Group of Microscopy and Spectroscopy of Surfaces

Department of Electron Microscopy

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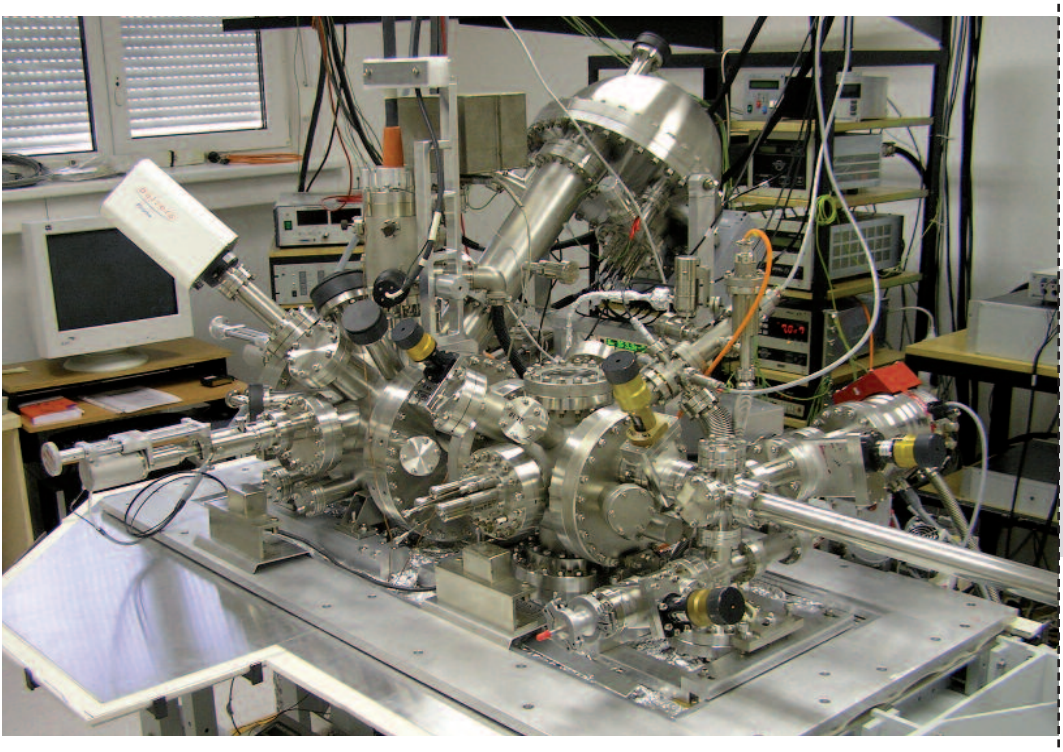


Ultrahigh vacuum apparatus for scanning microscopy with very slow electrons and for Auger electron spectromicroscopy of atomically clean or in-situ prepared surfaces

THEMATIC RESEARCH FOCUS

Research area

– Low energy scanning electron microscopy, Auger electron spectroscopy and spectromicroscopy, surface physics
– Micro- and nanostructure of advanced materials, thin films

Excellence

Contrast formation at low and very low energies in the scanning electron microscopy (SEM) both in the reflection and transmission mode with lateral resolution of units of nm, with an application to the study of advanced materials and biostructures

Mission

Development of advanced methods of scanning electron microscopy and their application in materials and biomedical sciences and technologies

DEVELOPED TECHNOLOGIES

Content of research

– Methodology for the formation of beams of very slow electrons and their manipulation aimed at the illumination of solid surfaces or free-standing films, including vortex electron beams

– Theory of interaction of slow electrons with matter, generation of signals released under impact of electrons, analysis of information carried by species emitted under electron bombardment
– Detection of electrons emitted from surfaces or transmitted through films, including multichannel detection of angular and energy distribution of emitted electrons in SEM, aiming at ultimate angular, energy and lateral resolution
– Interpretation of scanned electron beam micrographs and Auger electron spectromicrographs
– Quantitative scanning electron microscopy, especially at low energies

Main capabilities

Basic research

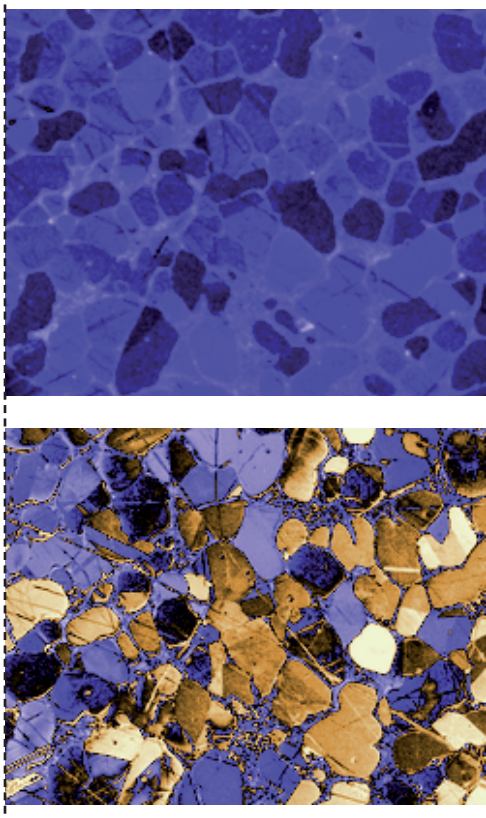
– Interaction of slow electrons with matter
– Reflectance and transmittance of slow and very slow electrons
– Generation of Auger electrons
– Theory of contrast mechanisms in scanning electron microscopy
– Relations between the state of surface and its response to electron bombardment
– Principles of detection of low energy electrons
– 3D distribution of electromagnetic fields and motion of charged particles in them

Applied research

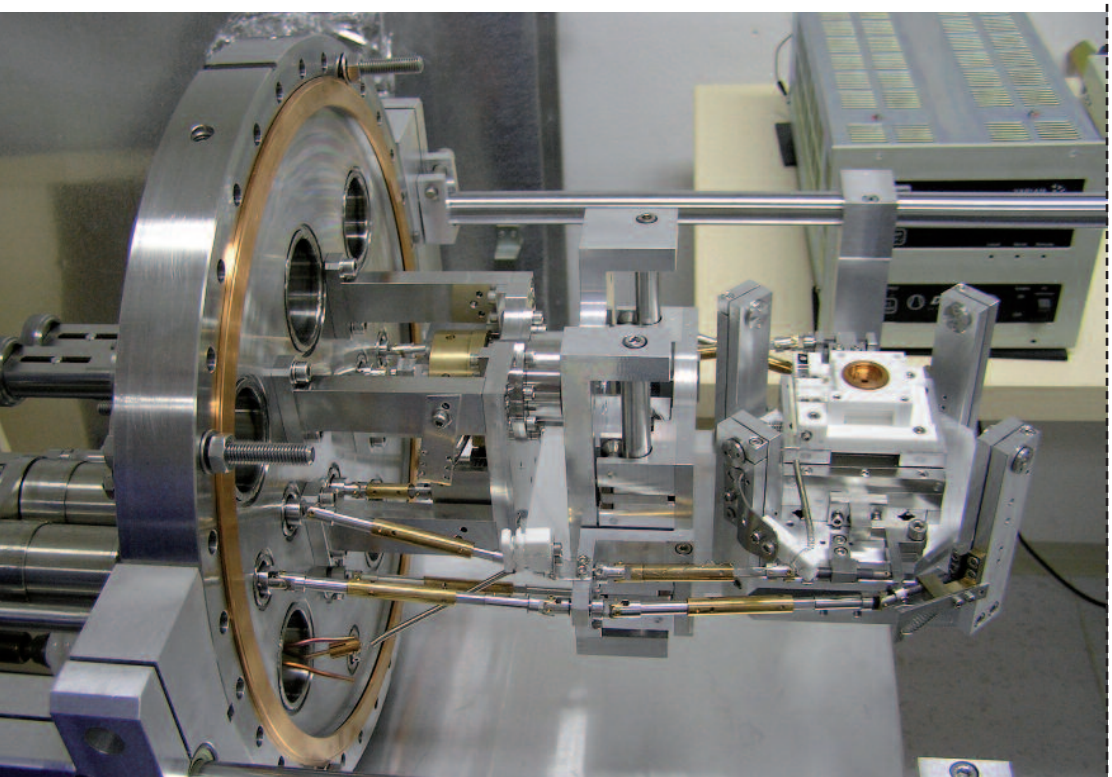
– Ultrahigh vacuum scanning low energy electron microscopy
– Analysis of phases in complex materials, e.g. steels
– Analysis of surface coatings and thin films, Auger electron spectromicroscopy
– Analysis of ultrathin tissue sections and free-standing films or 2D crystals
– Simulation of electron trajectories in electron optical elements and systems
– Design of multichannel electron detectors

Fields of research results application

– Materials science (micro- and nanostructure of materials)
– Life sciences (ultrathin tissue sections, biological crystals)
– Nanotechnologies
– Industry of scientific instrumentation
– Metallurgy
– Industry of polymers, composites, surface coatings, etc.
– Medicine



Structure of X210Cr12 stainless steel with the grain contrast enhanced due to a decrease of the incident electron energy: original micrograph taken at 6 keV (top), and at 500 eV using the cathode lens with a biased sample (bottom); pseudo-color images



Specimen stage of the ultrahigh vacuum scanning low energy electron microscope

KEY RESEARCH EQUIPMENT

- Ultrahigh vacuum scanning low energy electron microscope of in-house design consisting of the observation and preparation chambers, equipped with in-situ technologies for specimen treatment, namely ion beam cleaning, heating and deposition of thin films, and auxiliary techniques, namely Auger electron spectrometry and mass spectroscopy of released gases
- Attachments for several commercial scanning electron microscopes allowing sample observation with low and very low energy electrons
- Equipment for the preparation of clean, smooth or coated specimens

ACHIEVEMENTS

Study of transmissivity of ultrathin free standing foils at very low energies in scanning electron microscope with a high contrast and a high lateral resolution in the nm scale.

- Müllerová, I.; Hovorka, M.; Frank, F.; *A method of imaging ultrathin foils with very low energy electrons.* Ultramicroscopy 119, (2012), p. 7881

Characterisation of crystal orientation with a high lateral resolution from the reflectivity of electrons at impact electron energies between 50 eV and 1 eV. The information is proportional to the local density of electron states.

- Pokorná, Z.; Mikmeková, Š.; Müllerová, I.; Frank, L.; *Characterization of the local crystallinity via reflectance of very slow electrons.* Appl. Phys. Lett. 100, (2012), 261602, p. 1–4

Applications of low energy SEM in nanotechnology.

- Müllerová, I.; Hovorka, M.; Mika, F.; Mikmeková, E.; Mikmeková, Š.; Pokorná, Z.; Frank, L.; *Very low energy scanning electron microscopy in nanotechnology.* International Journal of Nanotechnology 9, (2012), p. 695–716

Development of new highly sensitive method for the determination of crystallographic orientation of grains from maximum anisotropy of reflected electrons. Tiny changes in the lattice constant caused by local deformations are made visible. The method has a very high sensitivity to the local 3D inner potential.

- Mikmeková, Š.; Hovorka, M.; Müllerová, I.; Man, O.; Pantělejev, L.; Frank, L.; *Grain contrast imaging in UHV SLEEM.* Materials Trans. 51, (2010), p. 292–296

A method for quantitative measurements of dopant level in semiconductors using the secondary electron yield at optimum primary beam energy.

- Frank, L.; Müllerová, I.; Valdaitsev, D.; Gloskovskii, A.; Nepijko, S.; Elmers, H.; Schönhense, G.; *The origin of contrast in the imaging of doped areas in silicon by slow electrons.* J. Appl. Phys. 100, (2006), 093712, p. 1–5
- Müllerová, I.; El Gomati, M. M.; Frank, L.; *Imaging of the boron doping in silicon using low energy SEM.* Ultramicroscopy 93, (2002), p. 223–243

An overview about the development of the scanning low energy electron microscopy.

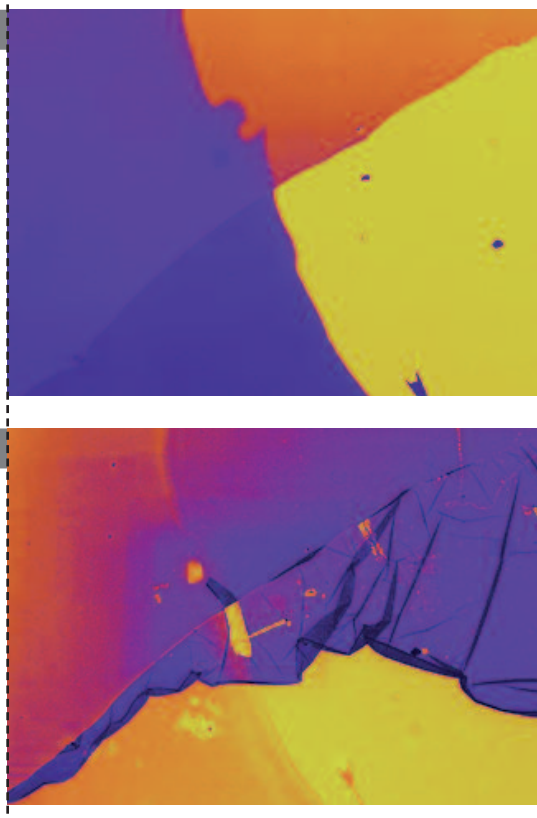
- Müllerová, I.; Frank, L.; *Scanning low energy electron microscopy.* Advances in imaging and electron physics 128, (2003), p. 309–443

An automatic method for the imaging of uncoated and nonconductive specimens by fine-tuning the primary beam energy so that the number of incoming and outgoing electrons from the specimen is equal.

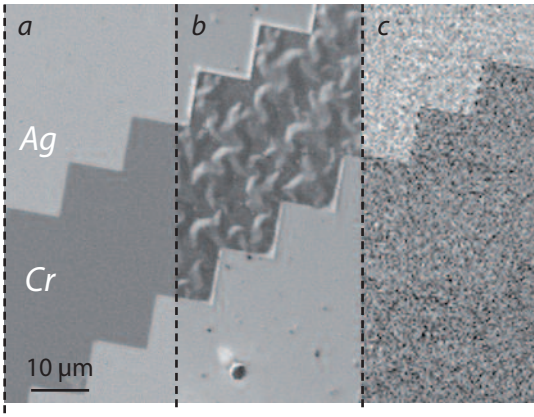
- Frank, L.; Zadražil, M.; Müllerová, I.; *Scanning electron microscopy of nonconductive specimens at critical energies in a cathode lens system.* Scanning 23, (2001), p. 36–50

Mini scanning low energy electron microscope with the total length of 10 cm was designed and tested for the in situ observation of surfaces by Auger electrons and by low energy electrons. Six channel detectors were designed for the collection of Auger and low energy electrons.

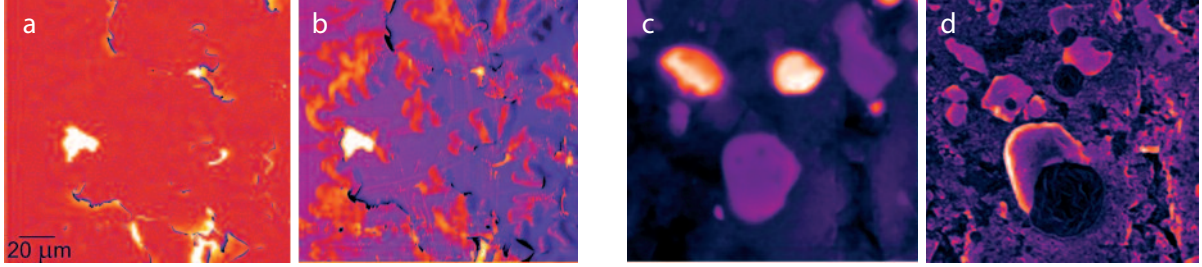
- Frank, F.; Müllerová, I.; El Gomati, M. M.; *A novel in-lens detector for electrostatic scanning LEEM mini-column.* Ultramicroscopy 81, (2000), p. 99–110



Structure composed of mutually overlapped thin layers of Au and GeSi on a silicon substrate, imaged with 9800 eV electrons (top) and with 850 eV electrons (bottom), showing the high surface sensitivity of low energy electrons



Surface analysis tools demonstrated on a 100 nm microcrystalline chromium foil, electron beam lithograph patterned on a silver-coated silicon wafer: a) scanning electron microscopy, image taken at 6 keV; b) scanning low energy electron microscope, image taken at 1 keV, surface crystallinity is clearly visible; c) elemental mapping using Auger electron spectroscopy, chromium LMM peak at 482 eV



Delaminated 200 nm CN_x layer on Si imaged with 4 keV electrons (a) and with 500 eV electrons (b) emitted from the sample biased to 4 kV

Composite of AlB_2, MgB_2, TiB_2 and ZrB_2 particles embedded in an Al matrix and imaged using 20 keV electrons (c) and 1 keV (d) with the sample biased to 10 kV

Pseudo-color images

- **First experiments showing the diffraction contrast in SEM and surface crystal orientation of clean Si were performed.**
  - Frank, F.; Müllerová, I.; Faulian, K.; Bauer, E.; *The scanning low-energy electron microscope: first attainment of diffraction contrast in the scanning electron microscope.* Scanning 21, (1999), p. 1–13
- **European and US patent**
  - El Gomati, M. M.; Frank, L.; Müllerová, I.; Electron detectors, EP1012867 (2006), US 6,570,163 (2003)
- **Applied result**
  - Müllerová, I.; Frank, L.; Klein, P.; Sýkora, J.; Detector of transmitted very low energy electrons for the ultra high vacuum scanning low energy electron microscope, functional module, (2012)

MAIN COLLABORATING PARTNERS

Collaboration with academic partners

- University of Toyama (Toyama, Japan)
- University of York (York, UK)
- University of Zürich (Zürich, CH)
- University of West Bohemia (Plzeň, CZ)
- Biology Centre of the ASCR (CZ)
- Institute of Macromolecular Chemistry of the ASCR (CZ)
- Brno University of Technology (Brno, CZ)
- Masaryk University (Brno, CZ)

Collaboration with companies

- JFE Steel Corporation (Tokyo, Japan)
- Voestalpine Stahl (Wien, Austria)
- Research and Testing Institute (Plzeň, CZ)
- Delong Instruments (Brno, CZ)
- FEI Czech Republic (Brno, CZ)
- FEI Company (Hillsboro, OR, USA)
- Crytur (Turnov, CZ)

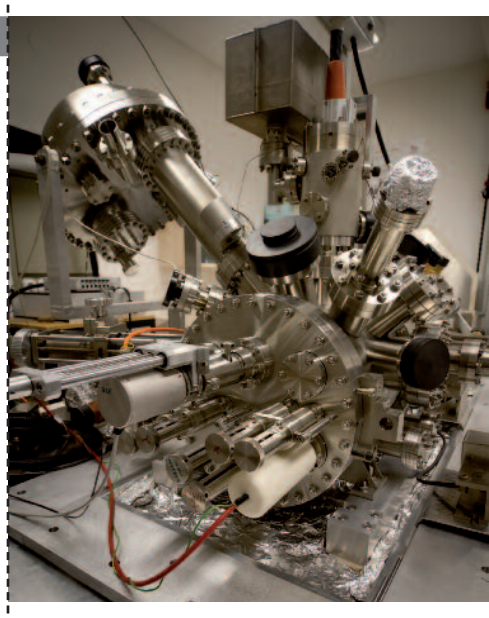
EXPECTATIONS

Offers

- Partnership in international projects
- Analysis of samples of advanced materials (imaging of various types of precipitates even under the sample surface, contrast measurement of graphene and other thin foils in reflected and transmitted signal; study of clean surfaces, crystallographic orientation of ultrafine grains; measurement of dopant concentration)
- Cooperation on nanostructure tasks difficult to solve with traditional electron microscopic methods (e.g. biological samples, polymers etc.)
- Design of detection systems for instruments using charged particles (electron and ion microscopes and lithographs)
- Contrast formation in electron and ion microscopes and lithographs, study of signal trajectories and interaction of charged particles with matter
- Design and manufacture of ultrahigh vacuum components and systems

Requirements

- Provision of samples of advanced materials
- Cooperation on nanostructure tasks difficult to solve with traditional electron microscopic methods
- Collaboration with industrial and academic partners
- Cooperation on vortex electron beams



Manipulators and feedthroughs controlling the specimen stage of the ultrahigh vacuum scanning low energy electron microscope