

## Electron microscopy\*

An international conference on electron microscopy (EMSI-2016) was held recently, coinciding with the year-long centennial celebration of the Banaras Hindu University (BHU), founded by Pt. Madan Mohan Malaviya in 1916. It is well known that BHU has contributed immensely to electron microscopy research since 1963 and has produced many renowned electron microscopists since then.

This conference was aimed at bringing together a large number of distinguished researchers, microscopists and technical experts from across the globe to discuss the latest advancements on the characterization of materials at the atomic level. For the benefit of the participants and young researchers, three pre-conference workshops were organized (30 May–1 June 2016) on: (i) Electron microscopy in materials science, (ii) Electron microscopy in biological sciences and (iii) Electron probe micro analyzer (EPMA). Nearly 100 participants, mostly young researchers and doctoral students from all over the country attended the workshops. Subsequent to the workshops, more than 300 participants from various countries attended the conference on electron microscopy. There were 14 plenary lectures by experts drawn from India and abroad, and 121 oral presentations of which 54 were invited lectures and the others contributory. Poster presentation sessions consisting of 95 papers and metallography contests with more than 30 entries were also conducted. Twenty technical sessions were planned over three days covering subjects such as nanomaterials, steel, nonferrous and complex metallic alloys as well as various techniques such as SEM-FIB, EBSD, electron diffraction, HREM, atom probe tomography, etc. Some sessions were also devoted to bio-

logical as well as earth and planetary sciences. Details of the conference are available at the following website: [www.emsi2016iitbhu.org](http://www.emsi2016iitbhu.org).

The two aspects emphasized during this conference were: (i) recent developments in the microscopy techniques, including instrumentation in terms of hardware and software, as well as data analysis and image processing, and (ii) applications of these techniques for scientific research and developments in materials science and biological sciences. Electron microscopy techniques are indeed essential to understand the submicron details of the structures, their chemical composition, atomic arrangements and other crystallographic details with very high spatial resolution. The conference, therefore, intended to cover several aspects of electron microscopy and also to provide glimpses of other complementary techniques. A technical exhibition, which is always an important part of these annual meetings and conferences, was organized to showcase the latest developments in terms of various electron microscopy techniques. In the following, some of the important lectures and discussions leading to issues emerging from this conference are highlighted.

In the inaugural session which included the award ceremony, R. K. Mandal (IIT-BHU, Varanasi), welcomed the delegates and urged them to engage in meaningful discussions during the conference. M. Vijayalakshmi (Kalpakkam) briefed about the theme of the conference and highlighted the importance of the meeting at Varanasi. G. C. Tripathi (BHU) expressed assurance for the successful growth of electron microscopy research in BHU. In the inaugural session, G. V. S. Sastry (IIT-BHU) elaborated the role of electron microscopy in the advancement of knowledge. He also shared his experience about the near-discovery of quasicrystalline phase in Al-Pd and Al-Mn-Ni in the early seventies along with G. Van Tendeloo (Belgium) during his visit to BHU<sup>1</sup>. The Chief Guest, R. Chidambaram (Principal Scientific Adviser to the Government of India (GoI)) in his inaugural address discussed about the importance of sophi-

sticated techniques such as electron microscopy for materials research. He pointed out the importance of new variants of microscopes in solving some of the challenging problems in biological sciences. Indigenous technology for manufacturing electron microscopes in the country was emphasized according to the mandate for technology development set out by the GoI. He stressed how the basic and deliverable research should be developed using advanced characterization techniques. He also suggested involving many more scientists from biological sciences to exploit these techniques. N. K. Mukhopadhyay (IIT-BHU) mentioned with examples how technology drives science<sup>2</sup> and quoted from an editorial by P. Balaram<sup>3</sup>, that 'Seeing is believing', which is highly relevant to the present context. He pointed out that the credit for discovery of several novel materials and advancement of our knowledge should be given to the discipline of 'Characterization science and technology'.

It is important to mention that the Electron Microscope Society of India (EMSI) was established on 2 December 1961 at the Saha Institute of Nuclear Physics, Kolkata by the distinguished scientist S. N. Bose. The Society has been continuing its affiliation with the International Federation of Electron Microscope Societies (now renamed as International Federation of Societies of Microscopy (IFSM)) since 1962. It is relevant to note that during 1946–48, N. N. Dasgupta, founder president of EMSI and student of C. V. Raman, led a team of scientists to construct a horizontal electron microscope for the first time for observation of biological sample. Unfortunately, no such initiatives have been taken to continue the effort of constructing electron microscopes in our country. However, to recognize this important and encouraging contribution, the Society established a Memorial Lecture to be delivered in its Annual Meeting. Accordingly every year, a renowned scientist is invited to deliver the Prof. N. N. Dasgupta Memorial Lecture. This year, Barry Carter (USA) delivered the lecture. He elaborated upon new techniques in transmission electron microscopy (TEM),

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including operando microscopy, tomography, orientation imaging, cryo-TEM, at as low as 4 K temperature (which is a significant development for microscopy for biological sciences)<sup>4,5</sup>. It was noted that temperature can be controlled and modified by the advanced instrumentation technique. Computer modelling and handling big data were pointed out to be a major challenge which is required to be solved in the near future. Van Tendeloo talked about the electron microscopy of soft materials using aberration-corrected instrumentation, monochromators, better X-ray and electron detectors. He demonstrated the results on beam-sensitive material, where the specimen could withstand very low electron beams for a few seconds. He showed successful imaging of light elements in Li-based batteries and embedding of Pt nanoparticles in metal organic frameworks<sup>6,7</sup>.

J. W. Steeds (UK) discussed the earlier work as well as advances in convergent beam electron diffraction (CBED) techniques for extracting crystallographic information such as point group, space group, Bravais lattice and lattice parameter determination of metals, ceramics, semiconductors and insulators, and phase identification in complex materials. He presented some results on coherent CBED and precision diffraction. He also pointed out that the Bloch wave approach to electron diffraction was effective in explaining the influence of dynamical diffraction. G. K. Dey (BARC, Mumbai) dealt with the effects of extreme environments on Zr-based materials, characterizing them through electron microscopy. The transformation occurring under pressure, thermal activation and also irradiation was identified through detailed electron microscopy techniques. This study essentially revealed the formation of omega phase under different activation conditions, including the ones in extreme conditions in pure Zr, Zr-based alloys probed through electron microscopy. Omega phase transformation is known to be one of the intriguing phase transformations in specially Ti- and Zr-based alloys.

Martina Luysberg (Germany) elaborated the technique of advanced (chromatic) aberration-corrected microscopy in materials science. She mentioned that aberration correction in TEM in conjunction with spectroscopic methods made it possible to measure the structure, composition and bonding properties on an

atomic scale. It was realized that the adjustment of chromatic aberrations for HREM at low voltages and energy selective imaging could open up a new direction in the study of the interface between scandates and titanates. In addition to the complex oxide interfaces, experimental observation of low-voltage high-resolution and energy-filtered images of thin layers and nanoparticles was presented from a newly built chromatic aberration-corrected microscope, designated as PICO. Emine Korkmaz (The Netherlands) presented 3D isotropic volume imaging and reconstruction of large tissue volumes in biological samples. It was shown that visualizing the three-dimensional architecture of cells and tissues was essential for understanding the relationships between structure and function in biological systems. Y. Kondo (Japan) demonstrated how the HREM images of SrTiO<sub>3</sub> could reveal Sr, Ti + O, O columns using analytical aberration-corrected microscopes with cold field emission gun (CEFG), advanced corrector, and double-detector analytical system. It was claimed to open up the possibilities of innovations in cutting-edge technologies in electron microscopy. Y. Zhou (Germany) dealt with the advances in ion beam microscopy such as focused ion beam (FIB) microscope. It was demonstrated that using neon and helium ion beams, it is possible to create delicate sub-10 nm structures requiring high machining fidelity.

V. K. Vasudevan (USA) advocated the effect on grain boundary engineering utilizing thermomechanical processing with the iterative cycles of cold working and strain annealing of metallic alloys. A clear correlation and mechanistic understanding relating grain boundary character, sensitization, carbide precipitation and susceptibility to corrosion and stress corrosion cracking was established using TEM, SEM, precision electron diffraction and electron backscatter diffraction mapping. Somnath Dasgupta (Delhi) discussed the multiple uses of EPMA in earth science, especially in petrology (i.e. study of rocks). It was successfully demonstrated that the EPMA study for geochronological aspects (e.g. chemical dating) is useful. It was recognized that many minerals were chemically zoned with respect to both major and minor (including trace) elements. Hence, it was possible to trace the pathways of evolution of rocks using experimentally determined diffusion parameters for sig-

nificant number of elements present in the minerals. K. Muraleedharan (CGCRI, Kolkata) illustrated how analytical electron microscopy techniques, diffraction and spectrometry were employed in characterizing fine-scale microstructure and for finding the correlation between processing, structure and properties of a wide range of materials such as glass, ceramics and composites including nanomaterials. He shared examples of development of materials from his own research.

R. Tandon (Univeristy of Delhi) elaborated on how discovery of electron microscopy led to the discovery of new and exotic materials. He shared his experience in the areas of electroceramics, sensors, conducting polymers, carbon nanotubes and other exotic materials. O. N. Srivastava (BHU) discussed the contribution of BHU since 1963 on electron microscopy research initiated by A. R. Verma in physics and T. R. Anantharaman in metallurgy. This significantly contributed to materials research at BHU with special reference to polytypes, metastable alloy phases, quasicrystals, metallic glasses and nanomaterials. Satyam Suwas (IISc, Bengaluru) dealt with the development of orientation imaging microscopy for understanding the micromechanisms operating during materials processing and materials performance. This technique based on the Kikuchi lines formed by the electron backscatter diffraction in SEM and the Kikuchi lines/diffraction spots in TEM, was effectively utilized to explain the mechanical behaviour of thermomechanically processed samples and also the evolution of microstructures in the nanocrystalline materials.

In conclusion, after three days of deliberation, it was realized that the issues related to aberration-corrected microscopy, operando microscopy (i.e. *in situ* microscopy), cryo-microscopy, atom probe tomography, FIB, high-resolution analytical microscopy among others were central to the discussions among the participants and experts. However, the cost of equipment is escalating continuously and may not be affordable by all the groups engaged in materials research. For the benefit of science and technology in the country, central and national facilities could be created by the Government as well as corporate sector so that researchers in the country can make use of the same at affordable charges. It also

emerged that our training on electron microscopy should be strengthened through several workshops and tutorials to be organized frequently by EMSI or other organizations in association with it, with emphasis on imparting education of electron microscopy, including skill development. The area of computation and simulation of electron microscopic images and diffraction patterns should be developed in a big way in order to reap the benefits of the advanced experimental techniques of electron microscopy. The indigenous technology for manufacturing of electron microscopes as well as the accessories should be given serious consideration and priority, so that dependence on foreign technology can gradually be reduced, as pointed out by Chidambaram in his inaugural address. It is also relevant to mention that in recog-

nition of the electron microscopy research by Indian scientists and researchers, the International Committee on Asia Pacific Society for Microscopy responded to the request of EMSI to organize the Asia Pacific Conference on Electron Microscopy in the year 2020 at Hyderabad. All the delegates were invited to the next conference to be organized by EMSI and IGCAR, Kalpakkam (EMSI-2017) in Mahabalipuram, Tamil Nadu in July 2017. Those interested may visit the EMSI website ([www.emsi.org.in](http://www.emsi.org.in)) for updates on the forthcoming conferences and workshops related to electron microscopy.

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