | October 1, 2014 | Technician "B"          | Technician "C"        |
| Mr. S. Narsinga Rao       | October 1, 2014 | Technician "A"          | Technician "B"        |
| Mr. Ch. Jangaiah          | October 1, 2014 | Technician "A"          | Technician "B"        |
| Ms. K. Shakunthala        | October 1, 2014 | Assistant "B"           | Assistant "B" (MACP)  |

## Superannuation

| Employee Name              | Designation Held        | Date of Superannuation |
|----------------------------|-------------------------|------------------------|
| Shri. S. Jagan Mohan Reddy | Security & Fire Officer | 30.05.2014             |

## Resignations

| Employee Name          | Designation Held            | Date of Reliving |
|------------------------|-----------------------------|------------------|
| Dr. T. Mohan           | Senior Scientist (Contract) | 11.04.2014       |
| Dr. Prabhu Delhi Babu  | Scientist (Contract)        | 03.06.2014       |
| Dr. Raju Prakash       | Senior Scientist (Contract) | 11.06.2014       |
| Dr. Srinivasan Anandan | Senior Scientist (Contract) | 11.06.2014       |
| Mr. T.K. Gireesh Kumar | Technical Assistant "A"     | 12.12.2014       |
| Dr. H. Purushotham     | Scientist "G"               | 21.01.2015       |

## Visit by Students and Others to ARCI

01. 75 B.Tech (Mechanical) students and faculty members from MVSR College of Engineering, Hyderabad visited ARCI on April 3, 2014.
02. 75 B.Tech (Mechanical) students and faculty members from MVSR College of Engineering, Hyderabad visited ARCI on April 11, 2014.
03. 20 Scientists from various DRDO laboratories who participated in Administrative Staff College of India (ASCI's) "Management Training Programme" visited ARCI on August 13, 2014.
04. 50 B.Tech (Mechanical) students and faculty members from Jyothi Engineering College, Kerala visited ARCI on August 20, 2014.
05. 30 B.Sc (Physics) final year students and faculty members from St. Francis College for Women, Hyderabad visited ARCI on September 03, 2014.
06. 25 Engineers from various Government Organizations who participated in Engineering Staff College of India (ESCI's) "Welding Technologies and NDT Techniques Programme" visited ARCI on September 25, 2014.
07. 30 Faculty and Student members of SAE India, Southern Section, Chennai visited ARCI on October 10, 2014.
08. 60 B.Tech. (Mechanical) students and faculty members from Christu Jyothi Institute of Technology and Science, Warangal visited ARCI on October 24, 2014.
09. 40 B.Tech. (Mechanical) students and faculty members from Vignan Institute of Technology and Science, Hyderabad visited ARCI on November 14, 2014.
10. 28 Engineers from various Government Organizations who participated in Engineering Staff College of India (ESCI's) "Creativity and Innovation Management in Research" visited ARCI on November 18, 2014.
11. 30 Scientists and Technical Staff from various DRDO Laboratories who participated in "DMRL's CEP course" visited ARCI on December 09, 2014.
12. 20 Scientists/Engineers from ISRO who participated in Administrative Staff College of India (ASCI's) "Management

Training Programme" visited ARCI on December 17, 2014.

13. 50 B.Tech. (Materials Science) students and faculty members from University of Petroleum and Energy Studies, Dehradun visited ARCI on January 07, 2015.
14. 20 M.Sc. (Physics) students and faculty members from Haribhai V Desai College, Pune visited ARCI on January 13, 2015.
15. 38 B.Sc. (Physics) students and faculty members from DBF Dayanand College of Arts & Science, Solapur visited ARCI on January 22, 2015.
16. 60 B.Tech. (Materials Science and Nanotechnology) students and faculty members from JNTU College of Engineering, Sultanpur visited ARCI on February 02, 2015.
17. 40 M.Sc. (Applied Electronics and Materials Science) students and faculty members from Solapur University, Solapur visited ARCI on March 03, 2015.

## Summer Research Programme

As in the previous years, this year too students from IITs, NITs, IIITs, Central Universities, various other state and private universities from all over the country were short-listed for availing Summer Research Programme (SRP) at ARCI. 55 students were selected for the Summer Research Programme, for a period of 45 – 60 days started from 5th May, 2014. The selected students initially underwent a week long orientation course so as to get familiarity with the activities being carried out at ARCI. Each student was guided by a scientist to carry out a mini project. The students were issued certificates on completion of the programme.

## Reservations and Concessions

The Reservations and Concessions for SCs/STs/OBCs and persons with disabilities are followed as per Government of India orders from time to time. At ARCI, the representation of employees under SC is 16.77%, ST is 3.73%, OBC is 24.84% and that of persons with disabilities is 1.86% as on March 31, 2015.



Dr. S.V. Joshi, Additional Director with Summer Training Students



## Indian and Foreign Visitors for Technical Discussion

1. Dr. V. Shrinet, Deputy Director (Technology), Electrical Research and Development Association (ERDA), Vadodara visited on April 15, 2014.
2. Dr. A.K.S. Bhujanga Rao, President (R&D and Technical), NATCO Pharma, Hyderabad visited on April 29, 2014.
3. Prof. Muralidhar, Assistant Professor, Mepco-Schlenck Engineering College, Sivakasi visited on May 27, 2014.
4. Dr. Gukan Rajaraman, Assistant Professor, PSG College of Technology, Coimbatore visited on June 09, 2014.
5. Mr. Pankaj Pujara, General Manager (GM) Operations & Project Implementation, Gujarat Alkalies and Chemicals Limited (GACL), Vadodara; Mr. Anil Dalvi, GM (O&ES), GACL; Mr. Pramod Parikh, Advisor (Corporate Strategy) to MD, GACL; Dr. Sunil Sinha, Assistant General Manager (AGM) R&D, GACL and Mr. Rakesh Desai, Chief Manager (O&ES), GACL visited on June 20, 2014.
6. Mr. Mayank Shaw, Director, Madhuchitt Industries, Mumbai visited on July 08, 2014.
7. Dr. Naveen Vashista, Scientist, Department of Science and Technology (DST), New Delhi visited on July 17, 2014.
8. Dr. Alok Singh, Chief Researcher, Microstructure Design Group, National Institute for Materials Science (NIMS), Japan, visited on August 01, 2014.
9. Prof. A. Suzuki, Yokohama National University, Japan visited on August 07, 2014.
10. Mr. Alok Sharma, Chief Manager R&D, Indian Oil Corporation Limited (IOCL), Faridabad and Mr. Sachin Chugh, Senior Manager R&D, IOCL, Faridabad visited on August 12, 2014.
11. Mr. M. Vekateswaralu, Manager (R & D), Amara Raja Batteries, Hyderabad visited on September 04, 2014.
12. Dr. Saptarshi Ghosh, Manager, Bharat Electronics Limited (BEL), Pune visited on September 04, 2014.
13. Dr. A. Rajendra Prasad, Dean R&D, Sai Ram College of Engineering, Chennai visited on September 09, 2014.
14. Dr. Gopi, Assistant Professor, National Institute of Technology (NIT), Warangal; Dr. Ravi Kumar, Assistant Professor, NIT, Warangal and Dr. P.V. Suresh, Assistant Professor, NIT, Warangal visited on September 16, 2014.
15. Mr. K. Chandran, Executive Vice President, RANE Holdings Ltd, Chennai visited on September 17, 2014.
16. Mr. V. R. Ramesh, Manager R&D, TVS Motors, Hosur and Ms. Jayamathy Mathialagan, Manager, R&D, TVS Motors, Hosur visited on September 22, 2014.
17. Mr. Shivam Tiwari, Director, Eastern Electrolyzers, New Delhi visited on October 08, 2014.
18. Mr. Tarun Reddy, Director, Sindhya Resources Pte. Ltd., Singapore and Mr. Shyam Sundar Raghupathy, Deputy General Manager, Indo National Ltd, Chennai visited on October 15, 2014.
19. Dr. Gukan Rajaram, Assistant Professor, PG College of Technology, Coimbatore visited on October 15, 2014.
20. Mr. K. Lakshman, Chief Executive Officer (CEO), RANE Holdings Ltd, Chennai visited on November 03, 2014.
21. Prof. N. Matsumi and Prof. Raman Vedarajan, Japan Advanced Institute of Science and Technology (JAIST), Japan visited during December 22-23, 2014.
22. Dr. U. Balachandran, Scientist, Argonne National Laboratory, USA visited on January 01, 2015.
23. Mr. S. Anand Kumar, Director Technical, CIRA Renewable Energy (P), Ltd, Hyderabad visited on February 04, 2015.
24. Dr. D. Elangovan, Assistant Professor, Vellore Institute of Technology (VIT), Vellore visited on February 09, 2015.
25. Dr. Frank Riedel, Head of Department, Thermal Joining, Fraunhofer Institute of Machine Tools and Forming Technology (IWU), Germany and Mr. Markus Puschmann, Group Leader, Processing, IWU, Germany visited during February 09-13, 2015.
26. Mr. R.K. Kashyap, Executive Director R&D, GAIL (India) Ltd., Noida; Ms. K. Barathi, Senior Manager R&D, GAIL (India) Ltd., Noida and Mr. Parashuram Chaurasia, Senior Manager R&D, GAIL (India) Ltd., Noida visited on February 11, 2015.
27. Dr. Shweta Shom, Research Associate, Solar Research Institute, Ministry of New and Renewable Energy (MNRE), Noida visited on March 19, 2015.
28. Dr. N.V. Choudary, GM, Hindustan Petroleum Corporation Limited (HPCL), Bengaluru, Dr. Peddy V C Rao, Deputy GM-R&D, HPCL, Bengaluru visited on March 23, 2015.
29. Dr. Praveer Asthana, Adviser, DST, New Delhi visited on March 31, 2015.

## Seminars by Indian and Foreign Visitors

1. Dr. Narendra B. Dahotre, Professor & Chairman, Department of Materials Science and Engineering and Professor of Mechanical and Energy Engineering, University of North Texas, USA delivered a lecture on "Laser Assisted Crystallization of Ferromagnetic Amorphous Ribbon" on April 04, 2014.
2. Dr. G. Veerappan, Post Doctoral Fellow, Sungkyunkwan Advanced Institute of Nanotechnology, Republic of Korea delivered a lecture on "Perovskite-based Next Generation Solar Cells" on April 10, 2014.

3. Dr. S. Marya, Emeritus Professor, Ecole Centrale Nantes, France delivered a lecture on "Revisiting Manufacturing Technologies – Focus on Powder Feedstock Laser based Additive Manufacturing" on April 16, 2014.
4. Dr. Neelkanth G. Dhere, Programme Director, Florida Solar Energy Centre (FSEC)-University of Central Florida, USA delivered an invited lecture on "CIGS Thin Film Solar Cell Research and Development at FSEC" on April 16, 2014.
5. Dr. Lokesh Kesavan, Post-Doctoral Researcher, Aalto University, Finland, delivered a lecture on "Designer Supported Metal Nanoparticles for Heterogeneous Catalysis Applications" on April 21, 2014.
6. Prof. Arvind Agarwal, FASM, Indian Institute of Technology (IIT), Delhi delivered a lecture on "Plasma Sprayed Hydroxyapatite-Carbon Nanotube Coating for Orthopedic Implants and Quantifying Adhesion Strength at Sub-Micron Scale using a Nano Scratch Based Technique" on May 01, 2014.
7. Dr. C. V. Gopal Reddy, Senior Research Scientist, AZ Electronic Materials, USA delivered a lecture on "Synthesis, Characterization, Applications of Metal, Metal Oxide Nanomaterials and Thin Films" on May 02, 2014.
8. Dr. Vishal Mahajan, Lead Engineer, Xalt Energy, USA delivered a lecture on "Lithium Ion Cell Technology - Challenges and Opportunities" on May 22, 2014.
9. Dr. Raj Sankar Cheriyaedath, Postdoctoral Fellow, Northwestern University, USA delivered a lecture on "Discovery and Development of New Materials for Thermoelectric Applications" on May 30, 2014.
10. Prof. Palani Balaya, National University of Singapore, Singapore delivered a lecture on "Translational Battery Research" on June 25, 2014.
11. Prof. Apparao M. Rao, Clemson University, USA delivered a lecture on "Scalable Nano-Manufacturing of Nano-Carbon-based Supercapacitors for Next Generation Energy Storage" on July 15, 2014.
12. Dr. Suwas Nikumb, Senior Research Officer, National Research Council (NRC) Canada delivered a lecture on "Laser Micromachining for Engineering Applications" on July 16, 2014.
13. Dr. Swati Ghosh Acharyya, Assistant Professor, University of Hyderabad, delivered a lecture on "Revisiting the Horizons of Corrosion Science" on July 31, 2014.
14. Dr. B. Varaprasad, Post Doctoral Fellow, NIMS, Japan delivered a lecture on "Future Recording Media and Read Head Technology" on August 13, 2014.
15. Prof. S.C. Misra, IIT, Guwahati delivered a lecture on "Porous Burners" on August 25, 2014.
16. Dr. Maikel Van Hest, Senior Scientist, National Renewable Energy Laboratory (NREL), USA delivered a lecture on "Atmospheric Processing of Photovoltaic Materials" on August 25, 2014.
17. Dr. Digambar Y. Nadargi, Senior Postdoctoral Fellow, Empa-Swiss Federal Laboratories for Materials Science and Technology, Switzerland delivered a lecture on "Sol-Gel Derived Advanced Materials: Aerogels and Coatings" on December 10, 2014.
18. Dr. Valeria Lauter, Lead Instrument Scientist, Oak Ridge National Laboratory (ORNL), USA delivered a lecture on "Magnetic Heterostructures for Spintronics: Spatial Characterization with Polarized Neutron Scattering" on January 30, 2015.
19. Dr. Dan Brett, Reader in Electrochemical Engineering, University College London, United Kingdom delivered a lecture on "Electrochemical Impedance Spectroscopy for Power Systems" on February 23, 2015.
20. Dr. Branko Matovic, Department of Materials Sciences, Belgrade, Serbia delivered a lecture on "Synthesis and Properties of Monolithic Nanocrystalline SiC Ceramics" on February 23, 2015.
21. Prof. Tamas Ungar, Department of Materials Physics, Budapest Eötvös University, Budapest, Hungary delivered a lecture on "The Substructure of Crystalline Materials as seen by X-ray Diffraction Patterns" on February 23, 2015.
22. Prof. Anthony Kucernak, Imperial College, London, United Kingdom delivered a lecture on "Assessing the Performance of Reactant Transport Layers Ad Flow Filed Towards Oxygen Transport – A New Imaging Method based on Chemi Luminescence" on February 24, 2015.
23. Dr Paul Shearing, Lecturer, University College London, United Kingdom delivered a lecture on "X-Ray Imaging of Li Ion Batteries" on February 25, 2015.
24. Prof. S.D. Mahanti, Michigan State University, USA delivered a lecture on "Thermoelectrics with Hierarchical Structures" on February 27, 2015.

## Visits Abroad

1. Mr. M. Ramakrishna visited Pleasanton, USA during April 04-14, 2014 to attend the 'Training School on Electron Energy Loss Spectroscopy (EELS) and Energy Filtered Transmission Electron Microscope (EFTEM) Techniques'.
2. Dr. Tata N. Rao visited Tokyo, Japan during April 20-May 01, 2014 to participate in technical discussions with faculty and students of Tokyo University of Science (TUS) and delivered a series of lectures at TUS.
3. Dr. Roy Johnson visited Potsdam, Germany during May 21-26, 2014 to attend the 'Indo-German Frontiers

- of Engineering Symposium 2014' and (a) delivered an introductory lecture on "Ceramics for Health, Energy and Environment" and b) made a poster presentation on "Ceramic Honeycombs: Unique Structures for Energy Absorption, Conservation and Generation".
4. Dr. G. Padmanabham visited Potsdam, Germany during May 21-29, 2014 to Co-chair the 'Indo-German Frontiers of Engineering Symposium 2014' and also to have technical discussion with Fraunhofer IWU, Chemnitz and Fraunhofer IWS, Dresden.
  5. Dr. S.V. Joshi visited Wiener Neustadt, Austria during May 26-29, 2014 to participate in the '2nd Austrian-India Symposium on Materials Science and Tribology' and delivered an invited lecture on "Composite Coatings Employing a Novel Hybrid APS + SPPS Technique for Tribological Applications".
  6. Dr. S.V. Joshi visited USA during May 30- June 16, 2014 to (a) participate and deliver an invited lecture on "Surface Engineering at ARCI and Preliminary ICP Field Trips" at the 'Spring 2014 Thermal Spray Consortium Meeting' held at Stony Brook, (b) attend the '40th IEEE Photovoltaic Specialists Conference' held at Denver and c) visit the Solar Energy Research Institute for India and United States (SERIUS), Denver for review and technical discussion.
  7. Mr. D. Srinivasa Rao, Dr. R. Gopalan and Dr. Raju Prakash visited Russia and Ukraine during May 31-June 06, 2014 for technical discussions with the Indo-Russian Science and Technology Centre (IRSTC).
  8. Dr. N. Rajalakshmi visited United Kingdom during June 17-25, 2014 to a) participate in the review meeting of the DST-EPSC project on 'Mind the Gap-Jumping the Hurdles for PEMFC Commercialization' and b) visit Trinity College, London., Imperial College, London and The Open University, Ireland for technical discussions.
  9. Dr. R. Subasri visited The Netherlands during June 23-26, 2014 to participate in the 'Coatings Science International (COSI 2014)' and presented a paper on "Sol-gel Derived Zeolite -MgF<sub>2</sub> Composite Antireflective Coatings with Improved Mechanical Properties on Polycarbonate".
  10. Ms. Prithi Jayaraj, Senior ARCI Fellow visited Rhode Island, USA during July 31-August 10, 2014 to participate in the 'Gordon Research Symposium and Conference on Fuel Cells' and presented a paper on "Studies on Sulfur Tolerance with Mesoporous Electrocatalysts".
  11. Dr. N. Rajalakshmi visited Japan during August 01-06, 2014 to (a) attend the DST- Japan Society for the Promotion of Science (JSPS) project on 'Organoboron Organic-Inorganic Hybrids as Solid Electrolyte for Li Batteries with Graphene based Anodes' and (b) to participate in the 'India-Japan Symposium on Automotive Technologies, Energy, Fuel and Plastics 2014' and delivered an Invited lecture on "Energy Conversion Devices for Automotive Applications".
  12. Dr. R. Gopalan visited Annapolis, USA during August 15-22, 2014 to participate in the 'International Conference on Rare Earth Permanent Magnets (REPM 2014)' and delivered an invited lecture on "Rare Earths: Present and Future, the Indian Scenario".
  13. Dr. S.V. Joshi visited University West, Sweden during August 29-September 15, 2014 and delivered invited lectures on "Opening New Vistas for Coating Deposition through Solution Precursor Plasma Spraying" and "Applications of High Power Lasers in Advanced Manufacturing".
  14. Dr. Sanjurani Chandaroy visited Kuala Lumpur, Malaysia during September 04-05 2014 to participate in the '4th International Conference on Energy and Environmental Science (ICEES 2014)' and presented a paper on "Effect of Nanotube Diameter on Photo-Electrochemical Properties of Carbon Quantum Dot Functionalized TiO<sub>2</sub>Nanotubes".
  15. Dr. G. Sundararajan visited Hamburg, Germany during September 09-12, 2014 to attend the '3rd Meeting of India-DESY Steering Committee'.
  16. Dr. Joydip Joardar and Mr. K. Ramesh Reddy visited Rigaku Corporation, Tokyo, Japan during September 15-21, 2014 for pre-dispatch inspection cum training on 'X-Ray Diffraction Analysis System'.
  17. Dr. G. Sundararajan visited China during September 25-28, 2014 to lead an Indian delegation of 13 experts in the capacity as President, MRSI to the '5th MRS Trilateral Conference' and delivered a keynote lecture on "Nanotechnology related Research in India and at ARCI".
  18. Dr. G. Padmanabham visited Canada and USA during September 28- October 12, 2014 to a) participate in the '53rd Annual Conference of Metallurgists 2014 (COM 2014)' held at Vancouver and delivered a talk on "Laser-MIG Hybrid Welding of Maraging Steel" and b) visited Boeing, Seattle, USA for technical discussion.
  19. Dr. P. Suresh Babu visited Zwick GmbH & Co.KG (Zwick/Reoll), Ulm, Germany during October 13-16, 2014 to attend the '23<sup>rd</sup> International Forum for Materials Testing (23 testXpo)'.
  20. Dr. H. Purushotham visited Malaysia during October 29-31, 2014 to participate in the 'Regional Open Innovation Forum (OIF) on Promoting Nanotechnology and Agriculture for Sustainable Development' and delivered a lecture on "Knowledge Management for Promoting Nanotechnology R&D, Innovation and Commercialization".



21. Dr. Raju Prakash visited Washington DC, USA during November 11-14, 2014 to participate in the '10th Annual Knowledge Foundation Conference on Lithium Battery Power and Battery Safety' and made a poster presentation on "Fabrication and Electrochemical Performance of Lithium Ion Batteries for EV/HEV Applications".
22. Mr. Kaliyan Hembram visited Boston, USA during November 28-December 07, 2014 to participate in the 'Symposium on Flame and High Temperature Nano Particle Synthesis at MRS Conference' and presented a paper on "Shape Control Synthesis of ZnO Nano Powders and for Varistors Application by Flame Spray Pyrolysis".
23. Dr. Dibyendu Chakravarty who was deputed to Rice University, USA under the Indo-US fellowship programme, participated in the 'Symposium on Graphene and its Applications at MRS Conference' held at Boston during November 29-December 06, 2014 and presented a paper on "Graphene based Foams as Potential Electrodes for Supercapacitor Electrodes".
24. Dr. K. S. Dhathathreyan visited Cape Town, South Africa during December 01-03, 2014 to participate in CARISMA 2014 and delivered an invited lecture on "Recent Advances in Hydrogen Technology Development at the Centre for Fuel Cell Technology, ARCI".
25. Dr. Tata N. Rao visited Malaysia during December 04-05, 2014 to attend the Committee of Asian Standardization for Photocatalytic Materials and Products (CASP) meeting and delivered a lecture on "The Status of Photocatalytic Work Carried out in India".
26. Dr. G. Sundararajan visited USA during January 25-February 02, 2015 a) to participate in the '39th International Conference on Advanced Ceramics and Composites' held at Daytona Beach, and delivered an invited lecture on "Processing, Structure and Thermal Properties of Solid-State Sintered SiC Foams by Aqueous Gelcasting" and b) visited Florida International University, Miami and delivered an invited lecture on "Nano Materials at ARCI : An Overview".
27. Dr. G. Sivakumar visited United Kingdom during March 21-April 05, 2015 to attend the quarterly progress meeting of the DST-EPSCRC joint project on 'Improvements in Gas Turbine Performance via Novel Plasma Spray Coatings Offering Protection Against Ingested Species' and also to conduct performance studies related to the project.
28. Dr. S.V. Joshi visited United Kingdom during March 28 - April 05, 2015 to attend the quarterly progress meeting of the DST-EPSCRC joint project on 'Improvements in Gas Turbine Performance via Novel Plasma Spray Coatings Offering Protection against Ingested Species' and also to visit University of Cambridge and Cranfield University for technical discussions.

## Lectures by ARCI Personnel in India

1. Dr. Sanjay R. Dhage delivered an invited lecture on "ARCI's Research and Technology Demonstration Initiatives for Solar Energy Applications" at a 'Programme on Research Directions in Solar Energy -2014' held at Bengaluru during March 31 - April 02, 2014.
2. Dr. T.N. Rao delivered an invited lecture on "Nanostructured Nano-porous High Performance Anodes for Li Ion Batteries" at the 'Indo-US workshop on Engineered Electrodes for Electrochemical Energy Storage' held at Chennai during April 03-04, 2014.
3. Dr. N. Rajalakshmi delivered an invited lecture on "Engineering of PEMFC Electrodes for Impure Fuel and Air" at the 'Indo-US Conference on Engineered Electrodes for Electrochemical Energy Storage' held at Chennai during April 03-04, 2014.
4. Dr. T.N. Rao delivered an invited lecture on "Nanomaterials for High Performance Fuels and Lubricants" at the 'International Symposium on Fuels & Lubricants- 2014 (ISFL-2014)', held at New Delhi during April 15-17, 2014.
5. Dr. N. Rajalakshmi delivered an invited lecture on "Activated Carbons - Promising Materials for Hydrogen Storage" at the '9th International Symposium on Fuels and Lubricants' held at Faridabad on April 16, 2014.
6. Dr. S.V. Joshi delivered an invited lecture on "Opening New Vistas for Coating Deposition through Solution Precursor Plasma Spraying" at the '2nd National Symposium on Polymers and Coatings' held at Hyderabad on April 25, 2014.
7. Dr. P. K. Jain delivered an invited lecture on "Synthesis of Carbon Nanomaterials for Electronics Application" at the 'National Faculty Development Programme on Recent Developments in Microwave & RF Engineering' held at University of Kurushetra, Kurushetra during May 05-10, 2014.
8. Dr. K. S. Dhathathreyan delivered an invited lecture on "Integration of Wind Energy with other Energy Sources - A Case of Sustainable Hydrogen through Wind Energy" at the '13th International Training Course on Wind Turbine Technology and Applications' held at Chennai during May 07-30, 2014.
9. Dr. K.S. Dhathathreyan delivered an invited lecture on "Nanomaterials in Energy Conversion Devices - Some Recent Developments at CFCT- ARCI" at the 'R&D Conclave -VIII on Emerging Technologies for Sustainable Growth' held at Kodaikanal during May 11-13, 2014.

10. Dr. H. Purushotham delivered an invited lecture on "Patinformatics for Assessing Global Trends in Nanotechnology Applications in Upstream Oil Industry" at the 'R&D Conclave –VIII on Emerging Technologies for Sustainable Growth' held at Kodaikanal during May 11-13, 2014.
11. Dr. H. Purushotham delivered an invited lecture on "Freedom to Operate Search : A Case Study" at the Thomson Innovation User Group Meeting" held at Hyderabad on May 21, 2014.
12. Dr. S. Sakthivel delivered an invited lecture on "Novel Type of Chemical Coatings for Explosive Applications" at the 'Workshop on Chemical Coatings on Explosives' held at Mumbai on May 21, 2014.
13. Dr. N. Rajalakshmi delivered an invited lecture on "Carbon- An Energy Material" at the 'Carbon – One-Day Workshop on Energy Materials' held at Chennai on May 23, 2014.
14. Dr. H. Purushotham delivered an invited lecture on "Global Nanotechnology Regulatory Frameworks - An Overview" at the 'National Summit NANO-India - Policy and Regulation & 3rd Innovation Excellence Awards' held at New Delhi on June 10, 2014.
15. Dr. R. Gopalan delivered an invited lecture on "Technological Significance of Lithium Ion Batteries, Magnets and Thermoelectric Materials for Energy Savings" at the 'Conference on Energy Storage Systems for Power Electronic Application' held at Hyderabad during June 12-13, 2014.
16. Dr. S.V. Joshi delivered an invited lecture on "Realizing Novel Coating Architectures for Property Enhancement using Powders and Solution Precursors" at the 'International Conference on Powder Metallurgy and Particulate Materials (PM-15)' held at Mumbai during June 19-21, 2014.
17. Dr. Neha Hebalkar delivered an invited lecture on "Surface Engineering of Nanomaterials" at the 'National Conference on Impact of Nanoparticles on Health and Environment' held at Hyderabad on June 23, 2014.
18. Dr. K. Suresh delivered a lecture on "Celebrating 100 Years of X-Ray Diffraction" at ARCI, Hyderabad on June 23, 2014.
19. Dr. S.V. Joshi delivered an invited lecture on "Advances in Surface Engineering for Wear Protection" at the '6th Summer School in Tribology conducted by Tribology Society of India' held at Gurgaon on June 26, 2014.
20. Dr. T. N. Rao delivered a contributory lecture on "Nanostructured Materials for Li-Ion Battery and Filtration Applications" at 'TEQIP Workshop on Fundamentals and Applications of Nanofibers' held at Hyderabad during July 04-05, 2014.
21. Dr. Sanjay Bhardwaj delivered invited lectures on "Forging Technological Alliances in the Advanced Materials Sector" and "Entry and Growth Strategies for Technology-based Enterprises" at the 'Entrepreneurship Development Programme (EDP)' held at Hyderabad on July 17, 2014.
22. Dr. R. Easwaramoorthi delivered an invited lecture on "Next Generation Solar Cells" at the 'National Conference on New Frontiers in Mechanical Engineering' held at Kakinada during July 18-19, 2014.
23. Dr. T. N. Rao delivered an invited lecture on "Nanomaterials-based Technologies for Energy and Water Applications" at the 'International Conference on Advancements in Materials, Health and Safety towards Sustainable Energy and Environment (MHS-2014)', held at Chennai during August 07-08, 2015.
24. Dr. S.V. Joshi delivered an invited lecture on "The Excitement of Developing Materials Technologies for Strategic and Industrial Applications" at the 'Annual NK Batra Quiz' held at IIT-Kanpur on August 10, 2014.
25. Dr. K. Ramya delivered an invited lecture on "Electrochemistry Applications in Fuel cells" at the 'DST SERB School on Fundamental Electrochemical Principles Applied to Problems in Science and Engineering' held Chennai during August 10-14, 2014.
26. Dr. K. S. Dhathathreyan delivered an invited lecture on "Sustainable Hydrogen through Wind Energy" at the '14th International Training Course on Wind Turbine Technology and Applications' held at Chennai during September 03-30, 2014.
27. Dr. T. N. Rao delivered an invited lecture on "Opportunities for Metal Matrix Nanocomposites in Structural and Electronic Applications" at the 'National Workshop on Challenges and Latest Trends in Nanocomposites' held at Visakhapatnam on September 04, 2014.
28. Dr. G. Padmanabham delivered an invited lecture on "Laser processing of Materials and Applications" at the 'National Welding Meet 2014 (NWM 2014)' held at Chennai during September 05-06, 2014.
29. Dr. N. Rajalakshmi delivered an invited lecture on "Passion for Science and Technology" at the Sri Sastha Engineering College, Chennai on September 09, 2014.
30. Dr. R. Gopalan delivered a plenary lecture on "A Survey on Rare Earths and their Role for Magnet Technology" at the International Conference on Magnetic Materials and Applications (ICMAGMA 2014)' held at Pondicherry during September 15-17, 2014.
31. Dr. D. Prabhu delivered an invited lecture on "Microstructural Engineering of Magnetic Materials"

- at the 'International Conference on Magnetic Materials and Applications (ICMAGMA 2014)' held at Pondicherry during September 15-17, 2014.
32. Dr. H. Purushotham delivered an invited lecture on "Transfer of Technology: R&D to Industry" at 'CEP on Techno-Managerial Challenges in R&D Organizations' held at Hyderabad during September 17- 19, 2014.
  33. Dr. Roy Johnson delivered an invited lecture on "Engineering Ceramic Materials: An Overview" at Karunya University, Coimbatore on September 21, 2014.
  34. Dr. R. Balaji delivered an invited lecture on the "Recent Trend in Hydrogen Production Technologies" at the 'National Seminar on Recent Research Trend in Chemistry' held at Vellore on September 26, 2014.
  35. Dr. K. S. Dhathathreyan delivered an invited lecture on "Relevance of Hydrogen and Fuel Cell Technologies in the Sustainable Energy Scenario" at the 'Workshop on Recent Trends in Fuel Cell Technologies' held at Guntur on September 26, 2014.
  36. Dr. K. S. Dhathathreyan delivered an invited lecture on "Fuel Cell Technology - Engineering Challenges" at Vel Tech University, Chennai on October 11, 2014.
  37. Dr. G. Padmanabham delivered an invited lecture on "Joining and Surface Engineering with Lasers and their Applications" at the 'One-Day Workshop on Advances in Welding and Surface Engineering (AWSE)' held at Hyderabad on October 17, 2014.
  38. Dr. S.V. Joshi delivered an invited lecture on "Surface Engineering: A Vast Playground to Pursue Cutting-Edge Research and Realize Exciting Applications" at the 'One-Day Workshop on AWSE' held at Hyderabad on October 17, 2014.
  39. Dr. G. Padmanabham delivered a special invited lecture on "Laser Assisted Joining of Materials and Applications" at 'International Welding Symposium (IWS 2k14)' held at Mumbai during October 28-30, 2014.
  40. Dr. N. Rajalakshmi delivered an invited lecture on "Advanced Materials for Energy Storage" at the 'National Seminar on Advance Materials and Renewable Energy (NSAMRE 2014)' held at Chennai on October 30, 2014.
  41. Mr. S. Sankar Ganesh delivered an invited lecture on "Role of Information and Communication Technology in Industries" at a 'Workshop on Science and Technology (SAT-2014)' held at Srikakulam on October 30, 2014.
  42. Dr. Malobika Karanjai delivered an invited lecture on "Friction Composite Materials" at the 'International Workshop on Carbon, Composites and Diamonds' held at Nashik during October 30-31, 2014.
  43. Dr. Sanjay Bhardwaj delivered invited lectures on "Research and Technology Organization (RTO) - Industry Partnership: Case Studies" and "Insights on Idea to Business Value Chain in the Advanced Materials Sector" at National Institute of Industrial Engineering (NITIE), Mumbai on October 31, 2014.
  44. Dr. K. S. Dhathathreyan delivered an invited lecture on "Recent Advances in Nanomaterials for Energy Conversion Devices at CFCT-ARCI" at the 'Indian Institute of Materials - 52nd National Metallurgists Day - 68th Annual Technical Meeting (IIM-NMD-ATM 2014)' held at Pune during November 12-15, 2014.
  45. Dr. S.V. Joshi delivered an invited lecture on "Solution Precursor Plasma Spraying: Opening New Vistas for Exciting Research and Niche Applications" at the 'IIM-NMD-ATM 2014' held at Pune during November 12-15, 2014.
  46. Dr. S. Sakthivel delivered an invited lecture on "Nano Functional Materials / Coatings for Solar Energy Harvesting Technologies" at the 'International Conference on Chemistry and Materials' held at Trichy during November 14-15, 2014.
  47. Dr. H. Purushotham delivered an invited lecture on "Knowledge Management for Promoting Nanotechnology R&D and Innovation" at '7th AP Science Congress (APSC)' held at Hyderabad on November 15, 2014.
  48. Dr. G. Sundararajan delivered an invited lecture on "Development of Nanostructured Products at ARCI" at PSG Institute of Advanced Studies, Coimbatore on November 17, 2014.
  49. Dr. K.S. Dhathathreyan delivered an invited lecture on "Wind Energy for Sustainable Hydrogen" at the '12th International Training Course on Wind Turbine Technology and Applications Specifically for African Countries' held at Chennai during November 19 - December 12, 2014.
  50. Dr. S. Sakthivel delivered an invited lecture on "Importance of Solar Functional Coatings for Solar Thermal and PV Applications" at the 'Conference on Recent Advances in NanoSciences (RANSS' 44)' held at Vellore during November 21-22, 2014.
  51. Dr. S. Sakthivel delivered an invited lecture on "A Role of Nanofunctional Coatings for Solar Application" at Vellore Institute of Technology (VIT) University, Vellore on November 22, 2014.
  52. Dr. G. Sundararajan delivered an invited lecture on "Techniques for the Assessment of the Integrity and Quality of Cold Sprayed Coatings" at the 'Asian Thermal Spray Conference (ATSC) 2014' held at Hyderabad on November 25, 2014.



53. Dr. L. Rama Krishna delivered an inaugural lecture on "Smart Materials: A Surface Engineering Perspective" at the 'National Seminar on Smart Adaptive Structures and Intelligent Systems' held at Vijayawada on November 28, 2014.
54. Dr. R. Vijay delivered an invited lecture on "Development of High Performance Materials" at the 'National Seminar on Smart Materials, Adaptive Structures and Intelligent Systems (SASI 2014)' held at Vijayawada during November 28-29, 2014.
55. Dr. S.V. Joshi delivered an invited lecture on "ARCI's Initiatives for Energy Applications: Straddling Research and Technology Demonstration" at Indian Institute of Science Education and Research, Bhopal on December 01, 2014.
56. Dr. G. Padmanabham delivered an invited lecture on "Metal Joining with Lasers" at the 'CEP for DRDO Scientists on Welding and Joining Technologies' held at Hyderabad on December 02, 2014.
57. Dr. G. Padmanabham delivered an invited lecture on "Laser based Joining and Surface Engineering", at 'QIP for Engineering Faculty on Welding Technologies' held at Coimbatore on December 03, 2014.
58. Dr. Malobika Karanjai delivered invited lectures on "Friction Materials and Composites" and "PM in Bioengineering Materials" at the 'Powder Metallurgy Short Course' held at Ahmedabad during December 03-05, 2014.
59. Dr. S.V. Joshi delivered an invited lecture on "High Performance Coatings for Aerospace Applications" at the 'Discussion Meeting on Structural Engineering Materials for the Future: The Way Forward' held at Coorg during December 03-06, 2014.
60. Dr. G. Padmanabham delivered an invited lecture on "Laser Processing for Light-weighting" at the 'International Conference on Light Weighting Technologies (LWT 2014)' held at Chennai during December 04-05, 2014.
61. Dr. Sanjay Bhardwaj delivered invited lectures on "Leveraging Intellectual Capital of Public-Funded R & D Laboratories" and "Materials Technology based Entrepreneurship: Case Studies and Opportunities" at 'Technology-based Entrepreneurship Development Programme' held at Hyderabad on December 05, 2014.
62. Dr. N. Rajalakshmi delivered an invited lecture on "Electrochemistry for Energy Systems" at '13th Euroasia Conference on Chemical Sciences' held at Bengaluru during December 14-18, 2014.
63. Dr. S.V. Joshi delivered an Plenary lecture on "High Performance Coatings for Aerospace Applications" at the '18th NASAS Conference with Focus on Aerospace and Advanced Materials' held at Nagpur on December 15, 2014.
64. Dr. R. Balaji delivered a guest lecture on "Hydrogen Energy Technology-An Overview" at Gandhigramam Rural Institute, Tamilnadu on December 15, 2014.
65. Dr. K. S. Dhathathreyan delivered an invited lecture on "Overview of Hydrogen Energy Systems & Challenges" at the 'International Conference on Environment and Energy (ICEE-2014)' held at JNTU, Hyderabad during December 15-17, 2014.
66. Dr. P.K. Jain delivered an invited lecture on "Synthesis of Carbon Nanostructured Carbons for Energy Storage Applications" at the 'International Conference on Environmental and Energy (ICEE)' held at Hyderabad during December 15-17, 2014.
67. Dr. R. Gopalan delivered a contributory lecture on "Nanostructure Control to Enhance Figure of Merit in Bulk Filled Skutterudite" at the '2nd Indo-US Workshop on Thermoelectrics' held at New Delhi during December 15-17, 2014.
68. Dr. S.V. Joshi delivered a valedictory address on "High Performance Coatings to Combat Wear" at the 'National Tribology Conference' held at Bengaluru on December 17, 2014.
69. Dr. P.H. Borse delivered an invited lecture on "Exploring Nanotechnology for Fabrication of Advance Materials" at the 'National Conference on Application of Bioprocess and Nanotechnology in Chemical Engineering' held at Nandel during December 18-22, 2015.
70. Dr. G. Sundararajan delivered the RN Mookerjee Memorial lecture on "Technology Transfer and Commercialization: ARCI's Experience" at the Institute of Engineers, Hyderabad on December 20, 2014.
71. Dr. T.N. Rao delivered an invited lecture on "Nanomaterials for Energy and Water Management" at 'International Conference on Frontiers in Nanoscience, Technology and Applications (FINSTA-2014)' held at Puttaparti during December 20-22, 2014.
72. Dr. G. Sundararajan delivered an invited lecture on "An Overview of ARCI's Contributions to Defence and Aerospace Systems" at the 'National Conference on Advanced Materials for Defence & Aerospace Applications' held at Hyderabad on December 22, 2014.
73. Dr. G. Padmanabham delivered an invited lecture on "Innovative Engineering Solutions with Lasers", at the 'One-day Workshop on Laser Assisted Materials Processing 2014 (LAMP 2K14)' held at Annamalainagar on December 27, 2014.
74. Dr. R. Balaji delivered an invited lecture on "Hydrogen

- and Fuel cell Technologies for Sustainable Future” at the ‘Workshop on Fuel cell Technology’ held at SCAD Engineering College, Tirunelveli, on January 05, 2015.
75. Dr. G. Sundararajan delivered the Institute of Nano Science and Technology (INST) Foundation Day lecture on “Nanomaterials at ARCI : An Overview” at INST, Mohali on January 08, 2015.
  76. Dr. G. Sundararajan delivered an plenary lecture on “Plasma Electrolytic Oxidation: A Unique Coating Technique” at the ‘3rd International Conference on Laser & Plasma Applications in Materials Science (LAPAMS-2015)’ held at Kolkata during January 15-17, 2015.
  77. Dr. G. Padmanabham delivered an invited lecture on “Materials Joining and Repair with Lasers” at the ‘LAPAMS-2015’ held at Kolkata during January 15-17, 2015.
  78. Dr. Gururaj Telasang delivered an invited lecture on “Evaluation of Laser Assisted Material Deposition for Refurbishment of Pressure Die Casting Components in Simulated and Actual Conditions” at the ‘LAPAMS 2015’ held at Kolkata during January 15-17, 2015.
  79. Dr. G. Padmanabham delivered an invited lecture on “Lasers Assisted Materials Joining” at the ‘One-day Workshop on Welding Technologies’ held at Hyderabad during January 20, 2015.
  80. Dr. K. S. Dhathathreyan delivered an invited lecture on “Advances in Hydrogen and Fuel Cell Technologies at ARCI” at Dynamic Scientific Research and Technologies (DSRT), Thirunelveli during January 24-25, 2015.
  81. Dr. T. N. Rao delivered an invited lecture on “Nanomaterials in Surface Treatment and Bulk Embedded Process Technologies” at the ‘NanoIndia-2015’ held at Tajnavur during January 29-30, 2015.
  82. Dr. N. Rajalakshmi delivered an invited lecture on “Materials for Energy Storage and Conversion”, at the ‘National Conference on Advanced Materials and their Applications’ held at Chennai during January 29-30, 2015.
  83. Dr. P.K. Jain delivered an invited lecture on “Carbon and their Composites for Aerospace Applications” at the ‘International Conference in Fiber Reinforced Plastics’ held at Hyderabad during January 29-31, 2015.
  84. Dr. R. Subasri delivered an invited lecture on “Multifunctional Sol-Gel Nanocomposite Coatings: Development, Demonstration and Commercialization” at the ‘National Seminar on Nanomaterials and Global Perspectives’ held at Anantapur on January 30, 2015.
  85. Dr. K.S. Dhathathreyan delivered an invited lecture on “Wind Energy for Sustainable Hydrogen” at the ‘15th International Training Programme on Wind Turbine Technology and Applications for Indian Technical and Economic Cooperation Programme (ITEC)/ Special Common Wealth Assistance for Africa Programme (SCAAP) Member Countries’ held at Chennai during February 04 - March 03, 2015.
  86. Dr. P.K. Jain delivered an invited lecture on “Synthesis of Nanostructured Carbon for Various Applications” at the ‘National Seminar on Role of Physics in Technology Development’ held at Srikakulam during February 05-06, 2015.
  87. Dr. G. Sundararajan delivered an plenary lecture on “Nano Materials at ARCI: An Overview” at the ‘3rd International Conference on Nanoscience& Nanotechnology (ICONN2015)’ held at Chennai on February 06, 2015.
  88. Dr. G. Padmanabham delivered a keynote lecture on “Laser Metal Deposition for Repair of Tools and Steel Components” at the ‘International Conference on Additive Manufacturing 3D printing and 3D Scanning (ICAM 3D)’ held at Chennai during February 06-07, 2015.
  89. Dr. S.M. Shariff delivered an invited lecture on “Surface Engineering with Lasers”, at ‘ICAM 3D’ held at Chennai during February 06-07, 2015.
  90. Dr. Gururaj Telasang delivered a contributory lecture on “Pressure Die Casting Tools Building and Refurbishment by Laser Assisted Alloy Powder (AISI H 13) Deposition” at ‘ICAM 3D’ held at Chennai during February 06-07, 2015.
  91. Dr. R. Subasri delivered the Materials Research Society of India (MRSI) Medal lecture on “Commercialization of Sol-Gel Nanocomposite Coating Technology: Challenges and Perspectives” at the ‘26th MRSI Annual General Meeting’ held at Jaipur during February 09-11, 2015.
  92. Dr. K. S. Dhathathreyan delivered an invited lecture on “Fuel Cells” at the ‘Workshop on High Temperature Ceramics ( HITEC 2015)’ held at Chennai during February 09-20, 2015.
  93. Dr. P.K. Jain delivered invited lectures on “Carbon for Energy Storage Applications” and “Importance of Rajbhasha and Challenges during their Implementation” at the ‘10th All India Joint Rajbhasha Scientific and Technical Seminar’ held at Hyderabad during February 12-13, 2015.
  94. Dr. Sanjay Bhardwaj delivered invited lectures on “Utilizing Patent Information in the Academic and Research Organizations” and “Nurturing Technological Entrepreneurship” at ‘Faculty Development Programme’ held at Osmania University, Hyderabad on February 13, 2015.

95. Dr. S. Sakthivel delivered an invited lecture on "Cost Efficient Nanofunctional Coatings for Solar Thermal and PV Applications" at the 'DST-SERI Workshop on Advanced Solar Thermal Technologies' held at Bengaluru during February 16-17, 2015.
96. Dr. T.N. Rao delivered an invited lecture on "Nanomaterials for Energy and Water Management: The Indian Perspective" at the 'National Conference on Advanced Materials' held at Coimbatore during February 18-19, 2015.
97. Dr. R. Gopalan delivered an invited lecture on "Necessity of Tuning the Functional Materials Properties for Automotive Sector Applications" at the 'International Conference on Advanced Functional Materials (ICAFM 2014)' held at Trivandrum during February 19-21, 2015.
98. Dr. M. B. Sahana delivered an invited lecture on "Batteries for Electric Vehicles: Present and Future" at the '7th Indo-German Frontiers of Engineering' held at Agra during February 19-22, 2015.
99. Dr. K. Ramya delivered an invited lecture on "Polymer Electrolytes in Electrochemical Devices" at the 'International Conference on Advancements in Polymeric Materials (APM-2015)' held at Bengaluru during February 20-22, 2015.
100. Dr. R. Subasri delivered an invited lecture on 'Nanocomposite Coatings on Plastics for Improved Optical and Mechanical Properties' at 'APM-2015' held at Bengaluru during February 20-22, 2015.
101. Dr. Malobika Karanjai delivered an invited lecture on "Resin -Inorganic Coated Bonded Magnets" at 'APM-2015' held at Bengaluru during February 20-22, 2015.
102. Dr. P.H. Borse delivered a keynote lecture on "A Hunt for Efficient and Stable Hydrogen Producing Photocatalytic Nano/Materials: A Ladder to Renewable Energy" at the 'National Seminar - Vision Nano15' held at Coimbatore on February 21, 2015.
103. Dr. K. S. Dhathathreyan delivered an invited lecture on "Fuel Cells - Science and Engineering" at NIT, Warangal on February 23, 2015.
104. Dr. P.H. Borse delivered an invited lecture on "Energy Materials- Need of the Hour" at Hyderabad Institute of Technology And Management (HITAM), Hyderabad on February 28, 2015.
105. Dr. T. N. Rao delivered an invited lecture on "Nanomaterials: Synthesis, Characterization and Applications" at the 'National Seminar on Green Environment by Nano Chemical Technology (GENCT-2015)' held at Srikakulam during March 01-02, 2015.
106. Dr. B. V. Sarada delivered an invited lecture on "Nanostructured Materials by Novel Electrochemical Synthesis and Applications in Solar Energy Conversion and Electronics" at 'GENCT- 2015' held at Srikakulam during March 01-02, 2015.
107. Dr. P.K. Jain delivered an invited lecture on "Carbon Nanomaterials for Targeted Drug Delivery System" at the 'Refresher Course for Faculties' held at Hyderabad during March 03-20, 2015.
108. Dr. R. Balaji delivered an invited lecture on "Electrochemistry and its Application-An Introduction" at the 'Workshop on Functional Coatings Recent Trend' held at Madurai on March 06, 2015.
109. Dr. Roy Johnson delivered an invited lecture on "Transparent Polycrystalline Alumina: A New Class of Functional Ceramics" at the '1st International Conference on Alumina and Functional Ceramics' held at Kolkata during March 11-13, 2015.
110. Dr. R. Easwaramoorthi delivered an invited lecture on "Nanomaterials for High-performance Solar Cells" at the 'National Seminar on Advanced Materials and their Applications' held at Coimbatore on March 12, 2015.
111. Dr. R. Subasri delivered a invited lecture on "Sol-Gel based Nanocoatings : Basic Principles of Processing and Characterization and Applications" at IIT Madras, Chennai on March 14, 2015.
112. Dr. R. Balaji delivered an invited lecture on "Material Aspects of Electrolytic Hydrogen Generation" at the 'National Conference on Advanced Materials in Energy and Environmental Applications' held at Coimbatore on March 20, 2015.
113. Dr. M. B. Sahana delivered an invited lecture on "Lithium -Ion Battery Materials: Present and Future" at the 'National Conference on Materials for Energy Conversion and Storage' held at Chennai during March 20-21, 2015.
114. Dr. S. Anandan delivered an invited lecture on "Core-Shell Structured Carbon Coated Electrode Materials for Improved Lithium -Ion Battery Performance" at the 'National Conference on Materials for Energy Conversion and Storage' held at Chennai during March 20-21, 2015.
115. Dr. N. Rajalakshmi delivered an invited lecture on "Advanced Energy Storage Systems- Fuel Cells, Batteries, Super-Capacitors" at the 'National Conference on Materials for Energy Conversion and Storage' held at Chennai during March 20-21, 2015.
116. Dr. R. Balaji delivered an invited lecture on "PEM Fuel Cell Technology for Sustainable Future" at the 'National Conference on Frontiers Chemistry and Environment' held at Vellore on March 28, 2015.
117. Dr. Krishna Valleti delivered an invited lecture on



"Synthesis, Characterization and Applications of Thin Films", at the 'National Seminar on Recent Trends in Physics' held at Cuddapa on March 29, 2015

## Papers Presented at Indian Conference/Symposia

1. Dr. R. Subasri made a poster presentation on "Water Conservation through Surface Engineering of Materials" at the 'Indo-American Frontiers of Engineering Symposium-2014' held at Mysore during May 18-21, 2014.
2. Dr. H. Purushotham presented a paper on "Patinformatics to Assess Global Technology Trends- A Case Study on Iron Oxide Nanoparticles for Biomedical Applications" at the 'International Conference on Nano Science and Engineering Applications (ICONSEA 2014)' held at Hyderabad during June 26-28, 2014.
3. Mr. S. Sudhakara Sarma made a poster presentation on "Nanofiber Coatings in Filtration Application" at 'TEQIP Workshop on Fundamentals and Applications of Nanofibers' held at Hyderabad during July 04-05, 2014.
4. Dr. K. Suresh presented a paper on "Microstructure and Micro-Texture of Pulsed Electron Deposited Ni" at the 'International Conference on Electron Microscopy and XXXV Annual Meeting of Electron Microscopy of India (EMSI)' held at New Delhi during July 09-11, 2014.
5. Mr. M. Ramakrishna presented a paper on "TEM Study on 18Cr ODS Steel Alloys" at the 'EMSI' held at New Delhi during July 09-11, 2014.
6. Dr. H. Purushotham presented a paper on "Incorporation of Nanomaterials in Li-Ion Batteries – A Worldwide Patent Analysis" at the 'International Conference on Energy Materials (ICEM-2014)' held at Chennai during July 28-30, 2014.
7. Ms. Shanmuga Priya (Dr. K.S. Dhathathreyan) presented a paper on "Photoelectrochemical Studies on TiO<sub>2</sub> and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> TiO<sub>2</sub>Rod-like Nanostructures at the 'International Conference on Electrochemical Science and Technology (ICONEST-2014)' held at Bengaluru during August 07-09, 2014.
8. Dr. Sanjay Bhardwaj made a presentation on "ARCI Technologies and Knowledgebase Available for Adaptation" at the 'Society for Technology Management (STEM) Annual Summit' held at Hyderabad during August 19-20, 2014.
9. Mr. R. Rajashekar (Dr. R. Gopalan) made a poster presentation "Synthesis of High Coercivity SrFe<sub>12</sub>O<sub>19</sub> Powders" at the 'ICMAGMA 2014' held at Pondicherry during September 15-17, 2014.
10. Mr. V. V. Ramakrishna (Dr. R. Gopalan) made a poster presentation on "Evolution and Growth of LTP MnBi in Mn-Bi System", at the 'ICMAGMA 2014' held at Pondicherry during September 15-17, 2014.
11. Ms. S. Nirmala presented a paper on "Computer Control of a Thermal Cycling System" at the 'National Symposium on Instrumentation (NSI -39)' held at Haridwar during October 15-17, 2014.
12. Ms. V. Uma presented a paper on "Design Automation for Circular Motion of Substrate during Electrophoretic Deposition (EPD) of Sol-Gel Coating Process" at 'NSI-39' held at Haridwar during October 15-17, 2014.
13. Mr. Ch. Sambasiva Rao presented a paper on "Development of an Arduino based Charge Controller for a Solar Panel" at 'NSI-39' the held at Haridwar during October 15-17, 2014.
14. Ms. L. Subashini (Dr.G. Padmanabham) made a poster presentation on "Investigation of Microstructure and Properties of Laser-MIG Hybrid Welded Maraging Steel" at the 'Workshop on Advances in Welding and Surface Engineering (AWSE) held at Hyderabad on October 17, 2014.
15. Mr. E. Anburasu presented a paper on "Dissimilar Material Joining of Aluminium to Steel: Study on Critical Parameters Influencing Joint Properties" at the 'Workshop on AWSE' held at Hyderabad on October 17, 2014.
16. Dr. Sanjay Bhardwaj presented a paper on "Application of Intellectual Property Development Indices (IPDIs) for Identifying R&D-Academia-Industry Collaboration Opportunities in Advanced Materials Sector" at the 'National Conference on Industrial Engineering and Technology Management (NCIETM)' held at Mumbai on October 30, 2014.
17. Mr. S. Vasu (Dr.R.Gopalan) presented a paper on "Investigation of Synthesis Pathways LiNi<sub>0.8</sub>CO<sub>0.15</sub>Al<sub>0.05</sub>O<sub>2</sub> Solid State Assisted Co-Precipitation" at the 'Indian Institute of Materials – 52nd National Metallurgists Day – 68th Annual Technical Meeting (IIM-NMD-ATM 2014) held at Pune during November 12-15, 2014.
18. Ms. K. Kumari (Dr. R. Gopalan) presented a paper on "The Effect of Electrode Thickness on Charge/Discharge Hysteresis of Lithium-Ion Cell" at the 'IIM-NMD-ATM 2014' held at Pune during November 12-15, 2014.
19. Dr. Gururaj Telasang presented a paper on "Evaluation of Laser Cladding Process on AISI H 13 Hot Work Tool Steel for PDC Die Repair Application" at the 'IIM-NMD-ATM 2014' held at Pune during November 12-15, 2014.
20. Dr. Nitin Wasekar presented a paper on "Corrosion Studies of Near Amorphous Ni-W Alloy Coatings" at 'IIM-NMD-ATM 2014' held at Pune during November 12-15, 2014.

21. Ms. L. Subashini (Dr. G. Padmanabham) presented a paper on "Fusion and Microstructural Characteristics of Laser-MIG Hybrid Welded Maraging Steel" at the 'IIM-NMD-ATM 2014' held at Pune during November 12-15, 2014.
22. Mr. N. Ravi presented a paper on "Effect of Nitrogen Pressure on Mechanical Properties of nc-Ti AlN/a-Si<sub>3</sub>N<sub>4</sub> Nanocomposite Coatings Deposited by CAPVD Process" at the 'Andhra Pradesh Academy of Sciences (APAS) Golden Jubilee Congress', held at Hyderabad during November 13-15, 2014.
23. Mr. M. Ramesh (Dr. Roy Johnson) presented a paper on "Correlation of Primary Particle Size with the Morphology and Flow Behaviour of Spray Dried Alumina Granules" at the 'APAS Golden Jubilee Science Congress' held at Hyderabad during November 13-15, 2014.
24. Ms. K. Bhargavi (Dr. Roy Johnson) presented a paper on "Effect of Pore Former Addition on the Thermal and Mechanical Properties of Zirconia Ceramics" at the 'APAS Golden Jubilee Science Congress' held at Hyderabad during November 13-15, 2014.
25. Dr. Shilochana Dudi (Dr. K.S. Dhathathreyan) made a poster presentation on "Modelling Studies in Alkaline Fuel Cells" at the 'COMSOL Conference 2014' held at Bengaluru during November 13-14, 2014.
26. Dr. L. Ramakrishna presented a paper on "Structure-Property Corelation in Cold Sprayed Splats of Copper and Copper Alloys with Different Stacking Fault Energies (SFE)" at the '6th Asian Thermal Spray Conference (ATSC 2014)' held at Hyderabad during November 24-26, 2014.
27. Dr. S. Kumar presented a paper on "Effect of Heat Treatment on the Mechanical Properties and Corrosion Performance of Cold Sprayed Tantalum Coating" at the 'ATSC 2014' held at Hyderabad during November 24-26, 2014.
28. Mr. Manoj Kumar (Dr. S. Kumar) made a poster presentation on "Development of Erosion-Corrosion Resistance Cold-Spray Nanostructured Ni-20Cr Coating for Coal Fired Boiler Applications" at the 'ATSC 2014' held at Hyderabad during November 24-26, 2014.
29. Mr. K.R.C. Soma Raju presented a paper on "Sol-Gel Derived Solar Selective Coatings for Solar Thermal Applications" at the 'International Conference on Environment and Energy' held at Hyderabad during December 15-17, 2014.
30. Ms. Alka Pareek (Dr. P.H. Borse), presented a paper on "Electrochemical Characterization of Ag-loaded Nano-Titania Modified CS/ Polysulphide Electrolyte Interface" at the 'International Conference on Environment and Energy' held at Hyderabad during December 15-17, 2014.
31. Dr. D. Sivaprahasam presented a paper on "Thermoelectric Characterization of Nano-Structured Cu<sub>1</sub>Pb<sub>18</sub>Sb<sub>1</sub>Te<sub>20</sub> and Ni<sub>1</sub>Pb<sub>18</sub>Sb<sub>1</sub>Te<sub>20</sub> Compounds" at the '2nd Indo-US Workshop on Thermoelectrics-Recent Trends in Thermoelectric Materials –Fundamentals to Applications' held at New Delhi during December 15-17, 2014.
32. Dr. Manjusha Battabyal (Dr. R. Gopalan) presented a paper on "Nanostructure Control to Enhance Figure of Merit in Filled Bulk Skutterudities" at the '2nd Indo-US Workshop on Thermoelectrics-Recent Trends in Thermoelectric Materials –Fundamentals to Applications' held at New Delhi during December 15-17, 2014.
33. Mr. Manish Tak presented a paper on "Study on the Influence of Pre-heating on Cracking Susceptibility in Laser-Clad Coatings of NiCr on Austenitic Stainless Steel" at the 'International Conference on Laser and Plasma Application in Materials Science' held at Kolkata during January 15-17, 2015.
34. Mr. K.V. Phani Prabhakar presented a paper on "Aluminium Steel-Weld Brazing by Cold Metal Transfer (CMT) Process-Influence of Filler Composition on Microstructure and Mechanical Properties" at the 'National Welding Seminar (NWS 2015-15) & Weld India 2014-15' held at Jamshedpur during January 22-24, 2015.
35. Mr. Manish Tak presented a paper on "Refurbishment of Grey Cast Iron Components using Laser Cladding Process using Ni based Powders" at 'International Conference on Additive Manufacturing, 3D Printing & 3D Scanning (ICAM 3D)' held at Chennai during February 06-07, 2015.
36. Mr. Kaliyan Hembram presented a paper on "Large Scale Manufacturing of Doped ZnO Nanopowders for Varistor Applications by Top-Down and Bottom-Up Approach" at the 'Regional Conference of Young Scientists on Nanoscience and Nanomaterials' held at Bengaluru during February 18-20, 2015.
37. Mr. G. Vijay Dev (Dr. K.S. Dhathathreyan) presented a paper on "Recovery of Waste Heat in a HT-PEMFC" at the (Select X5 meet ) held at Karaikudi during February 26-27, 2015.

### Participation in Indian Conferences/Symposia/ Seminars/ Workshops/Exhibitions

1. Dr. H. Purushotham, Mr. V. Balaji Rao, Dr. P.K. Jain, Dr. Roy Johnson, Mr. D. Srinivasa Rao, Dr. T.N. Rao, Dr. R. Vijay, Dr. G. Ravi Chandra, Mr. K.V.P. Phani Prabhakar, Dr. Malobika Karanjai, Dr. Sanjay Bhardwaj, Dr. G. Siva Kumar, Dr. I. Ganesh, Dr. S.M. Shariff, Dr. R. Subasri, Dr. Joydip Joardar, Dr. P.H. Borse, Ms. S. Nirmala, Mr. Kaliyan Hembram, Dr. Nitin P Wasekar, Dr. K. Murugan, Dr. P. Suresh Babu, Mr. R. Senthil Kumar, Dr. Neha Hebalkar,

- Dr. M. B. Suresh, Mr. R. Vijaya Chander, Mr. Pandu Ramavath, Ms. J. Revathi, Mr. Prasenjit Barick, Mr. Manish Tak, Mr. Naveen M. Chavan, Mr. M. Ramakrishna, Mr. S. Arun, Dr. S. Kumar, Dr. R. Easwaramoorthy, Mr. R. Prabhakara Rao, Mr. K. R.C. Somaraju, Mr. V. Mahender, Mr. P. Rama Krishna Reddy, Mr. V.C. Sajeev, Ms. V. Uma, Mr. G. Venkata Ramana Reddy, Mr. C. Karunakar, Mr. Ch. Sambasiva Rao, Mr. M. Srinivas, Mr. K. Srinivasa Rao, Mr. N. Venkata Rao, Mr. D. Sreenivas Reddy, Mr. J. Nagabhushana Chary, Mr. K. Ramesh Reddy, Mr. P.V.V. Srinivas, Mr. A.R. Srinivas, Mr. E. Anburasu, Mr. S. Sankar Ganesh, Mr. K. Naresh Kumar, Mr. G. M. Raj Kumar, Mr. A. Srinivas, Mr. Y. Krishna Sarma, Mr. G. Ramesh Reddy, Mr. B. Uday Kumar, Mr. P. Venugopal, Mr. P. Dharma Rao, Mr. G. Gopal Rao, Mr. Ravi Singh, Ms. K. Shakunthala, Mr. T. Venu, Mr. M. R. Renju, Mr. T.K. Gireesh Kumar, Mr. R. Anbarasu, Ms. K. Madhura Vani, Mr. Narendra K. Bhakta, Mr. P. Anjaiah, Mr. E. Konda, Mr. D. Krishna Sagar, Mr. D. Kutumba Rao, Mr. A. Praveen Kumar, Mr. A. Ramesh, Mr. K. Satyanarayana Reddy, Mr. K. Subba Rao, Mr. B. Subramanyeswara Rao, Mr. K.V.B. Vasantha Rayudu, Mr. B. Venkana, Mr. K. Venkata Ramana, Mr. Ch. Venkateswara Rao, Mr. G. Venkat Reddy, Mr. Govinda Kumar, Mr. A. Jagan, Mr. M. Satyanand, Mr. B. Hemanth Kumar, Mr. Sushanta Mukhopadhyay, Mr. P. Suri Babu, Mr. D. Manikya Prabhu, Mr. K. Ashok, Mr. E. Yadagiri, Mr. Ch. Jangaiah, Mr. Md. Sadiq, Mr. T. Satyanarayana, Mr. M.A. Fazal Hussain, Mr. J. Bansilal, Dr. K. Suresh, Mr. Vivek Patel, Dr. C.K. Nisha, Dr. Samba Sivudu, Ms. I. Sophia Rani, Mr. Ratnesh Kumar Gaur, Mr. Md. Shakeel Iqbal, Mr. Abhilasha Verma, Mr. N. Prasad, Mr. K. Ramesh, Mr. T. Panduranga Rao, Dr. P. Uday Bhaskar, Mr. B. Amol Chintaman, Mr. P. Sai Karthik, Mr. Siddartha Sankar Pal attended a 'Programme on New Rules on Sexual Harassment of Women at Workplace (Prevention, Prohibition and Redressal)' held at ARCI, Hyderabad during April 16-17, 2014.
2. Dr. Raju Prakash attended the 'ECS- India School on Advances in Batteries and Supercapacitors' held at Karikudi during May 13-15, 2014.
  3. Dr. G. Padmanabham, Dr. Sanjay Bhardwaj and Dr. Gururaj Telasang attended the Society of Automotive Engineers (SAE) INDIA-Hyderabad Division: monthly technical lecture on 'Additive Manufacturing for Automotive and Aerospace Applications' held at Hyderabad on May 30, 2014.
  4. Dr. N. Rajalakshmi and Dr. K. Ramya attended the 'One Day Seminar on Exploring the Current Issues in Sustainable Energy' held at Chennai on June 05, 2014.
  5. Dr. Sanjay Bhardwaj attended the SAE INDIA-Hyderabad Division: monthly technical lecture on 'Monitoring Technological Innovations in the Auto Sector through Patent Analytics' held at Hyderabad on June 20, 2014.
  6. Dr. G. Padmanabham and Dr. Gururaj Telasang attended the SAE INDIA-Hyderabad Division: monthly technical lecture on 'Laser Micromachining for Engineering Applications' held at Hyderabad on July 18, 2014.
  7. Ms. Priya Anish Mathews and Mr. S. Arun attended a 'Seminar on Protection of Intellectual Property Rights in India & Internationally' held at Hyderabad on July 18, 2014.
  8. Dr. I. Ganesh attended the 'International Conference on Electrochemical Science and Technology (ICONSET)' held at Bengaluru during August 07-09, 2014.
  9. Dr. G. Padmanabham and Dr. Gururaj Telasang attended the SAE INDIA-Hyderabad Division: monthly technical lecture on 'The Mobility Industry from the Eyes of an Organization Development Consultant' held at Hyderabad on August 22, 2014.
  10. Dr. G. Padmanabham, Dr. Sanjay Bhardwaj, Mr. Manish Tak, Dr. Gururaj Telasang and Mr. S. Arun attended the 'Entrepreneur and Student Interaction Meet at Club Officers Leadership Programme (COLT)' organized by SAE INDIA, Hyderabad Division at Dundigal, Hyderabad during August 30-31, 2014.
  11. Mr. M. Ramakrishna, Dr. K. Suresh and Dr. N. Padmavathi attended the 'National Seminar on Crystallography for Materials Scientists' held at Hyderabad during September 01-02, 2014.
  12. Dr. G. Padmanabham attended the '4th International Conference and Exhibition on Additive Manufacturing Technologies – AM 2014' held at Bengaluru during September 01-02, 2014.
  13. Dr. R. Subasri attended in the 'International Weathering Technology Symposium' held at Chennai on September 09, 2014.
  14. Mr. Arun Joshi, Ms. S. Nirmala and Mr. Ch. Sambasiva Rao attended a 'Programme on Key Measurement Insights' held at Hyderabad on September 11, 2014.
  15. Mr. G. Ramesh Reddy, Mr. P. Venu Gopal and Mr. T. Venu attended a 'Seminar on Public Procurement' held at Hyderabad on September 13, 2014.
  16. Mr. Manish Tak attended the 'Laser World of Photonics India 2014 Exhibition' held at Bengaluru during September 23-25, 2014.
  17. Mr. J. Nagabhushana Chary attended the 'Workshop on Advances in Welding and Surface Engineering' held at Hyderabad on October 17, 2014.



18. Mr. S. Arun participated in the 'AWSE Exhibition' held at Hyderabad on October 17, 2014.
19. Dr. G. Padmanabham, Dr. S.M. Shariff and Dr. Gururaj Telasang attended the SAE INDIA- Hyderabad Division: monthly technical lecture on 'Metro Rail Technology' held at Hyderabad on October 18, 2014.
20. Dr. V. C. Sajeev attended a 'Programme on Electrical Safety in India' held at Pune during November 19-21, 2014.
21. Mr. Balaji Rao attended the '8th International Conference on Capacitors' held at New Delhi during November 20-21, 2014.
22. Dr. Sanjay Bhardwaj and Mr. S. Arun attended the 'FICCI- Buyer-Seller Meet cum Exhibition' held at Hyderabad on November 24, 2014.
23. Dr. H. Purushotham, Dr. Sanjay Bhardwaj, Ms. Priya Anish Mathews, Mr. S. Arun, Mr. Samba Sivudu attended the 'Roving Seminar on Patent Corporation Treaty' held at Hyderabad on November 26, 2014.
24. Dr. S. M. Shariff attended the 'International Conference on Materials Engineering, Technology and Heat Treatment (MET-14)' held at Gandhi Nagar during December 04-06, 2014.
25. Mr. K. Naresh Kumar attended the '3rd Next-Generation Network Annual Workshop' held at Guwahati during December 15-17, 2014.
26. Mr. D. Sreenivas Reddy attended the 'International Conference on Environment and Energy', held at Hyderabad during December 15-17, 2014.
27. Dr. R. Vijay attended the 'One day Workshop on Failure Analysis in Industry' held at Hyderabad on January 20, 2015.
28. Mr. S. Nath Jana attended the 'Workshop on Emerging Applications of Laser Technology in Manufacturing' at Vellore on February 13, 2015.
29. Dr. S. Kumar attended the 'National Workshop on High Entropy Alloys: Prospects and Challenges' held at Chennai during March 28-29, 2015.
3. Mr. G.M. Raj Kumar attended the 'Management Training Programme on Accounting, Financial Management and Governance for Autonomous Bodies' held at Faridabad during July 21-25, 2014.
4. Mr. R. Prabhakara Rao attended a 'One-Day Training Programme on Right to Information Act, 2005' held at Chennai on August 04, 2014.
5. Mr. Y. Krishna Sarma attended the 'Technical Workshop on Enhancing Efficiency and Effectiveness of PS/PA and Personal Staff' held at New Delhi during August 07-09, 2014.
6. Mr. S. B. Chandrasekhar and Dr. Nitin P. Wasekar attended the 'Two-Day Training Programme on Reservation Policy' held at Chennai during September 12-13, 2014.
7. Dr. K. Ramya attended the 'Training Programme on Operation of Transmission Electron Microscope' held at IIT Madras, Chennai during January 12-16, 2015.
8. Dr. B. V. Sarada attended the 'NAIS-DST Training Programme for Women Scientists on Science for Progress in India: Innovations in Technologies for Scientists and Technologists' held at Bengaluru during February 23-27, 2014.
9. Mr. V. Balaji Rao, Mr. K.V. Phani Prabhakar, Dr. S.S. Sakthivel, Ms. S. Nirmala, Dr. Dibyendu Chakravarty, Dr. Nitin P. Wasekar, Dr. Neha Hebalkar, Dr. Sanjay Dhage, Mr. Prasenjit Barick, Mr. S. Arun, Mr. R. Vijaya Chander, Ms. A. Jyothirmayi, Ms. V. Uma, Mr. K. Srinivasa Rao, Mr. D. Sreenivas Reddy, Mr. A.R. Srinivas, Mr. S. Kalyanaraman and all security staff attended the 'One Day Training Programme on First Aid for Safety Coordinators' held at ARCI, Hyderabad on March 09, 2015.
10. Mr. D. Srinivasa Rao, Dr. R. Vijay, Mr. N. Ravi, Dr. Malobika Karanjai, Dr. Sanjay Bhardwaj, Dr. G. Siva Kumar, Dr. I. Ganesh, Dr. L. Rama Krishna, Dr. S.M. Shariff, Dr. Ravi N Bathe, Dr. Joydip Joardar, Dr. B.V. Sarada, Dr. P.H. Borse, Mr. Kaliyan Hembram, Dr. K. Murugan, Dr. Dibyendu Chakravarty, Dr. P. Suresh Babu, Mr. R. Senthil Kumar, Dr. Krishna Valleti, Dr. M. B. Suresh, Mr. S. Sudhakara Sarma, Mr. Pandu Ramavath, Ms. J. Revathi, Ms. Priya Anish Mathews, Mr. Manish Tak, Mr. Naveen M. Chavan, Mr. M. Ramakrishna, Mr. Balaji Padya, Ms. Papiya Biswas, Dr. Gururaj Telasang, Dr. S. Kumar, Mr. L. Venkatesh, Mr. S. Kalyanaraman, Mr. Debjyoti Sen, Mr. K. R.C. Somaraju, Mr. G. Venkata Ramana Reddy, Mr. P. Rama Krishna Reddy, Mr. Ch. Sambasiva Rao, Mr. C. Karunakar, Mr. M. Srinivas, Mr. N. Venkata Rao, Mr. M. Srihari, Mr. J. Nagabhushana Chary, Mr. A. Rajashekhar Reddy, Mr. E. Anburasu, Mr. S. Sankar Ganesh, Mr. K. Naresh Kumar and Mr. M. Ilaiyaraja attended the 'In-Plant Training on Industrial Safety' held at ARCI, Hyderabad during March 11-12, 2015.

### Participation in Training Programmes in India

1. Dr. Sanjay Bhardwaj and Mr. S. Arun attended the 'Training Programme on using Thomson Innovation Database for Patent Prior Art Search' held at Hyderabad on May 21, 2014.
2. Dr. Sanjay Bhardwaj and Mr. S. Arun attended the 'Training Programme on Espacenet Patent Database' held at Hyderabad on May 26, 2014.

# Patents' Portfolio

## Indian Patents Granted

| Title of Patent                                                                                                                                                                                                                                                              | Patent Application Number | Date of Filing | Patent Number | Date of Grant |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------|----------------|---------------|---------------|
| A Solar Drier                                                                                                                                                                                                                                                                | 487/<br>MAS/1994          | 08/06/1994     | 184674        | 23/09/2000    |
| A Process for Preparation of Reaction Bonded Silicon Carbide Components                                                                                                                                                                                                      | 1886/<br>MAS/1996         | 28/10/1996     | 195429        | 31/08/2006    |
| New Composite Material Having Good Shock Attenuating Properties and a process for the Preparation of Said Material                                                                                                                                                           | 976/<br>MAS/1998          | 06/05/1998     | 194524        | 02/01/2006    |
| Improved Process for the Preparation of Magnesium Aluminate Spinel Grains                                                                                                                                                                                                    | 29/MAS/1999               | 07/01/1999     | 200272        | 02/05/2006    |
| Ceramic Honey Comb Based Energy Efficient Air Heater                                                                                                                                                                                                                         | 30/MAS/1999               | 07/01/1999     | 200787        | 02/06/2006    |
| A Method and a Device for Applying a Protective Carbon Coating on Metallic Surfaces                                                                                                                                                                                          | 719/<br>MAS/1999          | 08/07/1999     | 211922        | 13/11/2007    |
| A Process for the Preparation of Improved Alumina Based Abrasive Material, an Additive Composition and a Process for the Preparation of the Composition                                                                                                                      | 122/<br>MAS/2000          | 18/02/2000     | 198068        | 16/02/2006    |
| A Process for the Production of Dense Magnesium Aluminate Spinel Grains                                                                                                                                                                                                      | 520/<br>MAS/2000          | 06/07/2000     | 198208        | 16/02/2006    |
| A Process for Preparing Ceramic Crucibles                                                                                                                                                                                                                                    | 806/<br>MAS/2000          | 26/09/2000     | 207700        | 20/06/2007    |
| An Improved Method for Making Honeycomb Extrusion Die and a Process for Producing Ceramic Honeycomb Structure using the Said Die                                                                                                                                             | 538/<br>MAS/2001          | 03/07/2001     | 198045        | 13/01/2006    |
| Device for Gas Dynamic Deposition of Powder Materials                                                                                                                                                                                                                        | 944/<br>MAS/2001          | 22/11/2001     | 198651        | 25/01/2006    |
| A Process for Forming Coatings on Metallic Bodies and an Apparatus for Carrying out the Process                                                                                                                                                                              | 945/<br>MAS/2001          | 22/11/2001     | 209817        | 06/09/2007    |
| An Improved Boronizing Composition                                                                                                                                                                                                                                           | 289/<br>MAS/2001          | 03/04/2001     | 220370        | 27/05/2008    |
| Process for Carbothermic Reduction of Iron Oxide in an Immiscible Flow with Constant Descent in Vertical Retort of Silicon Carbide                                                                                                                                           | 546/<br>CHE/2003          | 01/07/2003     | 205728        | 16/04/2007    |
| An Evaporation Boat useful for Metallization and a Process for the Preparation of Such Boats                                                                                                                                                                                 | 882/<br>CHE/2003          | 31/10/2003     | 201511        | 01/03/2007    |
| Titanium Based Biocomposite Material useful for Orthopaedic and other Implants and a Process for its Preparation                                                                                                                                                             | 2490/<br>DEL/2005         | 14/09/2005     | 228353        | 03/02/2009    |
| An Improved Method of Forming Holes on a Substrate using Laser Beams                                                                                                                                                                                                         | 3205/<br>DEL/2005         | 29/11/2005     | 239647        | 29/03/2010    |
| A Method of and an Apparatus for Continuous Humidification of Hydrogen Delivered to Fuel Cells                                                                                                                                                                               | 670/<br>CHE/2007          | 30/03/2007     | 247547        | 19/04/2011    |
| An Improved Process for the Preparation of Doped Zinc Oxide Nanopowder useful for the Preparation of Varistors                                                                                                                                                               | 1669/<br>DEL/2006         | 20/07/2006     | 254913        | 03/01/2013    |
| A Device for Controlling the On & Off Time of the Metal Oxide Semi Conductor Field Effect Transistor (MOSFET), A Device for Spark Coating the Surfaces of Metal Workpiece Incorporating the said Control Device and a Method of Coating Metal Surfaces using the said Device | 1610/<br>DEL/2005         | 21/06/2005     | 262189        | 05/08/2014    |

## Indian Patents Filed

| Title of Patent                                                                                                                                                                        | Patent Application No. | Date of Filing |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------|----------------|
| A Process for the Preparation of Nanosilver and Nanosilver-Coated Ceramic Powders                                                                                                      | 2786/DEL/2005          | 19/10/2005     |
| Novel Ceramic Materials Having Improved Mechanical Properties and Process for their Preparation                                                                                        | 3396/DEL/2005          | 19/12/2005     |
| An Improved Process for the Preparation of Exfoliated Graphite Separator Plates useful in Fuel Cells, the Plates Prepared by the Process and a Fuel Cell Incorporating the Said Plates | 1206/DEL/2006          | 17/05/2006     |
| An Improved Hydrophilic Membrane useful for Humidification of Gases in Fuel Cells and a Process for its Preparation                                                                    | 1207/DEL/2006          | 17/05/2006     |
| An Improved Process for Preparing Nanotungsten Carbide Powder useful for Fuel Cells                                                                                                    | 81/DEL/2007            | 12/01/2007     |
| A Hydrophilic Membrane Based Humidifier useful for Fuel Cells                                                                                                                          | 95/DEL/2007            | 16/01/2007     |
| Improved Fuel Cell having Enhanced Performance                                                                                                                                         | 606/DEL/2007           | 21/03/2007     |
| An Improved Method for the Generation of Hydrogen from a Metal Borohydride and a Device Therefor                                                                                       | 1106/DEL/2007          | 23/05/2007     |
| Improved Cylindrical Magnetron Cathode and a Process for Depositing Thin Films on Surfaces using the said Cathode                                                                      | 21/DEL/2008            | 03/01/2008     |
| Improved Electrode Membrane Assembly and a Method of Making the Assembly                                                                                                               | 631/DEL/2008           | 13/03/2008     |
| An Improved Catalyst Ink useful for Preparing Gas Diffusion Electrode and an Improved PEM Fuel Cell                                                                                    | 680/DEL/2008           | 18/03/2008     |
| A Process for Continuous Coating Deposition and an Apparatus for Carrying out the Process                                                                                              | 1829/DEL/2008          | 01/08/2008     |
| An Improved Gas Flow Field Plate for use in Polymer Electrolyte Membrane Fuel Cells (PEMFC)                                                                                            | 2339/DEL/2008          | 13/10/2008     |
| Improved Method of Producing Highly Stable Aqueous Nano Titania Suspension                                                                                                             | 730/DEL/2009           | 09/04/2009     |
| Novel Copper Foils having High Hardness and Conductivity and a Pulse Reverse Electrodeposition Method for their Preparation                                                            | 1028/DEL/2009          | 20/05/2009     |
| An Improved Method for Preparing Nickel Electrodeposited having Predetermined Hardness Gradient                                                                                        | 1455/DEL/2009          | 15/07/2009     |
| An Improved Composition for Coating Metallic Surfaces, and a Process for Coating Such Surfaces using the Composition                                                                   | 620/DEL/2010           | 17/03/2010     |
| An Improved Gas and Coolant Flow Field Plate for use in Polymer Electrolyte Membrane Fuel Cells (PEMFC)                                                                                | 1449/DEL/2010          | 22/06/2010     |
| Improved Process for the Preparation of Stable Suspension of Nano Silver Particles having Antibacterial Activity                                                                       | 1835/DEL/2010          | 04/08/2010     |
| Improved Method for Producing Carbon Containing Silica Aerogel Granules                                                                                                                | 2406/DEL/2010          | 08/10/2010     |
| Improved Scratch and Abrasion Resistant Compositions for Coating Plastic Surfaces, a Process for their Preparation and a Process for Coating using the Compositions                    | 2427/DEL/2010          | 12/10/2010     |
| An Improved Method for Producing ZnO Nanorods                                                                                                                                          | 2759/DEL/2010          | 19/11/2010     |



| <b>Title of Patent</b>                                                                                                                                       | <b>Patent Application No.</b> | <b>Date of Filing</b> |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------|-----------------------|
| Improved Process for the Preparation of Bi-Functional Silica Particles useful for Antibacterial and Self Cleaning Surfaces                                   | 3071/DEL/2010                 | 22/12/2010            |
| An Improved Method of Preparing Porous Silicon Compacts                                                                                                      | 912/DEL/2011                  | 31/03/2011            |
| An Improved Process for Preparation of Nanosilver Coated Ceramic Candle Filter                                                                               | 1249/DEL/2011                 | 28/04/2011            |
| An Improved Abrasion Resistant and Hydrophobic Composition for Coating Plastic Surfaces and a Process for its Preparation                                    | 1278/DEL/2011                 | 02/05/2011            |
| An Improved Method for Making Sintered Polycrystalline Transparent Sub-Micron Alumina Article                                                                | 1358/DEL/2011                 | 10/05/2011            |
| An Improved Hybrid Methodology for Producing Composite Multilayered and Graded Coatings by Plasma Spraying Utilizing Powder and Solution Precursor Feedstock | 2965/DEL/2011                 | 17/10/2011            |
| An Improved Composition for Solar Selective Coatings on Metallic Surfaces and a Process for its Preparation and a Process for Coating using the Composition  | 3324/DEL/2011                 | 22/11/ 2011           |
| A Process and a Multi-Piston Hot Press for Producing Powder Metallurgy Component, such as Cerametallic Friction Composite                                    | 3844/DEL/2011                 | 28/12/ 2011           |
| A novel Process for Produced IR Transparent Polycrystalline Alumina Article and the Article so Produced                                                      | 365/DEL/2012                  | 08/02/2012            |
| A Process for Preparing Nanocrystalline Olivine Structure Transition Metal Phosphate Material                                                                | 405/DEL/2012                  | 14/02/2012            |
| An Improved Aqueous Method for Producing Transparent Aluminium Oxy Nitride (ALON) Articles                                                                   | 1408/DEL/2012                 | 08/05/2012            |
| A Device for and A Method of Cooling Fuel Cells                                                                                                              | 1409/DEL/2012                 | 08/05/2012            |
| An Improved Solar Selective Multilayer Coating and a Method of Depositing the Same                                                                           | 1567/DEL/2012                 | 22/05/2012            |
| A Novel Method for the Synthesis of Tungsten Disulphide Nanosheets                                                                                           | 1703/DEL/2012                 | 04/06/2012            |
| Enhanced Thermal Management Systems for Fuel Cell Applications Using Nanofluid Coolant                                                                       | 1745/DEL/2012                 | 07/06/2012            |
| Process for Producing Anti-Reflective Coatings with Scratch Resistance Property                                                                              | 1777/DEL/2012                 | 11/06/2012            |
| Improved Method of Manufacturing Copper-Indium-Gallium Diselenide Thin Films by Laser Treatment                                                              | 2084/DEL/2012                 | 05/07/2012            |
| Electronically and Ionically Conducting Multi-Layer Fuel Cell Electrode and a Method for Making the Same                                                     | 2198/DEL/2012                 | 17/07/2012            |
| Fuel Cell System Equipped with Oxygen Enrichment System Using Magnet                                                                                         | 2985/DEL/2012                 | 25/09/2012            |
| A High Thermal Stable Selective Solar Absorber layer with Low Emissive Barrier Coating over a Substrate and a Process of Producing the Same                  | 3312/DEL/2012                 | 29/10/2012            |
| A Polymer Electrolyte Membrane (PEM) Cell and a Method of Producing Hydrogen from Aqueous Organic Solutions                                                  | 3313/DEL/2012                 | 29/10/2012            |
| A Novel Laser Surface Modification Technique for Hardening Steel                                                                                             | 3312/DEL/2012                 | 29/10/2012            |
| An Improved Test Control System useful For Fuel Cell Stack Monitoring and Controlling                                                                        | 337/DEL/2013                  | 06/02/2013            |

| Title of Patent                                                                                                                                                                         | Patent Application No. | Date of Filing |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------|----------------|
| An Improved Solar Selective Absorber Coating with Excellent Optical Absorptance, Low Thermal Emissivity and Excellent Corrosion Resistance Property and a Process of Producing the Same | 1129/DEL/2013          | 16/04/2013     |
| An Improved Composition for Coating Anodizable Metal Surfaces and a Process of Coating the Same                                                                                         | 1310/DEL/2013          | 03/05/2013     |
| A Method of Preparation of Supported Platinum Nano Particle Catalyst in Tubular Flow Reactor Via Polycol Process                                                                        | 1571/DEL/2013          | 24/05/2013     |
| An Improved Composition for Antireflective Coating with Improved Mechanical Properties and a Process of Coating the Same                                                                | 2330/DEL/2013          | 05/08/2013     |
| Process for Producing Anti-Reflective Coatings With Anti-Fogging (Super Hydrophilic), UV, Weather and Scratch Resistance Properties                                                     | 2919/DEL/2013          | 03/10/2013     |
| An Improved Process for Obtaining a Transparent, Protective Coating on Bi-Aspheric / Plano-Convex Lenses made of Optical Grade Plastics for use in Indirect Ophthalmoscopy              | 3072/DEL/2013          | 17/10/2013     |
| Exfoliated Graphite Separator based Electrolyzer for Hydrogen Generation                                                                                                                | 3073/DEL/2013          | 17/10/2013     |
| A Super Hydrophobic Coating with High Optical Properties having Easy to Clean Property, UV and Corrosion Resistance Properties, a Process of Preparation and Application of the Same    | 402/DEL/2014           | 12/02/2014     |
| High Temperature Polymer Electrolyte Membrane Fuel Cells with Exfoliated Graphite based Bipolar Plates                                                                                  | 494/DEL/2014           | 20/02/2014     |
| Method of Deposition of Double Perovskite of Sr-Fe Niobium Oxide Film on a Substrate by Spray Coating Technique and the Coated Substrate Thereof                                        | 1151/DEL/2014          | 29/04/2014     |
| An Improved Process to Make Coating Compositions for Transparent, UV Blocking on Glass and a Process of Coating the Same                                                                | 1152/DEL/2014          | 29/04/2014     |
| Method of Producing Multifunctional Self Assembled Mixed Phase Titania Spheres                                                                                                          | 3777/DEL/2014          | 19/12/2014     |
| Method of Producing Porous MgF <sub>2</sub> Nanoparticles, Antireflection Coating Suspension and Coatings for Solar Optical UV and IR Transparent Window Applications                   | 4041/DEL/2014          | 31/12/2014     |
| A Novel Electrochemical Method for Manufacturing CIGS Thin Film Containing Nanomesh Like Structure                                                                                      | 426/DEL/2015           | 16/02/2015     |

### International Patents Filed and Granted

| Title of Patent                                                                                                                                                                                                                                                              | Country      | Patent Number | Date of Grant | Date of Filing | Indian Patent Details |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------|---------------|---------------|----------------|-----------------------|
| Process for Forming Coatings on Metallic Bodies and an Apparatus for Carrying out the Process                                                                                                                                                                                | USA          | US6893551B2   | 17/05/2005    | 02/08/2002     | 209817                |
| A Device for Controlling the On & Off Time of the Metal Oxide Semi Conductor Field Effect Transistor (MOSFET), A Device for Spark Coating the Surfaces of Metal Workpiece Incorporating the said Control Device and a Method of Coating Metal Surfaces using the said Device | USA          | US8143550B2   | 27/03/2012    | 20/03/2006     | 1610/DEL/2005         |
| A Process for the Preparation of Nano Silver and Nano Silver-Coated Ceramic Powders                                                                                                                                                                                          | South Africa | 2006/8591     | 30/04/2008    | 13/10/2006     | 2786/DEL/2005         |
|                                                                                                                                                                                                                                                                              | Sri Lanka    | 14258         | 02/11/2011    | 17/10/2006     |                       |
|                                                                                                                                                                                                                                                                              | Indonesia    | P-00200600616 | ---           | 18/10/2006     |                       |
|                                                                                                                                                                                                                                                                              | Bangladesh   | 233/2006      | ---           | 18/10/2006     |                       |

| Title of Patent                                                                                                                                               | Country        | Patent Number    | Date of Grant | Date of Filing | Indian Patent Details |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------|------------------|---------------|----------------|-----------------------|
| A Process for Continuous Coating Deposition and an Apparatus for Carrying out the Process                                                                     | South Africa   | 2009/06786       | 26/05/2010    | 30/09/2009     | 1829/<br>DEL/2008     |
|                                                                                                                                                               | UK             | 2464378          | 15/05/2013    | 02/10/2009     |                       |
|                                                                                                                                                               | USA            | 8486237          | 16/07/2013    | 14/10/2009     |                       |
|                                                                                                                                                               | Japan          | 2009-237921      | 27/12/2013    | 15/10/2009     |                       |
|                                                                                                                                                               | Germany        | DE102009044256.1 | ---           | 15/10/2009     |                       |
|                                                                                                                                                               | France         | 0957102          | ---           | 12/10/2009     |                       |
|                                                                                                                                                               | Brazil         | PI0904232-6      | ---           | 15/10/2009     |                       |
| Improved Process for the Preparation of Stable Suspension of Nano Silver Particles having Antibacterial Activity                                              | United Kingdom | GB2496089        | 18/06/2014    | 19/07/2011     | 1835/<br>DEL/2010     |
|                                                                                                                                                               | Hong Kong      | 13107076.7       | ---           | 18/06/2013     |                       |
| An Improved Hybrid Methodology for Producing Composite, Multilayered and Graded Coatings by Plasma Spraying Utilizing Powder and Solution Precursor Feedstock | South Africa   | 2012/02480       | ---           | 05/04/2012     | 2965/<br>DEL/2011     |
|                                                                                                                                                               | Japan          | 2012-093888      | ---           | 17/04/2012     |                       |
|                                                                                                                                                               | United Kingdom | 1206843          | ---           | 18/04/2012     |                       |
|                                                                                                                                                               | Germany        | 102012218448.1   | ---           | 10/10/2012     |                       |
|                                                                                                                                                               | France         | 1259820          | ---           | 15/10/2012     |                       |
|                                                                                                                                                               | Brazil         | 102120221209     | ---           | 31/08/2012     |                       |
|                                                                                                                                                               | Canada         | 2784395          | ---           | 31/07/2012     |                       |
|                                                                                                                                                               | Canada         | 2784395          | --            | 31/07/2012     |                       |

## Discontinued Indian Patents

| Title                                                                                         | Patent Number with Date of Grant | Remarks                                 |
|-----------------------------------------------------------------------------------------------|----------------------------------|-----------------------------------------|
| A Solar Cooker                                                                                | 184675 -25/05/2001               | Discontinued from 11 <sup>th</sup> Year |
| An Indirect Heated Catalytic Converter for use with Vehicles                                  | 185433-10/08/2001                | Discontinued from 9 <sup>th</sup> Year  |
| A Process for the Preparation of Short Ceramic Fibres                                         | 186751-07/06/2002                | Discontinued from 11 <sup>th</sup> Year |
| A Process of Producing Chemically Treated Expanded Graphite and a Device having Such Graphite | 187654 -05/12/2002               | Discontinued from 11 <sup>th</sup> Year |



## Journal Publications

1. M Prekajski, M Stojmenovic, A Radojkovic, G Brankovic, H Oraon, R. Subasri and B Matovic, "Sintering and Electrical Properties of  $Ce_{1-x}Bi_xO_{2-\delta}$  Solid Solution", *Journal of Alloys and Compounds*, Vol.617, p 563-568, 2014.
2. P. Saravanan, V.T.P. Vinod, M. Cernik, D. Chakravarty, P. Ghosal and S.V. Kamat, "Exchange Coupled Rare-Earth Free Mn-Al/Fe Nanocomposite Magnets by Spark Plasma Sintering", *Materials Letters*, Vol.137, p 369-372, 2014.
3. Y. Krishna Priya, R.N. Bathe, K.V. Rajulapati, K.B.S. Rao and G. Padmanabham, "Fluxless Arc Weld-Brazing of Aluminium Alloy to Steel", *Journal of Materials Processing Technology*, Vol. 214(12),p 2949-2959, 2014.
4. K.S. Hong, Y.J. Cha, M.G. Ha, S. Choi, J.P. Kim, B.S. Lee, E.D. Jeong, H.G. Kim, and P.H. Borse, "Optical Properties and Glass-Forming Region of the  $K_2O-Sm_2O_3-TeO_2$  Glass System", *Journal of the Korean Physical Society*, Vol. 65 (9), p 1453-1456, 2014.
5. G. Sivakumar, R.O. Dusane, S.V. Joshi, "Understanding the Formation of Vertical Cracks in Solution Precursor Plasma Sprayed Ytria-Stabilized Zirconia Coatings", *Journal of the American Ceramic Society*, Vol. 97(11), p 3396-3406, 2014.
6. A.K. Haridas, C.S. Sharma and T.N. Rao, "Electrochemical Performance of Lithium Titanate Submicron Rods Synthesized by Sol-Gel/Electrospinning", *Electroanalysis*, Vol. 26 (11), p 2315-2319, 2014.
7. A. Pareek, P. Paik and P.H. Borse, "Characterization of Nano-Titania Modified CdS/Polysulfide Electrolyte Interface by Utilizing Mott-Schottky and Electrochemical Impedance Spectroscopy", *Electroanalysis*, Vol. 26(11), p 2403-2407, 2014.
8. M. Krishnan, B. Tiwari, S. Seema, N. Kalra, P. Biswas, K. Rajeswari, M.B. Suresh, R. Johnson, N.M. Gokhale, S.R. Iyer, S. Londhe, V. Arora and R.P. Tripathi, "Transparent Magnesium Aluminate Spinel: A Prospective Biomaterial for Esthetic Orthodontic Brackets", *Journal of Materials Science-Materials in Medicine*, Vol. 25 (11), p 2591-2599, 2014.
9. S.R. Dhage, M. Tak and S.V. Joshi, "Fabrication of CIGS Thin Film Absorber by Laser Treatment of Pre-Deposited Nano-Ink Precursor Layer", *Materials Letters*, Vol. 134, p 302-305, 2014.
10. R. Bathe, V.S. Krishna, S.K. Nikumb and G. Padmanabham, "Laser Surface Texturing of Gray Cast Iron for Improving Tribological Behavior", *Applied Physics A-Materials Science & Processing*, Vol. 117(1), p 117-123, 2014.
11. Lohia, G. Sivakumar, M. Ramakrishna and S. V. Joshi, "Deposition of Nanocomposite Coatings Employing a Hybrid APS Plus SPPS Technique", *Journal of Thermal Spray Technology*, Vol. 23(7), p 1054-1064, 2014.
12. P. Sai Pramod, K. Valleti, G. Ravi Chandra, R. V. Koteswararao, M. Ramakrishna, K. Suresh and S. V. Joshi, "Effect of Microstructure and Phase Constitution on Mechanical Properties of  $Ti_{1-x}Al_xN$  Coatings", *Applied Surface Science*, Vol. 313, p 936-946, 2014.
13. R. Kumar, S. Anandan, K. Hembram, and T. N. Rao, "Efficient ZnO-Based Visible-Light-Driven Photocatalyst for Antibacterial Applications", *ACS Applied Materials & Interfaces*, Vol. 6 (15), p 13138-13148, 2014.
14. M. S. Archana, G. Ravi Chandra, Y. S. Rao, V.V.S.S. Srikanth, S.V. Joshi and J. Joardar, "Rapid Consolidation of FeAl- $Fe_3AlC_x$  Ultrafine Composites by Mechanically Activated Field-Assisted Technique", *Materials Science and Engineering A-Structural Materials Properties Microstructure and Processing*, Vol. 611, p 298-305, 2014.
15. P. Jayaraj, P. Karthika, N. Rajalakshmi and K. S. Dhathathreyan, "Mitigation Studies of Sulfur Contaminated Electrodes for PEMFC", *International Journal of Hydrogen Energy*, Vol. 39 (23), p 12045-12051, 2014.
16. Y.J. Cha, J.S. Bae, T.E. Hong, J.H. Yoon, E.H. Chung, E.D. Jeong, H.G. Kim, P.H. Borse, and K.T. Lim, "Structural, Optical and Visible-Light Photocatalytic Properties of  $Sr_3FeNb_2O_9$  Oxide", *Journal of the Korean Physical Society*, Vol. 65(4), p 520-525, 2014.
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20. H. Kumar, K. V. P. Prabhakar, S. Sam, S. K. Albert, G. Padmanabham, A.K. Bhaduri, T. Jayakumar and E.R. Kumar, "Development of Laser Welding Process for Reduced Activation Ferritic Martensitic Steel for Indian Test Blanket Module Fusion Science and Technology", Vol. 66 (1), p 192-199, 2014.
21. R. Papitha, M. B. Suresh, R. Johnson and D. Dibakar, "High-Temperature Flexural Strength and Thermal Stability of Near Zero Expanding doped Aluminum Titanate Ceramics for Diesel Particulate Filters Applications", International Journal of Applied Ceramic Technology, Vol. 11(4), p 773-782, 2014.
22. J.S. Jang, P.H. Pramod, J.S. Lee, K.T. Lim, C.R. Cho, E.D. Jeong, M.G. Ha, M.S. Won and H.G. Kim, "Photocatalytic Performance of Nanocrystalline  $\text{Bi}_5\text{Ti}_3\text{FeO}_{15}$  Layered Perovskite Under Visible Light (vol 10, pg 5008, 2011)", Journal of Nanoscience and Nanotechnology, Vol. 14 (7), p 5596-5596, 2014.
23. N. K. Tewary, B. Syed, S. K. Ghosh, S. Kundu, S. M. and G. Padmanabham, "Microstructural Evolution and Mechanical Behaviour of Surface Hardened Low Carbon Hot Rolled Steel", Materials Science and Engineering A-Structural Materials Properties Microstructure and Processing, Vol. 606, p 58-67, 2014.
24. L. Rama Krishna and G. Sundararajan, "Aqueous Corrosion Behavior of Micro Arc Oxidation (MAO)-Coated Magnesium Alloys: A Critical Review", JOM, Vol. 66(6), p 1045-1060, 2014.
25. G. V. Ramana, B. Padya, V. V. S. Srikanth and P. K. Jain, "Rapid Mixing Chemical Oxidative Polymerization: An Easy Route to Prepare PANI Coated Small-Diameter CNTs/PANI Nanofibres Composite Thin Film", Bulletin of Materials Science, Vol. 37(3), p 585-588, 2014.
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27. P. Ramavath, P. Biswas, K. Rajeswari, M. B. Suresh, R. Johnson, G. Padmanabham, C.S. Kumbhar, T.K. Chongdar, N.M. Gokhale, "Optical and Mechanical Properties of Compaction and Slip Cast Processed Transparent Polycrystalline Spinel Ceramics", Ceramics International, Vol. 40(4), p 5575-5581, 2014.
28. A. Bhaskar, M. Deepa, M. Ramakrishna and T. N. Rao, "Poly (3,4-ethylenedioxythiophene) Sheath over a  $\text{SnO}_2$  Hollow Spheres/Graphene Oxide Hybrid for a Durable Anode in Li-Ion Batteries", Journal of Physical Chemistry C, Vol. 118(14), p 7296-7306, 2014.
29. G. Telasang, J.D. Majumdar, G. Padmanabham and I. Manna, "Structure-Property Correlation in Laser Surface Treated AISI H13 Tool Steel for Improved Mechanical Properties", Materials Science and Engineering A-Structural Materials Properties Microstructure and Processing, Vol. 599, p 255-267, 2014.
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33. S. Anandan, T. N. Rao, R. Gopalan and Y. Ikuma, "Fabrication of Visible-Light-Driven N-Doped Ordered Mesoporous  $\text{TiO}_2$  Photocatalysts and their Photocatalytic Applications", Journal of Nanoscience and Nanotechnology, Vol. 14(4), p 3181-3186, 2014.
34. P. Kathirvel, J. Chandrasekaran, D. Manoharan and S. Kumar, "Preparation and Characterization of Alpha Alumina Nanoparticles by In-Flight Oxidation of Flame Synthesis", Journal of Alloys and Compounds, Vol. 590, p 341-345, 2014.
35. A.Pareek, R. Purbia, P. Pradip, N. Y. Hebalkar, H. G. Kim and P. H. Borse, "Stabilizing Effect in Nano-Titania Functionalized CdS Photoanode for Sustained Hydrogen Generation", International Journal of Hydrogen Energy, Vol. 39 (9), p 4170-4180, 2014.
36. R. Bathe, A. K. Singh and G. Padmanabham, "Effect of Pulsed Laser Dressing of Metal-Bonded Diamond Wheels on Cutting Performance", Materials and Manufacturing Processes, Vol. 29(3), p 386-389, 2014.
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- Opportunities (A Review)", *Renewable & Sustainable Energy Reviews*, Vol. 31, p 221-257, 2014.
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  40. M. Sreekanth, B. V. Sarada, S. R. Dey and S. V. Joshi, "CuIn<sub>1-x</sub>Ga<sub>x</sub>Se<sub>2</sub> Thin-Film Absorber Layers for Solar Photovoltaics Fabricated by Two-Stage Pulsed Current Electrodeposition", *Materials Letters*, Vol. 118, p 158-160, 2014.
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## Awards and Honours

1. Dr. Sanjay Bhardwaj was inducted as a 'Fellow' of the Society of Technology Management (STEM) at the 'STEM Annual Summit' held in Hyderabad during August 19-20, 2014.
2. Dr. G. Sivakumar was awarded Best Ph.D. Thesis under "Innovative Student Project", by Indian National Academy of Engineering (INAE) at Birla Institute of Scientific Research, Jaipur on December 12, 2014.
3. Dr. R. Subasri was awarded the MRSI Medal 2015, in recognition of her contribution to the field of Materials Science and Engineering by Materials Research Society of India at University of Rajasthan, Jaipur on February 11, 2015.
4. Mr. G. Vijay Dev (Dr. K.S. Dhathathreyan) received the third prize for the 'Best Paper Presentation' at the 'Select X5 Meet' held at CECRI, Karaikudi during February 26-27, 2015.
5. Mr. M. Sreekanth (Dr. B. V. Sarada) received the 'National Level Padarth 3MT (3 Minute Thesis) Presentation Competition in PG category' held at IIT Bombay, Mumbai on March 15, 2015.
6. Mr. M. Sreekanth (Dr. B. V. Sarada) received the 'Excellence in Research Award 2014' for the academic year 2014-15 at IIT, Hyderabad on March 25, 2015.

## Books Chapters

1. A chapter on "Polymer Nanocomposites: Emerging Growth Driver for the Global Automotive Industry" authored by Vivek Patel and Y. R. Mahajan in the 'Handbook of Polymer Nanocomposites Processing, Performance and Application', Volume A: Layered Silicates', (eds.) J.K. Pandey, K.R. Reddy, A.K.Mohanty and M.Misra., Springer Verlag, p 511-538, 2014.
2. A chapter on "Sol-Gel Nanocomposite Hard Coatings" authored by K.R.C. Soma Raju and R. Subasri in the 'Handbook on Anti-abrasive Nanocoatings: Current and Future applications' (ed.) M. Aliofkhazraei, Woodhead Publishers, UK., p 105-136, 2015.
3. A chapter on "Improving Corrosion Resistance of Metals/Alloys using Hybrid Nanocomposite Coatings Synthesized through Sol-Gel Processing" authored R. Subasri in the book on 'Comprehensive Guide for Nanocoatings Technology: Properties and Development', (ed.) M. Aliofkhazraei, Nova Science Publishers Inc., New York, USA. Vol. 3, 2015.



Dr. Sanjay Bhardwaj making a presentation during STEM Annual Summit 2014



Dr. G Sivakumar being awarded Best Ph.D Thesis under 'Innovative Student Project'

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