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LABORATORY FOCUS

Here we begin a new feature for EMAS News. An opportunity to describe your own laboratory, its equipment, applications, and even, if you so wish, the staff who work there. This is not intended to be an advertisement of a facility's services but a reader-focussed article to describe how things work in the real world of our respective laboratories. EMAS retains the right for editorial control of the article that will be finalised with the authors before publication. Any member interested in making such a contribution please contact the editor.

THE INTERNATIONAL CENTRE OF ELECTRON MICROSCOPY FOR MATERIALS SCIENCE (IC-EM) AT THE AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY IN KRAKOW

The International Centre of Electron Microscopy for Materials Science (IC-EM; www.tem.agh.edu.pl) headed by Prof. Aleksandra Czyrska-Filemonowicz, continues a more than 50 years long tradition of electron microscopy at the AGH University of Science and Technology in Krakow. The Centre was created on 1st June 2010 as a non-faculty unit, acting in co-operation with foreign partners. The lead unit of the Centre at the AGH-UST is the Faculty of Metals Engineering and Industrial Computer Science, whereas the lead foreign partner is the Forschungszentrum Jülich in Germany.

The main activity of the IC-EM is the application and tuning of new methods of electron microscopy to materials investigation, mainly to quantitative characterisation of the micro-and nanostructure of innovative materials in order to achieve the desired properties. The main groups of materials investigated at IC-EM are materials for energy systems, aeronautics, graded and multilayered materials, biomaterials, nanomaterials, structural and functional materials.

The IC-EM team comprises 10 permanent researchers, 1 (permanent) visiting professor, 2 technicians, and several PhD students (Fig. 1). Their field of expertise covers quantitative characterisation of micro/nanostructure and properties of engineering materials, including structural defect analyses and phase identification in multiphase and multilayered materials down to the nanoscale (even atomic) scale by advanced electron microscopy, electron tomography and electron holography. The IC-EM staff is developing methods for nanophase identification and mapping by coupling fast EDS spectrometry and precession electron diffraction (PED) in the Cs-corrected STEM as well as STEM-EDX tomography.



Figure 1. The team of the International Centre of Electron Microscopy for Materials Science (IC-EM) at the AGH University of Science and Technology in Krakow during installation of new microscopes in 2011.

The Centre has developed its infrastructure to upgrade the expertise in several advanced techniques. The IC-EM is equipped with 4 transmission electron microscopes (TEM) and 2 scanning electron microscopes (SEM). In particular, FEI's 3rd ultimate performance analytical electron microscope, a Titan Cubed G2 60-300 for high-resolution analytical microscopy at high (300 kV) and low (60 kV) energy was installed in 2011. This unique microscope is equipped with a ChemiSTEM system consisting of the new X-FEG Schottky high brightness source with a monochromator, a high resolution STEM-HAADF unit with the new dodecapole DCOR probe Cs-corrector, and the new EDS microanalysis system based on 4 windowless silicon drift detector (SDD) technology with enhanced acquisition efficiency (0.7 sr) and speed for low dose, high spatial (atomic) resolution and fast chemical mapping. Other unique equipment on the Titan Cubed G2 60-300 microscope includes EELS GIF Quantum 693 spectrometer for edge shifts/fine structure (using monochromator and probe Cs-corrector), the new FEI PED precession electron diffraction, dual-axis tomography holder, Lorentz lens, rotatable bi-prism for off-axis electron holography and TARO for full remote access operation. Fig. 2 shows STEM-EDS elemental maps of CMSX-4 single crystal superalloy used for aero engine turbine blades acquired with Titan Cubed G2 60-300 with ChemiSTEM technology. Fig. 3a shows HRSTEM-HAADF image of the γ - γ'' interface, while Fig. 3b HRSTEM-EDX are elemental maps of Ni, Mo and Nb in the γ'' phase precipitated in Inconel 718 nickel-base superalloy.

The second transmission electron microscope installed in 2011 is Tecnai G20 Twin with LaB6 cathode, equipped with DigiStar precession diffraction and ASTAR for orientation and phase mapping as well as STEM-HAADF and EDS microanalysis system (TIA/EDAX).

Other two TEMs – JEOL JEM-2010ARP and JEM-200CX – are the workhorse microscopes for defect analysis and intermediate resolution phase identification for engineering materials.

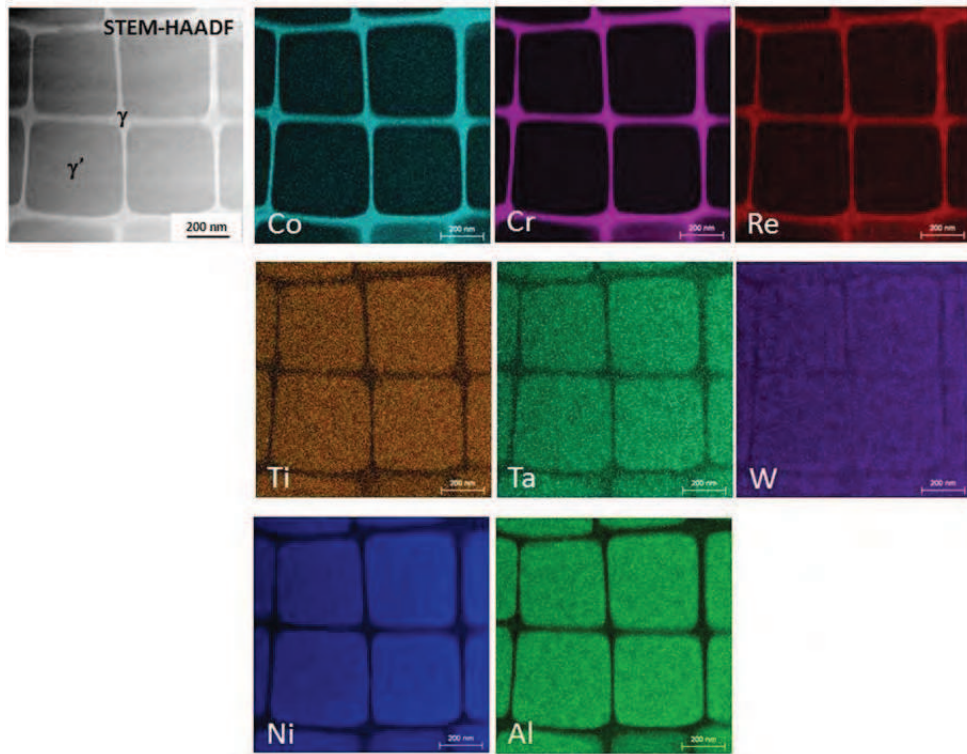


Figure 2. HRSTEM-EDS elemental maps of CMSX-4 single crystal superalloy used for aero engine turbine blades acquired with Titan Cubed G2 60-300 with ChemiSTEM technology.

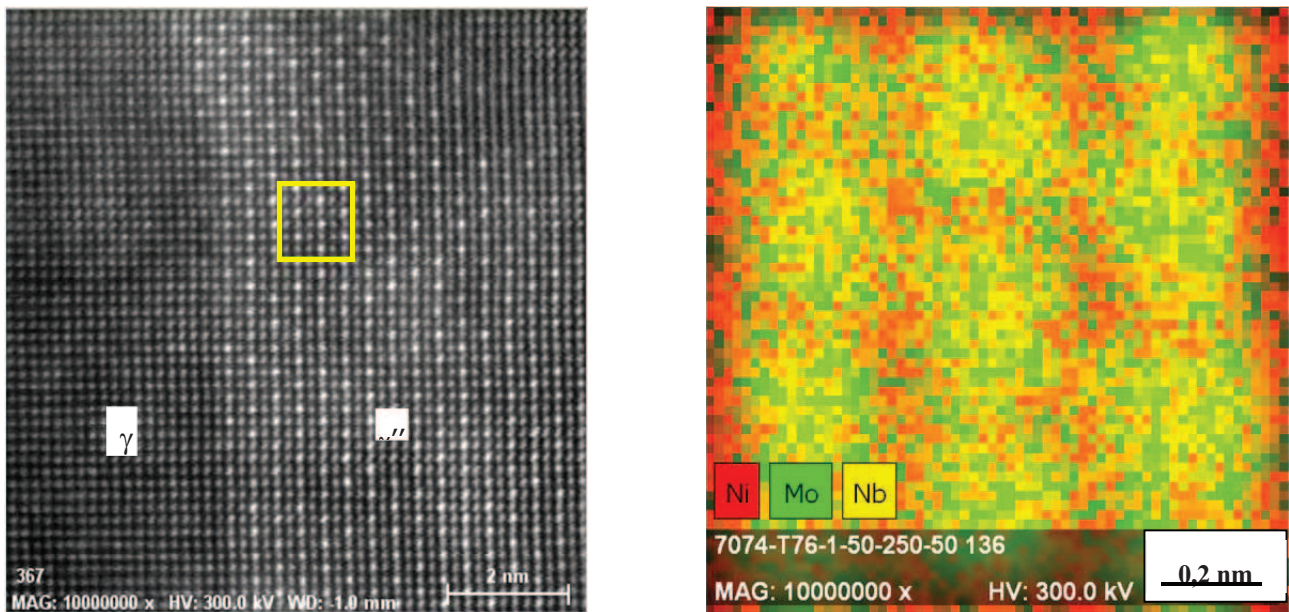


Figure 3. a) HRSTEM-HAADF image of the γ - γ'' interface in Inconel 718 nickel-base superalloy for aerospace application. b) HRSTEM-EDS elemental maps of Ni, Mo and Nb in the γ'' -phase precipitated in Inconel 718 nickel-base superalloy. The image shows the enlarged scanned area marked by a yellow square in Figure 3a.

The Centre possesses two SEM microscopes. Installed in 2009 the FIB-SEM dual beam NEON 40 EsB CrossBeam of Zeiss with EDS microanalysis system Quantax 200 (30 mm² SDD) of Bruker system, FEG SEM column, SE and BSE modes is used for SEM investigation as well as TEM sample preparation and FIB-SEM tomography. Recently, in the summer of 2012, the IC-EM launched a new SEM, a Merlin Gemini II equipped besides several detectors, with FEG, EDS microanalysis system Quantax 800, and EBSD Quantax CrystAlign 400.

The remaining scientific facilities of the Centre include a scanning probe microscope Dimension 3100 SPM, advanced light microscopes Axio Imager M1m and Stereo Discovery of Zeiss, computer-aided image analysis laboratory with several programmes and mechanical properties testing equipment.

The comprehensive TEM sample preparation laboratory is mainly dedicated to metals, alloys, ceramics, composites and coatings. The laboratory is equipped with two low temperature electropolishing Tenupols of Struers, two room and LN2 temperature PIPS (Gatan) ion mills with low voltage (100 V) and CCD camera for careful final polishing, as well as the new ultra-low energy ion beam mill NanoMill 1040 of Fischione for post-processing of FIB lamellae for ultra-thin, clean samples required for advanced TEM analyses.

The IC-EM team is very active in collaboration with research and industrial partners. The team was involved in more than 150 scientific projects. At present, the IC-EM acts as a TEM training site for Central and Eastern Europe and collaborates with many research partners. Within ESTEEM2 project (<http://esteem2.eu>), the IC-EM provides the transnational access to its facilities for scientists who do not have such modern research instruments and infrastructure.

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Krakow, December 2012

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