Group of Microscopy and Spectroscopy of Surfaces

Department of Electron Microscopy

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THEMATIC RESEARCH FOCUS

Research area

- Low energy scanning electron microscopy
- Low energy transmission electron microscopy and time-of-flight spectroscopy
- Auger electron spectroscopy and spectromicroscopy
- Surface physics
- Micro- and nanostructure of advanced materials
- Technology and diagnostics of 2D crystals and thin films
- Computer simulations of formation and detection of electron beams

Excellence

Contrast formation at low and very low energies in the scanning electron microscopy and spectroscopy 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; generation, acquisition and processing of electron micrographs

Mission

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

UP-TO-DATE ACTIVITIES

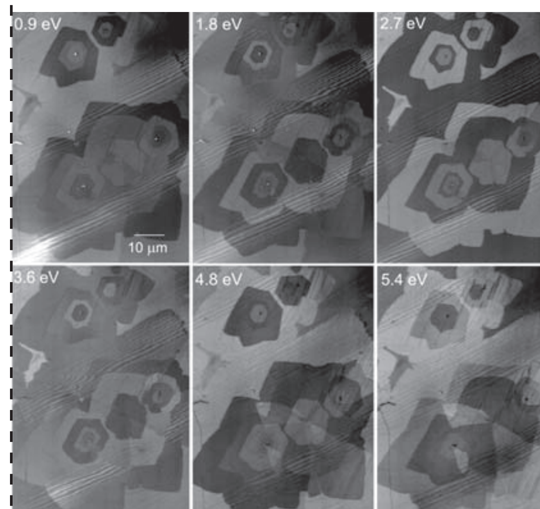
Research orientation

- 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, electron crystallography, examination of 2D crystals
- Detection of electrons emitted from surfaces or transmitted through films, including multichannel detection of angular and energy distribution of emitted electrons in SEM and STEM, aiming at ultimate angular, energy and lateral resolution, simulation of signal generation and detection
- Interpretation of scanned electron beam micrographs and Auger electron spectromicrographs
- Quantitative scanning electron microscopy, especially at low energies
- Examination of treatment of solid surfaces with slow electrons
- Questions of the coherence of electron beams

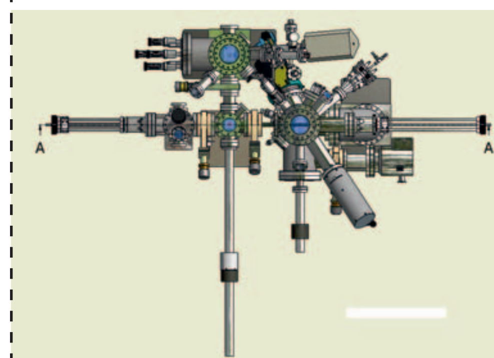
Main capabilities

Basic research

- Interaction of slow electrons with matter
- Reflectivity and transmissivity of slow and very low electrons from/through solids
- 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 field and motion of charged particles therein



Multilayer graphene deposited by the CVD technique on a Cu foil; variations in brightness of graphene islands originates in fluctuations of their reflectivity and number of reflectivity minima indicates the number of overlapped graphene layers



Design of the innovated preparation chambers and air-lock for the ultrahigh vacuum microscope, which is currently assembled

Applied research

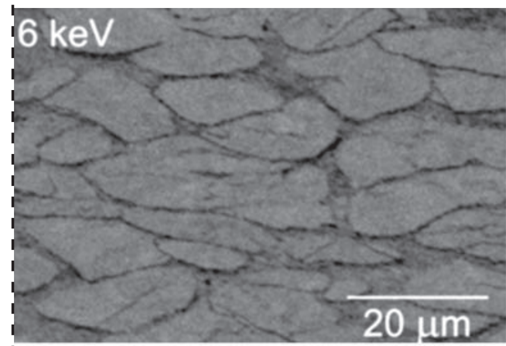
- Ultrahigh vacuum scanning low energy electron microscopy
- Analysis of phases in complex materials, e.g. steels
- Visualization of crystal orientation and internal stress
- Analysis of surface coatings and thin films, Auger electron spectromicroscopy
- Analysis of ultrathin tissue sections and free-standing films and 2D crystals
- Simulation of electron trajectories in electron optical elements and systems
- Design of multichannel electron detectors and the time-of-flight velocity analyser

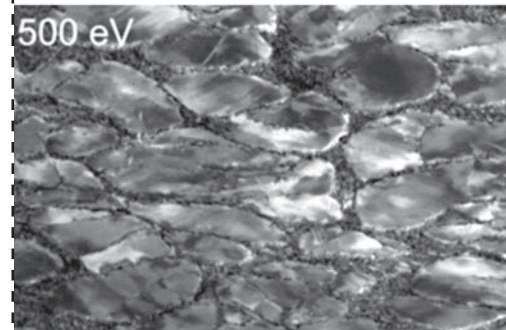
Innovations

- Extension of the scanning electrons microscopy to arbitrarily low energy in reflection and transmission modes
- Electron-beam-induced release of hydrocarbons from solid surfaces
- Counting of graphene layers upon reflection as well as transmission of slow electrons
- Acquisition of high contrast images of tissue sections not treated with any heavy metal species

Subfields of group activities

- 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



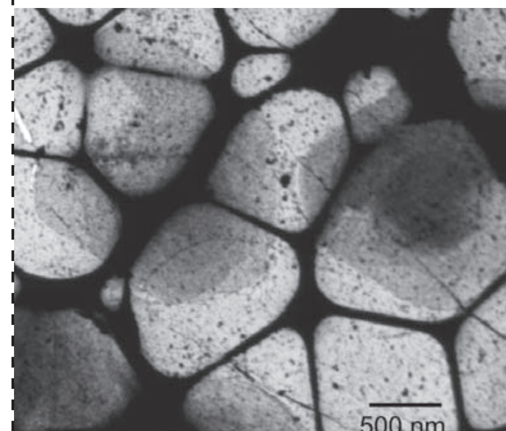


X210Cr12 ledeburitic steel heated to a semisolid state, heavily deformed and cooled; standard micrograph at 6 keV does not visualize the distribution of internal stress that is well shown in the cathode lens mode at 500 eV

KEY RESEARCH EQUIPMENT

List of devices

- 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 spectroscopy, mass spectroscopy of released gases and reflection high-energy electron diffraction from crystals
- Ultrahigh vacuum scanning low energy electron microscope of in-house design equipped with time-of-flight analysis of energies of electrons transmitted through ultrathin films and 2D crystals and two-dimensional position sensitive detection of the angular distributions of reflected electrons
- 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 and for the CVD technology grown thin films



CVD graphene samples deposited on lacey carbon lying on a copper mesh, commercially available sample declared as three- to five-layer graphene; image in transmitted electrons at 220 eV clearly visualizes sites differing by a single carbon atom in thickness

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**
  - I. Müllerová, M. Hovorka, L. Frank: "*A method of imaging ultrathin foils with very low energy electrons*", Ultramicroscopy **119**, 78–81, 2012

- **Imaging of graphene multilayers with a high contrast, counting the graphene layers upon reflectivity as well as transmissivity of very slow electrons, identification of the grow mechanism of graphene**
  - L. Frank, E. Mikmeková, I. Müllerová, M. Lejeune: *"Counting graphene layers with very slow electrons"*, Applied Physics Letters **106**, 013117:1-5, 2015
  - E. Mikmeková, L. Frank, I. Müllerová, B.W. Li, R.S. Ruoff, M. Lejeune: *"Study of multi-layered graphene by ultra-low energy SEM/STEM"*, Diamond and Related Materials **63**, 136-142, 2016

- **Characterisation of crystal orientation with a high lateral resolution from the reflectivity of electrons at impact energies below 40 eV**
  - Z. Pokorná, Š Mikmeková, I. Müllerová, L. Frank: *"Characterization of the local crystallinity via reflectance of very slowelectrons"*, Appl. Phys. Lett. **100**, 261602: 1–4, 2012
  - A. Knápek, Z. Pokorná: *"A method for extraction of crystallography-related information from a data cube of very-low-energy electron micrographs"*, Ultramicroscopy **148**, 52-56, 2015

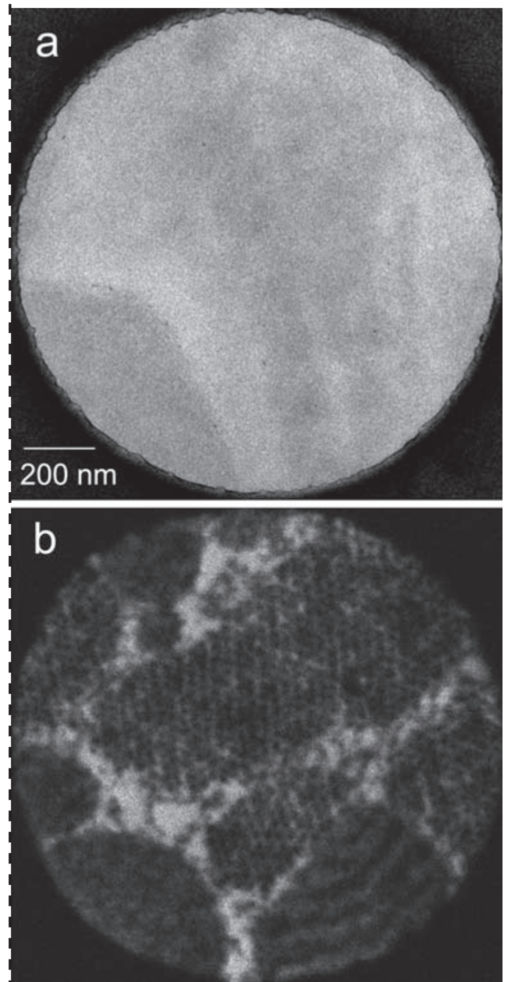
- **Imaging at high contrast and resolution of ultrathin tissue sections not treated with any agents containg heavy metal salts for contrast enhancement**
  - L. Frank, J. Nebesářová, M. Vancová, A. Paták, I. Müllerová: *"Imaging of tissue sections with very slow electrons"*, Ultramicroscopy **148**, 146-150, 2015
  - J. Nebesářová, P. Hozák, L. Frank, P. Štěpán, M. Vancová: *"The Cutting of Ultrathin Sections With the Thickness Less Than 20 nm From Biological Specimens Embedded in Resin Blocks"*, Microscopy Research Technique **79**, 512–517, 2016

- **Applications of low energy SEM in nanotechnology**
  - I. Müllerová, M. Hovorka, F. Mika, E. Mikmeková, Š. Mikmeková, Z. Pokorná, L. Frank: *"Very low energy scanning electronmicroscopy in nanotechnology"*, International Journal of Nanotechnology **9**, 695–716, 2012

- **Development of new highly sensitive method for the determination of crystallographic orientation of grains from maximum anisotropy of reflected electrons**
  - Š. Mikmeková, M. Hovorka, I. Müllerová, O. Man, L. Pantělejev, L. Frank: *"Grain contrast imaging in UHV SLEEM"*, Materials Trans. **51**, 292–296, 2010

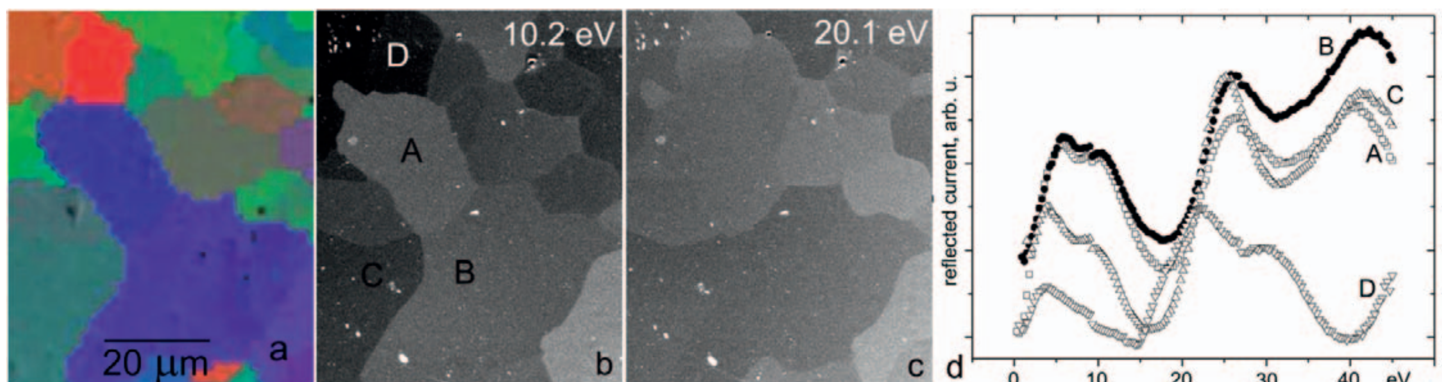
- **A method for quantitative measurements of dopant level in semiconductors using optimum primary beam energy**
  - L. Frank, I. Müllerová, D. Valdaitsev, A. Gloskovskii, S. Nepijko, H. Elmers, G. Schönhense: *"The origin of contrast in the imaging of doped areas in silicon by slow electrons"*, J. Appl. Phys. **100**, 093712: 1–5, 2006
  - I. Müllerová, M.M. El Gomati, L. Frank: *"Imaging of the boron doping in silicon using low energy SEM"*, Ultramicroscopy **93**, 223–243, 2002

- **An overview about the development of the scanning low energy electron microscopy**
  - I. Müllerová, L. Frank: *"Scanning low energy electron microscopy"*, Advances in imaging and electron physics **128**, 309–443, 2003



Section of mouse heart muscle, not fixed with osmium tetroxide and not stained; (a) 10 nm section imaged by conventional TEM at 80 keV, (b) micrograph taken at 500 eV by means of the cathode lens, showing drastically enhanced contrast

Identification of crystal grains in Al on the basis of reflectivity of very slow electrons; EBSD map (a), micrographs acquired using the cathode lens (b and c), and energy dependence of the reflectivity of selected grains (d)



- **An automatic method for non-charging imaging of uncoated and nonconductive specimens by fine-tuning the primary beam energy**
  - L. Frank, M. Zadražil, I. Müllerová: "*Scanning electron microscopy of nonconductive specimens at critical energies in a cathode lens system*", Scanning **23**, 36–50, 2001

M+AIN 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 CAS (CZ)
- Institute of Macromolecular Chemistry of the CAS (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)
- Thermo Fisher (FEI Czech Republic, Brno, CZ)
- Thermo Fisher (FEI Company, Hillsboro, OR, USA)
- Crytur (Turnov, CZ)

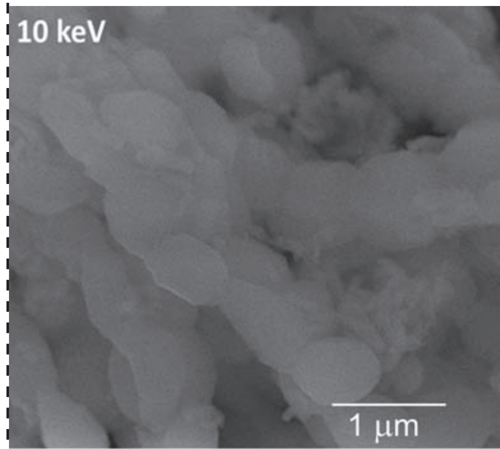
EXPECTATIONS

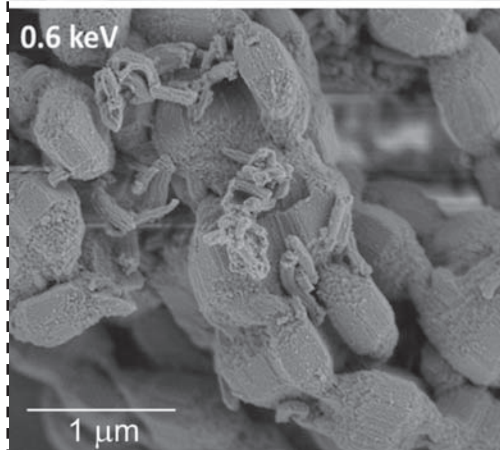
Offers

- Partnership in international projects
- Analysis of samples of advanced materials
- Cooperation on nanostructure tasks difficult to solve with traditional electron microscopic methods
- 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

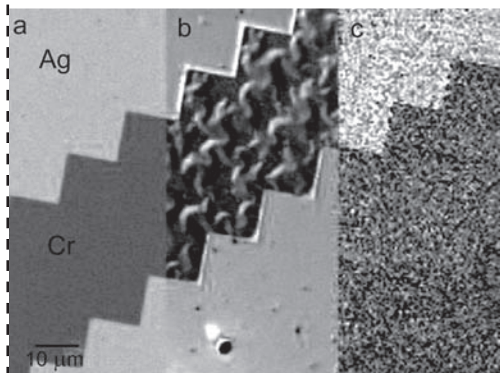
Requirements

- 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

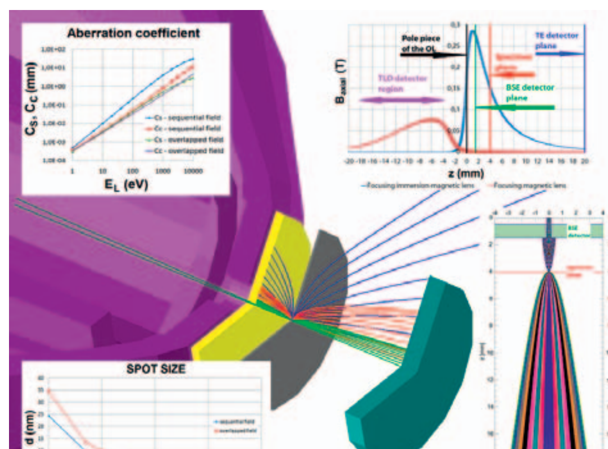




Mesoporous carbon nitride foam as a carrier for catalytic gold nanoparticles; when reducing the energy of electrons, we diminish the interaction volume from which we receive the image information so the image gets „sharper"



Surface analysis tools demonstrated on a 100 nm microcrystalline chromium foil, electron beam lithograph patterned on a silver-coated silicon wafer; (a) conventional image; (b) low energy image; (c) corrected Auger mapping in Cr



Examples of simulated configurations and results of simulations of electron-optical properties and trajectories of signal species