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Institute for Electron Microscopy and Nanoanalysis Graz University of Technology

Graz Centre for Electron Microscopy ACR Austrian Cooperative Research















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Austrian Centre for Electron Microscopy and Nanoanalysis Graz University of Technology

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Pilz, auf Hesperis Matronalis (gewöhnliche Nachtviole) by Sanja Simic

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65 Years Electron Microscopy in Graz

Sixty-five years ago, Graz University of Technology took the future oriented decision to establish an electron microscopy facility. This first electron microscope in Styria and the dedicated scientist -Fritz Grasenick – formed the nucleus of a rapidly developing facility. Our university can look back at these sixty-five years with some pride and satisfaction. The today's Institute of Electron Microscopy and Nanoanalysis stands at the forefront of microscopy research and represents a key resource for Austrian research institutions. I believe that the close collaboration with the Association for Electron Microscopy and Fine Structure Research is crucial to develop competences and to form the critical mass, which is necessary for ambitious endeavors such as electron microscopy at atomic resolution. Remarkably, the people in the institute have managed to gain an international reputation in the field of nanotechnology and advanced microscopy of materials. I know very well, that advanced electron microscopy needs expensive infrastructure and a long-term commitment. Graz University of Technology is ready to support future developments.



Univ.-Prof. Dipl.-Ing. Dr.techn. Dr.h.c. Harald Kainz © Lunghammer - TU Graz



Making Progress

Microscopy plays an essential role in modern sciences and technologies: be it materials and biological sciences or, increasingly, the development and production of smart devices, sensors and machines. Scientists and industrialists from Styria have always been front-runners of this development starting their efforts as far back as in the fifties of the last century. They established a collaborative research institute based on the Association for Electron Microscopy and Fine Structure Research: the Graz Centre for Electron Microscopy (ZFE), which developed into an internationally recognized centre for advanced materials characterisation focusing on high resolution electron microscopy. In the meantime, our recent success in the field of nanofabrication is increasingly important for industry and research institutes.

We take great pride in the substantial impact that the institute is having on research in this country; being essential to both, to industry and universities. We are convinced that the institute has a bright future and therefore we will continue to support the institute's progress with funding for leading-edge instrumentation and highly skilled scientists. Finally, we would like to thank the lab team for the hard work and never ending enthusiasm, our partners from the TU Graz and especially the team from the Austrian Cooperative Research.



Prof. Dipl.-Ing. Dr.-Ing.h.c. Helmut List President of the Association



KR Dipl.-Ing. Ulrich Santner Vice-President of the Association



Evolving Microscopy Research

65 years ago, the first electron microscope in Styria was installed at the former Technische Hochschule Graz; in the course of decades, this first nucleus has evolved into one of the leading microscopy facilities in Europe. Back then the reasoning behind was to build an institution to bridge the gap between basic and applied research on the one hand and to serve the needs of the Austrian industry on the other hand. Remarkably, this concept is still worthwhile and a main source of our success. Due to increasing costs for high-end electron microscopes and other state-of-the-art instrumentation, European microscopy laboratories are evolving into a similar direction. Another key factor is the active promotion of research collaborations with and services for other university institutes and enterprises on a European scale.

Advanced electron microscopy and nanofabrication is an expensive endeavour and needs support, not only from the University, but also from various other funding sources. We are in a very good position thanks to the support of the Association for the Promotion of Electron Microscopy and Fine Structure Research, helping us to develop a critical mass for worldwide visibility. Nevertheless, that is not enough.

Industry collaborations have always been a main source of income; still we are increasingly promoting advanced research projects with other university institutes and industry partners. With the shift to basic research, which came with the incorporation of the FELMI into the Faculty of Mathematics, Physics and Geodesy a decade ago, we enhanced our project collaborations and teaching activities to a high level.

In the reporting period, we successfully finished European research projects like the prestigious ESTEEM2 network and the Triple-S project. The project OPTIMATSTRUCT funded by the FFG was realised in collaboration with the Austrian Foundry Institute (ÖGI) in Leoben. However, in these past two years we had to accept that not every application for research funding was successful. Nevertheless, we launched new research projects such as the multi firm project "Quantitative analysis of internal interfaces" with important Austrian microelectronic companies, SOLABAT with the Institute of Chemical Technology of Materials of the TU Graz, SENTINEL with the Chinese Academy of Sciences in Beijing, SENTECH with the Institute of Physical Chemistry at the University of Leoben and the AEROPORE-project with the ofi in Vienna. Finally, the research project "Innovative Materials Characterization" with partners from the Austrian Cooperation Research (ACR) was funded in autumn 2016. A new approach has been chosen as far as the proposal for the infrastructure network ELMINET Graz is concerned, which is handled together with the Medical University of Graz and the University of Graz in 2016. Finally, a new project application for a Christian-Doppler Laboratory on Nanofabrication is on its way.

I am happy that the results of these projects are increasingly finding their way into leading scientific journals such as Nanoletters, Physics Review Letters, Nature Communications, Scientific Reports, and many others.

Our focus on electron microscopy and nanofabrication requires a highly skilled research team and high-end instrumentation to support not only our own research but also that of other institutes at the TU Graz and from industry. Here we could proceed with scientific infrastructure for *in situ* microscopy of materials and an SDD X-ray spectrometer funded by the ACR and the Ministry of Economics, Science and Research (BMWFW). In August 2017, we will install a brand new scanning electron microscope, which is equipped with an integrated Raman spectrometer for the parallel structural and chemical imaging of surfaces.

Finally, I would like to take this opportunity to thank all my colleagues in the institute for their hard work and enthusiasm in moving forward. Last but not least, I am very grateful to the Rector of the TU Graz, Harald Kainz, the Vice-rector of the TU Graz, Horst Bischof, the Dean of the Faculty of Mathematics, Physics and Geology, Wolfgang Ernst, the presidents of the Association, Helmut List and Ulrich Santner, and the board for their continuous support of our institution.



Ao.Univ.-Prof. Dipl.-Ing. Dr.techn. Ferdinand Hofer

The Institute

The Institute at a Glance

The Austrian Centre for Electron Microscopy and Nanoanalysis is the leading Austrian research institution in the field of advanced materials microscopy and nanotechnology. It consists of two independent organisations:

- the Institute of Electron Microscopy and Nanoanalysis (FELMI) at the Faculty of Mathematics, Physics and Geodesy.
- the Graz Centre for Electron Microscopy (ZFE), a member of Austrian Cooperative Research (ACR).

Having their own legal status and budget, both institutes work in close alliance to ensure efficient use of personnel and instrumental resources. The Institute is located in the heart of Graz, on the campus *Neue Technik* of the Graz University of Technology. It is organised in research groups, each dedicated to important research topics in advanced electron microscopy and functional nanofabrication. A unique feature of the Institute is its broad field of activities ranging from fundamental research to teaching an increasing number of students and service research for other institutes and industries.



Austrian Centre for Electron Microscopy and Nanoanalysis

Nanoanalysis in the TEM	High Resolution Electron Microscopy	Functional Nano- fabrication	SEM & <i>in situ</i> Methods	SEM & Microanalysis
Gerald Kothleitner	Werner Grogger	Harald Plank	Peter Pölt	Hartmuth Schröttner
Administration		Techn. Laboratory	Media	Communications

A History of Excellence

We support excellence in teaching and research for 65 years. In 1949 an industrial donation of 100,000 Schilling was the financial basis of the first electron microscope at the former Technische Hochschule, today's Graz University of Technology. Two years later a specialised research centre for electron microscopy, the Forschungsstelle für Elektronenmikroskopie was established. Fritz Grasenick became the first head of the Institute. The microscope Übermikroskop UEM100 by Siemens & Halske in Berlin was delivered and installed in March 1951. The inauguration ceremony of the research centre took place three month later on June 30th 1951. A symposium was held; many international experts participated, to name some of them: Ernst Ruska (Nobel Prize winner), Bodo von Borries (physicist and co-inventor of the electron microscope), Walter Glaser (theoretical physicist) or Otto Wolf (Siemens & Halske, Berlin).

Our unique position in the field of electron microscopy analyses is based on our leading-edge equipment ranging from the latest light and electron microscopes to high performance computing hardware. This, combined with the ambition of our staff, makes us the Austrian powerhouse of electron microscopy.

On this basis we are endeavouring to develop new microscopy methods and to improve special preparation techniques, which are especially used for materials and life sciences to characterise all kinds of materials and to provide efficient answers and solutions to scientific and industrial problems.

We have established strong links with research partners and special industries which is reflected by the fact that the development of personnel is increasingly influenced by funded research projects and contractual research.



Research Focus

Advanced electron microscopy provides unique insights into the micro- and nanoworld and is an important prerequisite and key to innovations, especially in materials research and nanotechnology. Therefore, we concentrate our research activities on the development of new microscopic techniques and their application to challenging themes in physics, chemistry, materials science, and nanotechnology. Typically, we try to push the boundaries of our understanding of nature by means of world-class microscopy research. We transfer the acquired knowledge and capabilities into collaborations with other university institutes and special industries.

Major areas of research are:

- Nanoanalysis of materials
- Functional nanofabrication
- 3D and in situ characterisation
- Soft matter and biomicroscopy



First head of the Institute: Fritz Grasenick (middle)

Collaborations

Each year we have been collaborating on average with around 30 university institutes and more than 120 companies – mainly from Austria, but 👥 thods. We believe that it is not only our responincreasingly also from other European countries. Two main directions are being followed during recent years:

Firstly, collaborations within the ACR group which are developing well and secondly, the incorporation into important European research networks such as the STREP project "CopPeR", the ENIAC or our scientific and technical support. initiative or the ESTEEM2 project.

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Being an interdisciplinary research institute we are constantly creating new knowledge and me-.... sibility to support knowledge creation but also to share generated know-how and expertise with others: each year we welcome some 200 visitors from other research groups and companies. dents from other institutions have profited from

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Europe

Belgium

University of Antwerp, Antwerp

Bulgaria

AMG Technology Ltd., Botevgrad Croatia

University of Zagreb, Zagreb

France

Université Paris Sud, Orsay

CEMES-CNRS, Toulouse

Germany

University of Kiel, Kiel Martin-Luther-University, Halle-Wittenberg Goethe University, Frankfurt a. Main Jülich Research Centre, Jülich Fritz-Haber-Institute (MPG), Berlin University of Tübingen, Tübingen

Greece

CPERI/CERTH, Thessaloniki

Hungary

Hungarian Academy of Sciences Centre for Energy Research, Budapest Centre for Energy Research, Budapest

Italy

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Politecnico di Milano, Milano University of Parma, Parma

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Poland

AGH University of Science and Technology, Kraków

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Russia

Ural Division of the Russian Academy of Sciences,

Ekaterinburg

Slovenia

University of Maribor, Maribor

Institute Jožef Stefan, Ljubljana

Spain

Instituto de Ciencia de Materiales, Sevilla

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Australia

University of Melbourne, Melbourne ESEM Research Laboratory, North Bondi

Association for the Promotion of Electron

Being a non-profit association the exclusive and direct purpose is to promote research and scientific teaching in the field of electron microscopy. Ever since its establishment in 1959 the association has been shaping the Austrian research community by fulfilling a crucial double task: it is providing special industries with the latest results of approved as well as newly designed microscopy techniques on the one hand; on the other hand it is keeping our scientists up to date as far as developments in the field are concerned. In doing so the Graz Centre for Electron Microscopy (ZFE) is operated by the association which plays an important role in developing and applying fundamentals as well as carrying out different projects. Cuttingedge instrumentation has been acquired and highly skilled and well trained permanent staff is doing research in different research areas. The last General Meeting of the association took place in 2014. The president has been re-elected for the next six years. The association has currently 36 members mainly from Austria.

Presidential Committee

President: Prof. Dipl.-Ing. Dr-Ing.h.c. Helmut LIST 1. Vice president: KR Dipl.-Ing. Ulrich SANTNER 2. Vice president: Ing. Hans HÖLLWART

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Microscopy and Fine Structure Research

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AUSTRIAN COOPERATIVE RESEARCH

Sensengasse 1 1090 Vienna www.acr.at

Austrian Cooperative Research (ACR)

The ZFE Graz is a member of the "Austrian Cooperative Research" (ACR). For more than 60 years ACR has been supporting Austrian SMEs in their innovation process offering specialized research and technology expertise. ACR stimulates and enables innovation within trade and industry thus improving the competitiveness of the Austrian economy.

The strength of ACR-members:

- Applied research, development and innovation
- Technology and know-how transfer
- Project management for R&D projects
- Efficient funding advice
- High-grade testing and measuring accredited at EU level
- Certification and audits
- Training courses, expert seminars, company events

Currently, ACR has 18 full members. In 2016 the full members with a total of more than 800 employees generated a turnover of EUR 61.4 million. The network performed 75% of their services for SMEs.

The ZFE Graz cooperates with the following ACR institutes:

- Austrian Foundry Institute (ÖGI), Leoben
- Austrian Research Institute for Chemistry and Technology (ofi), Vienna
- V-Research, Dornbirn

During a study trip in 2015 the ACR visited the Danish Technological Institute (DTI) offering so called innovation checks to companies where fresh minds may be helpful to develop prospects and detect potential for innovation.

Two Danish trainers held a five day seminar to introduce the concept of **"Innovation Agents"** to the ACR network.



ZFE as 3rd Place Winner of the Carbonium Prize 2015

ACR President Dr. Martin Leitl presented the Carbonium Prize to the ZFE, an award which decorates those ACR members who contribute the most as far as internal networking and external communication of the ACR umbrella organisation are concerned. © Foto: ACR



The Cooperation Award 2015 goes to Harald Plank In close cooperation with SCL-Sensor.Tech. Fabrication GmbH (Vienna), Harald Plank and his team developed a new generation of nanoprobes allowing the electric and thermal material characterisation in the sub-10 nm range. © Foto: ACR

Research Expert for Austrian SMEs



For more than 65 years the Institute has supported Austrian companies in their innovation process. We have made a considerable contribution to the competitiveness of the Austrian economy thanks to numerous projects and temporary activities. The strengths of the Institute is our research and development competence with excellent links to the academic and business world, strong and flexible research groups in close contact with SMEs and expert knowledge of the national and international funding landscape.

The Institute in Figures

We seek to foster and sustain a creative and supportive working environment based upon an ethos of respect and rigorous scientific enquiry. We value the diversity of our staff and student population and want to promote equality as an essential element.

Staff FELMI (TU) ZFE



Scientific Results and Impact

FELMI-ZFE is facing a number of different tasks due to the fact that we are not only a research institute, but also devoted to teaching and services. Nevertheless, we could reach a satisfactory number of peer reviewed publications (99 papers in two years). This high number comes from manifold collaborations with other university institutes and industry; we are increasingly publishing in high impact journals such as Nature Communications, Scientific Reports and Nano Letters. The number of invited talks remains at a very high level (21 in two years). All publications of the last two years are listed in the chapter Publications (p. 68).

Budget

The budget of both institutions increasingly depends on the income from public research funding and contractual research with Austrian and European companies. The last two years have been very difficult and the main reason for this development lies in the substantial and rapid increase of the service costs for the microscopy After a reorganisation of the research groups in 2016 we plan to extend the number of scientists and PhD students in the next two years. This will occur in both institutions and will depend on our success in funded research projects and contractual research work for industry. Since we are hosting more and more master students, the space pressure we face remains very tight.







equipment. However, these costs cannot be avoided in an electron microscopy laboratory with leading-edge instrumentation. Consequently, we had to cut our investments of new research infrastructure. The budget development of the ZFE also shows very high expenses for the ongoing renovation of the Institute and the Steyrergasse 17 premises. On the FELMI side the income comes mostly from LLL-courses and publically funded research projects.



Quality Assurance

The Institute works under an advanced quality management system according to the rules of ISO 9001:2008. The aim is to maintain the outstanding quality of our work and analysis results and to improve continuously our organisation and management structures. The system was first certified in 2004 by the TÜV Austria and has since been under continuous development. It remains valid until May 2018 and covers "Research and teaching in the field of microstructure research and materials characterisation by electron microscopy, micro- and nanoanalysis and the development of analysis and preparation methods".



New Instrumentation

New Methods for *in situ* Microscopy

In situ microscopy investigations are of rapidly growing importance in materials and biological sciences. Images or spectral data recorded sequentially are used to track the changes caused by heating, cooling, straining, chemical reactions, or beam damage. In order to keep track of recent

developments, new specimen holders for SEM and TEM microscopy were in urgent need. In 2016 we introduced an advanced tomography specimen holder and a MEMS heating stage for TEM (up to 1300°C) and a heating holder for SEM-studies (up to 1050°C). This infrastructure was funded by the Austrian Cooperative Research (ACR) and the Austrian Ministry for Science, Economy and Research (BMFWF) in Vienna.





Cryo-Preparation Unit

The new grid-plunger system Leica EM GP considerably improves the quality our cryo-TEM investigations. It is used for the vitrification of thin samples (up to a few micrometers) like nanoparticles, colloids and biomolecular structures, such as proteins and viruses with subnanometre resolution. Samples in suspension are applied onto TEM grids in a humidity controlled environmental chamber and plunge frozen into liquid ethane after removing excess fluid by automatic blotting. This instrument was purchased with funding from a HRSM-project of the Austrian Ministry of Science, Economy and Research (BMWFW) and by the Association for Electron Microscopy and Fine Structure Research.



















Prof. Dr. Werner Grogger

holds a PhD in Physics and defended his habilitation in 2004. He is head of the research group for high-resolution analytical transmission electron microscopy. In the course of his scientific career he carried out research at the National Center for Electron Microscopy, Berkeley.

Research Groups

High Resolution Electron Microscopy

Cool (and also low temperature) experiments, accurate elemental and structural analysis, and supporting image simulations.

The group of Prof. Grogger works in the field of analytical transmission electron microscopy (TEM). The methods we use mainly focus on quantitative elemental analysis using X-ray spectrometry (EDXS), Cryo-TEM, high resolution STEM, and image simulation. These techniques are applied to different materials ranging from hard coatings, perovskites and nanoparticles to organic materials and pharmaceuticals.

Cryo-TEM of Symmetric and Asymmetric Lipid Vesicles

Cryo-TEM is an essential tool to answer questions which remain open when using small- and wide-angle X-ray scattering (SWAXS) as well as small-angle neutron scattering (SANS). F.i. measurements on lipid vesicles show especially in the gel phase properties, which cannot be explained with a symmetric model of the lipid membranes. Experimental data give reasons for the assumption that the surface of vesicles might not be perfectly round. Cryo-TEM measurements give an insight into the actual shape of vesicles in the liquid phase as well as in the gel phase and furthermore an essential contribution to modelling of SAXS and SANS data. Not only for symmetric but also for asymmetric lipid vesicles (inner and outer shell of the bilayer of the liposomes have different chemical composition) is Cryo-TEM fundamental to visualise the shape of individual vesicles which allows conclusions about the type of impurities.



Figure: Liposome particles in vitreous ice



Group Leader: Prof. Dr. Werner Grogger

Senior Researchers:

Dr. Evelin Fisslthaler Dr. Christian Gspan Dr. Ilse Letofsky-Papst Dr. Franz Schmidt

PhD Student: Mag. Johanna Kraxner DI Judith Lammer

Technical Assistants: Manuel Paller

Film and Edge Plasmons

Metallic nanostructures can bind light close to their surface, leading to highly localized and strong electromagnetic fields. Combining single nanostructures leads to even stronger fields due to coupling of so-called surface and edge plasmons. A detailed understanding was achieved by resolving plasmon fields of single and coupled nanoparticles using electron energy loss spectroscopy (EELS). In ref. 1 we studied film and edge plasmons and their corresponding field distribution on a metallic thin film taper. In case of a single silver nanocuboid we observed an energy splitting of plasmon edge modes, proofing for the first time the existence of plasmon coupling within a single nanoparticle (ref. 2). In another work (ref. 3) we addressed the problem of signal identification from EELS data and the assignment to specific plasmon modes. The rich plasmon mode spectrum of metallic nanoparticles often impedes a clear identification of specific plasmon modes due to signal overlap, therefore post-processing techniques like data deconvolution are required to unveil otherwise hidden information. We developed a data analysis routine for advanced data analysis, an appropriate display of the measured data on the screen in order to facilitate the handling of large data sets to get the best out of the data.

simulations we tried to find out the type of atomic

species inside the channels. There is a pretty good

agreement between the experimental and the si-

mulated image when we put water molecules into

the channels (simulated image / right). Additional

measurements using Raman and UV-Vis spectro-

metry support this finding.



Figure: EEL maps of the dipolar plasmon eigenmode on a 30 nm thick gold nanostar using different plot view options available in the "Spectrum Image ananlysis tool".

Atomic Resolution Microscopy Investigations

Aquamarine is the blue variety of Beryl and shows an interesting crystal structure with channels with a diameter of about 0.5 nm. By looking along the c axis one can see the hexagonal structure of the natural mineral (HAADF-STEM image / left). A closer inspection of the channels reveals that some of them are filled. With the help of image



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2. F. Schmidt, H. Ditlbacher, U. Hohenester, A. Hohenau, F. Hofer, J. Krenn, "Edge Mode Coupling within a Plasmonic Nanoparticle", Nano Letters 16 (2016), 5152–5155.

3. F. Schmidt, F. Hofer, J. Krenn, "Spectrum image analysis tool – A flexible MATLAB solution to analyze EEL and CL spectrum images", Micron 93 (2017), 43–51.



Prof. Dr. Gerald Kothleitner

earned his PhD in Chemistry, worked abroad as an international product manager for analytical instruments in electron microscopy and received his habilitation in Physical Chemistry. His focus is on analytical techniques and methodical developments for materials characterisation.

Nanoanalysis in the TEM

From Chemical Analysis to Plasmonics

Gaining detailed knowledge about the atomic arrangement and the chemical composition is paramount for a deeper understanding of the properties of matter and their potential applications in technology. In the past two years, the AEM group has lifted electron microscopic imaging into the third dimension, innovating in analytical tomography and emphasizing simulations that shed light on atomic scale phenomena.

Nanoanalysis in ...

Spherical-aberration (CS) corrected electron microscopy, available with the Austrian scanning transmission electron microscope (ASTEM), has developed into a powerful tool, for studying matter with unprecedented spatial resolution and chemical sensitivity. While the structural characterisation of a material often relies on electrons having undergone large angles of scatter, element-specificity is obtained by studying the inelastic interactions between the microscope electrons with the sample atoms. The ability "to see", however, necessitates a thorough understanding of the underlying physical and chemical processes, which require in-depth simulations. Furthermore, effects that occur in the bulk of a specimen and differ depending on the viewing angle, can only be recovered from tilted projection images, leading to tomographic reconstructions. Consequently, in the past the group has focused its efforts towards theoretical calculations and novel analysis and reconstruction schemes.

... 2D

In a study of Aluminium-Silicon cast alloys, we looked at the nucleation and growth mechanisms of the eutectic Si phase as well as twinning mechanisms in the presence of dopant atoms. Experimentally, evidence was found that dopant Sr atoms arrange only along certain crystallographic directions and at twin boundaries. Backed up by density functional theory ab-initio total energy calculations, carried out in a collaboration with the theory group of Prof. R.C. Picu at Rensselaer Polytechnic Institute, Troy, USA a new concept for twin-formation was proposed and published in a Nature research journal 2016 [1].

... and 3D

The workgroup also deals with the complex problem of optimizing and adjusting reconstruction algorithms for the respective microscope signals and how to correlate theoretical information with 3D data such that more physically meaningful information can be extracted from the experiment. This shall be exemplified by two cooperations with the Institute of Experimental Physics, TU



Group Leader: Prof. Dr. Gerald Kothleitner

Senior Researchers: Dr. Mihaela Albu Dr. Georg Haberfehlner Dr. Karin Wewerka

PhD Students:

DI Daniel Knez Mag. Lukas Konrad DI Angelina Orthacker

Technical Assistants: Margit Brunegger Martina Dienstleder Ing. Claudia Mayrhofer

Apprentices: Arnela Blažević Karina Mollich Jakob Schaller Graz (Wolfgang Ernst) and with the Institute of Physics, KFU Graz (Ulrich Hohenester):

A study, published in Nature Communications 2015 [2], is dealing with the synthesis and characterisation of nanoclusters in superfluid Helium, consisting of just a few thousand gold and silver atoms. The issues in question were the growth mechanism of these clusters as well as their structure and chemical composition after deposition on a carrier. To answer this question a novel acquisition scheme that minimized image noise and scan distortions, as well as algorithms that allowed to extract atomic positions plus the chemical make-up from the core-shell object were developed. From the 3D visualization, valuable conclusions about material properties and possible uses could be derived.

Another interesting application for electron tomography lies in the field of plasmonics. Light or electromagnetic radiation couples to nanometersized structures (for instance of gold or silver). Depending on shape, size, environment, and chemistry of the object, so-called surface plasmons are forming, which are resonances of the metal electrons, responding to the incoming radiation. While characterising such particles and their associated resonances with optical methods is often complicated, the TEM offers enough spatial and spectral resolution to resolve such modes. Microscope beam electrons, running by these structures in close proximity, loose energy, which can be measured spectroscopically. The full 3D energetic distribution of the modes can be reconstructed and related to theoretical calculations (NanoLetters 2015) [3].



Figure: Synthesis and characterisation of single- and multi-core nanoclusters. (a) Schematic of the experimental setup for cluster synthesis. (b, c) Elemental maps of a single-core/shell Ag-Au cluster (b) and of a dual-core/shell cluster (c). (d) Calculated occurrence probability of single-, double- and triple-core clusters as a function of final cluster size. Scale bars, 2 nm.

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Functional Nanofabrication



DI Dr. Harald Plank

earned his PhD in Technical Sciences. Since 2010 he has been coordinating the work group S³ for Focused Ion Beams (FIB) and Atomic Force Microscopy (AFM). In the course of his research activities he spent several months in the USA, South Corea, France, Turkey, and Switzerland.

Direct-Write Fabrication of Functional Nanostructures

Focused Electron Beam Induced Processing (FEBID)

Additive direct-write manufacturing has become an integrative part in science and technology over recent years. The main advantage of such methods is the ability to deposit complex structures in a single step eliminating preliminary templating as typically used in resist-based processes, which expands the applicability to even non-flat surfaces. By that, such direct-write methods are of complementary character for situations where alternative methods approach their limitations. Within the pool of emerging technologies in this field, focused electron beam induced deposition (FEBID) attracts increasing interest as it evolved into a reliable, predictable and highly flexible bottom-up fabrication method which is applicable on virtually any given surface. In principle, FEBID uses nanometer-sized and widely non-invasive particles for highly localized chemical nano-synthesis of gaseous precursor molecules which physisorb on the specimen surfaces (see Figure 1a). As this method requires neither pre- nor post-processing treatments, it indeed represents a direct-write technology for additive manufacturing with spatial nanometer resolution. The scientific focus of FELMI's workgroup S³ is the fundamental understanding of the deposition process and in particular the correlation between fabrication procedures and their implication on chemistry, morphology and final functionality. Particular emphasis lies on fundamental resolution limitations, which finally paves the way towards high-fidelity structures down to the lower nanoscale. Here, the S³ group answered the long lasting debate about unwanted broadening effects during FE-BID. In more detail, systematic studies decoupled electron related trajectory effects from precursor surface dynamics [1]. By that, we could pinpoint the broadening effects to distinct electron species called forward- and backscattering electrons in dependency on the primary electron energies as a main parameter during FEBID. The combined outcome led to a parameter guide in dependence on the aimed applications.

In a further study, we then used the gained knowledge about unwanted broadening effects and focused on the morphology of the fabricated structures with emphasis on surface flatness and edge rounding effects. Both are of high interest in the field of resonant optics where highly defined shapes are a key element for the highest performance. The study revealed several different morphological implications in dependency on the FE-BID parameter space [2]. The essential outcome of this study, however, was the successful classification of appearing morphologies in dependence on the precursor working regime including their mutual transition regimes. By that, we could again derive a parameter guide towards nanoflat highfidelity structures with height variations in the sub-nm regime.



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PhD Students: DI Thomas Ganner (AFM|FEBIC) DI Jürgen Sattelkow (FEBID)

DI Robert Winkler (FEBID)

Technical Assistant: Sebastian Rauch (FIB)

Structuring Biomaterials

While the fabrication of functional nanostructures via FEBID mostly focuses on metallic materials, the direct-write fabrication of biological material is much more complicated. In the past, FELMI's workgroup S3 focused on the design and fabrication of cellulosic multiphase materials [3], however, with the aim to produce comparable large layers for integrative biochemical experiments. Together with the background in FEBID we therefore focused on the highly localised fabrication of cellulosic nanostructures. In more detail, we have introduced a technology which we called Focused Electron Beam Induced Conversion (FEBIC). Here we start with a thin film of a cellulose precursor, which is then chemically transferred into pure cellulose by the focused electron beam [4]. By that we demonstrated the fabrication of cellulosic structures in the sub-100 nm regime with fully maintained cellulose properties for the first time. The main advantage of this technology, however, is the enormous flexibility concerning the structure shapes as shown in Fig. 1.X. By that, we have successfully introduced a direct-write technology for the fabrication of biological nanostructures, which will exploit the full potential.



Figure: (a) working principle of Focused Electron Beam Induced Deposition (FEBID) which allows highly precise fabrication of planar, and freestanding nanostructures even on non-flat surfaces as representatively shown by a light fiber tip modification in (b). (c) shows cellulosic nanostructures with feature sizes down to the sub-100 nm regime fabricated via a new technology called Focused Electron Beam Induced Conversion (FEBIC) [4].

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R. Winkler, A. Szkudlarek, J.D. Fowlkes, P.D Rack, I. Utke, H. Plank, "Towards Ultraflat Surface Morphologies During Focused Electron Beam Induced Nanosynthesis: Disruption Origins and Compensation", ACS Applied Materials & Interfaces 7 (2015), 3289.

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SEM & in situ Methods



Dr. Peter Pölt

works at the Institute since 1980. He holds the Venia docendi since 2008. Being group leader in the field of scanning electron microscopy, his current research concentrates on materials characterisation including *in situ* experiments in the ESEM and electron backscatter diffraction.

Microscopy - much more than Images

Scanning electron microscopy can provide in many cases information about the 3-dimensional structure of a sample, its chemical composition, its crystallinity, and also the distribution of stresses and dislocations in the material down to the sub-micrometer scale. Thus, a full characterisation of a material is often possible.

Basics

Persons who are not very familiar with microscopy regard it as a method delivering nice images about surface topography and its structure; and not much more else. As long as these images are two-dimensional, even the information you can gain about the structure of a sample is limited. For example, apart from very regular structures, no direct information about the size and distribution of the single phases in a multi-phase material can be extracted. But the properties of materials may also be strongly dependent on their chemical composition, their crystallinity and internal stresses.

Yet, a scanning electron microscope (SEM) equipped with an x-ray detector (EDS, WDS) and an electron backscatter diffraction (EBSD) detector enables, within certain limits of course, the determination of the morphology, chemistry and crystallinity of individual structures / phases / grains down to a size of around 20 – 100 nm.

Nevertheless, to get reliable quantitative results, flat (polished, cut) specimens have to be used. The 3rd dimension is still missing. To get the 3-dimensional structure and composition of a sample it has to be sliced and subsequently a 3D reconstruction has to be performed using the respective stack of 2-dimensional images. Dependent on the method used for slicing slice thicknesses down to a few nm are possible. Automated slicing and imaging is, amongst other methods, possible with an ultra-microtome mounted in the specimen chamber of a SEM. For hard materials a dual beam system (combination of a SEM and a focused ion beam [FIB]) can be used.

But additional questions might arise: if a material is exposed to stress, temperature changes, aggressive agents etc., how does the microstructure of the material respond to these strains? *In situ* experiments in the environmental scanning electron microscope enable the simultaneous recording of macroscopic parameters like stress, strain, temperature etc. and of morphological and microstructural changes in the sub-µm range. The correlation of both can provide a deeper understanding of the basic processes.



Figure 1: Experimental setup (home-made) for the characterisation of microfiltration membranes mounted at the stage in the specimen chamber of an ESEM.



Figure 2: Measurement of the temperature changes at the membranes surfaces (top) during wetting and drying of the membrane, with images of the membrane surface at various stages (bottom).



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PhD Student: Mag. Manfred Nachtnebel

Technical Assistants: Daniel Schreiner Sanja Šimić

R&D and Knowledge Transfer

The research of the group comprised, as far as techniques are concerned, *in situ* experiments in the SEM, 3D reconstructions of the inner structure of materials and electron backscatter diffraction. The investigations concentrated in the last two years on the characterisation of polymeric microfiltration membranes, the corrosion of Cobase superalloys and the fracture behaviour of polymers.

Degradation of multi-layered flat sheet microfiltration membranes can happen both during the operation of the membrane and the chemical cleaning of the membranes. Contrary to conventional techniques the new method is able to provide information whether the degradation is homogeneous across the whole cross-section or happens mainly in just one of the layers and in which one.

Cobalt-base superalloys are potential materials for various high-temperature applications. One of the still unsolved problems is their resistance to high temperature corrosion. The very first stages of the oxidation process were investigated by use of *in situ* experiments in the ESEM and EBSD. This enables the continuous observation of the start and progress of the corrosion in dependence on the temperature, the surface roughness and the grain orientation at the sample surface.

Most of the research was performed in cooperation with companies or other university institutes, thus providing knowledge transfer in both directions. The cooperation partners provided mainly materials and related information, whereas we ourselves were active in the development of new characterisation methods and their applications. These new methods should provide a deeper understanding of the inner structure of the material itself and also of possible deterioration mechanisms: how and where does degradation start, how does it develop and how does it finally lead to material failure.

This should open new possibilities for both, the improvement of already existing materials and the design of new materials with tailored properties and new applications. Thus the basic research can hopefully lead to a higher competitiveness of our industrial partners.

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Ing. Hartmuth Schröttner

works at the Institute since 1991. Being group leader in the field of scanning electron microscopy (since 2005) his focus is on the development of preparation techniques and methods in microanalysis. His expertise is directly incorporated into industria' collaborations and applications.

Surface & Materials Characterisation with SEM/IR/Raman

Troubleshooting and Problem Solving

Thanks to our experience, materials know-how and good networking we can act as a solution provider for your problems. We offer a Multi-Scale-Analysis of surfaces and materials with a wide variety of methods from the mm to nm range for quality assurance, current problems and future developments.

Joined Focus on Microanalysis

In the beginning of 2015 the Institute's leadership decided to join the former separated groups SEM-II and IR- & Raman- Microscopy to form one task force focussing on Microanalysis – SEM/IR/ Raman. Using synergy between Scanning Electron Microscopy- and X-Ray-Spectroscopy-Techniques as well as Infrared- and Raman-Microscopy we are able to solve our customer requests with the highest efficiency.

FT-IR- and Raman-imaging techniques were strengthened and became a powerful tool to gain additional chemical and molecular information. In the course of his doctoral thesis Harald Fitzek is doing research on the fundamentals of Surface Enhanced Raman Spectroscopy (SERS). This is a surface-sensitive technique that enhances Raman scattering by molecules adsorbed on rough metal surfaces or by nanostructures. The enhancement factor can be as much as up to 10¹¹, which means this technique may detect even single molecules (see Figure 1).

The work group is working hard to bridge the gap between development, optimisation of preparation methods and versatile microscopy techniques on the one side and their industrial applications to provide a sustainable creation of value and know-how for our cooperation partners on the other side.



Figure 1



Figure 3



Figure 2

EI/E



Figure 4



Group Leader: Ing. Hartmuth Schröttner

Senior Researchers: Dr. Boril Chernev Dr. Johannes Rattenberger Dr. Julian Wagner

PhD Student: DI Harald Fitzek

Technical Assistants: Christian Brandl Sabrina Mertschnigg Anita Rossmann

Research Partner for University

Research projects in cooperation with institutes from universities and competence centres are aligned on Solid Oxid Fuel Cells (SOFC) [1] (Figure 2), lithium ion accumulators [2], various catalysts as well as on different aspects of pharmaceuticalengineering [3] (Figure 3) and soft matter characterisation.

Research Partner for Industry & ACR-Institutes

Being very experienced the work group positioned itself as a reliable and capable cooperation partner in a lot of different R&D projects. For example within the Austrian Cooperative Research (ACR) and its FFG-COIN projects – "Optimatstruct" [4] (Figure 4) and "AMCC" (see brief description S. 39). The projects are focused on generating comprehensive knowledge on modern magnesium- and aluminium-based light-weightalloys. We want to transfer this know-how and expertise to further industrial projects and applications. Currently there are two projects engaged with surface defects, the structure of cast aluminium wheels and the corrosion behaviour of aluminium condensers of automotive air conditioning systems. In November 2016 the joint cooperation ACR laboratory project "Innovative Materials Characterisation" started under the lead of Johannes Rattenberger. In its centre the future possibilities of the Universal Pressure Scanning Electron Microscope (UPSEM). (see brief description S. 38).

Figure 1: Simulation of the electric nearfield strength around Au nanoislands from an AFM image; the red high intensity regions are the origin of the signal enhancement in surface enhanced Raman spectroscopy (SERS).

Figure 2: Secondary Electron (SE) Image of a Broad-Ion-Beam (BIB) Cut through an SOFC electrode showing the formation of carbon nanotubes.

Figure 3: Secondary Electron (SE) Image of a modified glass carrier particle.

Figure 4: Backscatter Electron Image (BSE) Image of a AlCu₄Y₁ alloy.

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Research Highlights

Direct-Write Fabrication of Cellulose Nano-Structures via Focused Electron Beam Induced Nano-Synthesis

SCIENTIFIC REPORTS

Scientific Reports 6, (2016) Article number: 32451

T. Ganner, J. Sattelkow, B. Rumpf, M. Eibinger, D. Reishofer, R. Winkler, B. Nidetzky, S. Spirk, and H. Plank

In many areas of science and technology, patterned films and surfaces play a key role in engineering and development of advanced materials. In this study, we introduced a new generic technique for the fabrication of cellulose nano-structures via focused electron beam induced conversion (FEBIC). For the proof of principle, organosoluble trimethylsilyl-cellulose (TMSC) thin films have been deposited by spin coating on SiO, / Si and exposed to a nano-sized electron beam. It turns out that in the exposed areas an electron induced desilylation reaction takes place converting soluble TMSC to rather insoluble cellulose. While the unwanted TMSC can easily be removed by a fast, wet-chemical step, the converted cellulose patterns remain on the surface. A systematic study on the process parameters revealed an exclusively electron dose dependent behaviour with three working regimes: incomplete conversion, ideal doses and over exposure. These investigations

are complemented by a theoretical model, which suggests a two-step reaction process by means of TMSC \rightarrow cellulose and cellulose \rightarrow non-cellulose material conversion in excellent agreement with experimental data. The extracted, individual reaction rates allowed the derivation of design rules for FEBIC parameters towards highest conversion efficiencies and highest lateral resolution. According downscaling experiments revealed that converted areas below 100 nm can be achieved for ideal settings. By that, this study introduced a new approach for the defined structuring of cellulose with sub-100 nm resolution for the combination with electronic devices, microfluidic arrays, small scale bio-sensors or diagnostic tools. Finally, it should be mentioned that the structuring of chitin based films is now feasible which show a slightly different but highly interesting chemistry66 for fundamental research.



Figure: The FEBIC principle allows the e-beam induced conversion of a cellulose precursor (TMSC) into pure cellulose (a). The nano-sized e-beam together with the high flexibility of its movement allows the fabrication of complex negative (b) or positive (c) cellulose nanostructures with line widths down to the sub-100 nm scale.

Formation of Bimetallic Clusters in Superfluid Helium Nanodroplets Analyzed by Atomic Resolution Electron Tomography

G. Haberfehlner, P. Thaler, D. Knez, A. Volk, F. Hofer, W.E. Ernst, G. Kothleitner

Structure, shape and composition are the basic parameters responsible for properties of nanoscale materials, distinguishing them from their bulk counterparts. Electron tomography, can reveal these parameters in three dimensions at the nanoscale, reconstructing a volume from a tilt series of (S)TEM images. Electron tomography can be advanced to atomic resolution in an aberration-corrected transmission electron microscope, but this remains challenging and requires optimal samples, experiments and data processing methods. Previous demonstrations of atomic resolution electron tomography by other research groups either used prior information about the sample or employed extensive filtering to allow visualizing atomic positions in three dimensions.

This paper demonstrates atomic resolution electron tomography on silver/gold core/shell nanoclusters. These nanoclusters are grown in superfluid helium nanodroplets, a method which allows creating particles from a wide range of materials with high purity. We reveal morphology and composition of a cluster identifying gold- and silverrich regions in three dimensions and we estimate atomic positions without using any prior information and with minimal filtering. With the full three-dimensional information down to the atomic scale we are able to understand the growth and deposition process of the nanoclusters and demonstrate an approach that may be generally applicable to all types of nanoscale materials.



Nature Communications 6, (2015) Article number: 8779



Figure: 3D reconstructions of Ag-Au nanoclusters. Reconstruction showing structure (a) and composition (b) of the cluster. For each reconstruction, a volume-rendered 3D view and three orthogonal 0.23 Å-thick slices through the reconstruction are shown. Scale bars, 2 nm.

Check also the related video: goo.gl/h2LiSU





Nano Letters, 15 (11) (2015), 7726–7730

Correlated 3D Nanoscale Mapping and Simulation of Coupled Plasmonic Nanoparticles

G. Haberfehlner, A. Trügler, F.P. Schmidt, A. Hörl, F. Hofer, U. Hohenester, G. Kothleitner

Plasmonics allow confinement of light at the nanoscale. This is achieved by binding light to coherent electron charge oscillations at the boundary of metallic nanoparticles, so-called particle plasmons, which come together with strong and tightly focused electromagnetic near-fields. While light confinement to extreme subwavelength dimensions holds promise for many applications, it hinders direct observation of the plasmonic nearfields by optical means because of the diffraction limit of light. An alternative measurement scheme is provided by electron energy loss spectroscopy (EELS) in a scanning transmission electron microscope (STEM), where the electrons can lose a small fraction of their kinetic energy through excitation of particle plasmons. In this paper electron tomography in combination with EELS experiments and simulations was used to unravel the interplay between structure and plasmonic properties of a silver nanoparticle dimer. With electron tomography the precise 3D geometry of the particles was reconstructed, and the full three-dimensional information was used as an input for simulations of energy-loss spectra and plasmon resonance maps. Excellent agreement between experiment and theory was found, bringing the comparison between EELS imaging and simulations to a quantitative and correlative level. In addition, interface mode patterns, normally masked by the projection nature of a transmission microscopy investigation, could be unambiguously identified through tomographic reconstruction. This work overcomes the need for geometrical assumptions or symmetry restrictions of the sample in simulations and paves the way for detailed investigations of realistic and complex plasmonic nanostructures.



Figure: 3D reconstructions of the silver nanoparticle dimer (top left). Simulated and experimental energy loss spectra for electron beam positions on the sample and plasmon resonance map for a specific energy loss (right). 3D reconstruction of interface modes (bottom left).

Self-Organized Sr Leads to Solid State Twinning in Nano-Scaled Eutectic Si Phase

M. Albu, A. Pal, C. Gspan, R. C. Picu, F. Hofer, G. Kothleitner

The past decades have witnessed a noticeable increase in research and improvement of light Al-Si cast alloys to be used in domestic, military, automotive, and aerospace components. Given the widespread (80%) use of this alloy system, the optimiszation of the microstructure is a crucial matter to ensure superior mechanical properties for the as-cast finished products. Of particular importance are the nucleation and growth mechanisms of the eutectic Si phase, which can be manipulated by changes in the impurity concentration – by bringing in atomic modifiers such as Ba, Ca, Na, Sc or rare earth elements - or kinetics of the eutectic reaction. Several theories have been proposed to explain the mechanisms leading to refinement and twinning, but none of them is able to conclusively explain microstructural refinement and twinning induced by all atomic modifiers or trace impurities.

This paper proposes a new mechanism for the twin nucleation in the eutectic Al-Si alloy with trace Sr impurities. Observations made by subangstrom resolution scanning transmission electron microscopy and X-ray probing proved the presence of <110> Sr columns located prreferentially at twin boundaries. Density functional theory simulations indicate that Sr atoms bind in the Si lattice only along the <110> direction, with preferential positions at first and second nearest neighbours for interstitial and substitutional Sr, respectively. Density functional theory total energy calculations confirm that twin nucleation at Sr columns is energetically favorable. Hence, twins may nucleate in Si precipitates after solidification, which provides a different perspective to the currently accepted mechanism, which suggests twin formation during precipitate growth.



Scientific Reports; 6: 31635 (2016)



Figure: (left) HAADF STEM high resolution image of an eutectic Si particle tilted in the [110] zone axis. Yellow arrows indicate first order twins in $\{111\} < 112>$ direction associated with Sr columns (high intensity at interstitial position). (right) Charge density difference plot for the Si-Sr system, with Si atoms (blue) and a <110> interstitial Sr column (marked by a red dot in the center of the image) in the {110} projection. The charge perturbation is aligned with the <1–12> direction, while atomic displacements (red arrows) indicate incipient twinning.

0	DURINE OF INMOTOLINGUIGH
	ocused particle beam- induced processing

Beilstein J. Nanotechnology, 6 (2015) 462–471

Fundamental Edge Broadening Effects during Focused Electron Beam Induced Nano-Synthesis

R. Schmied, J.D. Fowlkes, R. Winkler, P.D. Rack, and H. Plank

Focused Electron Beam Induced Deposition (FE-BID) is known as a mask-less, direct-write technology, which allows fabrication of functional nanostructures with spatial nanometer resolution. Although powerful in its capabilities, the working principle is rather complex as the electrons can travel in manifold ways through the deposit and the substrate. In this study, we explored the origins of highly unwanted, lateral broadening effects in solid 3D structures. In particular, the scaling behaviour of proximity effects as a function of primary electron energy and the deposit height itself was investigated by experiments and validated by simulations. In addition, correlated Kelvin force microscopy and conductive atomic force microscopy measurements identified conductive and non-conductive proximity regions. The essential finding of this study was the fundamental

understanding of edge broadening formation, which implied that a certain degree of side wall growth is absolutely unavoidable during FEBID. On the other hand, we could demonstrate that there exists always a parameter window, which lead to strong side wall convolution induced by the deposit footprint itself and therefore should be entirely avoided. By combining these findings, we could derive a strategy in dependency on FE-BID process parameters which allows to estimate the expected morphological (non-functional) and functional (fully functional) side wall broadening tendencies. By that, the edge sharpness can be tuned to the desired application, which is indispensably needed when aiming for very small and defined structures fabricated via FEBID.



Figure: The left scheme gives a cross section of a deposited structure (grey) fabricated on the a solid substrate (green). The arrows indicate the manifold electron trajectories which can ultimately lead to additional deposition events (encircled red). The study investigated the unwanted side wall broadening (black curve around the grey deposit) in dependency on the most important FEBID parameter as summarized in the right graph. Additionally, we could successfully divide the broadening in non-functional, morphological and fully functional broadening effects (see legend) which acts as strategy guide during FEBID based fabrication towards most precise micro- and nano-fabrication.
Towards Ultraflat Surface Morphologies during Focused Electron Beam Induced Nano-Synthesis: Disruption Origins and Compensation

R. Winkler, A. Szkudlarek, J.D. Fowlkes, P.D. Rack, I. Utke, and H. Plank

Emerging applications for nanoscale materials demand precise deposit shape retention from design to deposition. This study investigated the effects that disrupt high-fidelity shapes during focused electron beam induced deposition (FEBID). We have shown in detail how process parameters, patterning strategies and deposit shape itself can induce unwanted topographic artefacts based on lateral precursor coverage gradients during FEBID. The study could successfully classify the evolving surface shapes into four general types by means of 1) flat, 2) concave, 3) slanted and 4) patterning dependent. The latter can be further categorized into different shapes such as trenches, chair-like and tunnel-shapes for spiral, raster and serpentine scanning, respectively. In addition, continuum model calculations and simulations expand the experimental results to provide a comprehensive insight to understand the disruption mechanism. The findings demonstrate that the wellestablished concept of growth regimes has to be expanded by its lateral gradients as they strongly influence final shape fidelities. Finally, the study is complemented by a compensation strategy, which improves the edge fidelity on the lower nanoscale to further push this technique towards the intrinsic limitations. By that, the study paves the way towards high-fidelity fabrication of functional nano-structures via FEBID on the lower nanoscale, which is of decisive importance specifically for applications such as resonant optics or electronic nanostructures.



ACS Applied Material Interfaces, 7 (5) (2015) 3289–3297

spatial gradients of the growth regime



Figure: In FEBID, the surface morphology of functional deposits are often disrupted due to spatial precursor gradients (left) of by the applied fabrication strategy (right). The detailed investigations of this study provided a fundamental insight in the dynamic formation of unwanted surface disruption, which in turn allowed to overcome these issues. The results are highly defined deposits as shown in the center with surface height variations in sub-nm range as indispensably required for diverse applications.



Ultramicroscopy, 174 (2017) 1–7

Transformation Dynamics of Ni Clusters into NiO Rings under Electron Beam Irradiation

D. Knez, P. Thaler, A. Volk, G. Kothleitner, W. E. Ernst, F. Hofer

We report the transformation of nickel clusters into NiO rings by an electron beam induced nanoscale Kirkendall effect. The Kirkendall effect generally denotes void formation due to diffusivity differences at solid interfaces. Usually this effect is thermally activated, but the activation energy for the oxidation can also be provided by highenergy electrons, enabling localized triggering of the process even at non-elevated temperatures in the electron microscope.

We used high-purity nickel clusters consisting of a few thousand atoms as precursors, which were synthesized with the superfluid helium droplet technique at the Institute of Experimental Physics of the TU Graz. We applied aberration-corrected, analytical scanning transmission electron microscopy to oxidise and simultaneously analyse the clusters. We studied the transient dynamics of the oxidation by time-lapse series using high-angle annular dark-field imaging and electron energyloss spectroscopy. In our work we identified a two-step oxidation mechanism and we found that water adsorbed adjacent to the clusters acts as oxygen source for the electron beam induced oxidation. With quantitative EELS measurements combined with molecular dynamics simulations, we were also able to estimate a size-dependent oxidation rate. Our findings could serve to better control sample changes during examination in an electron microscope, and might provide a methodology to generate other metal oxide nanostructures.



Figure: (a) Selected images of the time-lapse series from particle 1 in Fig. a, showing the transformation to a hollow and finally ring-shaped NiO cluster. (b) Corresponding illustration of the transformation process according to the Kirkendall effect. An electric field inside the oxide drives the diffusion of Ni cations to the NiO surface and O anions to the Ni/NiO interface, while voids aggregate in the cluster centre.

Check also the related video: goo.gl/mWzQXA



Spatial Localization of Membrane Degradation by *in situ* Wetting and Drying of Membranes in the Scanning Electron Microscope

M. Nachtnebel, H. Fitzek, C. Mayrhofer, B. Chernev, P. Pölt

Sustainable development is a major challenge in all fields of technology, including water treatment and health management, leading to progressive innovation in separation membrane development and production. A widely used membrane type are flat sheet microfiltration membranes. They are often based on polymers or their blends, because of their wide range of applications and their excellent resistance to many physical and chemical treatments. Nevertheless, regular membrane cleaning is necessary, which often leads to membrane degradation and thus a gradual reduction in performance. In our study we investigated the impact of three typical cleaning agents on multilayered PES/PVP (polyethersulfone/ polyvinylpyrrolidone) membranes used for water filtration by *in situ* wetting and drying experiments in an environmental scanning electron microscope (ESEM). It has been proven that this method enables the detection of membrane degradation and is able to identify the layer which is most affected by these chemical treatments. The results were verified by Fourier transform infrared spectroscopy (FT-IR) and a method based on the imbibition of water droplets at the membrane surface. This ESEM based method is the only method that involves a large sample volume and can thus provide statistically significant results.



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Figure: *In situ* ESEM investigation revealed a distinct decrease in drying time in the region responsible for the drying at the membrane surface after the treatment with NaClO, shown in figure a). Figure b) shows the PES/PVP distribution over the cross-section of a pristine membrane obtained by FT-IR. The appropriate result for a treated membrane is shown in c).



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Synergy of SEM and Ultramicrotomy SBEM Spreading from Life Sciences to Materials Science

A. Zankel, M. Nachtnebel, S. Wernitznig

In 2004 a groundbreaking paper was published by Denk and Horstmann introducing a new technique called serial block-face scanning electron microscopy, abbreviated SBFSEM or SBEM. Here an ultramicrotome is mounted in the specimen chamber of a scanning electron microscope which enables the investigation of electrically non-conductive samples, either a variable-pressure scanning electron microscope (VPSEM) or an environmental scanning electron microscope (ESEM). Automated slicing and imaging of the block-face of a specimen is performed using a diamond knife for cutting. This serial imaging leads to a stack of micrographs which are used for the 3D reconstruction of specimens. The first decade of SBEM was primarily characterised by publications in the field of life sciences. As an example from neuroscience a 3D reconstruction of a bundle of neurons of the visual system of a grasshopper is shown in

Fig. 1 (cooperation between FELMI-ZFE and Medical University of Graz). In 2009 first results in materials science were presented by FELMI-ZFE authors, describing applications on different soft matter materials. Fig. 2 shows several steps in the 3D analysis of a polymer blend (polypropylene with EPR particles) after a tensile test. Furthermore SBEM is capable of generating 3D images of different engineering materials at nanoscale resolution over large volumes. Fig. 3a shows the block face of an aluminium specimen and Fig. 3b a 3D reconstruction delivering e.g. the distribution of precipitates. Fig. 3c shows schematically the setup of the SBEM system. While imaging is typically done with the BSE detector, the combination of SBEM and energy dispersive x-ray spectroscopy (EDS) was first published as 3D-elemental mapping in 2011, again by authors from the FELMI-ZFE.





Figure 1: 3D reconstruction of neurons in the grasshopper's visual system. The different colours correlate with different single neurons. At the bottom a typical block face with several cells can be seen (the rear edge of the micrograph has a total length of 25 μ m)

Figure 2: 3D reconstruction of rubber particles (yellow), the main crack (blue) and the crack distribution (light blue) after a tensile test ; (a) original SEM images, (b) segmented slices, (c) 3D reconstruction, (d) distance map of the filler particles (volume: $20.5 \times 20.5 \times 10.0 \ \mu\text{m}^3$) **Figure 3 :** (a) The block-face of an aluminium specimen, (b) 3D reconstruction (unit: microns), (c) Setup of the SBEM system

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TEM Investigation of Multi-Walled Hollow Fibres Produced by Tri-Axial Electrospinning

I. Letofsky-Papst, B.S. Okan, J.S.M. Zanjani, M. Yildiz, Y. Menceloglu, W. Grogger

Hollow structured nanofibers with exceptional properties such as low density, high specific surface area, and tuneable surface properties have found essential applications as catalyst supports, drug delivery systems and membranes. Different production techniques were reported for production of hollow structured fibres. In a first approach, conventional electro-spun polymeric fibres, utilized as template followed by coating the electro-spun fabric and removal of the template, were used to fabricate hollow structured fibres with different wall materials. The limiting factors of this method are complexity in coating and efficiency of the template removing processes. The second method is the co-axial electrospinning process, where core-shell fibres are produced by using two different solutions followed by a selective removal of the core material which also results in hollow structured fibres. Herein, the multi-axial electrospinning technique was employed to produce hollow structured fibers with different wall materials in a single step process without any post treatments. In the fabrication process of multi-axial electro-spun nanofibers, a strong electric field is applied between a nozzle containing concentric tubes allowing for the extrusion of different fluids to tip of the nozzle and grounded metallic plate as a collector. The diameter, surface morphology and layered structure of multi-walled hollow electrospun fibres are controlled by tailoring the solvent properties, degree of miscibility of solutions, polymer concentration, applied voltage, electrospinning distance, and flow rate.

In order to be able to show that the hollowness and structural integrity of the fibers can be controlled, we have investigated tri-axial electro-spun fibers using two different outer layer polymers, namely PMMA and PS while keeping the inner layer material the same, PAAm. The TEM image in Figure 1a) shows a PMMA/PAAm hollow fiber with a diameter of about 330 nm, where the inner part – corresponding to a hollow core – is about 125 nm in diameter. The fiber (500 nm in diameter) in Figure 1b) shows that using PS as an outer layer instead of PMMA leads to an increase of the inner diameter up to 260 nm.

From these results we can conclude the diameter of hollowness can be adjusted by changing the type of polymer in the outer layer. This controllability can provide an easy encapsulation of functional materials with different viscosities if required, and increase the life-time of encapsulated materials through circumventing leakage.

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Figure: a) TEM image of PMMA/PAAm multi-walled hollow fiber; b) TEM image of PS/PAAm multi-walled hollow fiber.



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High-Quality Imaging in Environmental Scanning Electron Microscopy – Optimizing the Pressure Limiting System and the Secondary Electron Detection of a commercially Available ESEM

H. Fitzek, H. Schröttner, J. Wagner, F. Hofer, J. Rattenberger

In Environmental Scanning Electron Microscopy (ESEM) applications in the kPa regime are of increasing interest for the investigation of wet and biological samples, because neither sample preparation nor extensive cooling are necessary. Unfortunately, the applications are limited by poor image quality.

In this work, the image quality at high pressures for the FEI ESEM Quanta Series Line is greatly improved by optimizing the pressure limiting system and the secondary electron (SE) detection system.

The scattering of the primary electron beam strongly increases with pressure and thus the image quality vanishes. A new aperture holder is presented that significantly reduces the amount of imaging gas, which must be overcome by the primary electron beam on the way to the sample. It is possible to achieve considerable improvements in image quality while maintaining the same field-of-view as the original design. By decreasing the pressure limiting aperture size, the available pressure range can be extended at the expense of a smaller field-of-view. With increasing pressure the amplification efficiency decreases because secondary electrons do not gain enough energy between collisions to ionize the gas. However, nearby a blade or needle detector with very small tip radius ($R < 10 \mu m$) the electric field is strong enough for amplification even at very high chamber pressure and by positioning the needle on the sample table it operates at ideal conditions regardless of pressure and working distance. A by-product of this design is that the conventional position of the backscatter electron detector (BSE) at the end of the column is no longer blocked by the SE detector and secondary electron and backscatter images can be simultaneously obtained. The figure shows a comparison between the original design (left) and the modified system (right). It can clearly be seen that low acceleration voltages are necessary to be surface sensitive but only with the optimized system an adequate image quality can be achieved.

With this optimized high pressure design the limits of conventional ESEM technology can be crossed. Imaging at higher chamber pressures, investigations of wet samples and living organisms and much more are possible.



Figure: Secondary electron micrographs of a MicroPES 2F membrane (Membrana) Left: FEI [GSED + Bullet] Right: Needle Detector (100 μ m radius) Aperture Holder (200 μ m PLA1) Microscopy Parameters: SE; 800 Pa H₂O; I ~ 0,8 nA; Pixel: 1024*943; Frame Time 9,47 s

Effect of Trace Elements on the Material Properties of an Aluminium Casting Alloy

H. Schroettner, A. Rossmann-Perner, S. Mertschnigg, T. Pabel, T. Petkov

The aim of this project is to determine the fundamental effects of impurities and individual micro-alloying elements (eg. vanadium, titanium, calcium, zirconium, potassium, phosphorus) or combinations of these trace elements in aluminum alloys and their impact on the quality of aluminum castings. As a result, limit values and tolerances for individual impurities (trace elements) are defined and determined. Further practical study methods are developed to support a reliable series production of high-quality alloys and castings on the one hand and the procurement of aluminum alloys by the foundries on the other hand.

The examinations were performed on the base of a high purity alloy AlSi7Mg0.3 with systematic addition (30 ppm, 300 ppm, 3000 ppm) of microalloying elements.

Based on phase calculations by ThermoCalc-Software the formation of intermetallic phases and their impact on the microstructure were

analysed by virtual additions of trace elements. Subsequent casting trials in industry-related standards with systematic addition of micro-alloying elements were performed. ZFE Graz made SEM/ EDXS/EBSD-measurements for the micro-characterisation of the structure; the composition of the intermetallic phases are made visible via cross sections (Figures 1, 2). Out of phases of interest TEM-lamellas are prepared with the FIB-technique (Figure 3) and transferred to TEM for using STEM/EDXS/EELS/EFTEM and diffraction methods for the nano-characterisation of these phases (Figure 4). The examinations show a significant correlation between the thermodynamic calculations and the actual casting trials close to industrial conditions. The casting characteristics worsen significantly with an increase of trace elements.



EMC 216, Lyon



Figure 1



Figure 2



Figure 1: Backscattered Electron (BSE) Image of AlSi, Mg doped with 3000 ppm zirconium - diagonal crossed (St. Andrew's cross shaped) Al₂SiZr-phase

Figure 2: Backscattered Electron (BSE) Image of AlSi⁷Mg_{0.3} doped with 300 ppm zirconium. Fine needles form this coralliform shaped Al3Zr-phases. The blue marker shows the area for the FIB-lamella.

Figure 3: HAADF image of the FIB-lamella with the blue marked area for HREM.

Figure 4: STEM HAADF-image of the eutectic Si-phase with twins.



Aberration Correction in Electron Microscopy

Being able to see and analyse materials at atomic resolution, has been a strong motivation for improving the electron microscope. However, the resolution of the conventional transmission electron microscope (TEM) was limited to around 0.2 nanometre, which is slightly larger than the diameter of atoms. This limitation comes from the spherical aberrations of the magnetic lenses, which lead to a blurring of images. Although this deteriorating effect was already recognised 80 years ago, it was only in the late 1990s that the first spherical aberration corrector improved the resolution of the TEM. The following development of commercial aberration-corrected TEMs was a revolution for microscopy research and helped to answer many open questions in physics, chemistry and materials and biological sciences.

The Challenge in Austria

Immediately after their commercial introduction in 2005 aberration corrected TEMs became the standard in high resolution electron microscopy. However, due to high costs there was no realistic chance to establish such an advanced instrument in Austria. Finally, the Association for Electron Microscopy and Fine Structure Research succeeded in 2011 via a complicated project mixture, which was supported by several funding organisations (FFG, ACR & BMWFW, Land Steiermark, WKO-STMK) and the Graz University of Technology. This breakthrough allowed us to build one of the best aberration corrected microscopes in the world – delivered by FEI Company (Eindhoven, The Netherlands) – the Titan cubed 60–300 (Austrian Scanning Transmission Electron Microscope = ASTEM).

The Operation of the ASTEM

The Austrian Scanning Transmission

In order to exploit all the benefits of the new instrument we had to improve the quality of our specimen preparation and to deal with theoretical modelling of the experimental results. Firstly, it was necessary to introduce a new method for low-energy ion milling (Nanomill, Fischione). Secondly, the atomically resolved STEM-images had to be interpreted with the help of advanced simulation tools such as QSTEM (Christoph Koch, Berlin, Germany) or μ STEM (Lesley Allen, Melbourne, Australia); collaborations in the field of first principles calculations have been started (Catalin Picu, Troy, USA).



Scientific impact of the ASTEM: Peer-review publications of the FELMI-ZFE (2011-2016).

Electron Microscope – The Story Behind

The Impact of the ASTEM

With the ASTEM the Institute has become the leading research institution in the field of advanced materials microscopy in Austria. The ASTEM plays a major role in the research of the FoE Advanced Materials Science of the TU Graz and increasingly also for the NAWI faculty of the University of Graz and TU Graz. Indeed, it developed into a national resource with intensive collaborations with the Universities of Graz, Leoben, Linz, and Innsbruck. Its impact on research collaborations with Austrian companies is manifold, particularly for semiconductor, ceramic and metal industries. Finally, it enabled the access to the leading European research network on advanced electron microscopy (ESTEEM2, FP7).

Main Results

Quantitative analysis at atomic resolution

Elemental mapping using EELS and EDXS in a STEM, a well-established method for precision elemental concentration analysis at nanometre level, was firstly demonstrated at atomic resolution in 2010. However, until 2015 these atomically resolved elemental maps have only been interpreted qualitatively, because elastic and thermal scattering of the electron probe confound quantitative analyses. Thanks to the work of Gerald Kothleitner elemental maps could be quantified. He was one of the first to perform absolute scale comparisons between simultaneous EELS and EDXS-experiments and quantum mechanical calculations thus yielding absolute volumetric concentrations at atomic resolution. Werner Grogger and his team study the influence of the four-quadrant X-ray detector (Super-X) of the ASTEM on the quantification of X-ray elemental mapping.

 Imaging of crystals at atomic resolution (aquamarine)

The mineral beryl is a precious gem exhibiting a wide range of colours ranging from the green emerald and the blue aquamarine to the yellow heliodor. These colours are connected with the complex crystal structure of beryl exhibiting crystal channels along the hexagonal axis. Although it is well known that these channels can be filled with various molecules and ions, however, it was only recently possible to image the crystal structure of beryl at atomic resolution. In 2015, Christian Gspan investigated an aquamarine sample with the ASTEM showing the individual atom columns (Al, Si and O) and even revealing the channel constituents such as water molecules for the first time.

The Future of the ASTEM

The ASTEM is surely a stable platform for the years to come, nevertheless upgrades and extensions are mandatory. Presently, we are working on projects for funding the introduction of new methods such as magnetic and electric imaging via differential phase contrast and radically improved detectors for imaging and spectroscopy (direct electron detection).



Aluminium EDS map of a Al/Sc structure (simulation), revealing less Al atoms at the defect side than expected.

Collaborations: Lesley Allen and co-workers, University of Melbourne, Australia; Budhika Mendis, Durham University, U.K



STEM-HAADF image of Beryl viewed in [001] direction, Al-columns appear as bright dots. Collaboration:

Karl Gatterer, Institute of Physical and Theoretical Chemistry, TU Graz. Collaborations: ams, Premstätten; AT&S, Leoben-Hinterberg; EPCOS, Deutschlandsberg; LAM Research, Villach; Infineon Technologies, Villach.



STEM-HAADF image of the interface between Si and SiGe

Collaborations: Ulrich Hohenester, Joachim Krenn, Institute of Physics, University of Graz; Cecilia Poletti, Institute of Materials Science and Welding, TU Graz; Mathieu Kociak, University of Paris-Sud, France.



3D-reconstruction of core-shell precipitates in an AIMg alloy



Tracking of a platinum atom on the surface of a thin silicon crystal

• Interfaces in semiconductors

Interfaces formed between semiconductors, metals and oxides are of crucial importance to solid state technology. A general problem is the continuous shrinkage of semiconductor devices, in which critical device dimensions already may approach the size of an atom. Therefore, it is important to understand the precise atom arrangements at interfaces and how they influence device properties. Thanks to its excellent resolution, the ASTEM is the ideal tool for studying semiconductor device interfaces. Evelin FissIthaler took up this challenge and established a research collaboration with leading Austrian microelectronic companies with a focus on the structural and chemical characterisation of special semiconductor interfaces.

Imaging of nanostructures in three dimensions

TEM or STEM images are only two-dimensional (2D) projections of three-dimensional (3D) objects. In order to reveal the real structure, we have to acquire a tilt series of TEM or STEM images and combine them in a three dimensional reconstruction. This method is called electron tomography and has been introduced at the institute by Gerald Kothleitner and Georg Haberfehlner. Thanks to the excellent stability of the ASTEM and the very advanced spectroscopic methods included, it was possible to record the crystal structure of an individual nanoparticle atom-by-atom. Recently, our tomographic methods were extended to study the photonic environment of plasmonic nanoparticles at sub-nanometre resolution.

• In situ studies of nanoparticles and moving atoms

Time-resolved experiments using the aberrationcorrected STEM allow direct observations of atomic motions. Recent examples from literature stay with a qualitative description of atom motions induced by the electron beam. However, in a breakthrough experiment, Daniel Knez tracked single Pt atoms on the surface of a very thin silicon crystal. Collaborating with Paul Midgley from Cambridge, we presently analyse the surface dynamics of the ad-atoms in a quantitative way, thus revealing all the influences from absorption, reactions and diffusion. The mechanisms observed may also inspire new ideas for electron beam nanostructuring.

Collaborations:

Paul Midgley, University of Cambridge, U.K.; Wolfgang Ernst, Institute of Experimental Physics, TU Graz; Asuncion Fernandez, CSIC e Univ. Sevilla.

Atomically resolved STEM investigations of alloys and steels

The properties of structural materials strongly depend on the occurrence and distribution of secondary phases, i.e. impurities, precipitates and grain boundary phases on a microscopic scale. With the rise of aberration corrected (S)TEMs it became immediately clear that it will open radically new insights especially for aluminium cast alloys. Mihaela Albu and Angelina Orthacker were successful with atomic resolution studies of precipitates in Al-Si cast alloys and Mg-alloys. They revealed the twinning mechanism in Sr-doped Al-Si cast alloys and explained the self-stabilization of core-shell precipitates in AlMgSc-alloys. However, until recently, high resolution studies of steels have been very rarely reported in literature. Here we have been successful with one of the first atomically resolved STEM investigations of precipitates in a high chromium steel.

Surface plasmon polaritons

Electron energy-loss spectroscopy (EELS) has recently emerged as the ideal method for the study of plasmonic nanoparticles and nanostructures. When high energy electrons pass or penetrate through a noble metal nanoparticle, surface plasmons are excited. They can be observed only in TEM/STEM systems, which are equipped with a monochromator for the electron source and a high resolution imaging filter/spectrometer. This is the case for the ASTEM and in combination with its high brightness gun we have excellent conditions for studying surface plasmons at high spatial and high energy resolution. In the last two years we have been using the ASTEM for studies of plasmon modes of a silver thin film taper and a detailed investigation of edge mode coupling within rectangular nanoparticles.



STEM-HAADF image of a precipitate in Al-Si alloy; bright spots are Ag- and Cu rich atom columns.

Collaborations: Gerhard Schindelbacher, Austrian Foundry Institute (ÖGI), Leoben; Christof Sommitsch, Institute of Materials Science and Welding, TU Graz; Elisabetta Gariboldi, Politecnico Milano, Italy; Jiehau Li, Institute of Casting Research, MU Leoben.



STEM-EELS investigation of a silver nanostar with surface plasmon polaritons. Collaborations: Ulrich Hohenester, Joachim Krenn, Institute of Physics, University of Graz; Mathieu Kociak, University of Paris-Sud, France.

Research Grants

Peer-reviewed research grants acquired or active from 2015 to 2016.

FELMI Projects

European Network for Electron Microscopy (ESTEEM2)

FELMI-ZFE leader: Ferdinand Hofer Coordinator: Etienne Snoeck, CEMES-CRNS, Toulouse, France EC-FP/ Infrastructures, Brussels, 10/2012–9/2016

Modelling of a Collision Sensor

FELMI-ZFE leader: Peter Pölt Coordinator: Gerd Leitinger, Medical University of Graz Provincial Government of Styria, Graz, 9/2012– 8/2015

Cellulose

FELMI-ZFE leader: Harald Plank Coordinator: Bernd Nidetzky, Institute of Biotechnology and Biomedical Engineering, TU Graz Austrian Science Fund (FWF), Vienna, 7/2012– 10/2015

Nanolithography

FELMI-ZFE leader: Harald Plank Coordinator: Joachim Krenn, Institute of Physics, University of Graz HRSM-project of the Federal Ministry of Science, Research and Economy, Vienna, 1/2014–12/2018

Cooperation Transmission Electron Microscopy

FEMI-ZFE leader: Ferdinand Hofer Coordinator: Kurt Hingerl, Center of Surface and Nanoanalytics, University of Linz HRSM-project of the Federal Ministry of Science, Research and Economy, Vienna, 1/2014–12/2018

Aluminium and Magnesium Processing Optimization (AMOREE)

FELMI-ZFE leader: Stefan Mitsche Coordinator: Austrian Institute of Technology (AIT), Vienna K-project COMET, Austrian Research Promotion Agency (FFG), Vienna, 7/2014–6/2018

Self-Sensing Nanoprobes for Electric and Thermal *in situ* Characterization in Electron Microscopes (SENTINEL) Leader: Harald Plank

Coordinator: Institute of Electron Microscopy and Nanoanalysis (TU Graz), Produktion der Zukunft 11. AS CHINA CAS Austrian Research Promotion Agency (FFG), Vienna, 10/2015–9/2017

ZFE Projects

Optimization of the Environmental SEM (ESEM)

Leader: Johannes Rattenberger Coordinator: ZFE Graz Austrian Research Promotion Agency (FFG), Vienna, 3/2013–6/2015

Quantitative Electron Microscopy of Hard Metals

Leader: Gerald Kothleitner Coordinator: ZFE Graz Sandvik, Stockholm, Sweden, 10/2013–9/2017

In situ Infrastruktur für die mikroskopische Materialforschung

Leader: Ferdinand Hofer Coordinator: ZFE Graz Austrian Cooperative Research (ACR) and Federal Ministry of Economy, Family and Youth (BMWFJ), Vienna, 11/2015–11/2016

TRIPLE-S

Leader: Harald Plank Coordinator: ZFE Graz FP7 EUROSTARS and Austrian Research Promotion Agency (FFG), Vienna, 10/2013–9/2016

Bio Scanning Atomic Force Microscope (Bio AFM)

Leader: Harald Plank Coordinator: ZFE Graz Styrian Business Promotion Agency (SFG), Graz, 5/2014–4/2015

Gefügeabhängige Verarbeitungs- und Applikationseigenschaften innovativer Leichtbausysteme (OPTIMATSTRUCT)

FELMI-ZFE leader: Hartmuth Schröttner Coordinator: Austrian Foundry Research Institute (ÖGI), Leoben Austrian Research Promotion Agency (FFG), Vienna, 3/2013–2/2017

Development Competence Center for Quality Control of Aluminium Melts (AMCC)

FELMI-ZFE leader: Hartmuth Schröttner Coordinator: Austrian Foundry Research Institute (ÖGI), Leoben Austrian Research Promotion Agency (FFG), Vienna, 3/2013–2/2017

Quantitative Analyse innerer Grenzflächen

Leader: Evelin FissIthaler Coordinator: ZFE Graz Austrian Research Promotion Agency (FFG), Vienna, 4/2015–9/2017

Innovative Materialcharakterisierung

Leader: Johannes Rattenberger Coordinator: ZFE Graz Austrian Cooperative Research (ACR) and Federal Ministry of Economy, Family and Youth (BMWFJ), Vienna, 11/2016–11/2018

Seltenerdnickelate für zukünftige Energietechnologien (SENTECH)

FELMI-ZFE leader: Werner Grogger Coordinator: Montanuniversität Leoben (MUL), Energieforschung e!mission 2.AS Austrian Research Promotion Agency (FFG), Vienna, 3/2016–2/2019

Solarzelle trifft Batterie: Realisierung eines Hybrid-Energiesystems (SOLABAT)

FELMI-ZFE leader: Ferdinand Hofer Coordinator: Institute of Chemistry and Technology of Materials (TU Graz), Austrian Research Promotion Agency (FFG), Vienna, 3/2016–2/2019

Trilple-S Microscope

Harald Plank

During the last decade, the field of advanced microscopy shows strong trends towards correlated analyses by combining different but complementary microscopic and spectroscopic approaches for a comprehensive insight into material properties down to the lowest nanoscale. In the large pool of microscopy, scanning electron microscopes (SEM) represent a particularly important class of instruments as it provides resolution down to the nanoscale together with the capability to identify material with laterally resolved character. On the other hand, the application of focused ion beams (FIB) allows to open up surfaces in a highly localized manner, which makes subsequent analyses via electron beam possible. By that, such combined dual beam microscopes (DBM) allow surface, sub-surface or even 3D analyses of materials in a unique way. While meanwhile well established in research and development, such DBMs approach their intrinsic limitations in situations where a quantitative height information is indispensably required. In this context, atomic force microscopy (AFM) is the complementary technique of choice as it provides true 3D metrology with sub-nm resolution. The problem, however, is that there are only very few systems which combine all three microscopes within one system without limiting their individual capabilities. Based on this motivation, the FELMI-ZFE started the EU EUROSTARS project TRIPLE-S in 2013 together with the Vienna AFM company GETec Microscopy GmbH and further experts in Switzerland and Bulgaria. The aim of this project was to fuse together three of the most powerful high-resolution microscopes. In essence, the TRIPLE-S microscope is a flexible platform, which allows all SEM based studies including the chemical analyses, further complemented by AFM based 3D morphology, phase mode imaging, nano-mechanical analyses and functional characterisation such as conductive-AFM. Furthermore, the application of the FIB together with the AFM high-speed capabilities enables true 3D tomography to access 3D material properties, which are complicated or even impossible to extract from SEM / FIB investigations. By that, the here anticipated TRIPLE-S microscopes opens up new possibilities in the area of correlated in situ nananalysis in yet unknown ways.



Figure: The Triple-S microscope is a flexible platform which combines a scanning electron microscope (SEM, e-), a focused ion beam (FIB, Ga2+) and an atomic force microscope (AFM) without limiting their individual capabilities. By that, this platform provides individual access to different information such as SEM imaging, chemical analyses via energy dispersive X-ray scattering (EDX), 3D height information (AFM), qualitative and quantitative mechanical properties (phase, mechanical) or functional material parameters (conductive). Furthermore, functional nanofabrication using the FIB sputtering or particle induced nano-fabrication can be combined with the strengths of AFM in a straightforward manner.

Quantitative Analysis of Interfaces

Evelin Fisslthaler

The FFG-funded project "Quantitative Analyse innerer Grenzflächen/Quantitative analysis of internal interfaces" is a cooperative research project that unites scientific challenges from five companies – ams, AT&S, EPCOS, Lam Research and Infineon – in order to forward both, knowledge about the microstructure of electronic devices and methods for quantitative nanoanalysis using the highly advanced TEM infrastructure available at the ZFE.

The scope of this project is the high-resolution analysis of internal interfaces in multilayer materials for electronic devices via aberration corrected STEM combined with HR EELS and EDS. For this purpose, a variety of different approaches for both, data acquisition and data analysis, is consequently refined to provide reliable and reproducible datasets with high accuracy in spatial and energetic resolution as well as in terms of quantitative reliability. At the same time, TEM sample preparation methods are sufficiently enhanced and modified to provide specimens with adequate quality.

One of the project's aims is the detection and analysis of interfacial layers, ranging from a few atomic layers to the sub-monolayer dimension. The properties and expanse of these transition regions are of major interest, since both can be crucial to device performance. Therefore, the EELS and EDS signals of the materials of interest are traced with high spatial and energetic resolution to yield detailed information about the chemical composition and the structure of those few atomic layers that form the interface between two different layers. For the subsequent processing of the acquired data, various signal optimisation and fit procedures are refined, finally leading to a more detailed comprehension of the nature of transition regions.



Figure: Atomically resolved HRSTEM image of an interface between silicon (bottom layer) and germanium (top layer), showing the effect of the slight discrepancy between the two materials lattice parameters – the germanium lattice in the upper part of the image shows crystal defects that were formed during deposition to compensate for the mismatch.

Aluminium and Magnesium Processing Optimization (AMOREE)

Stefan Mitsche

In this project a consortium of six scientific partners (LKR, ETH Zürich, TU Graz, TU Wien, University of Salzburg and University of Rostock) and seven companies (AMAG casting, AMAG rolling, HAI, HPI, Magna Steyr, MAS and non ferrum) focus on the research and development of two light metals: aluminium and magnesium. The goal of our part in this project is to investigate the so called roping effect in cooperation with AMAG rolling and LKR. This effect is relevant for Al-Mg-Si alloy sheets that are to be used for outer car bodies. Roping manifests itself as a series of macroscopic ridges and valleys on the surface. These valleys and ridges are still visible after painting, so that such sheets cannot be used for exterior automotive parts just for visual effects. The influence on mechanical properties can be neglected. As starting point two surfaces of aluminium alloy sheets were investigated by electron backscattered diffraction (EBSD) in the scanning electron microscope: one which tends to show roping and one without roping. The EBSD orientation maps are displayed (see Figure) and nicely depict the correlation between crystal orientation and appearance of roping. The surface of the "bad roping" sample (top) shows strips of similar crystal orientation whereas the "good" sample has no such areas. In further steps, cross sections at different states of the rolling process were investigated to establish a correlation between the process parameters and the developed texture of the sheets. At the end of this project part it should be possible to simulate the microstructure and texture of a rolled aluminium alloy sheet in dependence of the rolling parameters and to be able to predict roping probability of this sheet.



Figure: Inverse pole figure map of the surface of two different sheets with (top) pronuonced roping and (bottom) no roping. (sheetplane correspond to rolling direction - vertical, colour code refers to the crystal direction parallel to the normal direction.

Rare Earth Nickelates for Future Energy Technologies (SENTECH)

Christian Gspan

The SENTECH project is in collaboration with the chair of physical chemistry, Montan University Leoben (MUL) and the Max-Plank-Institute for solid state research (MPI) in Stuttgart.

The aim of the project is to get new substituted rare earth nickelates with better mass and charge transport properties with regard to applications in future energy technologies (electrodes for high temperature fuel and electrolyser cells). For this purpose a deeper understanding of mass and charge transport properties, defect chemistry and structure property relations is necessary. Rare earth nickelates A $_{_{\rm n+1}}$ B $_{_{\rm n}}O$ $_{_{\rm 3n+1}}$ (A = La, Pr, Nd; B = Ni; n = 1,2,3 etc.) are currently known as those materials with the highest diffusivities and ionic conductivities; they show good electronic conductivities. The substitution of these compounds on the A- and B-lattice sites offers the opportunity to tailor the material properties. Promising new compositions of A- and B-site substituted rare earth nickelates, which will be selected on the basis of structural-chemical considerations, are synthesized and characterized with respect to structure-property relationships. MUL will focus on the preparation of the materials and their characterisation with respect to phase purity, oxygen nonstoichiometry, oxygen exchange kinetics, and ionic/electronic conductivities. MPI will complement these activities by the experimental determination of the oxygen and proton exchange properties of thin film electrodes and the investigation of the underlying reaction mechanisms. The participation of ZFE allows the correlation of the mass and charge transport properties, investigated by MUL and MPI, with nanostructural properties. For this purpose accompanying analyses with high resolution scanning transmission electron microscopy (STEM) including in situ TEM analyses of atomic structural changes induced by variations in the oxygen content of new rare earth nickelates.



Figure: STEM HAADF-image of $La_{n+1}Ni_nO_{3n+1}$ with regions of different values for n: n=1 (La_3NiO_4) and n=3 ($La_4Ni_3O_{1n}$).



Services to Industry and Research Institutes

We are committed to keeping up to date with materials research and to provide the best service to our industrial/research partners. We also promote and work in collaborative Research&Development Projects (R&D); we facilitate procedures (planning, training, data acquisition etc.) and have access to other facilities as well as networks within the research community that could be of benefit to our clients.

• Grant-aided Partnerships

We work in collaborative long term research projects which are granted by Austrian and European funding organisations.

• Contract Research

Industrial partners fund the costs of research (instrument fees, consumables and salaries for staff or scholarships).

Testing and Consultancy

We assist the client to make well-informed decisions – be it failure analysis, materials testing, identification of contaminants or quality assurance.

• Training Courses

We provide training on in-house instruments for SMEs who wish to purchase certain microscopes or use these instruments on a regularly basis and see the need to upskill their staff to become selfsufficient with their analyses.

• Know-how Transfer

SEVENTH FRAMEWORK

We offer apprenticeships and work experience to young people who are interested in finding out more about work in different sectors of our Institute, from physical and chemical laboratories to public relations and media engineering.



Being an (inter)national resource of expertise our skills are in high demand. Since international collaboration, discussions and exchange of ideas are creating a productive and sustainable environment to develop new approaches – and subsequently projects – we are engaging in two different areas: European research programmes (FP7) such as ESTEEM2 in Infrastructures or S³ in Eurostars on the one hand; and world-wide collaborations focussing on specialised research tasks on the other hand.

ESTEEM2

ESTEEM2 is a European Integrated Infrastructure of electron microscopy facilities providing access for the academic and industrial research community in the physical sciences to the most powerful TEM installations in Europe (amongst them the ASTEM at Graz) and to some of the best characterisation techniques available at the nanoscale. ES-TEEM2 benefited from European Union support until September 2016.

FELMI-ZFE concentrates on nanoanalytical characterisation techniques such as EELS and EDX as well as studies of surface plasmons of nanostructures.

Events and Workshops



AFM Workshop

The first Atomic Force Microscopy Workshop took place at the Institute of Electron Microscopy and Nanoanalysis at the Graz University of Technology from 18th to 20th February 2015. The workshop was organised in close cooperation with the Bruker Nano Surfaces Division and focused on life sciences and bio applications, advanced materials sciences and automated AFM in industry.

ACR Workshop: Soft Matter Microscopy

In December 2015 we focused on biological and polymer samples. Together with collegues from the OFI (Österreichisches Forschungsinstitut für Chemie und Technik) possibilities and developments in the field of soft matter microscopy were presented.



Lange Nacht der Forschung 2016

The night's topic was *Microworlds* – *Nanoworlds*. Some 180 visitors participated in guided tours, covering light, scanning and transmission electron microscopy, and asked lots of questions.

FEBIP 2016 in Vienna

During the last decade, Focused Electron Beam Induced Processing (FEBIP) has developed from a flexible bottom-up and top-down nano-fabrication method into a versatile 3D nano-printing tool which increasingly attracts attention from industry. The workshop was organised in close cooperation with the TU Wien; four intense days were filled with tutorials, talks and poster presentations.





65 Years Electron Microscopy at Graz

Back in 1951 the first electron microscope was installed at the *Technische Hochschule Graz*. On the 10th November 2016, in the course of one afternoon, different speakers highlighted the latest developments in the field of electron microscopical materials characterisation at our institute as well as at enterprises and institutes collaborating with our team.







Academic Education

LLL Courses



LIFE LONG

European EELS & EFTEM-School in Cooperation with Gatan, USA

(G. Kothleitner, W. Grogger)

The joint FELMI/GATAN European EELS & EFTEM-School is a four days hands-on laboratory workshop taking participants step-by-step through the use of an integrated FEI energy-filtering / EELS system (CM20/GIF, TF20/HR-GIF and TITAN/GIF Quantum).

Our qualified staff familiarizes participants with the latest EELS & EFTEM equipment as well as with fundamental principles and methods. Participants learn to apply practical techniques, how to use hardware and software systems as well as advanced EELS and EFTEM applications in a very efficient manner. The techniques are applicable to fields ranging from biological to materials research. Prior experience with transmission electron microscopy is beneficial, in the conventional (TEM) and in the scanning (STEM) mode; a basic familiarity with EELS and EFTEM is an advantage. The lectures are highly informative filled with detailed discussions with experts in the field and hands-on experience with various techniques. By the end of the course participants have the knowhow to optimise the performance of their GIF as

well as their EELS and EFTEM experimental setups in order to capture and extract the maximum amount of information from TEM samples.

Problem Solving with Scanning Electron Microscopy and X-ray Microanalysis

(S. Mitsche, P. Pölt, H. Schröttner)

Once a year, scientists, engineers as well as technicians profit from this course to solve their analytical problems. We take interested participants step-by-step through the use of advanced scanning electron microscopes during an intense three days hands-on workshop so that they gain deep insight into essential principles and methods in the field of scanning electron microscopy and xray microanalysis of materials.

We are using the latest equipment and know-how to assure that the participants successfully take images, spectra and elemental maps. The techniques are applicable to fields ranging from materials research (steel, ceramics, semiconductors, polymers, etc.) to biological research. Participants are welcome to bring their own samples to analyse them with our help.

Further Activities

Urania-Course

(W. Grogger, C. Mayrhofer)

Elektronenmikroskopie: Einblicke in den Mikrokosmos – Erzeugung und Manipulation von Mikrostrukturen

In the course of a couple of evenings our team gives a general introduction to electron microscopy covering development, different preparation techniques, ultramicrotomy, transmission electron microscopy, scanning electron microscopy, and atomic force microscopy. The course is held in German.





Presentations and Lab Tours

We are active to promote the TU Graz among pupils from local schools and interested groups from Austria and abroad. Presentations at and tours through the Institute including lectures and demonstrations have been organised for groups of physics and chemistry teachers and for students of the TU Graz, schools and local universities. Around 250 pupils, teachers and students from other institutions visited the Institute during the period 2015–2016.

2015

19 Jan. 2015, Secondary School BRG Petersgasse, Graz (Mayrhofer)

03 Mar. 2015, FH Joanneum, University of Applied Sciences, Graz (Zankel)

14 Apr. 2015, Secondary School BRG Seebacher, Graz (Reichmann)

24 Apr. 2015, Working Group Hard Metal, Germany and Austria (Grogger)

08 Jun. 2015, Master's Degree Bionik, University of Applied Science Carinthia, Villach (Pölt)

19 Jun. 2015, Secondary School BRG Carnerigasse, Graz (Zankel)

29 Jun. 2015, Secondary School Konrad Lorenz Gymnasium, Gänserndorf (Zankel)

04 Dec. 2015, Fresenius from India, Indonesia and Brasil (Schröttner)

2016

21 Jan. 2016, Geosciences group, Graz (Šimić) 29 Feb. 2016, Secondary School BRG Kepler, Graz (Zankel)

30 Mar. 2016, Kendrion, Eibiswald (Schröttner) 20 Apr. 2016, Zellcheming, Technical committee, COAT, Germany (Schröttner)

22 Apr. 2016, Lange Nacht der Forschung (Letofsky-Papst, Mayrhofer, Zankel, Gspan, Fisslthaler, Haberfehlner, Gissing, Wallner)

11 Jul. 2016, Oak Ridge National Laboratory Group, USA (Plank)

Man hat ja sonst kaum Gelegenheit auf die TU zu gehen und die Mitarbeiter haben sogar aus dem "Nähkästchen" erzählt.

Eine der interessantesten Exkursionen, die wir im Studium bisher hatten.

05 Aug. 2016, Lenzing AG, Lenzing (Mayrhofer) 23 Aug. 2016 Apprentices of the TU Graz (Letofs-

20 Oct. 2016, Secondary School BRG Pestalozzi,

07 Nov. 2016, Chemieingenieurschule Graz (Le-

15 Nov. 2016, Advantage Austria: Marshallplan -

30 Nov. 2016, Medical University of Graz (Mayr-

15 Dec. 2016, Austrian Cooperative Research (Ho-

18 Nov. 2016, Joanneum Verein (Hofer)

ky-Papst)

Graz (Zankel)

tofsky-Papst)

hofer)

fer)

USA (Fisslthaler)











Harald ROSE, University of Ulm



Robert SINCLAIR, Ai Leen KOH, Stanford



Guest Speakers at the Institute

2015

02 Feb. 2015, Michael HUTH Goethe University, Frankfurt a.M., Germany, "Nano on the Spot"

10 March 2015, Ute KOLB

Johannes Gutenberg University of Mainz, Mainz, Germany, "Advances in Automated Electron Diffration Tomography"

27 March 2015, Catalin R. PICU Rensselar Polytechnic Institute, New York, USA, "To Cluster or not to Cluster: Improving Material Behavior by Multiscale Heterogeneity Control" 19 Jun. 2015, Christian TEICHERT Montanuniversität Leoben, Leoben, Austria, "Ad-

vanced Graphene Characterization via AFM" 21 Aug. 2015, Gianluigi BOTTON

McMaster University, Hamilton, Canada, "From Catalysis and Plasmonics: Probing the Structure of Nanoscale Materials with the TEM and EELS" 07 Sep. 2015, Robert SINCLAIR & Ai Leen KOH Stanford University, Stanford, USA, "An up-date on *in situ* and Environmental High Resolution Electron Microscopy of Materials Reactions" 16 Oct. 2015, Heinz AMENITSCH

TU Graz, Graz, Austria, "Small Angle Scattering and Electron Microscopy: Two Sides of the Same Coin" 06 Nov. 2015, Gernot VOITIC

TU Graz, Graz, Austria, "Production of pure pressurized hydrogen by the Reformer Steam Iron Process"

13 Nov. 2015, Heiko GROISS

Johannes Kepler University Linz, Linz, Austria, "Dislocation characterization in solids by TEM and LAC-BED"

20 Nov. 2015, Gregor TRIMMEL

TU Graz, Graz, Austria, "Metal sulfides: nanoparticles, nanocomposites and more"

27 Nov. 2015, Anton HÖRL

TU Graz, Graz, Austria, "Tomography of particle plasmon fields using EELS"

15 Dec. 2015, Alexander PFLEGER

TU Graz, Graz, Austria, "In situ Untersuchung der Hochtemperaturkorrosion von Co-basierenden Legierungen im ESEM"

Guest Professor at the Institute

Prof. Helen M. Chan, former chair of the Department of Materials Science and Engineering at Lehigh University (Bethlehem, USA) was awarded a Fulbright U.S. Scholar Program grant to teach in Austria in the 2016–2017 winter semester. Chan pursued her research on ceramics and gave a lecture about materials characterisation. Chan is the New Jersey Zinc Chaired Professor at Lehigh. She

2016

12 Jan. 2016, Wolfgang JÄGER University of Kiel, Kiel, Germany, "Advanced transmission electron microscopy for the development of high-efficiency solar cells" 04 Mar. 2016, Vanja SUBOTIC TU Graz, Graz, Austria, "Festoxidbrennstoffzellen - eine zukunftsträchtige, umweltfreundliche und dezentrale Energietechnologie" 14 Mar. 2016, Harald ROSE University of Ulm, Ulm, Germany, "Efficient linear phase contrast in STEM" 15 Mar. 2016, Harald ROSE University of Ulm, Ulm, Germany, "Prospects and Results of Aberration-corrected Low-Voltage Electron Microscopy - the SALVE Project" 03 Jun. 2016, Martin STERRER University of Graz, Graz, Austria, "Charge Transfer in Organic/Metal and Metal/Thin-Dielectric-Film Systems - Influence on Self-Assembly and Reactivitv" 14 Jun. 2016, Walid HETABA Fritz-Haber-Institut der Max-Planck-Gesellschaft, Berlin, Germany, "Electron Microscopy at the FHI-Berlin - Chemical and Structural Analysis for Heterogeneous Catalysis"

26 Jun. 2016, Sarah ZELLNITZ

Research Center Pharmaceutical Engineering (RCPE), Graz, Austria, "Particle engineering in dry powder inhalers"

07 Oct. 2016, Helen M. CHAN

Lehigh University, Bethlehem, USA, "Applications of Electron Microscopy to Research on Novel Composites and High Temperature Oxidation Behavior"

21 Oct. 2016, Martin FACCINELLI

TU Graz, Graz, Austria, "Defects in Proton Implanted Silicon"

04 Nov. 2016, Peter PUSCHNIG

University of Graz, Graz, Austria, "Structural and Electronic Properties of Organic/Metal Interfaces investigated by Photoemission Tomography"

is a Fellow of the American Ceramic Society, and has previously served as Chair of the Gordon Research Conference on Solid State Ceramics (2008). Dr. Chan has served as an Associate Editor for the Journal of the American Ceramic Society since 1999, and is a member of the Editorial Board of the Journal of Materials Science.

DIPLOMAT

Master & Doctoral Theses at the Institute

Finished PhD Theses

MEINGAST Arno (2015), Analytical TEM Investigations of Nanoscale Magnetic Materials. UUSIMÄKI Toni (2015), Electron Tomography of Pourous Materials and Magnetic Nanoparticles. GANNER Thomas (2016), Enzymatic Cellulose Degradation Visualized by Atomic Force Microscopy.

PhD Theses in Progress

FITZEK Harald, Understanding surface enhanced Raman spectroscopy using accurate nearfield-simulations.

HARTLER Christian, Cu interconnects for 3D applications.

KNEZ Daniel, Analytical high resolution transmission electron microscopy of metallic nano clusters.

KONRAD Lukas, How Multiple Scattering Simulations help for EELS Compositional Analysis of Hard Metals and Ceramics.

KRAXNER Johanna, Analytical TEM of organic electronics with a special focus on EDXS and geometry aspects.

LAMMER Judith, Analytical TEM: Influence of Specimen Holder Geometry on EDXS Quantification. NACHTNEBEL Manfred, *In situ* experiments with soft materials in the environmental scanning electron microscope.

ORTHACKER Angelina, Focused Ion Beam Processing of Polymers: The Influence of Material Properties.

SATTELKOW Jürgen, Direct-Write Fabrication of Electric and Thermal High-Resolution Nanoprobes on Self-Sensing AFM Cantilever.

WINKLER Robert, Fabrication of functional, freestanding 3D nano-architectures via Focused Electron Beam induced Deposition.

Finished Master Theses

ARNOLD Georg (2015), Quasi-1D Nano-Resonators for Ultra-Sensitive Gas Sensing Applications. MELISCHNIG Alexander (2015), DC Magnetron Sputtered Metallic Thin Films for High Resolution Scanning Electron Microscopy.

PLATZER Julia (2015), Optimization of sample preparation techniques for transmission electron microscopy of Al-Cu and Eco Mg alloys.

SATTELKOW Jürgen (2015), Semicrystalline Thin

Film Cellulose: Close-to-Nature Substrates for *in situ* Atomic Force Microscopy.

FRÖCH Johannes (2016), Focused Ion Beam Assisted Deposition of Metals: Process-Parameters and Functionality Relationships.

HASELMANN Ulrich (2016), Direct-Write Fabrication of Pure Metal Structures for Nano-Probing and Plasmonic Application.

LAMMER Judith (2016), Experimental Determination of the Solid Angle of Energy-Dispersive X-ray Detectors.

SEIDL Regina (2016), Specimen Preparation for Transmission Electron Microscopy by Focused Ion Beam: Refinement of Post-Treatment using Focused Low Energy Argon Ion Milling.

Master Theses in Progress

ACHTSNIT Tobias, Evaluating New Applications and Optimizing the Environmental Scanning Electron Microscope.

FALTHANSL Paul, High-Resolution Fabrication of Quasi-Planar Gold Structures for Plasmonic Applications using Focused Electron Beam Induced Deposition.

FRÖCH Johannes, Mechanical Properties of Free-Standing 3D Nanostructures Fabricated via Focused Electron Beam Induced Deposition.

POSTL Andreas, Contrast Analysis of Simulated and Experimental High Resolution Scanning Transmission Electron Microscope Images of Gold-Nickel Specimens.

RADESCHNIG Ulrich, Combining Atomic Force Microscopy with scanning electron microscopy and focused ion beam microscopy for *in situ* rapid prototyping.

RASTEL Michael, Untersuchung der Riss- und Bruchverhaltens von Polymeren mittels licht- und elektronenmikroskopischer Methoden / 3-D.

SPALT Sebastian, Multilayer optimization of power diodes.

STRIEMITZER Robert, Improving confocal Raman spectroscopy for advanced 3D-imaging.

TRUMMER Cornelia, Low voltage post treatment of mechanical and ion milled transmission electron microscopy samples. DISSERTATION

Criens In

MASTER TOOD

COSTORAL THEES

Master and Doctoral Students from Other University Institutes

With its advanced microscopy competences the Institute supports Master and PhD students from other institutes of the TU Graz and even other universities and research organisations. These activities range from short term services to enduring scientific collaborations.

Graz University of Technology

- Institute of Inorganic Chemistry Freskida GONI, Master Thesis
- Institute of Physical and Theoretical Chemistry Rupert DRAXLMAYR, Master Thesis
- Institute of Experimental Physics Maximilian LASSERUS, Master Thesis

Institute of Chemistry and Technology of Materials

Denise BACHER, Master Thesis Christine BUCHMAIER, PhD Thesis Christoph BRUDL, Master Thesis Simone LAIMGRUBER, PhD Thesis Katrin NIEGELHELL, PhD Thesis Verena PERNER, PhD Thesis Lukas SCHAFZAHL, PhD Thesis Christian ZEIGER, PhD Thesis

Institute of Analytical Chemistry and Food Chemistry

Josef EHGARTNER, PhD Thesis

Institute of Chemical Engineering and Environmental Technology

Georg BALDAUF-SOMMERBAUER, PhD Thesis Michael GIEBLER, Master Thesis Daniel VARGA, PhD THesis

Institute of Solid State Physics Martin FACCINELLI, PhD Thesis Gernot GRUBER, PhD Thesis Olivia KETTNER, PhD Thesis Andrea KRAXNER, PhD Thesis Stefan PACHMAJER, Master Thesis

Institute of Materials Physics Jaromir KOTZUREK, PhD Thesis

Eva Maria STEYSKAL, PhD Thesis Institute of Applied Geoscience

Florian KONRAD, PhD Thesis Alexandra STAMMEIER, PhD Thesis

Institute of Materials Science and Welding Rishi BODLOS, Master Thesis Petra CHRISTOFL, PhD Thesis Bernadete GSELLMANN, PhD Thesis Romana KÖPPL, Master Thesis Claudia RAMSKOGLER, PhD Thesis Corinna SABITZER, PhD Thesis Zahra SILVAYEH, PhD Thesis Maximilian STUMMER, PhD Thesis Johannes TÄNDL, PhD Thesis Simonet THIERRY, PhD Thesis Christopher WEIDNIG, PhD Thesis

Institute of Thermal Engineering Raphael NEUBAUER, PhD Thesis Alexandra MITTEREGGER, PhD Thesis

University of Graz

Institute of Physics Anton HÖRL, PhD Thesis Gernot SCHAFFERNAK, PhD Thesis

Institute of Mathematics Richard Martin HUBER, Master Thesis

Institute of Molecular Biosciences Barbara EICHER, PhD Thesis



Institute of Biotechnology and Biochemical Engineering, Graz University of Technology



Institute of Chemistry, University of Graz

University of Vienna

Physics of Nanostructured Materials Stefan NOISTERNIG, Master Thesis

Montan University Leoben

Chair of Physical Chemistry Christian BERGER, PhD Thesis Nina SCHRÖDL, PhD Thesis

MCL Materials Center Leoben Johanna KRAINER, PhD Thesis Eva LACKNER, PhD Thesis Robert WIMMER-TEUBENBACHER, PhD Thesis

Chemie-Ingenieurschule Graz

Bernhard RUMP, Diploma Thesis

Martin Luther University Halle-Wittenberg, Germany

Marta Rosario de Jesus Asturias Fuentes, PhD Thesis

Christian Albrechts University Kiel, Germany

Technical Faculty of CAU Kiel Julian STROBEL, PhD Thesis (ESTEEM2)

University of Tübingen, Germany

Nikolas Hagemann, PhD Thesis

Philipps-Universität Marburg, Germany

Department of Chemistry Tibor MÜLLNER, PhD Thesis

Ben-Gurion University, Israel

Eran Amsellem, PhD Thesis

Technion, Israel

Israel Institute of Technology Hadas Sternlicht, PhD Thesis

Institute of Solid State Chemistry, Russia

Ural Branch of the Russian Academy of Sciences Yulia Kuznetsova, PhD Thesis



Christian-Albrechts-University Kiel



University of Graz, Institute of Physics



Outreach

The Institute in the News



NEWS 2015

TU GRAZ RESEARCH, 2015-1, "Neue Materialien aus der Quantenwelt" DIE PRESSE/WISSEN, 28 Feb. 2015, "Forschung, die ans Limit geht" TU GRAZ PEOPLE, März 2015, "Methoden-Mix am Elektronenmikroskop" TU GRAZ PEOPLE, März 2015, "Nanotechnologie der Zukunft" KLEINE ZEITUNG, 10 Jun. 2015, "Rosa canina, Hedera helix & Co"

LOGIK EXPRESS, 19 Aug. 2015, "Harald Plank vom FELMI-ZFE habilitiert sich an der TU Graz" STANDARD/KARRIEREN, 22 Aug. 2015, "EIN-UM-AUFSTIEG: Harald Plank"

TU-AUSTRIA, 24 Aug. 2015, "Kooperation mit China: Nanotechnologie für die Zukunft" APA/SCIENCE, 15 Sep. 2015, "Maßgeschneiderte

Materialien: Physikerin der TU Graz ausgezeichnet"

derSTANDARD.at, 15 Sep. 2015, "US Wissenschaftspreis für Physikerin der TU Graz" FACTORYNET.at, 15. Sept. 2015, "Physikerin der TU Graz ausgezeichnet"

APA/SCIENCE, 15 Sep. 2015, "Maßgeschneiderte Materialien: Physikerin der TU Graz ausgezeichnet"

KLEINE ZEITUNG, 17 Sep. 2015, "Halbleiter: Jagd an den Grenzflächen"

CHEMIE.de, 18 Sep. 2015, "Methoden-Mix am elektronenmikroskop für neue Wege der Materialforschung"

DER GRAZER, 20 Sep. 2015, "Angelina Orthacker, Physikerin"

DIE KLEINE ZEITUNG, 8 Oct. 2015, "Kooperationspreis geht in die Steiermark"

DIE PRESSE/WISSEN, 10 Oct. 2015, "Eisblumen gezielt splittern lassen"

DIE DIGITALE TAGESZEITUNG, 29 Oct. 2015, "Grazer Forscher machen Atome in 3D sichtbar" DIE PRESSE/INOVATION, 31 Oct. 2015, "3D-Schnappschüsse von Atomen"

Orthacker erhielt den "Presidential Scholar Award" © M. Kanizaj/TU Graz

Maßgeschneiderte Materialien: Physikerin der TU Graz ausgezeichnet

NEWS 2016

TU GRAZ RESEARCH, 2016-1, "Nanowelt in 3D" LONDON INSTITUTE OF MEDICAL SCIENCES, 23 Jan. 2016, "Jailhouse Mock" KRONEN ZEITUNG, 26 Mai 2016, "Grazer baut weltweit kleinste Kirche" APA/SCIENCE, 15 Sep. 2016, "3D-Drucker für Nanostrukturen" DER STANDARD, 28 Sep. 2016, "Der Louvre als Nanometermodell" DIE KLEINE ZEITUNG, 5 Nov. 2016, "Workshop: Elektronenmikroskopie" TU GRAZ PEOPLE, 2016-4, "FELMI-ZFE: 65 Jahre, Durchblick an der TU Graz" TU GRAZ PEOPLE, 2016-4, "Very good News: Fullbright-Professur" PFARRBLATT HERZ JESU GRAZ, 2016, "Die kleinste Kirche der Welt: Die Herz-Jesukirche im Nanoformat"

Der Louvre als Nanometermodell

Der Physiker Robert Winkler macht das Elektronenmikroskop zum 3-D-Drucker

Die Herz-Jesu-Kirche im Nano-Format "Die kleinste Kirche der Weit"

"Tausend Seelen der gerechten Menschen im Himmel finden auf einer Stecknadel Platz" behauptet eine Gelehrtenaussage aus dem 14. Jahrhundert.

Zugegeben, die Richtigkeit dieser Aussage ist nicht überprüfbar, die Frage, wie viele Herz-Jesu-Kirchen auf eben dieser Stecknadel gebaut werden können, kann jedoch eindeutig geklärt werden, nämlich 2500! Zumindest werden, nämlich 2500! Zumindest wenn man von dem Nano-Modell der Kirche ausgeht, das am Institut für Elektronenmikroskopie um Nanoanalytik (FELMI) der TU Graz hergestellt



Nano-Modell der Herz-Jesu Kirche im Maßstab 1:4000000. Die Einzelstrukturen sind zwischen 20 und 100 Nanometer dick und bestehen aus Platin und Kohlenstoff, herge-



Halbleiter: Jagd an den Grenzflächen

Wie ein beispielhaftes Forschungsprojekt mit dem steirischen Supermikroskop fünf Top-Halbleiter-Firmen in die Zukunft katapultiert.

SCIENCE O ORF.at



III DELATE grafie" durchschaut Winzigerte Oklate deter sind in neue Dimensionen vorg 14 WISSEN Detrosennitzorden. Sie nennen de Detrosennitzordek fein reich - ein Nannenser ist ein Million

FELMI-ZFE: 65 Jahre "Durchblick" and an der TU Graz

Vor mittlerweile 65 Jahren wurde an der TU Graz das erste Elektronenmikroskop aufgebaut und demit das Fundament für jahrelange erfolgreiche Forschungsarbeit gelegt. TU GRAZ 2015 | RESEARCH & TECHNOLOGY

Awards

Fritz Grasenick Award

The Austrian Society for Electron Microscopy (ASEM) awards the Fritz Grasenick Prize to young scientists in the field of electron microscopy since 2007. In 2016 **Dr. Georg Haberfehlner** was awarded at the ASEM's annual general meeting.

Presidential Scholar Award

Angelina Orthacker won the M&M Meeting/ Presidential Scholar award for her paper entitled "Physical and Practical Challenges in Analytical Electron Tomography of Aluminum Alloys". The application pool was extremely competitive. Proceedings papers from more than 300 students were reviewed, of which only a small number were selected for M&M Meeting Awards. The award is sponsored by the Microscopy Society of America (MSA).

Best Young Lecturer Award

The 12th Multinational Congress on Microscopy took place in Eger, Hungary from 23–28th August 2015. The aim of MCM conferences is to bring together leading experts and emerging young researchers to stimulate scientific communication. Two of our junior scientists, **Harald Fitzek** and **Daniel Knez**, were awarded the best young lecturer award.

WKO Scholarship

In 2015 Georg Arnold received a scholarship sponsored by the Styrian Chamber of Commerce. 20 students were awarded as part of a programme to provide financial support for economically relevant Master Thesis projects.

Best Picture Award

Robert Winkler won the FEBIP 2016 Image Competition in Vienna. He fabricated a miniature model of the pyramides in front of the Louvre in Paris via FEBID technique; the scale to the real pyramides is 1:8 000 000.

EIPBN MicroGraph Contest

Robert Winkler, who is focusing on 3D nanofabrication via focused electron beam induced deposition (FEBID), won the 2015 EIPN MicroGraph Contest in San Diego in the category "aSpiring". He fabricated a miniature model of the neo-Gothic cathedral "Herz-Jesu-Kirche" in Graz on a dual beam system (FIB FEI Nova 200) via FEBID technique. The structure is made out of 60 nm thick Platinum-Carbon rods; the scale to the real cathedral is 1:10 000 000.



Around the World

Austrian Delegation Visits China

The increasing importance of nanoscience in today's scientific society is strongly reflected by the growth of international cooperation. An Austrian delegation of the Chamber of Commerce travelled for five days through China from Guangzhou over Shenzhen and Shanghai to Beijing. The intentions of this visit were bilateral discussions in the field of nanoscience and nanotechnology with high-ranking Chinese representatives from industry and top universities. A member of this delegation was Harald Plank who coordinates the nanofabrication and nanoapplication activities at the FELMI-ZFE. In particular, his work group is involved in the Austro-Chinese project "SENTINEL" which focuses on the development of hypersensitive nano-sensors for in situ characterisation in high resolution electron microscopes. The project is funded by the Austrian Research Promotion Agency (FFG) and carried out in collaboration with the instrument manufacturer GETec KG and the Institute of Physics at the Chinese Academy of Science. After five highly interesting days Harald Plank participated as invited speaker at the 6th International Conference on Nanoscience and Technology in Beijing.

Research at the OIST

Christian Gspan, one of our senior researchers, started spring 2015 with wales and jungle impressions. He followed an invitation to Japan where he was doing research at the Okinawa Institute of Science and Technology, an interdisciplinary graduate school offering a PhD programme in Science. Over half of the faculty and students are recruited from outside Japan – Christian found himself in a highly international environment.

During three weeks he conducted challenging experiments focussing on *in situ* investigations of nanoparticles as well as nanowires via ETEM (under oxygen pressures from 1–20 mbar and temperatures up to 550 °Celsius) and on the purification of platinum carbon layers with water vapour.

Experiments at Melbourne University

After attending the barbie (= Australian BBQ) Christmas party at MCEM (Monash Centre Monash for Electron Microscopy) our PhD student Angelina Orthacker experienced a January and early February 2015 where the microscope rooms at Monash University served as a shelter from the incredible heat of a Melbourne summer. At Monash University she got acquainted with the experimental aspects of guantitative STEM, while she gathered important insider information on the frozen phonon multi-slice simulation code µSTEM at Melbourne University. By combining experiment and simulation not only TEM, STEM, EELS, and EDX data interpretation at the atomic level is improved, but via the PACBED (position averaged convergent beam electron diffraction) method sample thicknesses can be determined with an accuracy of 1-2 nm.







FEBID in Cooperation with the Oak Ridge National Labs

Robert Winkler, PhD student in the workgroup "Functional Nanofabrication" was invited to scientific partners in the US in summer 2015. In the course of four weeks at the Oak Ridge National Laboratories (ORNL) and the University of Tennessee (UT) he exported the direct-write 3D-nanoprinting technique via Focused Electron Beam Induced Deposition as developed at the FELMI to the US-partners, while the simulation specialists at ORNL created a software/simulation package based on his conducted experiments at their Focused Ion Beam microscope. Beside the resulting publication in the journal ACS Nano, fruitful discussions and a further strengthening of the collaboration were the delightful outcome of this visit.

Cambridge Calling

Daniel Knez, one of our PhD students, followed an invitation to England where he was doing research at the Institute of Materials Science of the University of Cambridge. This was a great opportunity to gather valuable experience at one of the leading institutes on both, electron microscopy and material science. Within a cooperative research project, he studied the movement of single platinum atoms on silicon surfaces via high-resolution transmission electron microscopy. This includes the application of highly sophisticated data processing techniques, such as machine learning and drift correction, as well as atomistic simulations using DFT and molecular dynamics.

Cooperation with the Fritz Haber Institute

In Berlin, at the Fritz Haber Institute of the Max Planck Society (FHI) Johannes Rattenberger met international colleagues who are focusing on the in situ characterisation of catalytic growth processes in ESEM technology. Johannes who identified and improved crucial elements to optimise the image quality, the pressure limiting system and the secondary electron (SE) detection system. His efforts resulted in the possibility to use higher pressure regimes in the specimen chamber, the so called UPSEM - Universal Pressure Scanning Electron Microscope was born. He went to Berlin to test and to install the new equipment for the first time outside of our Institute. The researchers hope that higher pressure regimes will help to find deeper insight into catalytic growth processes of graphene and carbo nanotubes on heated copper and nickel substrates.

Sandvik – Winter in Stockholm

Lukas Konrad, PhD student at our institute, is dealing with the characterisation of hard metals and ceramics that are produced by Coromant. He experienced not only the ultimate snow chaos in Stockholm, when he followed the invitation of AB Sandvik Coromant in Västberga in November 2016. During his 2 weeks stay in Sweden he had also the opportunity to present his latest research results as well as exchange knowledge with R&D experts in the field of tooling materials and materials characterisation carrying on the cooperation with one of the leading tooling suppliers.






Publications







Publishing Activities

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Schrödl, N.; Bucher, E.; Gspan, Ch.; Egger, A.; Ganser, Ch.; Teichert, Ch.; Hofer, F.; Sitte, W.: "Phase decomposition in the chromiumand silicon-poisoned IT-SOFC cathode materials $La_{0.6}Sr_{0.4}CO_3-\delta$ and $La_3NiO_4+\delta$ " in: *Solid State Ionics* B 288 (2016) 14–21.

Steyskal, E.-M.; Qi, Z.; Pölt, P.; Albu, M.; Weissmüller, J.; Würschum, R.: "Electrochemically Tunable Resistance of Nanoporous Platinum Produced by Dealloying" in: *Langmuir* B 32 (2016) 7757–7764.

Subotić, V.; Schluckner, Ch.; Strasser, J.; Lawlor, V.; Mathe, J.; Rechberger, J.; Schröttner, H.; Hochenauer, Ch.: "*In situ* electrochemical characterization methods for industrial-sizedplanar solid oxide fuel cells Part I: Methodology, qualification and detection of carbon deposition" in: *Electrochimica Acta* B 207 (2016) 224–236.

Subotić, V.; Schluckner, Ch.; Stöckl, B.; Lawlor, V.; Schröttner, H.; Hochenauer, Ch.: "Strategy for Carbon Gasification from Porous Ni-YSZ Anodes of Industrial-Sized ASC-SOFCs and Effects of Carbon Growth" in: *Journal of the Electrochemical Society* B 163 (2016) 14, F1515–F1522.

Subotić, V.; Schluckner, Ch.; Schröttner, H.; Hochenauer, Ch.: "Analysis of possibilities for carbon removal from porous anode of solid oxide fuel cells after different failure modes" in: *Journal of Power Sources* B 302 (2016) 378–386.

Tändl, J.; Orthacker, A.; Amenitsch, H.; Kothleitner, G.; Poletti, M. C.: "Influence of the degree of scandium supersaturation on the

precipitation kinetics of rapidly solidified Al-Mg-Sc-Zr alloys" in: *Acta Materialia* B 117 (2016) 43–50.

Voitic, G.; Nestl, S.; Malli, K.; Wagner, J.; Bitschnau, B.; Mautner, F.-A.; Hacker, V.: "High purity pressurised hydrogen production from syngas by the steam-iron process" in: *RSC Advances* B 6 (2016) 53533–53541.

Volk, A.; Thaler, Ph.; Knez, D.; Hauser, A.; Steurer, J.; Grogger, W.; Hofer, F.; Ernst, W.: "The impact of doping rates on the morphologies of silver and gold nanowires grown in helium nanodroplets" in: *Physical Chemistry, Chemical Physics* B 18 (2016) 1451–1459.

Wiednig, Ch. A.; Plesiutschnig, E.; Mitsche, S.; Beal, C.; Enzinger, N.; Lochbichler, C.: "Dissimilar Electron Beam Welds of Nickel Base Alloy A625 with a 9% Cr-Steel for High Temperature Applications" in: *Mat. Sci. Forum* B 879 (2016) 2100–2106.

Wernitznig, S.; Sele, M.; Urschler, M.; Zankel, A.; Pölt, P.; Rind, F. C.; Leitinger, G.: "Optimizing the 3D-reconstruction technique for serial block-face scanning electron microscopy" in: *Journal of Neuroscience Methods* B 264 (2016) 16–24.

Xing, L.; Brink, G. H; Schmidt, F.-Ph.; Haberfehlner, G.; Hofer, F.; Kooi, B. J.; Palasantzas, G.: "Synthesis and morphology of iron–iron oxide core–shell nanoparticles produced by high pressure gas condensation" in: *Nanotechnology* B 27 (2016), 215703.

Zankel, A.; Wernitznig, S.; Nachtnebel, M.: "Synergy of SEM and Ultramicrotomy : SBEM Spreading from Life Sciences to Materials Science" in: *Imaging & Microscopy* B 18 (2016) 4, 34–36.

Zeb, F.; Sarwer, W.; Kamran, M. ; Mumatz, M.; Krenn, H. ; Letofsky-Papst, I.: "Surface spin-glass in cobalt ferrite nanoparticles dispersed in silica matrix" in: *Journal of Magnetism and Magnetic Materials* B 407 (2016) 241–246.

Zojer, K.; Rothländer, T.; Kraxner, J.; Schmied, R.; Palfinger, U.; Plank, H.; Grogger, W.; Haase, A.; Gold, H.; Stadlober, B.: "Switching from weakly to strongly limited injection in self-aligned, nano-patterned organic transistors" in: *Scientific Reports 6* (2016) 31387.

Lectures

2015

Hofer F.; "Atomic Resolution Electron Microscopy of Complex Nanostructured Materials", Nano and Photonics 2015, Mauterndorf, Austria, 19 Mar. – 21 Mar. 2015 **invited.**

Grogger W.; "Quantitative Cs Corrected EDXS: Influence of Detector Geometry", PICO 2015 in Vaals, The Netherlands, 23 Apr. 2015 **invited.**

Hofer F.; "Fundamentals of Electron Energy-loss Spectroscopy", European Workshop on Modern Developments and Applications in Microbeam Analysis, Portoroz, Slovenia, 3 May – 7 May 2015 **invited.**

Schröttner H.; "Oberflächencharakterisierung vom Millimeter- in den Nanometerbereich", V-Research Expertenseminar TriboDesign "Oberflächlich-Tiefgründig", Gebhardsberg Bregenz, Austria, 7 May 2015 **invited**.

Plank H.; "Focused Electron Beam Induced Processing: An Application Perspective", International Conference on Electron, Ion and Photon Beam Technology and Nanofabrication, San Diego, USA, 26 May 2015 **invited**.

Hofer F.; "Scanning transmission electron microscopy at atomic resolution", International Workshop on Advanced and *in situ* Microscopies of Functional Nanomaterials and Devices, IAMNano 2015, Hamburg, Germany, 9 Jun. 2015 **invited.**

Plank H.; "Focused Electron Beam Induced Processing: an Additive 3D Nano-Printing Method at its Turning Point Towards Applications", FIB Arbeitskreismeeting, Halle, Germany, 28 Jun. – 30 Jun. 2015 invited.

Kothleitner G.; "Compositional Quantification of Inelastic Atomic Resolution STEM Images", MMC 2015, Manchester, UK, 29 Jun. – 2 Jul. 2015 **invited**.

Haberfehlner G.; "Analytical electron tomography: Methods and applications", Microscopy & Microanalysis 2015, Portland, USA, 2 Aug. 2015 invited.

Hofer F.; "Scanning Transmission Electron Microscopy at Atomic Resolution", 17th Annual Conference YUCOMAT 2015, Herceg Novi, Montenegro, 31 Aug. – 4 Sep. 2015, **invited.**

Kothleitner G.; "Quantitative atomic scale inelastic STEM imaging", FEMMS 2015, Lake Tahoe, USA, 14 Sep. 2015 invited.

Kothleitner G.; "Advance in quantitative inelastic STEM imaging", 37. DGE Tagung 2015, Göttingen, Germany, 6 Sep. – 11 Sep. 2015 **invited.**

Kothleitner G.; "The Quest for Quantitative Figures on atomic resolution inelastic STEM images", Microscopy Conference 2015, Göttingen, Germany, 6 Sep. 2015 **invited**.

Hofer F.; "Scanning transmission electron microscopy at atomic resolution", Eröffnungsfeier Labor für Elektronenmikroskopie, University of Linz, Linz, 16 Sep. 2015 **invited.**

Grogger W.; "Quantitative Nanoanalysis in an Aberration Corrected (S)TEM", European Congress and Exhibition on Advanced Materials and Processes, Warsaw, Poland, 24 Sep. 2015 **invited**.

Grogger, W.; "Quantitative Nanoanalysis on a probe corrected (S) TEM", EPFL Lausanne, Switzerland, 2 Oct. 2015 **invited.**

Hofer F.; "Atomic resolution electron microscopy of complex materials", Vienna University of Technology, Vienna, 28 Oct. 2015 **invited.**

Orthacker A.; "Ausflug in die Nanowelt in 3D und Farbe" Untertitel: "Wenn Licht nicht ausreicht um zu sehen", VHS Wien, 25 Nov. 2015 invited. Mitsche S.; "High spatial and energy resolution x-ray analysis of thin specimens by scanning electron microscopy", European Workshop on Modern Developments and Applications in Microbeam Analysis, Portoroz, Slovenia, 3 May – 7 May 2015.

Reichmann A.; "Orientation imaging and EBSD analysis of the crack zone of fatigued Lead-Zirconate-Titanate", European Workshop on Modern Developments and Applications in Microbeam Analysis, Portoroz, Slovenia, 3 May – 7 May 2015.

Schmied R.; "Focused Ion Beam Processing of Polymers: Pushing the Limits by Alternative Patterning Strategies", International Conference on Electron, Ion and Photon Beam Technology and Nanofabrication, Manchester, UK, 25 May 2015.

Winkler R.; "Focused Ion Beam Processing of Polymers Pushing the Limits by Alternative Patterning Strategies", International Conference on Electron, Ion and Photon Beam Technology and Nanofabrication, San Diego, USA, 26 May – 29 May 2015.

Zankel A.; "New investigation methods of fibres and cellulose materials by conventional and environmental scanning electron microscopy", Die Österreichische Papierfachtagung, Graz, Austria, 11 May – 12 May 2015.

Schmidt F.-Ph.; "Analytical investigations on various metal", New type of spectroscopy (ESTEEM2 Meeting), Orsay, France, 1 Jun. 2015.

Grogger W.; "Energy dispersive X-ray spectrometry: from fundamentals to quantitative analysis", EMAT Workshop on Transmission Electron Microscopy, Antwerpen, Belgium, 17 Jun. 2015 **invited.**

Nachtnebel, M.; "Characterisation of microfiltration membranes by *in situ* wetting in the ESEM and FT-IR", 5th ASEM Workshop, Graz, Austria, 7 Jun. – 8 Jun. 2015.

Schmidt F.-Ph.; "SI analysis tool – a flexible MATLAB tool to analyze spectrum images", New type of spectroscopy (ESTEEM2 Meeting), Orsay, France, 1 Jun. – 2 Jun. 2015.

Orthacker A.; "Analytical Electron Tomographic Investigation of an Aluminium Alloy with Nano-Precipitates", 5th ASEM Workshop, Graz, Austria, 7 Jun. – 8 Jun. 2015.

Ganner T.; "Artificial Substrates as Key Element towards Single Enzyme Tracking via High Speed Atomic Force Microscopy in Enzymatic Degradation of Cellulose", 5th ASEM Workshop, Graz, Austria, 7 Jun. – 8 Jun. 2015.

Winkler R.; "Beyond Current SEM – AFM Solutions: A Highly Flexible *in situ* AFM for Correlated Microscopy", 5th ASEM Workshop, Graz, Austria, 7 Jun. – 8 Jun. 2015.

Konrad L.; "EEL and EDX spectroscopic sensitivity factors for the quantitative analysis of hard metals", 5th ASEM Workshop, Graz, Austria, 7 Jun. - 8 Jun. 2015.

Knez D.; "Electron beam induced oxidation of nickel nanoclusters", 5th ASEM Workshop, Graz, Austria, 7 Jun. – 8 Jun. 2015.

Winkler R.; "High-Fidelity Shapes and Disruption Mechanism during Focused Electron Beam Induced Deposition", 5th ASEM Workshop, Graz, Austria, 7 Jun. – 8 Jun. 2015.

Schmidt F.-Ph.; "Plasmon coupling on silver cuboids revealed by fast electrons", 5th ASEM Workshop, Graz, Austria, 7 Jun. – 8 Jun. 2015.

Kraxner J.; "Reliable quantification of X-ray spectra using ζ-factors: from standards to geometry", 5th ASEM Workshop, Graz, Austria, 7 Jun. – 8 Jun. 2015.

Fitzek H.; "Simulating the pressure limiting system of Environmental Scanning Electron Microscopes using the direct simulation Monte-Carlo method", 5th ASEM Workshop, Graz, Austria, 7 Jun. - 8 Jun. 2015.

Chernev B.; "FTIR-spectroscopic investigations on the absorption of metamaterial multi-arrays built by sub-micron single-structures", ICAVS8, Vienna, Austria, 12 Jul. – 17 Jul. 2015.

Orthacker A.; "Analytical Electron Tomographic Investigation of Aluminium Alloys with Nano-Precipitates", 18. Tagung Festkörperanalytik, Vienna, Austria, 6 Jul. – 8 Jul. 2015.

Dienstleder M.; "Focused Ion Beam Preparation for Transmission Electron Microscopy", 12th Multinational Conference on Microscopy 2015, Eger, Hungary, 23 Aug. 2015.

Fitzek H.; "Optimizing the environmental scanning electron microscope – getting high image quality above 1000 Pa", 12th Multinational Conference on Microscopy 2015, Eger, Hungary, 23 Aug. 2015.

Knez, D.; "Electron Beam Induced Oxidation of Nickel Nanoclusters" 12th Multinational Conference on Microscopy 2015, Eger, Hungary, 23 Aug. 2015.

Knez D.; "Electron beam induced oxidation of nickel nanoclusters", 12th Multinational Conference on Microscopy 2015, Eger, Hungary, 23 Aug. 2015.

Kraxner J.; "Reliable quantification of x-ray spectra using ζ -factors: from standards to geometry", 12th Multinational Conference on Microscopy 2015, Eger, Hungary, 23 Aug. 2015.

Nachtnebel M.; "3D-reconstruction of cracks in polymer blends", 12th Multinational Conference on Microscopy 2015, Eger, Hungary, 23 Aug. 2015.

Orthacker A.; "Physical and Practical Challenges in Analytical Electron Tomography of Aluminum Alloys", Microscopy & Microanalysis 2015, Portland, USA, 2 Aug. – 7 Aug. 2015.

Plank H.; "3D Nanoprinting via Focused Electrons: New Pathways towards Functional Nanostructures on the Lower Nanoscale", Nano-Delegationsreise, China, 24 Aug. – 28 Aug. 2015.

Hofer F.; "Scanning Transmission Electron Microscopy at Atomic Resolution", 17th Annual Conference YUCOMAT 2015, Herceg Novi, Montenegro , 31 Aug. – 4 Sep. 2015 invited.

Plank H.; "Focused Ion Beam Induced Soft Matter Processing Beyond Current Limitations", ChinaNANO, Beijng, China, 2 Sep. – 5 Sep. 2015.

Pölt P.; "In Situ investigation of hot Corrosion in the ESEM", 12th Multinational Conference on Microscopy 2015, Eger, Hungary, 23 Aug. 2015.

Fitzek H.; "Optimizing the Environmental Scanning Electron Microscope for the investigation of wet samples", Materials Day 2015, Graz, Austria, 28 Sep. 2015.

Knez D.; "Electron Beam Induced Oxidation of Nickel Nanoclusters", Materials Day 2015, Graz, Austria, 28 Sep. 2015.

Kothleitner G.; "Quantitative atomic scale inelastic STEM imaging", Letofsky-Papst I.; "TEM investigation of multi-walled hollow fibres produces by tri-axial electrospinning", Microscopy Conference 2015, Göttingen, Germany, 6 Sep. 2015.

Nachtnebel M.; "In situ analysis of PES microfiltration membrane degradation in the ESEM", Microscopy Conference 2015, Göttingen, Germany, 6 Sep. 2015.

Orthacker A.; "Analytical Electron Tomography of Aluminium Alloys with Nano-Precipitates and its Physical Challenges", Materials Day 2015, Graz, Austria, 12 Sep. 2015.

Plank H.; "SENTINEL Nano-Probes: Highly Advanced Nanoprobes for Electric and Thermal *in situ* Nano-Characterization in SEM's and TEM's", ChinaNANO, Beijng, China, 2 Sep. – 5 Sep. 2015.

Orthacker A.; "Die Nanowelt in 3D und Farbe", Science Slam, Mumuth, Graz, Austria, 10 Nov. 2015.

Fitzek H.; "IR/Ramanmikroskopie: Anwendungen zur chemischen Analyse von mm bis μm ", Soft Matter Workshop 2015, Graz, Austria, 2 Dec. 2015.

2016

Plank H.; "Fabrication of Cellulose Structures via Focused Electron Beam Induced Conversion: Approaching the Nanoscale", ACS National Meeting, San Diego, USA, 12 Mar. – 16 Mar. 2016 **invited**.

Zankel A.; "3View spreading from life science to materials science", 3View Users Forum, Heidelberg, Germany, 16 Mar. – 18 Mar. 2016, **invited.**

Hofer F.; "Elektronenmikroskopie mit atomarer Auflösung", Imaging Workshop, Karl-Franzens University of Graz, Graz, Austria, 04 Apr. 2016 **invited**.

Hofer F.; "Principles of scanning transmission electron microscopy and materials science applications", Politecnico Milano, Italy, 22 Apr. 2016, **invited.**

Hofer F.; "Analytical Scanning Transmission Electron Microscopy at Atomic Resolution", EMAG 2016, Durham, UK, 6 Apr. 2016, invited.

Hofer F.; "Analytical scanning transmission electron microscopy at atomic resolution", JEELS Conference, Tarragona, Spain, 29 Jun. 2016, **invited.**

Kothleitner G.; "Quantitative atomic scale inelastic STEM imaging", 4th Dresden Nanoanalysis Symposium 2016, Dresden, Germany, 15 Jun. 2016 **invited.**

Kothleitner G.; "Analytical Electron Microscopy", CenErgy 2016, Lyngby, Denmark, 14 Aug. – 26 Aug. 2016 invited.

Haberfehlner G.; "Formation of bimetallic clusters in superfluid helium nanodroplets analysed by atomic resolution electron to-mography", 16th European Microscopy Congress, Lyon, France, 28 Aug. – 2 Sep. 2016 **invited.**

Haberfehlner G.; "Tomography of particle plasmon fields by electron energy-loss spectroscopy", 16th European Microscopy Congress, Lyon, France, 28 Aug. – 2 Sep. 2016 **invited**.

Mayrhofer C.; "Querdenker in der Ultramikrotomie", Ultramikrotomie Workshop: Neue Möglichkeiten der Ultramikrotomie, Halle, Germany, 8 Nov. – 11 Nov. 2016, **invited.**

Kothleitner G.; "Elemental analysis with EELS and EDXS in 2D and 3D", 37. IAMNano 2016, Port Elizabeth, South Africa, 07 Nov. – 09 Nov. 2016 **invited.**

Mayrhofer C.; "In situ Ultramicrotomy in Medicine and Life Sciences-a Challenge for Preparation", Ultramikrotomie Workshop: Neue Möglichkeiten der Ultramikrotomie, Halle, Germany, 8 Nov. – 11 Nov. 2016, invited.

Hofer F.; "Nanoanalytik und Elektronenmikroskopie", NANONET Austria Meeting, 17 Mar. 2016.

Plank H.; "Towards Pure Metal Deposits via E-beam Assisted Purification using H_2O Vapor at Room Temperature", CELINA – Workshop on Perspectives of C-containing FEBID precursors, Bremen, Germany, 29 Feb. – 2 Mar. 2016.

Fitzek H.; "Looking for the origin of surface enhanced Raman spectroscopy using an atomic force", ASEM Workshop, Leoben, Austria, 28 Apr. – 29 Apr. 2016.

Haberfehlner G.; "Tomography of particle plasmon fields by electron energy-loss spectroscopy", ASEM Workshop, Leoben, Austria, 28 Apr. – 29 Apr. 2016.

Haselmann U.; "Pure FEBID materials for plasmonic and probing applications", ASEM Workshop, Leoben, Austria, 28 Apr. – 29 Apr. 2016.

Hofer F.; "Electron microscopy at atomic resolution", Science Discussions, Austria, 4 Apr. 2016.

Hofer F.; "Electron microscopy at atomic resolution", Graz Imaging Workshop 2016, Graz, Austria, 4 Apr. 2016.

Konrad L.; "How Multiple Scattering Simulations help for EELS Compositional Analysis of Hard Metals and Ceramics", ASEM Workshop, Leoben, Austria, 28 Apr. – 29 Apr. 2016.

Lammer J.; "Experimental Determination of the Solid Angle of EDXS Detectors", ASEM Workshop, Leoben, Austria, 28 Apr. – 29 Apr. 2016.

Orthacker A.; "Analytical Electron Tomographic Investigations Revealing Self Stabilization of Core-Shell Precipitates through Opposing Diffusion Processes", ASEM Workshop, Leoben, Austria, 28 Apr. – 29 Apr. 2016.

Plank H.; "AFM Based High-Speed Tomography in Electron and Ion Beam Microscopes", MRS Spring Meeting 2016, Boston, USA, 28 Mar. – 2 Apr. 2016.

Schmidt F.; "How dark are dark plasmon modes – a correlative EELS and CL study on lithographed silver nanodisks", ASEM Workshop, Leoben, Austria, 28 Apr. – 29 Apr. 2016.

Seidl R.; "Optimization of post-treatment of TEM lamella by lowenergy ion", ASEM Workshop, Leoben, Austria, 28 Apr. – 29 Apr. 2016.

Albu M.; "Analytical Sub Angstrom Scanning Transmission Electron Microscopy of Alloys and Steels", THERMEC, Graz, Austria, 30 May – 3 Jun. 2016

Pölt P.; "Analysis of the fracture behavior of polymers – 2D compared to 3D", PolyMerTec 2016, Halle-Merseburg, Germany, 15 Jun. – 18 Jun. 2016.

Zankel A.; "Advanced microscopic methods for fracture analysis and characterisation of polymers", PolyMerTec 2016, Halle-Merseburg, Germany, 15 Jun. – 18 Jun. 2016.

Haberfehlner G.; "Tomography of particle plasmon fields by electron energy-loss spectroscopy", JEELS Conference, Tarragona, Spain, 28 Jun. – 30 Jun. 2016.

Winkler R.; "FEBID Based 3D Nano-Printing of Plasmonic Gold Structures: Beyond Current Limitations", FEBIP , Vienna, Austria, 4 Jul. – 6 Jul. 2016.

Winkler R., "Plasmonic activity of freestanding and purified 3D-FEBID architectures; Novel investigation possibilities on FEBID deposits by combining dual-beam capabilities with *in situ* high-speed AFM", FEBIP, Vienna, Austria, 4 Jul. – 8 Jul. 2016.

Knez D.; "Atoms in Motion: Electron beam induced dynamics in experiment and simulation", 16th European Microscopy Congress, Lyon, France, 28 Aug. – 2 Sep. 2016.

Haberfehlner G.; "Electron tomography for 3D nanoscale materials characterization", Advanced Materials Day 2016, Graz, Austria, 30 Sep. 2016.

Konrad L.; "How Multiple Scattering Simulations help for EELS Compositional Analysis of Hard Metals and Ceramics", Advanced Materials Day 2016, Graz, Austria, 30 Sep. 2016.

Orthacker A.; "Analytical Electron Thomographic Investigations Revealing Self Stabilization of Core-Shell Precipitates through Opposing Diffusion Processes", Advanced Materials Day 2016, Graz, Austria, 30 Sep. 2016.

Rattenberger J.; "Pushing the limits of environmental Scanning Electron Microscopy", 16th European Microscopy Congress, Lyon, France, 28 Aug. – 2 Sep. 2016.

Sattelkow J.:"Direct-Write Fabrication of Electric and Thermal High-Resolution Nanoprobes on Self-Sensing AFM Cantilever", Micro and Nano Engineering 2016, Austria, 19 Sep. – 23 Sep. 2016.

Winkler R.; "Fabrication of Cellulose Structures via Focused Electron Beam Induced Conversion", Micro and Nano Engineering 2016, Vienna, Austria, 19 Sep. – 23 Sep. 2016.

Books & Conference Abstracts

2015

Dienstleder, M.; Gspan, C.; Kothleitner, G.; Hofer, F.: "Focused Ion Beam Preparation for Transmission Electron Microscopy", Multinational Congress on Microscopy, (2015) PO–135–101–103, Eger, Hungary.

Ganner, T.; Sattelkow, J.; Rosker, S.; Eibinger, M.; Chernev, B. S.; Fitzek, H. M.; Kraxner, J.; Mayrhofer, C.; Aschl, T.; Nidetzky, B.; Plank, H.: "Artificial Substrates as Key Element towards Single Enzyme Tracking via High Speed Atomic Force Microscopy in Enzymatic Degradation of Cellulose", 5th ASEM Workshop (2015) 30, Graz, Austria.

Granitzer, P.; Rumpf, K.; Tian, Y.; Coffer, J.; Akkaraju, G.; Pölt, P.; Reissner, M.: "Assessment of Cytocompatibility and Magnetic Properties of Nanostructured Silicon Loaded with Superparamagnetic Iron Oxide Nanoparticles", Meeting of the Electrochemical Society (2015) 1–7, Cancun, Mexico.

Granitzer, P.; Rumpf, K.; Gonzales-Rodriguez, R.; Coffer, J.; Pölt, P.; Reissner, M.: "Magnetic studies of iron oxide nanoparticles encapsulated within nanostructured silicon", Meeting of the Electrochemical Society (2015) 79–85, Phoenix, USA.

Granitzer, P.; Rumpf, K.; Koshida, N.; Pölt, P.; Michor, H.: "Morphology controlled magnetic interactions of porous silicon embedded nanostructures", Meeting of the Electrochemical Society (2015), 9–14, Cancun, Mexico.

Hobusch, U.; Wernitznig, S.; Gütl, D.; Zankel, A.; Pölt, P.; Rind, F. C.; Leitinger, G.: "3D-reconstructions of a neuronal Motion Detecting Sensor with Serial block Face Scanning Electron Microscopy (SBEM)", 5th ASEM Workshop (2015) 26, Graz, Austria.

Hofer, F.; Kothleitner, G.; Schmidt, F.; Grogger, W.: "Fundamentals of Electron Energy-loss Spectroscopy", European Workshop on Modern Developments and Applications in Microbeam Analysis (2015) 163, Portorož, Slovenia. Knez, D.; Volk, A.; Thaler, P.; Grogger, W.; Ernst, W.; Hofer, F.: "Electron beam induced oxidation of nickel nanoclusters", Multinational Congress on Microscopy, (2015) PO–126–188–190, Eger, Hungary.

Knez, D.; Volk, A.; Grogger, W.; Ernst, W.; Hofer, F.: "Electron beam induced oxidation of nickel nanoclusters", 5th ASEM Workshop (2015) 12, Graz, Austria.

Konrad, L.; Lattemann, M.; Kraxner, J.; Knez, D.; Orthacker, A.; Grogger, W.; Kothleitner, G.: "EELS and EDX spectroscopic sensitivity factors for the quantitative analysis of hard metals ", 5th ASEM Workshop (2015) 19, Graz, Austria.

Konrad, L.; Lattemann, M.; Jorissen, K.; Rehr, J.; Kraxner, J.; Knez, D.; Orthacker, A.; Grogger, W.; Kothleitner, G.: "EELS and EDX spectroscopy for the quantitative analysis of hard metals", Multinational Congress on Microscopy, (2015) PO–273–280–282, Eger, Hungary.

Kornmueller, K.; Letofsky-Papst, I.; Leitinger, G.; Amenitsch, H.; Prassl, R.: "Peptide-based architectures: morphology of a supramolecular selfassembled designer-peptide double helix", 5th ASEM Workshop (2015) 18, Graz, Austria.

Nachtnebel, M.; Mayrhofer, C.; Pölt, P.: "3D-Reconstruction of cracks in polymer blends", Multinational Congress on Microscopy (2015) O–216–34–35, Eger, Hungary.

Nachtnebel, M.; Fitzek, H. M.; Mayrhofer, C.; Brandl, C.; Chernev, B. S.; Pölt, P.: "Characterization of microfiltration membranes by *in situ* wetting in the ESEM and FT-IR mapping ", 5th ASEM Workshop (2015) 32, Graz, Austria.

Orthacker, A.; Haberfehlner, G.; Tändl, J.; Poletti, M. C.; Kothleitner, G.: "Analytical Electron Tomographic Investigation of an Aluminium Alloy with Nano-Precipitates", 5th ASEM Workshop (2015) 39, Graz, Austria.

Pölt, P.; Reichmann, A.; Weiser, M.; Virtanen, S.: "In situ corrosion of Co-based high temperature alloys in the ESEM", European Workshop on Modern Developments and Applications in Microbeam Analysis (2015) 387, Portoroz, Slovenia.

Pölt, P.; Nachtnebel, M.; Fitzek, H. M.; Chernev, B. S.: "*In situ* analysis of PES microfiltration membrane degradation in the ESEM", Microscopy Conference, (2015) 501–502, Göttingen, Germany.

Pölt, P.; Reichmann, A.: "*In situ* investigation of hot corrosion in the ESEM", Multinational Congress on Microscopy, (2015) PO–193–164–166, Eger, Hungary.

Reichmann, A.; Pojprarai, S.; Deluca, M.; Pölt, P.; Reichmann, K.: "Orientation imaging and EBSD analysis of the crack zone of fatigued Lead-Zirconate-Titanate", European Workshop on Modern Developments and Applications in Microbeam Analysis (2015) 388–389, Portorož, Slovenia.

Reichmann, K.; Reichmann, A.; Pölt, P.: "Microstructural analysis of functional ceramics by orientation contrast imaging", European Workshop on Modern Developments and Applications in Microbeam Analysis (2015) 390–391, Portorož, Slovenia. Wimmer-Teubenbacher, R.; Steinhauer, S.; von Sicard, O.; Magori, E.; Siegert, J.; Gspan, C.; Grogger, W.; Köck, A.: "High spatial and energy resolution x-ray analysis of thin specimens by scanning electron microscopy", European Workshop on Modern Developments and Applications in Microbeam Analysis (2015) 373, Portorož, Slovenia.

Winkler, R.; Arnold, G.; Schmied, R.; Szkudlarek, A.; Orthacker, A.; Fowlkes, J. D.; Timilsina, R.; Kothleitner, G.; Rack, P. D.; Utke, I.; Plank, H.: "High-Fidelity Shapes and Disruption Mechanism during Focused Electron Beam Induced Deposition", 5th ASEM Workshop (2015) 36, Graz, Austria.

Zankel, A.; Nachtnebel, M.; Müllner, T.; Pölt, P.: "*In situ* ultramicrotomy in the variable pressure/environmental scanning electron microscope for the assessment of polymeric materials", Microscopy Conference, (2015) 507, Göttingen, Germany.

Zupanic, F.; A Nunes, C.; Coelho, C. G.; Cury, P.; Lojen, G.; Gspan, C.; Boncina, T.: "Microstructure of a Continuously Cast Ni-based Dental Alloy", 55th International Foundry Conference (2015) 43–50, Portorož, Slovenia.

2016

Bucher, E.; Schrödl, N.; Gspan, C.; Höschen, T.; Hofer, F.; Sitte, W.: "Chromium and silicon poisoning of La0.6Sr0.4CoO3-δ IT-SOFC cathodes at 800°C", 12th European SOFC & SOE Forum (2016) 237–239, Luzern, Switzerland.

Fitzek, H. M.; Pölt, P.: "Where does the signal come from? – An easy-to-use spatial characterization method of the laser intensity and detection efficiency for confocal Raman spectroscopy", International Raman Fest (2016) 50–51, Berlin, Germany.

Fitzek, H. M.; Sattelkow, J.; Pölt, P.: "Looking for the origin of surface enhanced Raman spectroscopy using an atomic force microscope", 6th ASEM-Workshop (2016) 18, Leoben, Austria.

Haberfehlner, G.; Hörl, A.; Schmidt, F.; Trügler, A.; Hohenester, U.; Kothleitner, G.: "Thomography of particle plasmon fields by electron energy-loss spectroscopy", 6th ASEM-Workshop (2016) 9, Leoben, Austria.

Haselmann, U.; Winkler, R.; Schmidt, F.; Schwalb, C. H.; Winhold, M.; Strunz, T.; Deutschinger, A.; Fantner, G. E.; Fantner, E. J.; Plank, H.: "Pure FEBID Metal Materials for Plasmonic and Probing Applications", 6th ASEM-Workshop (2016) 19, Leoben, Austria.

Hofer, F.; Schmidt, F.; Grogger, W.; Kothleitner, G.: "Fundamentals of electron energy-loss spectroscopy", IOP Conf.Series: Materials Science and Engineering (2016) 012007, EMAS 2015, Portorož, Slovenia.

Konrad, L.; Lattemann, M.; Kothleitner, G.: "How Multiple Scattering Simultations help for EELS Compositional Analysis of Hard Metals and Ceramics", 6th ASEM-Workshop (2016) 31, Leoben, Austria. Kraxner, J.; Grogger, W.: "Experimental Determination of the Solid Angle of EDXS Detectors", 6th ASEM-Workshop (2016) 21, Leoben, Austria.

Lammer, J.; Kraxner, J.; Grogger, W.: "Experimental Determination of the Solid Angle of EDXS Detectors", 16th European Microscopy Congress (2016) IM08–431, Lyon, France.

Leitinger, G.; Wernitznig, S.; Pölt, P.; Zankel, A.; Rind, C.: "Using serial sectioning and imaging in the SEM to elucidate the wiring of collision-detecting neurons in the brain of a grashopper", 6th ASEM-Workshop (2016) 5, Leoben, Austria.

Orthacker, A.; Haberfehlner, G.; Tändl, J.; Poletti, M. C.; Sonderegger, B.; Kothleitner, G.: "Analytical Electron Tomographic Invesigations Revealing Self Stabilization of Core-Shell Precipitates through Opposing Diffusion Processes", 6th ASEM-Workshop (2016) 8, Leoben, Austria.

Padure, I. M.; Zernig, K.; Heber, G.; Simic, S.: "Morphological aspects and SEM studies of pollen and seed in Hesperis matronalis subsp. matronalis and H. matronalis subsp. candida (Brassicaceae) from Austria", 17. Treffen der Österreichischen Botanikerinnen und Botaniker (2016) 83, Graz, Austria.

Pölt, P.; Nachtnebel, M.; Zankel, A.; Mayrhofer, C.; Gahleitner, M.: "Analysis of the fracture behavior of polymers – 2D compared to 3D", PolyMerTec 16 (2016) 104, Merseburg, Germany.

Rumpf, K.; Granitzer, P.; Michor, H.; Pölt, P.: "Synthesis and magnetic characterization of (porous silicon/"hard-soft" magnetic) nanocomposites", ESC Transactions 75 (2016) 50–60, Honolulu, USA.

Schmidt, F.; Losquin, A.; Hofer, F.; Krenn, J. R.; Kociak, M.: "How dark are dark plasmon modes – a correlative EELS and CL study on lithographed silver nanodisks", 6th ASEM-Workshop (2016) 27, Leoben, Austria.

Schrödl, N.; Egger, A.; Bucher, E.; Gspan, C.; Höschen, T.; Hofer, F.; Sitte, W.: "Phase decomposition of La₂NiO₄+ δ under Cr-and Sipoisoning conditions", 12th European SOFC & SOE Forum (2016) 35/337, Luzern, Switzerland.

Seidl, R.; Dienstleder, M.; Grogger, W.; Fisslthaler, E.: "Optimization of post-tratment of TEM lamella by low-energy ion milling", 6th ASEM-Workshop (2016) 20, Leoben, Austria.

Subotić, V.; Schluckner, C.; Stöckl, B.; Schröttner, H.; Hochenauer, C.: "Carbon removal from the fuel electrode of ASC-SOFC and regeneration of the cell performance", 12th European SOFC & SOE Forum (2016) Luzern, Switzerland.

Zupanic, F.; Gspan, C.; Boncina, T.: "Orientation Relationships of Precipitates with the Matrix in an Aluminium Quasicrystalline Alloy", The TMS Annual Meeting & Exhibition (2016) 5–9, Neshville, Tennessee, USA.

Poster Presentations

2015

Fitzek, H. M.; Lembacher, C.; Chernev, B. S.: "Preventing oil contamination during immersion confocal Raman", ICAVS 8, Vienna, Austria, 12 Jul. 2015.

Ganner, T.; Sattelkow, J.; Aschl, T.; Kraxner, J.; Eibinger, M.; Mayrhofer, C.; Chernev, B. S.; Bubner, P.; Nidetzky, B.; Plank, H.: "The Visualization of Enzymatic Cellulose Degradation Using High Speed Atomic Force Microscopy", EBSA 2015, Dresden, Germany, 18 Jul. 2015.

Haberfehlner, G.; Schmidt, F.; Hörl, A.; Orthacker, A.; Trügler, A.; Hohenester, U.; Kothleitner, G.: "3D imaging of particle plasmon fields by electron energy-loss spectroscopic tomography", Nano and Photonics 2015, Mauterndorf, Austria, 19 Mar. 2015.

Konrad, L.; Lattemann, M.; Kraxner, J.; Knez, D.; Orthacker, A.; Grogger, W; Kothleitner, G.: "EELS and EDX spectroscopy for the quantitative analysis of hard metals", FKA, Vienna, Austria, 6 Jul. 2015.

Konrad, L.; Lattemann, M.; Jorissen, K.; Rehr, J.; Kraxner, J.; Knez, D.; Orthacker, A.; Grogger, W.; Kothleitner, G.: "EELS and EDX spectroscopy for the quantitative analysis of hard metals", MCM2015, Eger, Hungary, 23 Aug. 2015.

Letofsky-Papst, I.; Okan, B. S.; Zanjani, J. S. M.; Yildiz, M.; Menceloglu, Y.; Grogger, W.: "TEM investigation of multi-walled hollow fibres produced by tri-axial electrospinning", Microscopy Conference 2015, Göttingen, Germany, 6 Sep. 2015.

Mitsche, S.; Haberfehlner, G.; Dienstleder, M.; Pölt, P.: "High spatial and energy resolution x-ray analysis of thin specimens by scanning electron microscopy", European Workshop on Modern Developments and Applications in Microbeam Analysis, Portorož, Slovenia, 3 Mai 2015.

Nachtnebel, M.; Fitzek, H. M.; Mayrhofer, C.; Brandl, C.; Chernev, B. S.; Pölt, P.: "Spatially Resolved Characterisation of Microfiltration Membranes by two Different Microscopic Methods", Euromembrane 2015, Aachen, Germany, 6 Sep. 2015.

Reichmann, A.; Weiser, M.; Virtanen, S.; Pölt, P.: "In situ corrosion of Co-based high temperature alloys in the ESEM", European Workshop on Modern Developments and Applications in Microbeam Analysis, Portorož, Slovenia, 3 Mai 2015.

Reichmann, A.; Pojprarai, S.; Deluca, M.; Pölt, P.; Reichmann, K.: "Orientation imaging and EBSD-analysis of the crack zone of fatigued Lead-Zirconate", European Workshop on Modern Developments and Applications in Microbeam Analysis, Portorož, Slovenia, 3 Mai 2015. Schmidt, F.; Hohenester, U.; Hohenau, A.; Hofer, F; Krenn, J. R.: "Plasmon coupling on silver cuboids revealed by fast electrons", Nano and Photonics 2015, Mauterndorf, Austria, 19 Mar. 2015.

Winkler, R.; Arnold, G.; Schmied, R.; Orthacker, A.; Fowlkes, J. D.; Timilsina, R.; Szkudlarek, A.; Utke, I.; Rack, P. D.; Kothleitner, G.; Plank, H.: "High-Fidelity Shapes and Disruption Mechanism during Focused Electron Beam Induced Deposition", EIPBN 2015, San Diego, USA, 25 Mai 2015.

Zankel, A.; Nachtnebel, M.; Müllner, T.; Pölt, P.: "*In situ* ultramicrotomy in the variable pressure/environmental scanning electron microscope for the assessment of polymeric materials", Microscopy Conference 2015, Göttingen, Germany, 6 Sep. 2015.

2016

Arnold, G.; Winkler, R.; Stermitz, M.; Sax, S.; Klug, A.; Noh, J-H.; Fowlkes, J. D.; Rack, P. D.; Plank, H.: "Quasi-1D Nano-Resonators for Highly Sensitive Gas and Mass-Sensors", FEBIP, Vienna, Austria, 4 Jul. 2016.

Fitzek, H. M.; Pölt, P.: "Where Does the Signal Come From? An Easy-to-use Spatial Characterization Method of the Laser Intensity and Detection efficiency for Confocal Raman Spectroscopy", Ramanfest, Berlin, Germany, 18 Mai 2016.

Fitzek, H. M.; Sattelkow, J.; Pölt, P.: "Investigation of the near fields of sputtered Au thinfilms used for SERS, using the AFM and DDA", 16th European Microscopy Congress, Lyon, France, 28 Aug. 2016.

Haselmann, U.; Winkler, R.; Schmidt, F.; Plank, H.: "Pure FEBID Gold Deposits for Plasmonic Applications", FEBIP, Vienna, Austria, 4 Jul. 2016.

Lammer, J.; Kraxner, J.; Grogger, W.: "Experimental Determination of the Solid Angle of EDXS Detectors", Advanced Materials Day 2016, Graz, Austria, 30 Sep. 2016.

Nachtnebel, M.: "Spatial characterisation of PES/PVP based membranes by FT-IR", Permea & Melpro 2016, Prague, Czech Republic, 15 Mai 2016.

Nachtnebel, M.; Fitzek, H. M.; Brandl, C.; Loulergue, P.; Teychene, B.; Pölt, P.: "Spatial characterization of PES/PVP based membranes by FT-IR", PERMA- MELPRO, Czech Republic, 15 Mai 2016.

Radeschnig, U.; Haselmann, U.; Winkler, R.; Sattelkow, J.; Thomson, M. D.; Sachser, R.; Huth, M.; Plank, H.; Bürkle, F.; Wiecha, M.; Walla, F.; Roskos, H. G.: "Direct-Write Fabrication of Pure Gold Bi-Ring Systems for Plasmonic THz Applications", FEBIP, Vienna, Austria, 4 Jul. 2016.

Rattenberger, J.; Fitzek, H. M.; Schröttner, H.; Wagner, J.; Hofer, F.: "Optimizing the Environmental Scanning Electron Microscope for *In Situ* Applications", Materials Science and Engineering 2016, Darmstadt, Germany, 27 Sep. 2016.

Rattenberger, J.; Fitzek, H. M.; Schröttner, H.; Wagner, J.; Hofer, F.: "Pushing the Limits of Environmental Scanning Electron Microscopy", 16th European Microscopy Congress, Lyon, France, 28 Aug. 2016.

Reichmann, A.; Weiser, M.; Virtanen, S.; Pölt, P.: "*In Situ* investigation of high temperature corrosion of Co-based alloys in the ESEMthe very first stages", 16th European Microscopy Congress, Lyon, France, 28 Aug. 2016.

Sattelkow, J.; Fröch, J.; Winkler, R.; Schwalb, C. H.; Winhold, M.; Deutschinger, A.; Strunz, T.; Stavrov, V.; Fantner, G. E.; Fantner, E. J.; Plank, H.: "Direct-Write Fabrication of Electric and Thermal High-Resolution Nanoprobes on Self-Sensing AFM Cantilever", FEBIP, Vienna, Austria, 4 Jul. 2016.

Sattelkow, J.; Fröch, J.; Winkler, R.; Schwalb, C. H.; Winhold, M.; Deutschinger, A.; Strunz, T.; Stavrov, V.; Fantner, G. E.; Fantner, E. J.; Plank, H.: "Direct-Write Fabrication of Electric and Thermal High-Resolution Nanoprobes on Self-Sensing AFM Cantilever", Micro and Nano Engineering 2016, Austria, 19 Sep. 2016.

Schröttner, H.; Albu, M.; Rossmann, A.; Mertschnigg, S.; Pabel, T.; Petkov, T.: "Effect of trace elements on the material properties of an aluminium casting alloy", 16th European Microscopy Congress, Lyon, France, 28 Aug. 2016.

Winkler, R.; Yang, C.; Michelitsch, S. G. W.; Plank, H.; Schwalb, C. H.; Winhold, M.; Deutschinger, A.; Fantner, G. E.; Fantner, E. J.: "Novel Investigation Possibilities On FEBID Deposits By Combining Dual-Beam Capabilities With In-Situ High-Speed AFM", FEBIP, Vienna, Austria, 4 Jul. 2016.

Winkler, R.; Schmidt, F.; Haselmann, U.; Fowlkes, J.; Rack, P.; Plank, H.: "Plasmonic Activity of Freestandingand Purified 3D-FEBID Architectures", Micro and Nano Engineering 2016, Vienna, Austria, 19 Sep. 2016.

What else ?

Habilitation: Harald Plank

Our warmest congratulations go to Harald Plank who impressively demonstrated his expertise in the field of "Focused Electron Beam Induced Nanofabrication" in summer 2015. While functional nanostructures represent an essential part of modern technologies, their fabrication remains challenging. In the course of seven years he carried out research concentrating on both: a better understanding of theoretical and technical limitations as well as the application-oriented implementation of gained research results.



Focused Electron Beam Induced Nanofabrication: From Fundamentals Towards Applications

During the last decades, focused electron beam induced nanofabrication has attracted increasing attention in fundamental and applied research. These techniques represent flexible top-down and bottom-up capabilities as they allow mask-less, direct-write 3D nanofabrication and meet challenges when classical resist based lithography methods can barely be applied or not applied at all. Harald Plank's habilitation thesis summarized the scientific contribution to this field made at our Institute since 2008; a deep insight into fundamental resolution limitations, proximity broadening effects, high-fidelity shapes, and efficiency aspects has been gained which led to a strong performance improvement in this field. Furthermore, the unique material properties of FEBID materials based on the nanoscale metal-matrix composition are regarded which allow functional tuning as well as full purification towards pure metallic nanostructures. These aspects led to several new application concepts which rely on material properties, nanoscale capabilities and the mask-less direct-write 3D character which gives this technology the status of a 3D nanoprinter. In short this thesis covers a broad range from fundamentals towards applications and opens new pathways for industrial applications.



New Corporate Design and Website

In the course of the past to years we have seen some changes as long as marketing and communications are concerned.

We successfully designed and published a **new** website and started a Facebook profile. Once a month we are publishing a new video concerning electron microscopy, preparation techniques, analysis methods and much more on our YouTube Channel.

Please feel free to have a look and find some interesting facts!



FIT: Frauen in die Technik 2015

In summer 2015, our trainee Anna Janderka spent a month at the Institute and got a lot of insight in the different aspects of work in a research facility. Thanks to the project "FIT: Frauen in die Technik" she gathered experience in the physical and chemical sample preparation, participated in photo projects, did some administrative work and performed minor online research.







Exhibition: Rosa canina, Hedera helix & Co Four Ways to Look at Plants

Bringing together photographs, sculptures, drawings, and SEM images this exhibition offered a "complete" view of how to look at plants. This was a rare opportunity to encounter the work of highend technologies and the creations of artists at the same time. The private view received much interest; some 80 people met on a summer evening to admire the art works and to listen to the opening speech held by Walter Titz accompanied by music of the Armenian singer Anush Apoyan and her Montenegrin guitarist Filip Gavrinović.

The exhibition was organised by Marita Janka (photographs) and Krista Titz-Tornquist (sculptures), in collaboration with Josef Taucher (drawings) and Sanja Šimić (SEM images).



Laboratory Facilities

Cutting-edge in-house instrumentation is one of the keys to our success. Thanks to a broad range of different microscopes and the excellent preparation equipment our well trained staff is able to take top quality images, spectra and elemental maps. We house several scanning and transmission electron microscopes, as well as atomic force and light microscopes. The NanoMill and the Focused Ion Beam are amongst others crucial to sample preparation.



TEM Philips CM20 Analytical TEM, LaB6, STEM, EDX-spectrometer (Noran) and Imaging Filter (Gatan)



Bio-TEM FEI Tecnai12 Transmission electron microscope, LaB6, 120 kV, low-dose CCD (Gatan), cryo-transfer system (Gatan) and Freeze plunger (Leica)



ASTEM FEI Titan³ 60–300 Atomic resolution STEM, 60–300kV, X-FEG with monochromator, HAADF- and ABF-detectors, CS-probe corrector (DCOR), Super-X detector, Imaging filter Quantum ERS (Gatan), MEMS heating stage up to 1300°C (DENSsolutions), tomography holder (Fischione)



HR-TEM FEI Tecnai F20 High-resolution TEM, 80–200 kV, FEG with monochromator, HAADF-detector, EDX-spectrometer, Imaging Filter Quantum HR (Gatan), with cryo-holder



ESEM FEI Quanta 200 Environmental SEM (ESEM), W-cathode, EDX-spectrometer and heating stage (Kammrath & Weiss)



ESEM FEI Quanta 600 Environmental SEM (ESEM), FEG, EDX-spectrometer, tensile testing stage, 3View ultramicrotome (Gatan)



HR-SEM ZEISS Gemini DSM986 Analytical high resolution SEM, FEG, EDX-spectrometer



Analytical high resolution SEM, FEG, STEM-, EsB-, AsB- and in-lens detectors, EDX- and WDX-spectrometers (EDAX), EBSD-detector (EDAX), heating stage up to 1050°C (Kammrath & Weiss)





Siemens XRD D5005 X-ray diffractometer with heating stage up to 1200°C (An-



Horiba LabRAM microscope Confocal Raman microscope with Olympus BX41, red, blue and green lasers and DuoScan imaging system



Fischione Nanomill 1040 Ultra-low energy Argon ion mill with cryo-stage



Alicona Infinite Focus G3 Focus variation light microscope



Focused Ion Beam FEI Nova 200 DualBeam FIB-SEM, FEG and Ga-ion source, Omniprobe manipulator, various gas injection systems, XFlash X-ray spectrometer (Bruker), AFM *in situ* SEM (GETec)



Bruker FT-IR microscope Infrared microscope Hyperion 3000 with Tensor 27 spectrometer, ATR and GIR objective, FPA and MCT detectors



FELMI-ZFE Calendar 2016

A pool of fascinating micrographs is generated via an internal image competition; the best micrographs are chosen once a year shortly before Christmas. The most interesting images are usually combined under a general theme and published in our biannual calendar. However, in 2016 we decided to focus on 3D Nanoprinting and Focused Ion Beam treatments of samples and surfaces.





Team



Administration

Central Services

Techn. Laboratory



Nanoanalysis in the TEM











High Resolution Electron Microscopy





Nanofabrication S³



SEM & in situ Methods



















Nanoanalysis in the TEM Head

- Prof. Gerald Kothleitner
- Key Researchers
- , Dr. Mihaela Albu
- Dr. Karin Wewerka
- ٠ Dr. Georg Haberfehlner
- Technicians Margit Brunegger
- Martina Dienstleder *
- Ing. Claudia Mayrhofer Jakob Schaller
- Apprentices
- Karina Mollich
- Arnela Blažević
- PhD Students
- DI Daniel Knez
- Mag. Lukas Konrad

High Resolution Electron Microscopy

- Head Prof. Werner Grogger
- **Key Researchers**
- Dr. Christian Gspan
 Dr. Ilse Letofsky-Papst
- Dr. Evelin Fisslthaler
- PhD Students DI Judith Lammer

SEM & in situ Methods Head

- Doz. Dr. Peter Pölt
- **Key Researchers**
- , Dr. Stefan Mitsche п
- Dr. Armin Zankel
- Technicians
- Sanja Šimić Daniel Schreiner
- PhD Students
- Mag Manfred Nachtnebel

SEM & Microanalysis

- Head Ing. Hartmuth Schröttner
- **Key Researchers**
- Dr. Johannes Rattenberger
- Technicians
- Sabrina Mertschnigg
 - Anita Rossmann-Perner
- Christian Brandl
- PhD Students

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Funktional Nanofabrication

- Head
- Prof. Harald Plank
- Technicians Sebastian Rauch
- PhD Students

- DI Robert Winkler
- DI Jürgen Sattelkow

TU-Global

TU Drittmittel

Verein

- DI Harald Fitzek

Out of Office

2015

Life in the Institute is not only about industrial needs, microscopes, students, or articles. To be creative and foster a productive work environment one sometimes needs a change in time and place to break out of the daily routine and see problems with a fresh eye.

In spring 2015 we admired the partial solar eclipse (1). The company outing started with a degustation of different sorts of beer (2), we learned how whiskey is produced (3), we heard a lot about organic farming (4) and enjoyed Austrian delights at the magnificent garden terrace of a *Buschenschank* (wine tavern) (5). The more active ones among us meet every Tuesday in summer to play beach volleyball (6). The last highlight of the year is the Christmas Party, in 2015 we stayed at the Institute and decorated our seminar room (7).

2016

To be continued in 2016: Ferdinand Hofer celebrated his 60th anniversary (8). In spring the TU apprentices visited the impressive medieval castle Riegersburg which is situated on a dormant volcano (9). The company outing started with













the visit of the cave "Katerloch" (10), followed by the exhibition "Radiation and Mankind" at the European Centre for the History of Physics (11). A running competion for teams is always organised by the sports' centre of the University of Graz. Of course, we've sent our winning teams! (12) The Christmas Party gives Mr. Hofer the opportunity to sum up what progress has been made in the past months and to honour the achievements of his staff. In 2016 our visiting professor Helen Chan is partying with us (13) at the Rooftop Mia and Mason.











