# Performance Report 2017 | 2018

Institute of Electron Microscopy and Nanoanalysis
Graz University of Technology

Graz Centre for Electron Microscopy
ACR Austrian Cooperative Research

TU Graz

ACR
AUSTRIAN COOPERATIVE RESEARCH
*KOOPERATION MIT KOMPETENZ*

FELMI-ZFE

**Published by**

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**Cover Image**

Field of 3D-nanoprinted platinum/carbon flowers deposited via focused electron beam induced deposition by Robert Winkler

# Table of Contents

# Rector of the TU Graz

It's my great pleasure to present this performance report of the Institute for Electron Microscopy and Nanoanalysis (FELMI) of the Graz University of Technology. The following pages impressively document the Institute's successful mission that has led to much notable national and international impact and reputation.

Today, the Institute significantly contributes to several Fields of Expertise (FoE) at our own University but also across partner universities particularly within the NAWI-initiative. The powerful capabilities of new electron microscopes have thereby enabled spectacular scientific insight in almost any area of nanoscience and nanotechnology. The research and service activities of the Institute range from physics, chemistry, pharmacy and materials science even to biology and medicine.

Developing science and promoting tech advance are two cornerstones of the Graz University of Technology. World class research assets like the Austrian Centre for Electron Microscopy and Nanoanalysis support this philosophy to a great deal and therefore represent a fundamental constituent of our University's research efforts. It is only consequent to promote the FELMI-ZFE to the greatest extend possible.

Finally, I would like to take this opportunity to thank all the colleagues in the Institute for their hard work and enthusiasm in moving forward.

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

# A long-term engagement

Sixty years ago, five Austrian companies and the government of Styria took the far-sighted decision to set up the "Association for the Promotion of Electron Microscopy and Fine Structure Research". Under the guidance of the first president of the Association – Governor of Styria Josef Krainer senior - the team around Fritz Grasenick built up the Graz Centre for Electron Microscopy (ZFE), which immediately became a member of the Austrian Cooperative Research (ACR).

We can look back at the first sixty years with pride and satisfaction. The constant commitment of the Association, not only provides electron microscopy at the highest level in Styria and throughout Austria, but also finds worldwide recognition.

We are convinced that the close collaboration with the TU Graz and the ACR was paramount for our long-term success. The today's Institute stands at the forefront of microscopy research and is a key resource for Austrian companies and research institutes.

Desite all the positive news, however, there is a great challenge lying ahead: obsolete scientific infrastructure, dating back to the nineties, quickly needs to be renewed. This action has to be taken in order to maintain and enhance the position for such a valued research organisation. On behalf of the Board of the Association, it goes without saying that we will continue to support this ongoing endeavour.

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

# News from the Team

Ten years ago, various research proposals have laid the basis to set up the first atomic resolution electron microscope in Graz. Successful applications denoted the starting point to raise the Austrian Scanning Transmission Electron Microscope (ASTEM), thoughtfully specified by Gerald Kothleitner and Werner Grogger. The ASTEM was assembled from 2010 to 2011 and at this time it represented one of the most powerful electron microscopes worldwide.
Why do I mention that? This large-scale investment, financially massively backed up by the Association's own funds, has imposed enormous stress onto the institute that could only be tackled by the team effort across all employees. Beyond this point, great progress could be made in many ways: numerous collaborations with industry and research institutes, first-class publications in high-impact journals like Nature, Nature Materials, Nanoletters and similar could be realised. The ASTEM also served as an entrance ticket to high-ranking European projects such as ESTEEM 2 and now also ESTEEM 3, where Gerald Kothleitner leads the Austrian knot.
In other areas, further extremely important achievements could be made: In 2018 Harald Plank initiated the Christian-Doppler Laboratory for Nanofabrication (DEFINE) together with the companies Getec and Anton Paar. This Laboratory is a milestone for the Institute, as it adds a new dimension to electron microscopy: we are now not only able to analyse with electrons, but also use them to build up functional nanostructures.
In 2017 another exciting project was started: In cooperation with the Medical University Graz and the Karl-Franzens-University Graz, a network for electron microscopy (ELMINET) was set up. Grace to the initiatives of Peter Pölt and Hartmuth Schröttner, the worldwide first RISE microscope was installed at the Institute, combining scanning electron microscopy with Raman microscopy. This new correlative microscopy approach already provides many new applications in the physical and biological sciences.
Finally, I would like to take a look into the near future: Leading edge instrumentation in the field of electron microscopy comes at a price – literally: Great financial efforts must be made to maintain the Institute's competitiveness, and 20 or 30 year old obsolete instrumentation needs to be replaced quickly.
With this report, we invite you to explore the micro- and nano-cosmos, the gateway to exciting discoveries and innovations. See how the work of our dedicated team contributes to the further development of the Graz Universities and how we deliver economic benefits for Austrian companies.

Ao.Univ.-Prof. Dipl.-Ing. Dr.techn.
Ferdinand Hofer
Head of the Institute

Univ.-Prof. Dipl.-Ing. Dr.techn.
Gerald Kothleitner

# 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 nanofabrication. It consists of two independent organisations:

FELMI-ZFE

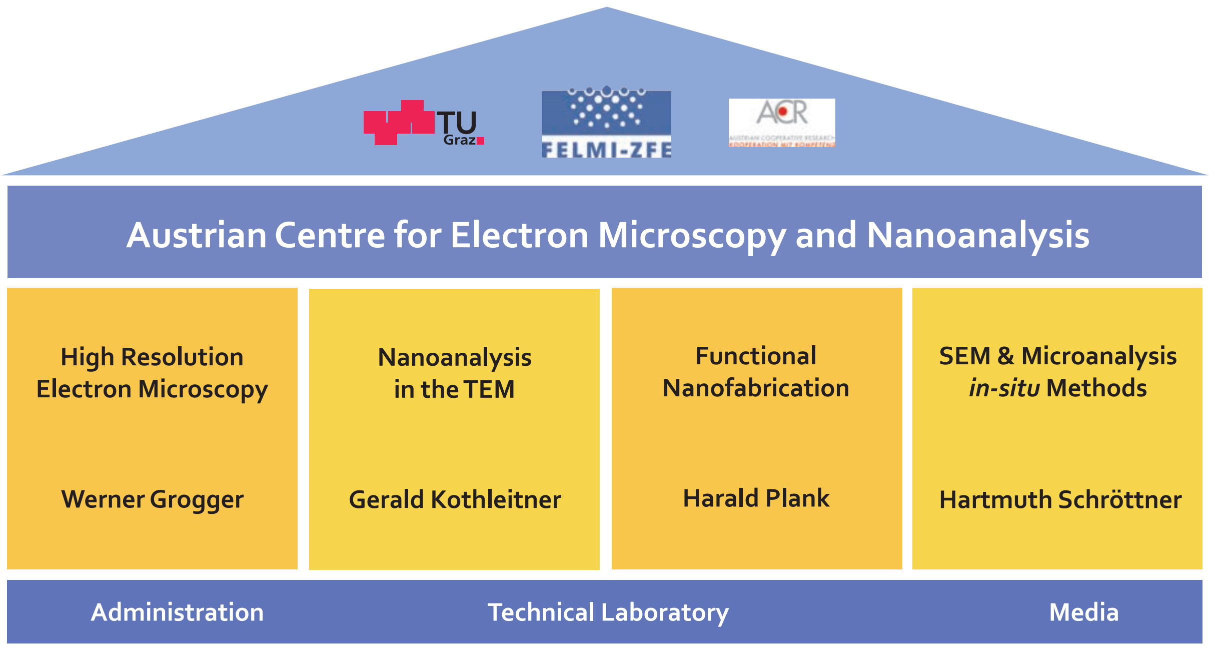
**Institute of Electron Microscopy and Nanoanalysis** (FELMI) at the Faculty of Mathematics, Physics and Geodesy of the TU Graz.

FELMI-ZFE

**Graz Centre for Electron Microscopy** (ZFE) under the guidance of the Association for the Promotion of Electron Microscopy and Fine Structure Research, and a member institute 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 Austrian Centre is organised in presently four groups, each dedicated to research topics in advanced electron microscopy and functional nanofabrication.

The Institute in the building Steyrergasse 17 is located on the 2nd and 3rd floor and the microscopy centre in the basement.

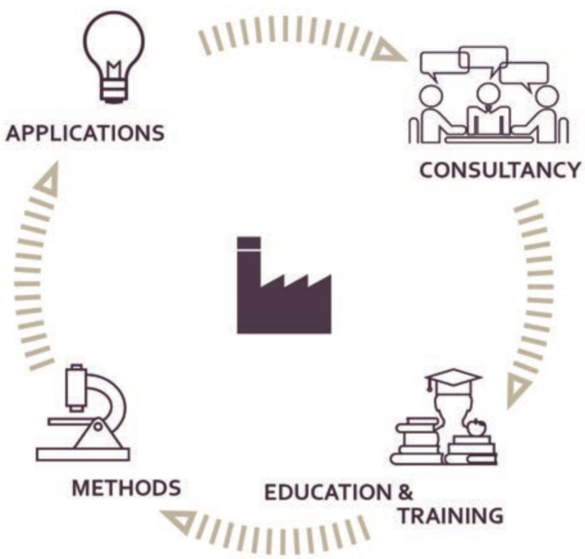


## Our Mission

The Institute's mission is to advance the understanding and control of matter at the nano- and atomic scale in a multi-disciplinary approach. We contribute to society through teaching, training and research in a collaborative way.

## Our Strengths: Uniting Fundamental and Applied Research

With a continuing tradition of excellence, we are trying to bridge the gap between academic research and practical problem solving as well as industrial needs; for 67 years we work in close cooperation with university institutes and enterprises in Europe and increasingly worldwide. Our unique position in the field of advanced microscopy is based on our leading-edge equipment and the expertise and ambition of our staff, thus making us the Austrian powerhouse of electron microscopy.




## Our Key Figures:

**120+** INDUSTRY CLIENTS

**14** MICROSCOPES

**50+** STAFF

**300** PEER REVIEW PAPERS SINCE 2010

**11.000** HRS BEAMTIME

**21** RESEARCH PROJECTS

**>900** STUDENTS IN COURSES

**42** COLLABORATIONS WITH RESEARCH INSTITUTES*

*in Austria

# Facts and Figures

In the meantime, the Institute has become the leading institution for advanced microscopy in Austria. More than 50 employees work with an annual average budget of 2.4 million euros, which is raised exclusively through public funded research projects and contractual research with about one hundred companies. A major problem are the rapidly increasing service costs for the scientific infrastructure, but such costs cannot be avoided in a well-equipped electron microscopy laboratory, especially if the microscopes are kept continuously operational.

In the last years the number of employees has remained constant, the percentage of women is 46% including PhD students. The graphs show the number of employees as of december 2018, with FELMI employees marked in red and ZFE employees in blue (head counts).

The institute has several focal points; in addition to teaching and research, we also carry out service work for other institutes and industry. Despite the variety of tasks, we were again able to achieve a high number of scientific publications in peer-reviewed journals, 99 publications in the years 2017-2018, which are also the result of numerous co-operations. To an increasing extent, we were also able to publish in very high-impact journals, including Nature Materials, Nature Communications, Scientific Reports and Nano Letters. The number of invited talks also remained at a high level (21 in 2 years). All publications of the years 2017 and 2018 are presented at the back of this report.

One more comment on teaching: Here we were able to expand the number of supervised courses and the number of students in these courses has even increased slightly compared to previous years.

6/6 Technical staff

4 Professors

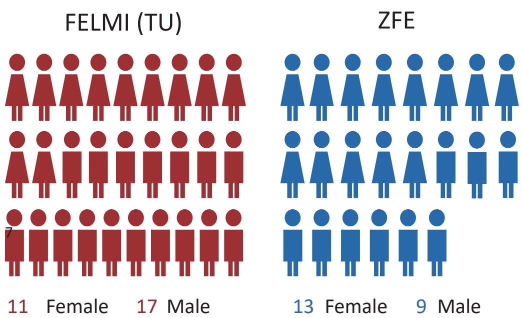
1/3 Managing staff

8+ Master students

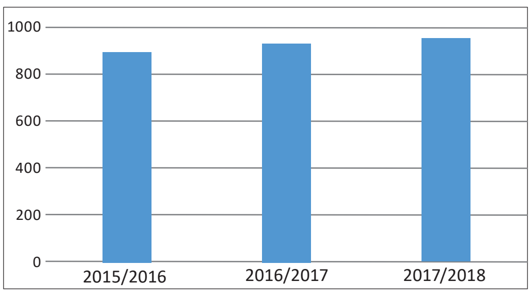
7/6 Senior scientists

7/4 PhD students

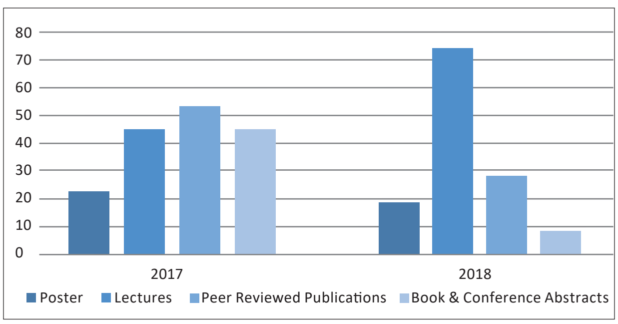
3 Apprentices




Teaching




Number of Publications

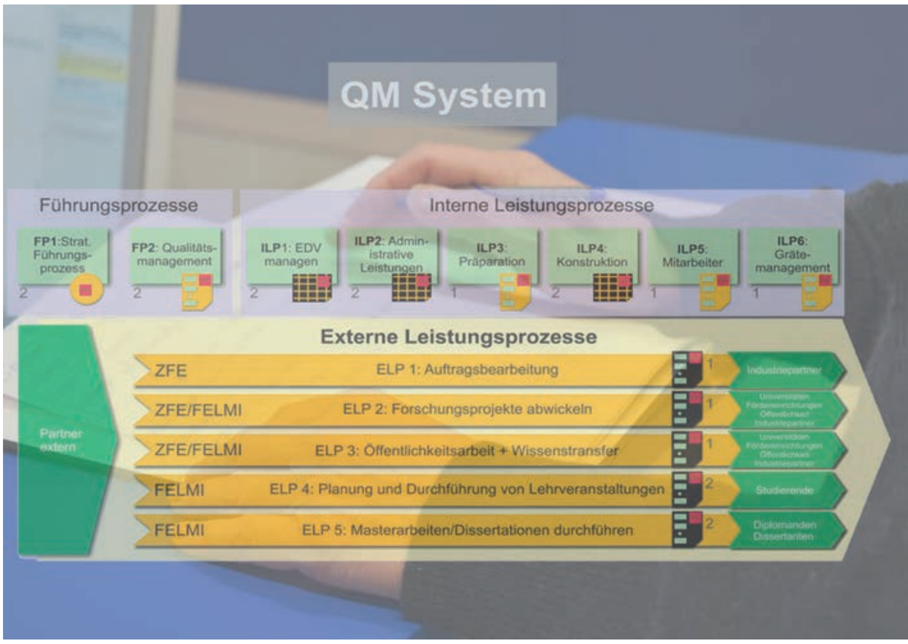


## Research Expert for Austrian SMEs

Over the past 65 years, the institute has supported numerous Austrian companies in their innovation efforts. We have contributed to the competitiveness of Austrian economy thanks to numerous projects and services. The strength 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 European funding landscape.


## Quality Assurance

The Institute works under an advanced quality management system according to the rules of EN ISO 9001:2015. The aim is to keep the outstanding quality of our work and analysis results and to improve our organisation and management structures. The system has been first certified in 2004 by the TÜV Austria and has since been under continuous development. It remains valid until December 2020 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".

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# National/International Impact

As an interdisciplinary research institution, we believe that our responsibility is not only to develop knowledge, but also to share our know-how with others.
Every year, we work together with about 40 institutes in Austria, in addition with around 20 institutes worldwide and with 120 companies.
In recent years, two main directions have been followed: Firstly, collaborations within the ACR group and local universities which are developing well and secondly, the incorporation into important European research networks such as the ESTEEM2 and ESTEEM3 projects.

ESTEEM is a special cornerstone in international relations: on the one hand, we offer European scientists access to our high-quality instrumentation (in ESTEEM3 now also for non-European scientists), and on the other hand we can build and maintain close links with the world's leading laboratories.
These international connections (see below) also lead to an increase in the number of visiting professors and post-docs staying for more than one month in the Institute and generally increase our attractiveness for excellent Master and PhD students.

## Europe

**Belgium**
EMAT, University of Antwerp

**Croatia**
Institute of Biology, University of Zagreb

**Czech Republic**
Department of Condensed Matter Physics, Charles University Prague

**France**
Institute of Chemistry, University of Poitiers
Laboratory of Solid State Physics, CNRS and University of Paris-Sud, Orsay

**Germany**
Center for Applied Geosciences, Eberhard-Karls-University Tübingen
Center of Engineering Sciences, Martin-Luther-University Halle-Wittenberg
Department of Chemistry, Phillips University Marburg
Institute of Inorganic Chemistry and Analytical Chemistry, Johannes-Gutenberg-University Mainz
Institute of Materials Science, Christian-Albrechts-University Kiel
Institute of Nanotechnology, Karlsruhe Institute of Technology
Max-Planck-Institute for Solid State Research, Stuttgart
Physics Institute, Johann-Wolfgang-Goethe-University, Frankfurt/Main

**Great Britain**
Department of Materials Science & Metallurgy, University of Cambridge
Department of Physics, Durham University
School of Physics and Astronomy, University of Glasgow

**Greece**
Department of Solid State Physics, Aristotle University of Technology

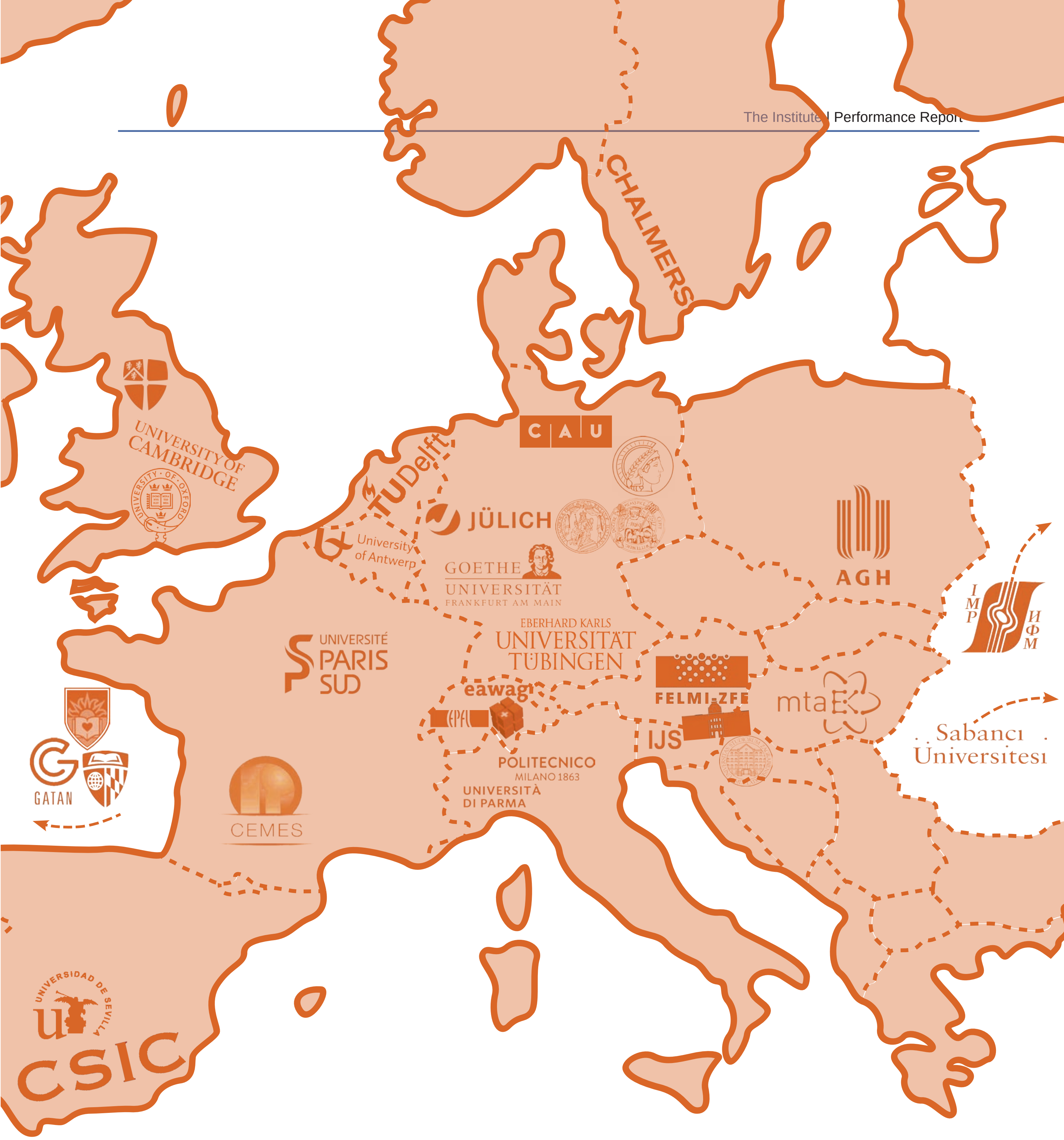
**Hungary**
Research Institute for Technical Physics and Materials Science, Hungarian Academy of Sciences, Budapest

**Italy**
Department of Mechanics, Institute Politecnico Milano
CNR-Institute of Condensed Matter Chemistry and Technology, Lecco

**Slovenia**
Faculty of Mechanical Engineering, University of Maribor
Institute Josef-Stefan, Ljubljana

**Sweden**
AB Sandvik Coromant, Stockholm
Department of Physics, Chalmers University of Technology, Gothenburg

**Switzerland**
Laboratory for Bio- and Nano-Instrumentation, L'Ecole Politechnique Federale de Lausanne




## America

**Brazil**

National Institute of Metrology, Quality and Technology, Duque des Caxias

**USA**

Gatan Inc. Pleasanton

Center for Nanophase Materials Science, Oak Ridge National Laboratory, Tennessee

Department of Materials and Engineering, University of Tennessee, Knoxville

Department of Materials Science and engineering, Lehigh University, Betlehem

Department of Physics, University of Washington

## Asia

**China**

Institute of Physics, Chinese Academy of Sciences, Beijing

**Singapore**

National University of Singapore

## Australia

School of Mathematical and Physical Sciences, University of Newcastle

OIST

# 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 advanced 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 industries with latest results of approved as well as newly designed microscopy techniques on the one hand; on the other hand it is keeping our staff up to date as far as developments in the field concerned.

The association leads the **Graz Centre for Electron Microscopy** (ZFE) which plays an important role in developing and applying fundamentals and carrying out different research projects with universities and industry.

**Professor Helmut List** is the president of the association which has currently 34 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**

### Managing Committee

Head:
KR Dipl.-Ing. Ulrich **SANTNER**

Vice-Head:
Mag. Christian **KNILL**

Financial referee:
Ing. Dr. Jürgen **STEINECKER,** MBA

Representative of the Styrian Universities:
O.Univ.-Prof. DI Dr. Horst **BISCHOF**

Head of ZFE:
Ao.Univ.-Prof. DI Dr. Ferdinand **HOFER**

### Accounting Controller

1. Controller:
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2. Controller:
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Graz University of Technology

Univ.-Prof. Dr. Roland **WÜRSCHUM**,
Graz University of Technology

## Microscopy and Fine Structure Research

# Members

# Our Networks

# Austrian Cooperative Research (ACR)


**The Woman Award 2017 goes to Evelin Fisslthaler**
ACR President Dr. Martin Leitl presented the ACR Woman Award to Evelin Fisslthaler for leading the FFG-funded project "Quantitative Analyse innerer Grenzflächen/Quantitative analysis of internal interfaces".

The ACR is a network of private research institutes offering applied R&D for companies. The demand-oriented services are tailored to meet especially the needs of SMEs, in order to support their innovation efforts, to provide the necessary know-how and to strengthen their competitiveness.

Presently, the ACR has 18 non-university cooperative research institutes with 64.4 million euro overall turnover and 775 employees.

The Association has been a full member of the ACR for 60 years and it collaborates with other institutes of the ACR. The Association benefits greatly from the co-operation with the ACR group, for example via special research funding coming from the Austrian Ministry of Digital and Economic Affairs. Since 1995, the ZFE has received funding of more than 5 million euros, including special funding for scientific instrumentation of around 1.2 million euros.

The ZFE is presently involved in strategic ACR projects such as "Microstructure of 3-D printed metallic devices", "Dust analysis of inner space air", "Tribology of polymers" and "Corrosion of materials".

Since 2008, the ZFE has received a total of 6 ACR Awards for collaborations with Austrian SMEs, and three female scientists have won the ACR Woman Award.

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*KOOPERATION MIT KOMPETENZ*

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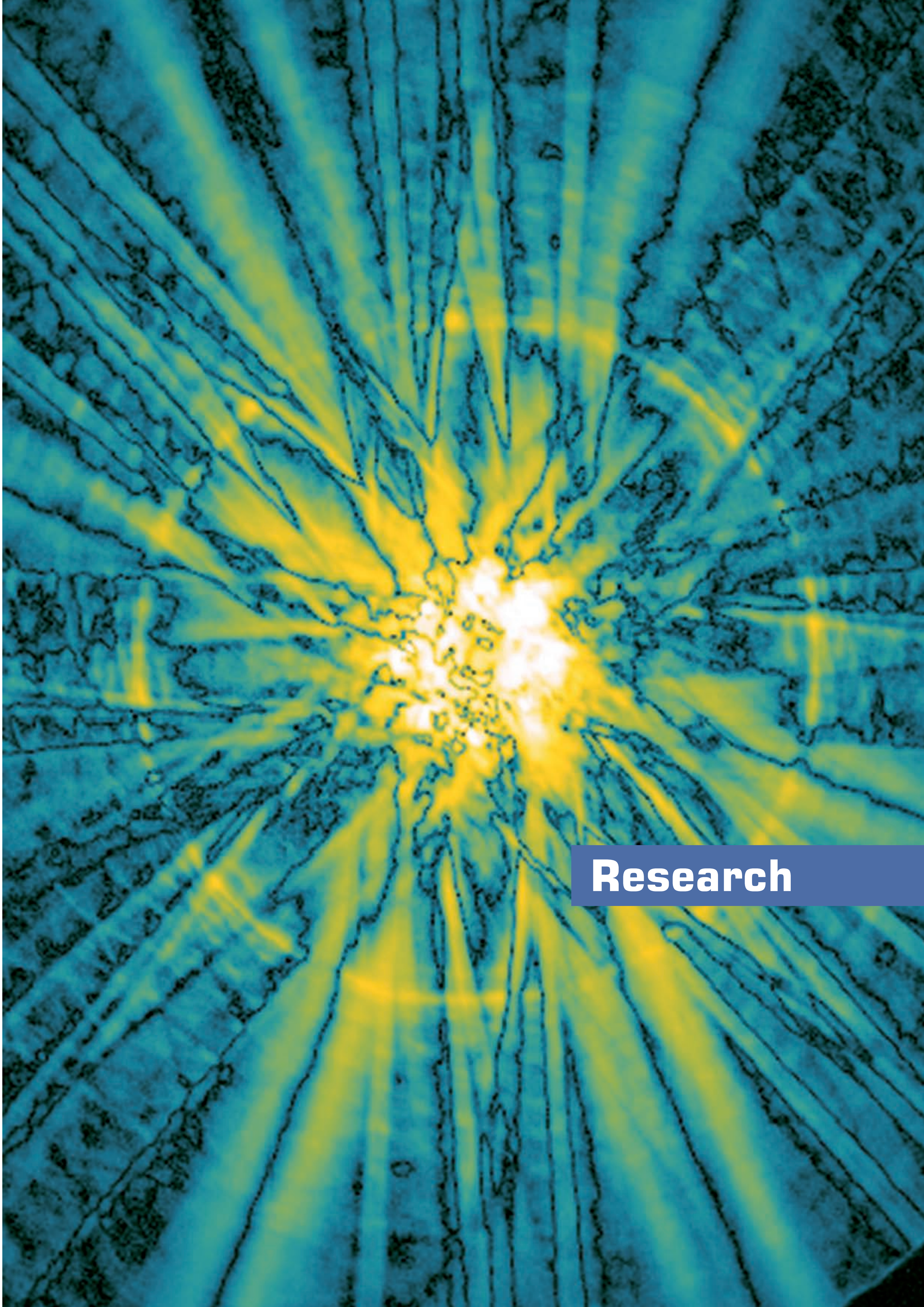
# Research



# Research Groups

## High Resolution Electron Microscopy

**Univ.-Prof. DI 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.

### From soft matter to inorganic compounds

The group of Werner Grogger works in the field of analytical transmission electron microscopy (TEM). The methods we use mainly focus on quantitative elemental analysis using X-ray spectroscopy (EDXS), high resolution STEM, diffraction and image simulation. These techniques are applied to materials ranging from hard coatings, perovskites and nanoparticles to organic materials and pharmaceuticals. In addition, the group focuses on experiments at low and elevated temperatures.

### Cryo-TEM as a tool to visualize hybrid nanoparticles

When polymers are anchored to solid surfaces at sufficiently high grafting densities, they form polymer brushes. If the surface is provided in the form of an inorganic nanoparticle (INP), so called hybrid nanoparticles emerge. This class of nanomaterials has gained significant attention mainly because (i) INPs are recognized as building units for the construction of materials with new collective properties, and because (ii) polymers are ideally suited to provide colloidal stability, ease of processability, and additional functionality. To visualize hybrid core–shell type nanoparticles from gold nanoparticle cores and poly(N-isopropylacrylamide) shells samples were stained with phosphorous tungsten acid and subsequently plunge frozen before carefully imaged at low electron dose under cryo conditions in the TEM to enable the visualization of the polymer patches in their native state by minimizing electron induced damage of the polymers [1].

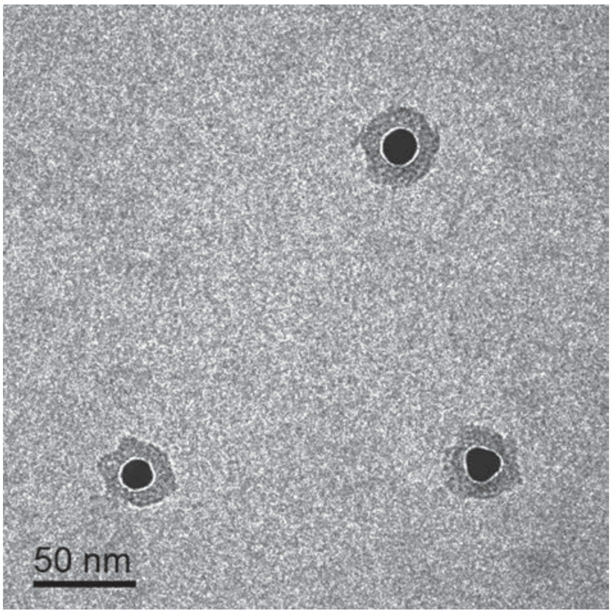



**Figure 1:** Cryo TEM: hybrid nanoparticles with Au core and poly(N-isopropylacrylamide) shell.

**Group Leader:**
Prof. Dr. Werner Grogger

**Senior Researchers:**
Dr. Evelin Fisslthaler
Dr. Christian Gspan
Dr. Ilse Letofsky-Papst

**PhD Students:**
DI Johanna Kraxner
DI Judith Lammer
DI Robert Krisper

## EDX(S) at Elevated Sample Temperatures

Over the last years, *in-situ* experiments in transmission electron microscopy (TEM) have significantly increased thanks to the introduction of specimen holder systems using micro-electro-mechanical system (MEMS) chips to mount the samples. EDX semiconductor detectors are naturally susceptible to infrared radiation (IR). Thus the influence of the emitted heat on background signal, energy resolution and elemental quantification in EDXS is of great interest. We simulated the detector illumination for characteristic X-rays based on Kraxner et al. [2] and compared the results with infrared-induced counts in the detector system over temperature and holder tilt. With our windowless SDD system, EDXS works up to up to 750 °C for deposited nano-particles or FIB-lamellae. Finding a convenient trade-off between X-ray shadowing and collected IR emission is still necessary as heat induced counts reduce the energy resolution.

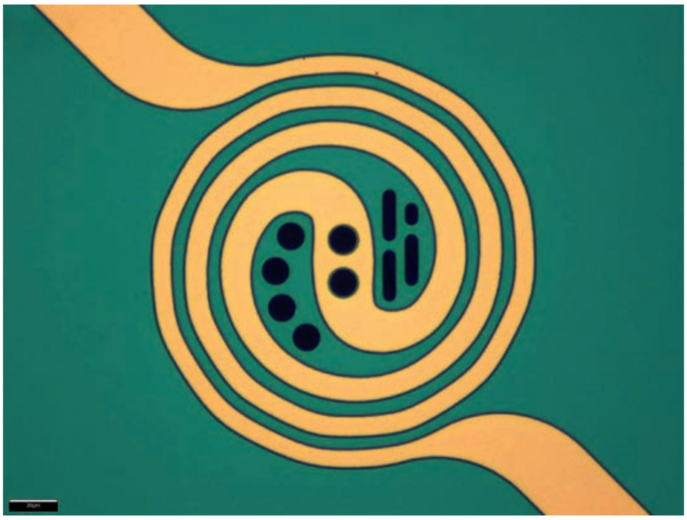


**Figure 2:** DENSsolutions Wildfire™ S3 nano-heater, resistively heated metallic spiral layer with a diameter of approx.150 µm. Light microscopy image (Alicona IFM).

## Ordering Phenomena in Rare-Earth Oxides

Rare-earth nickelates and ferrates are promising candidates for high temperature solid oxide fuel cell cathodes. However, the synthesis of the pure material is not straightforward. The characterization of the microstructure especially of crystal defects via electron microscopy is essential for the correlation with material properties. In praseodymium nickelate, high-temperature phase transitions, depending on the oxygen partial pressure, lead to the formation of domains (Figure 3 left). The appearance and the shape of these domains change with temperature. Rare-earth oxides also tend to form out-of-phase boundaries, which describe misalignments of the unit cell in one direction. Even though they are mainly reported in thin films, Lanthanum barium ferrate forms such misalignments within the bulk (Figure 3 right).

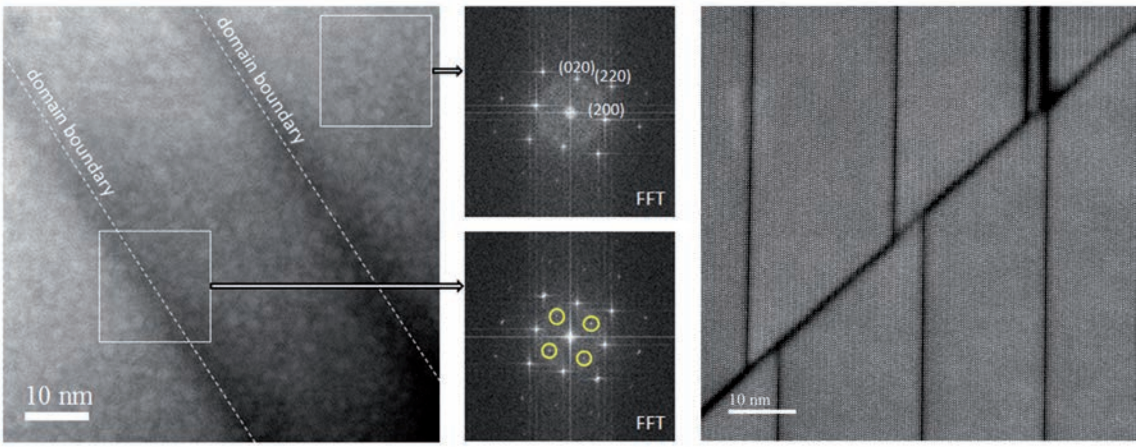



**Figure 3**: High resolution image (left) of the domains in praseodymium nickelate. FFTs show super structure reflections at domain boundaries (centre). The high-resolution image to the right reveals out of phase-boundaries in lanthanum barium ferrate.


**References:**

[1] C. Rossner, I. Letofsky-Papst, A. Fery, A. Lederer, G. Kothleitner, “Thermoreversible Surface Polymer Patches: A cryogenic transmission electron microscopy investigation”, Langmuir 34 (2018) 8622-8628.

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[3] E. Bucher, C. Gspan, T. Höschen, F. Hofer, W. Sitte, “Oxygen exchange kinetics of $La_{0.6}Sr_{0.4}CoO_{3-\delta}$ affected by changes of the surface composition due to chromium and silicon poisoning”, Solid State Ionics 299 (2017) 26–31.

**Univ.-Prof. DI 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.

# Analytical Electron Microscopy

## The many uses of electron tomography in physics and materials science research

Structural details and physical phenomena studied in a transmission electron microscope from a thin and transparent specimen sample often remain obstructed, when observed from a single view only. A sufficient number of projections, taken from different viewing angles, however, enable the three-dimensional reconstruction of yet unseen object features, greatly enhancing our knowledge about the properties of matter and their potential applications in technology.

### Electron tomography for ...

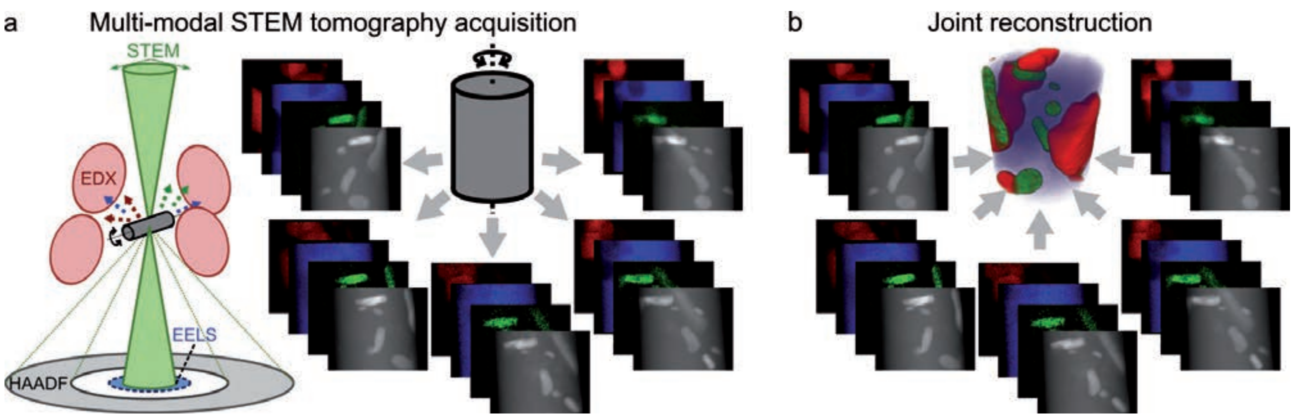
Analytical electron microscopy is a versatile tool in physical and materials science research and offers many ways to characterize exciting materials. The spherical-aberration corrected Austrian scanning transmission electron microscope (ASTEM), has given access to study matter with unprecedented spatial resolution and chemical sensitivity. Electrons passing through the sample encode a wealth of information ranging from physico-chemical parameters to nano-optical quantities. Yet, their visualization and the understanding of their impact in terms of properties imposed onto a certain class of materials, remains elusive. Consequently, over the past years, the AEM group has put tremendous effort into new data acquisition, analysis and reconstruction schemes, to better understand some of the so far uncovered effects. This shall be exemplified by two investigations, published in the high-impact journals Nature Materials and Nature Communications.

### ... decoding diffusion processes in nano particles...

Alloy elements such as scandium and zirconium are often added to an aluminum matrix to improve the strength, corrosion resistance and weldability of aluminum alloys. After further treatment, nano-precipitates only a few nanometers in size, are formed. Their form, atomic structure and the 'struggle' of the scandium and zircon atoms for the 'best place' in the crystal lattice are decisive for the properties and usability of the material. Knowing the exact precipitate composition is fundamental in the process of controlling the precipitation microstructure, and for optimizing the relevant heat treatments and alloy compositions to reach the desired properties.

However, defining whether a solvent atom belongs to the precipitate or the matrix is somewhat arbitrary and can only be answered by tomography. Such a tomographic analysis surprisingly revealed anomalies in the generated core-shell structures; something that could not be interpreted according to the previous level of knowledge. On the one hand, higher quantities of aluminum were found in the nano-precipitates but on the other hand, a zircon-enriched core was also discovered as well as border zones between the core and shell with an almost perfect composition and crystal structure.

Supported by quantum mechanical calculations and simulations, it turned out that a consequence of this stoichiometry deviation is that solute diffusion inside the precipitates becomes possible through Aluminium channels. This has changed the picture of precipitate formation and required subsequent heat treatments dramatically [1].



**Group Leader:**
Prof. Dr.
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**Senior Researchers:**
Dr. Mihaela Albu
Dr. Georg Haberfehlner
Dr. Daniel Knez
Dr. Karin Wewerka

**PhD Students:**
Mag. Lukas Konrad
DI David Moser
DI Angelina Orthacker
DI Cornelia Trummer

**Technical Assistants:**
Margit Brunegger
Martina Dienstleder
Ing. Claudia Mayrhofer

**Apprentices:**
Arnela Blažević
Paul Fastian

## ...and measuring light fields in 3D

Light as a carrier of information is indispensable to modern communication technology. The controlled manipulation of light quanta, so-called photons, form the basis for wireless transmission or data transfer in optical glass fibers. Due to the wave-like nature of light and its diffraction limit, however, optical components can only focus light down to the micron scale.

Coupling light onto metallic nanoparticles can overcome this problem and is subject of the field of plasmonics. With the ASTEM microscope it was possible for the first time to study the resulting resonating electron clouds along all three directions. The introduction of a novel tomography scheme allowed us to retrieve the complete local density of states of plasmonic nanoparticles with nanometer spatial and sub-eV energy resolution, a quantity of utmost importance in nano-optics.

The capability of mapping the 3D photonic environment is expected to be of great interest for a large variety of other nanophotonic systems, such as dielectric spheres or localized cavity modes in photonic crystals [2].

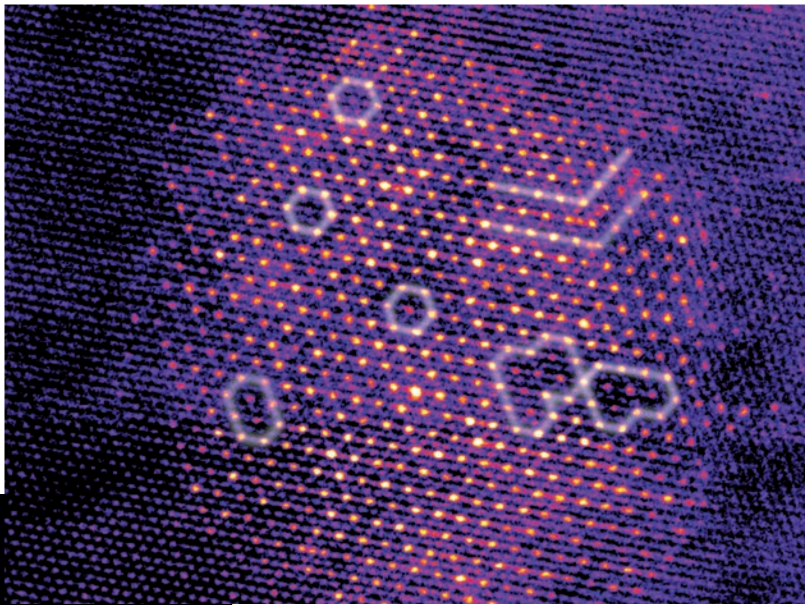

Fig. 1 HR-STEM HAADF image of nano-precipitate in an aluminium alloy with atom-sized diffusion channels, artificially colored to highlight the diffusion pathways [1].

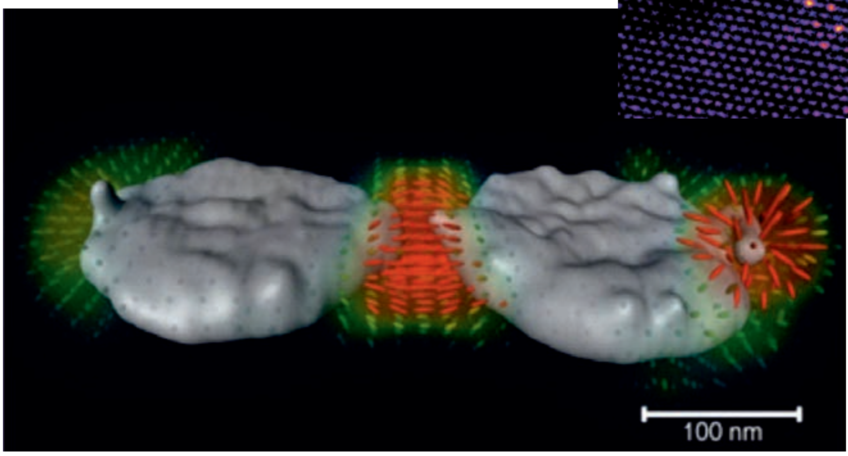


Fig. 2 3D tomographic reconstruction of the photonic density of states of two Ag nanoparticles [2].

**References:**
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# Functional Nanofabrication

**Ass.-Prof. Priv.-Doz. DI Dr. Harald Plank**

earned his PhD in Technical Sciences. Since 2010 he has been coordinating the work group S3 for "Functional Nanofabrication". In the course of his research activities he spent several months in the USA, Germany, Switzerland, South Korea, France and Turkey.

## From Fundamentals Towards Applications

### Focused Electron Beam Induced Processing (FEBID)

During the last decade, additive direct-write manufacturing has not only attracted significant attention in research but also found its way into commercial applications. An increasingly relevant aspect in this field is the possibility to fabricate three-dimensional architectures in a single step, which, for instance, is of high relevance if design complexity gets very challenging or classical multi-element assembly becomes even impossible. While fabrication down to the millimeter range and slightly below has meanwhile technologically matured, industry is currently working on reliable fabrication tools with resolution on the lowest microscale. To address the future needs, direct-write approaches for the sub-micro scale are currently in the research focus of many work groups worldwide.
Our institute is going a step further and has worked for a decade on the development of a true 3D nano-printing technology, called focused electron beam induced deposition (FEBID). This technique uses a nanometer-sized, precisely movable electron beam for highly localized dissociation of functional precursor molecules, delivered by a gas injection system as schematically shown in figure 1. The advantages of electrons, compared to other particles, are the widely non-invasive character, low temperature rises in the material or an implantation free situation after fabrication.

The activities of the workgroup S3 are focused on functional nanofabrication using the FEBID technology and range from fundamental studies towards applications in collaboration with industry. The former is the essential element for gradually leveraging FEBID from a more scientifically used technique into a predictable and reliable nanofabrication tool. This not only includes morphological aspects of fabricated structures but also material properties, which are decisive for the final functionality. Both together paved the way for the development of real applications, which attracted the interest of industrial partners. Joint efforts led to the Christian Doppler Laboratory for "Direct-Write Fabrication of 3D Nano-Probes" (CDL DEFINE), which started operation in March 2018 together with the Austrian company GETec Microscopy Inc. (see chapter Research Projects).

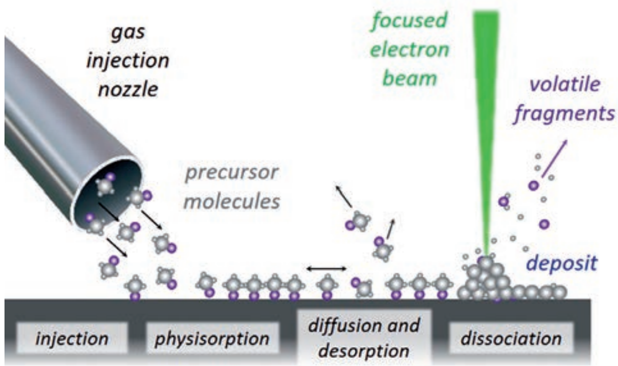


**Figure 1**: Working principle of focused electron beam induced deposition (FEBID) for direct-write fabrication of functional nanostructures.

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DI Jürgen Sattelkow
(FEBID Applications)
DI Mag. Dr.
Robert Winkler
(3D FEBID/Applications)

**Technical Assistant:**
Ing. Sebastian Rauch
(FIB)

## 3D Nano-Printing

Over the last five years, the group has focused on FEBIDs 3D possibilities. Although FEBID fabrication of freestanding features has been first demonstrated around the millennium, the quality lacked of precision, predictability and reproducibility. The reason might be found in the very complex process dynamics, which was barely understood at that time. As a natural consequence of our fundamental studies, we unraveled the process determining parameters and demonstrated that fabrication of freestanding 3D nanostructures is possible in a controlled way [1]. In a collaboration with the Oak Ridge National Laboratories in the US, we built up a profound understanding by combined experiments, theory and simulations [2]. Those efforts lead to a computer aided design (CAD) software package, called 3BID, which allows a reliable upfront design of even complex 3D nanostructures [3]. Figure 2 shows a few, post-colored examples, fabricated via FEBID based, direct-write 3D nano-printing. In the following, we demonstrated novel applications such as 3D plasmonics [4], quasi-1D nano-resonators for selective gas sensing [5] or magnetically frustrated 3D systems in a collaboration with the Goethe University Frankfurt in Germany. Our current emphasis is put on application oriented basic science in the frame of the CDL with focus on new 3D nano-probe concepts for application in GETec's in situ atomic force microscopy platform. At the same time, our research is currently focused on the expansion towards closed and semi-closed 3D architectures to expand FEBIDs flexibility, which will enable new, yet unexplored possibilities.

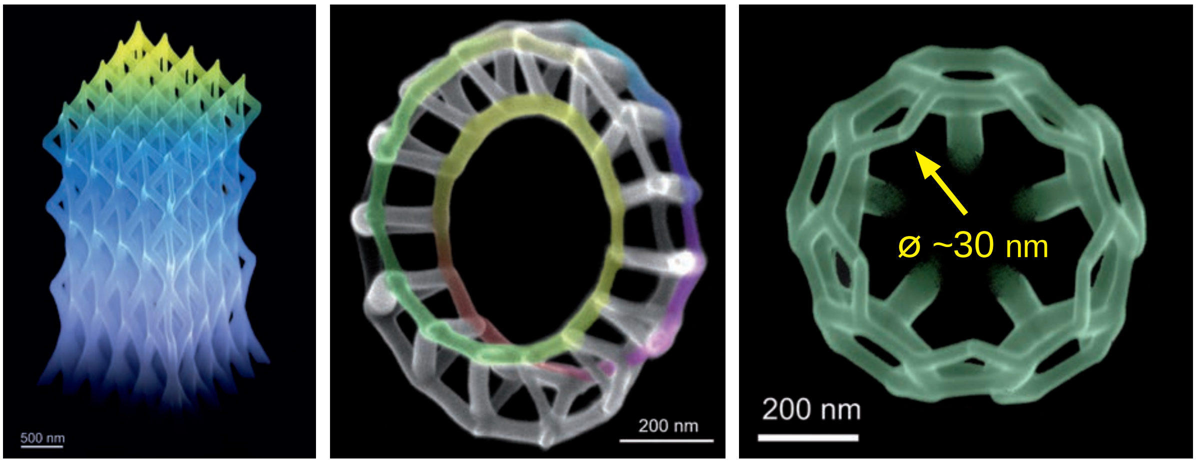


**Figure 2:** Selected examples of FEBID based 3D architectures. The possibilities range from comparable large 3D structures (left, SEM side view) over more complex architectures such as a Moebius strip (center, SEM top view) towards very small structures with features sizes down to about 30 nm (right, SEM top view). All examples were fabricated with a Pt based precursor in a single step on $SiO_2$ surfaces without any further modification. All Images are post-colored.

**References:**

[1] R. Winkler, B. B. Lewis, J. D. Fowlkes, P. D. Rack, and H. Plank, "High-fidelity 3D-nanoprinting via focused electron beams: growth fundamentals", ACS Appl. Nano Mater. 1 (2018) 1014.

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[5] L. Keller, M. K. I. Al Mamoori, J. Pieper, C. Gspan, C. Schröder, S. Barth, R. Winkler, H. Plank, M. Pohlit, J. Müller, and M. Huth, "Direct-write of free-form building blocks for artificial magnetic 3D lattices", Sci. Rep. 8 (2018) 6160.

# SEM/IR/Raman

**Hartmuth Schröttner**

Works at the FELMI-ZFE since 1991.
Group and project leader in the field of scanning electron microscopy (SEM) and micro-analysis since 2005.
Hartmuth Schroettner performes in the role of a first to contact person for cooperative research, quality control and troubleshootig with other institutes from universities, SMEs or industrial partners.

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## Advanced Materials Characterization

Due to our many years of experience and excellent materials know-how we can act as a solution provider for practical research problems. We offer multi-scale analysis of surfaces and materials with a wide variety of microscopy methods ranging from micrometer to nanometer resolution.

### A powerful team of experts

In 2017, the institute decided to merge the two previous SEM-groups SEM/IR/Raman and SEM & in-situ methods (due to the retirement of Peter Pölt) and to focus the new team on advanced methods of materials characterization.
The main areas of the group are now: 1) surface analysis, 2) correlative microscopy and 3) ESEM & *in-situ* microscopy.
The team is a main pillar of the institute and acts successfully in national and international cooperations in the fields of industrial and university research, failure analysis, trouble shooting, problem solving and quality control.
The group focuses its work on the border area between development and optimisation of advanced sample preparation methods and versatile microscopy techniques and its applications for providing a sustainable creation of value and know-how for our cooperation partners.

The ongoing development of modern materials demands the progressive advancement of existing methods [1] and new method combinations (for example: SEM & EBSD & heating stage by Stefan Mitsche, in figure 1).

### Joined focus on modern and new techniques

The worldwide first installation of the combination of high resolution SEM imaging, fast elemental analysis by EDXS and chemical mapping by Raman opens exciting opportunities for correlative microscopy and a wide variety of applications.

A team of specialists was formed around the new Raman integrated scanning electron microscope (RISE) system (Zeiss-Witec system, see page XX). Harald Fitzek focused his research on the near-field simulation of the substrate geometry for a better understanding of surface-enhanced Raman spectroscopy (SERS) (see page XX).

The new extremely fast EDXS system (Oxford Instruments) enables elemental mapping and spectrum imaging of very large sample areas and the automated particle analysis system can be applied searching small particles on filters or in dust samples. Large area elemental mapping allows to search the needle in the haystack in many applications (example in figure 2).

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**PhD Student:**
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**Technical Assistants:**
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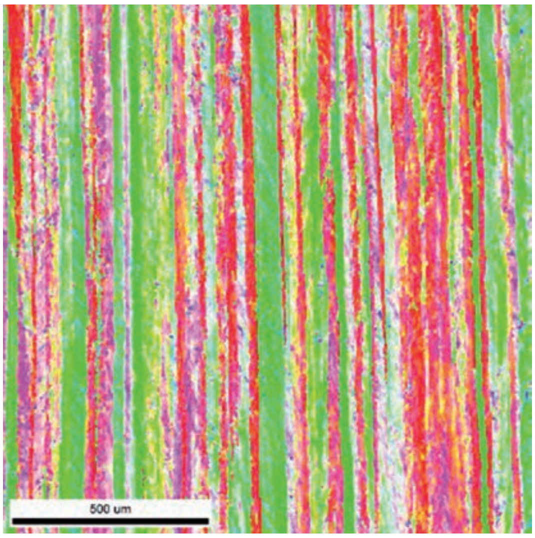
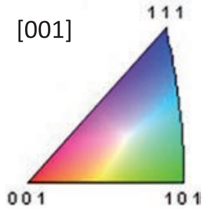
Sabrina Mertschnigg
Anita Rossmann-Perner
Šanja Simić



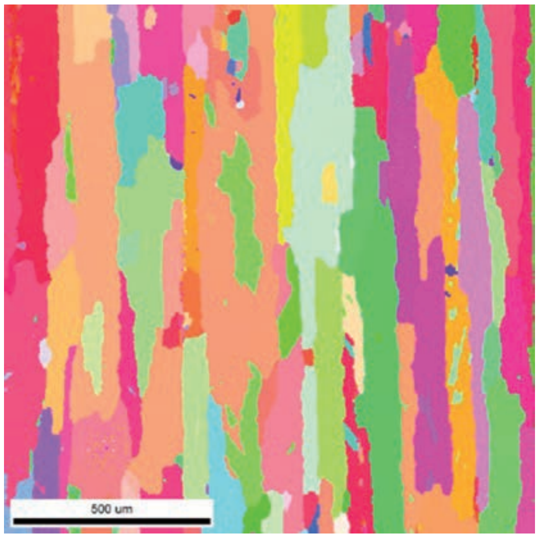




**Figure 1**: New method combination of SEM & EBSD & heating stage: AlSi7Mg automotive alloy - heavily rolled. Left: EBSD IPF microstructure after rolling, right: EBSD IPF microstructure after *in-situ* heat treatment.

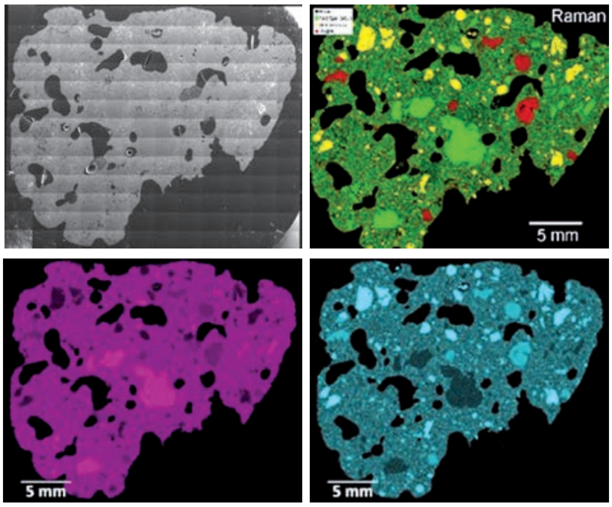
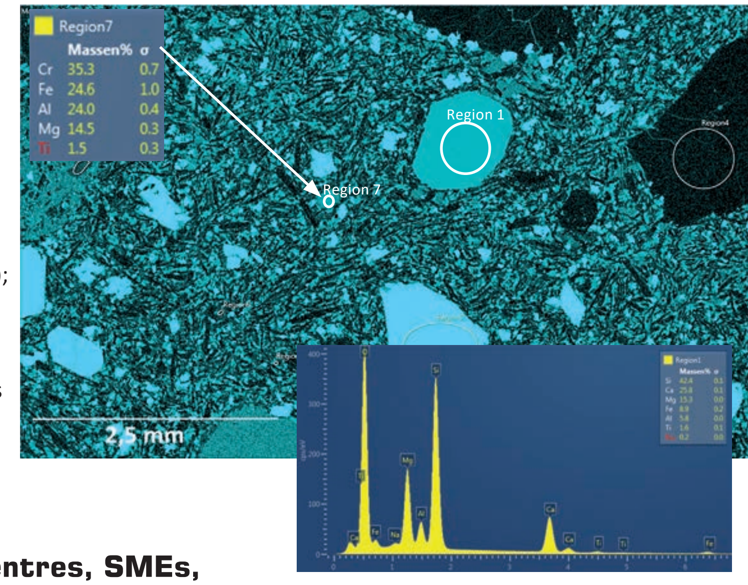



**Figure 2:** Large area mapping of a geological sample - cross-section - 156 fields with 2777*2295 pixel => 6.3 Mpixel.
Left: Stitched SE-image; Mg-elemental map (magenta); Si-elemental map (cyan); correlative Raman map (RGB),
right: Detail from the large area map shows the full spectral information in every pixel.




## Research Partner for Universities, COMET -Centres, SMEs, Industry and ACR-Institutes

Research projects were performed in cooperation with ACR institutes, institutes from universities and COMET-Centres and concentrated on light weight magnesium and aluminium alloys (figure 1), nitrocarburised and oxidised steel surfaces [2], solid oxide fuel cells (SOFC) [3], state-of-the-art Li-ion cells, various catalysts as well as on different aspects of pharmaceutical engineering, paper and polymer-characterisation.

An important example of our research is the co-operation with the OFI in Vienna: In the FFG funded project "Aeropore“ Johannes Rattenberger and Manfred Nachtnebel focus on the efficiency of filters and filter systems for allergens and pathogenic germs. They use a sophisticated method, immunogold labelling, to locate specific proteins responsible for an allergic reaction. This technique was adapted for environmental scanning electron microscopy (ESEM) (see page XX).

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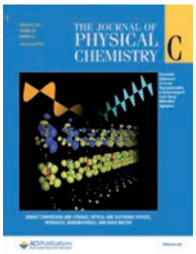
# Research Highlights

## Accurate Near-Field Simulations of the Real Substrate Geometry-A Powerful Tool for Understanding Surface-Enhanced Raman Spectroscopy

**Harald Fitzek, Jürgen Sattelkow, Harald Plank, Peter Pölt**



J. Phys. Chem. C, 122, 12, 6826–6834, (2018)

Surface-enhanced Raman spectroscopy (SERS) is apowerful analytical technique that is used in a wide range of research areas, such as analytical chemistry, lifescience and material science. The fundamental property of SERS is the enhancement (by several orders of magnitude) of the Raman signal from molecules close to or adsorbed on the surface of nanostructure. The bulk of this enhancement is attributed to the amplification of the electric fields near the surface of suitable nanostructures. Therefore characterizing these electric near fields precisely would be highly desirable for any SERS experiment.

The calculation of the electric near fields was accomplished in this work by a new approach combining precise microscopic measurements with a homemade MATLAB implementation of the discrete dipole approximation (DDA). A schematic summary of the approach is shown in the figure below. The main advantage of this combination is that the real substrate geometry is taken into account in the simulation, whereas most comparable studies use some approximated geometry.

In addition the whole approach was tested by calculating the average electromagnetic enhancement factors for two batches of substrates and comparing the results to measurements of the enhancement factors on the same batches. Using our approach it was possible to predict the average enhancement factor with good accuracy. Thus the combination of precise microscopy measurements with the DDA can offer further interesting insights into the origin of SERS and for the characterization of SERS substrates.

Further research developing SERS substrates with defined and characterized electric near fields as well as applications of those substrates and the approach developed in this work is currently ongoing at the FELMIZFE.

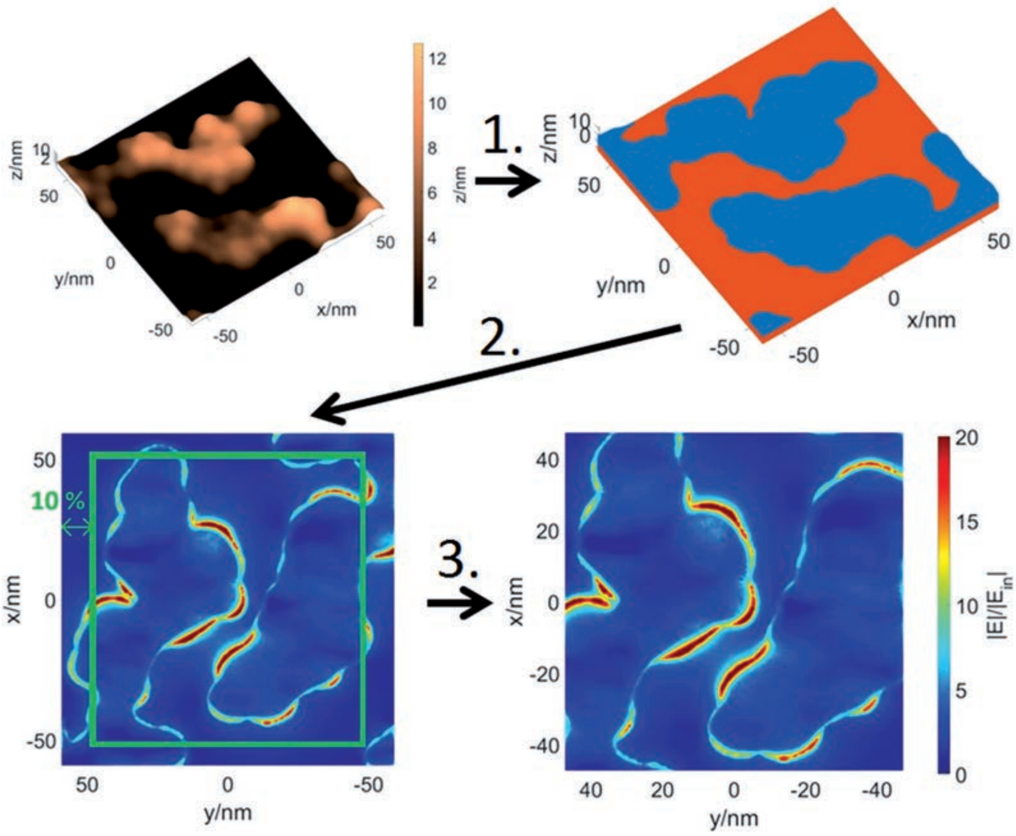



**Figure:** Schematics of the sequence for generating a simulation result. Step1: Discretization of an AFM measurement of the surface. Step 2: Solution of the electromagnetic scattering problem by the DDA-algorithm. Step 3: Reduction of the simulated area to exclude edges effects and other artefacts.

# Quantitative EDXS: Influence of Geometry on a Four Detector System

**J. Kraxner, M. Schäfer, O. Röschel, G. Kothleitner, G. Haberfehlner, M. Paller, W. Grogger**


Ultramicroscopy 172 (2017), 30–39

In energy dispersive X-ray spectrometry (EDXS), the illuminated X-ray detector area plays an important role when it comes to quantification of light elements. Advanced quantification methods (e.g. the ζ-factor method) assume that the detector illumination is a constant for each detector system. This is usually the case, when the sample is tilted towards the detector and the sample holder does not shadow the detector area. High-performance detectors nowadays do not consist of only one detectors, but of various detectors mounted symmetrically around the specimen. Therefor tilting towards one detector, immediately causes shadowing of other detectors and influences the quantification result. In this work the influence of the geometry on quantitative EDXS analysis is determined for a four-detector-system in combination with a low-background double-tilt specimen holder. For the first time a combination of experimental measurements with simulations is used to determine the positions of the individual detectors of a Super-X system. These positions allow us to calculate the detector's solid angles and estimate the amount of detector shadowing and its influence on quantitative EDXS analysis, including absorption correction using the ζ-factor method. Both shadowing by different parts of the holder (the brass portions and the beryllium specimen carrier) severely affect the quantification of low to medium atomic number elements. A multi-detector system is discussed in terms of practical consequences of the described effects, and a quantitative evaluation of a Fayalit sample is demonstrated. Corrections and suggestions for minimizing systematic errors are discussed to improve quantitative methods for a multi-detector system.

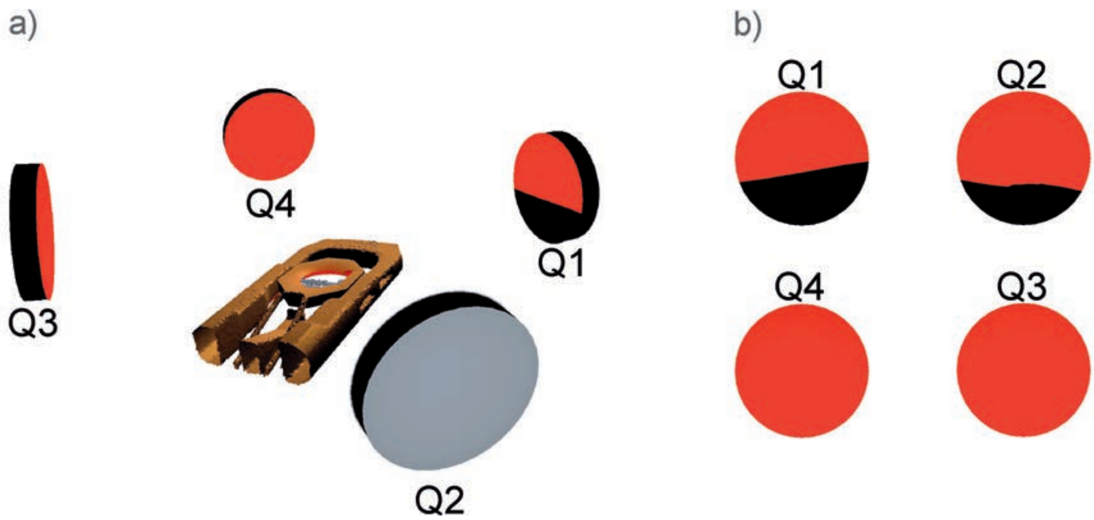
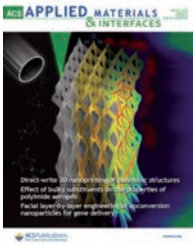



**Figure:** a) Simulated setup of the four detectors and the sample holder. b) Shadow images of the quadrants Q1, Q2, Q3, and Q4 at a specimen tilt of α=−10° and β=3°

# Direct-Write 3D Nanoprinting of Plasmonic Structures



ACS Appl. Mater. Interfaces 9, 8233 (2017).

**R. Winkler, F.-P. Schmidt, U. Haselmann, J.D. Fowlkes, B.B. Lewis, G. Kothleitner, P.D. Rack, and H. Plank**

During the last decade, resonant optics attracted enormous interest in science and technology as this research field provides deep insights in fundamental physics but also enables an increasing number of applications ranging from filters over waveguides towards sensor devices. While several techniques for the fabrication of metallic structures have been introduced the direct-write fabrication of highly defined structures on the nanoscale, especially for complex three-dimensional geometries on non-flat surfaces is still an intractable challenge.

Recently, Focused Electron Beam Induced Deposition (FEBID) has taken a huge step forward in terms of fabrication of predictable and complex three-dimensional geometries, leveraging this technique into the status of a nano-printer not only for 2D but also in real 3D on almost any substrate material and morphology.

Beside the reliable shape performance on the nanoscale high purity of the material is essential for plasmonic activity. Therefore, the direct usage of FEBID structures for plasmonic investigations is impossible due to the high carbon impurities of about 90 at.%.

In this work, we first demonstrate the suitability of FEBID Au-materials for plasmonics after an electron beam assisted purification approach in water vapor, which provides pore- and crack-free high-fidelity shapes with a pure metal character.

Then we demonstrate the improvements of 3D-nanoprinting via FEBID by the fabrication so far unrivaled multibranch nanoarchitectures with a high degree of complexity. After the purification of freestanding 3D-geometries (Figure) with sizes down to 20 nm we finally prove their plasmonic activity via STEM-EELS (Scanning Transmission Electron Microscopy based Electron Energy Loss Spectroscopy), which opens up new opportunities for advanced applications in research and development.

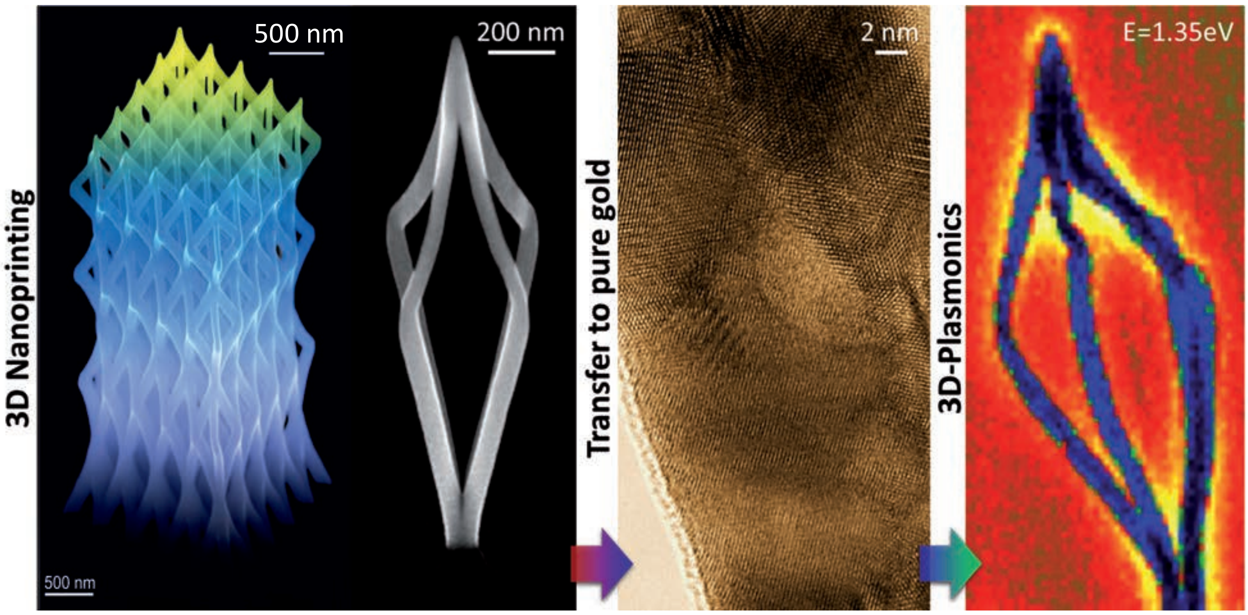



**Figure:** 3D-nanoprinting of plasmonic active FEBID-structures: First, complex 3D-nanoarchitectures are reliably fabricated (left). After that a purification step utilizing electron stimulated reactions with water vapor is introduced to transfer the Au-C deposition into pure gold as shown via TEM characterization (center). Finally, STEM-EELS investigation revealed plasmonic activity (right)

# Tunable 3D Nanoresonators for Gas-Sensing Applications

**G. Arnold, R. Winkler, M. Stermitz, A. Orthacker, J.-H. Noh, J. D. Fowlkes, G. Kothleitner, M. Huth, P. D. Rack, and H. Plank**


Advanced Functional Materials, 28, 1707387 (2018).

The detection of gas species with high sensitivity is a significant task for fundamental sciences as well as for industrial applications. Similarly, the ongoing trend for device miniaturization brings new challenges for advanced fabrication including on-demand functionality tuning. Following this motivation, functional and small, we here use Focused Electron Beam Induced Deposition (FEBID) for the additive, direct-write fabrication of freestanding 3D nanoarchitectures. In more detail, we deposit quasi 1D nanostructures, which can be brought into mechanical resonance via electric AC fields (see Figure 1.a and 1.c). Once gas molecules are introduced, they attach to the nano-resonators, which changes the intrinsic resonance behavior (Figure 1.b). This, in turn, entails an eventual stop of the resonance as shown in Figure 1.d. By that, such a concept can be act as highly sensitive gas sensor.

To exploit the full potential of this basic idea, this study focuses on the synthesis of such 3D nanostructures, then determines the Young modulus and demonstrates a postgrowth procedure, which can precisely increase the material modulus. As-fabricated resonators reveal a Young's modulus of 9–13 GPa, which can be increased by a factor greater than 5. To enable an electric readout of the resonance behavior, the design is then changed from straight pillars into 3D nano-arches, which bridge two electrodes. Such devices are then characterized in various conditions, which confirms an electric readout of the resonance behavior. Finally, the implications of gas-physisorption and gas-chemisorption on the resonance frequencies are studied, representing a proof-of-principle for sensing applications by the here presented approach.

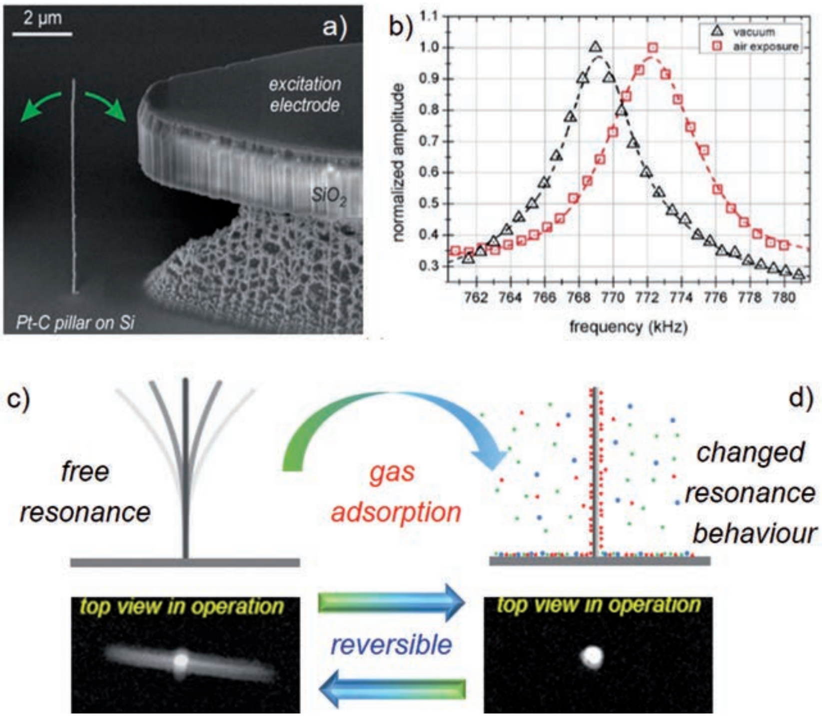



**Figure:** Nanoresonator basic principle. (a) shows a tilted SEM image with a quasi-1D Pt-C nanopillar next to an electrode. Once, an electric AC field is applied to the latter, the nanopillar can be brought into mechanical resonance as shown in (c) by a side view scheme and a top view SEM image. When such a resonator is exposed to gas molecules, they attach on the nanopillar (upper scheme in d), which change the resonance behavior, leading to a halt of the resonance as shown by the SEM top view images in (d). (b) shows the according resonance curve, which clearly confirm that basic idea. Based on this concept, the study explores fabrication, mechanical tunability, optimization, electric operation and pyhsical / chemical details during gas exposure to provide a comprehensive picture.

# Spectrum Image Analysis Tool - A Flexible MATLAB Solution to


Micron 93, 43-51 (2017)

**F.-P. Schmidt, F. Hofer, J. Krenn**

The combination of imaging and spectroscopy in a (scanning) transmission electron microscope ((S)TEM) has a high potential as it correlates structural information with a variety of materials properties. In particular electron energy loss spectroscopy (EELS) and cathodoluminescence (CL) in a STEM, where a highly focused electron beam (in aberration corrected TEMs down to 0.05 nm) is scanned over the specimen, are of great interest for many application fields. For example, the comparison of EELS and CL provides unique and complementary information on the optical properties of plasmonic systems. The data are recorded in the form of spectrum images, so-called three dimensional data cubes, consisting of images of the sample (x-y plane) at different energies (z direction).
However, correlative EEL/CL spectrum images require suitable and careful processing to extract information of the dataset. In the article the authors introduce a MATLAB based software that uses three-dimensional data (EEL/CL spectrum image in DM3 format (Gatan Inc.'s DigitalMicrograph®)) as input. A graphical user interface enables a fast and easy mapping of spectral dependent images and position dependent spectra. First, data processing such as background subtraction, deconvolution and denoising, second, multiple display options including an EEL/CL moviemaker and, third, the applicability on a large amount of data sets with a small work load makes this program an interesting tool to visualize otherwise hidden details. The method has been successfully used for the investigation of plasmon eigenmodes of a gold nanostar and a silver nanotriangle.

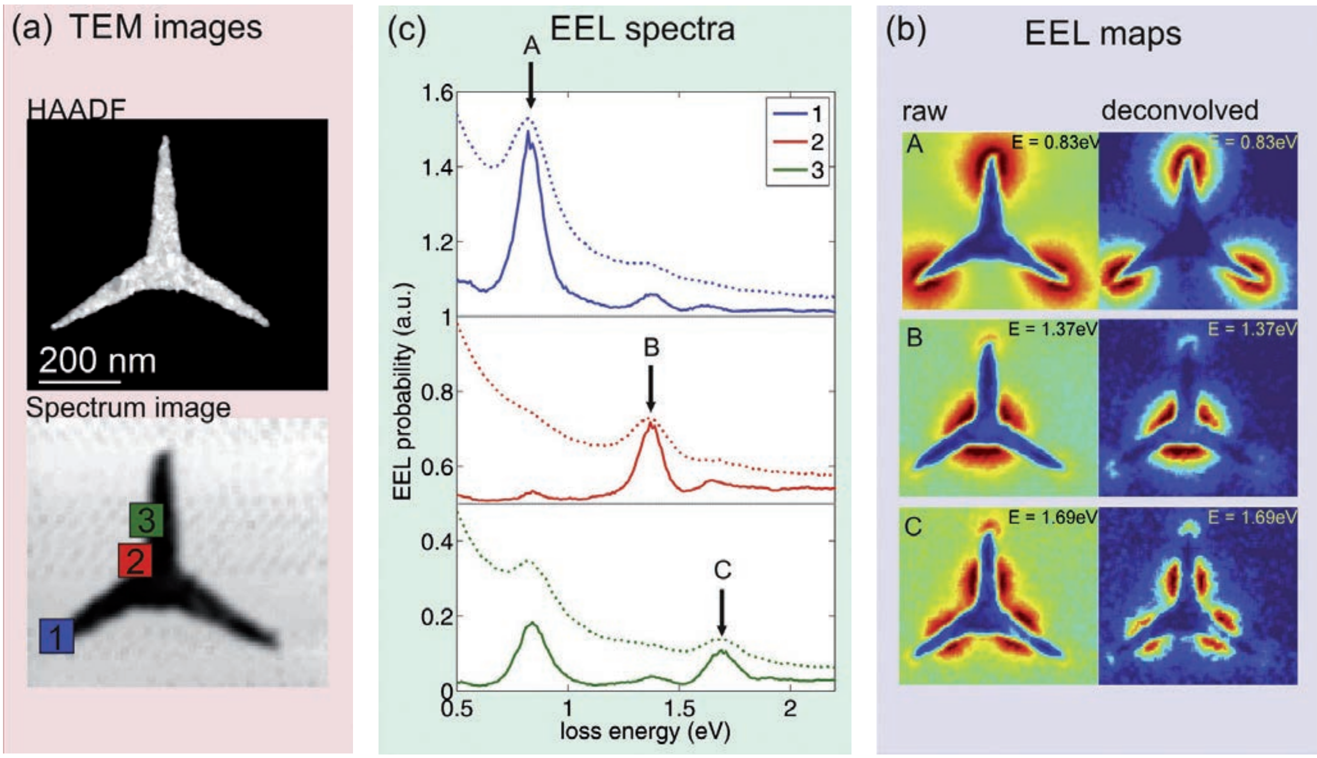
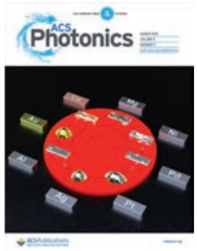



**Figure:** EELS maps and spectra of a gold nanostar, (a) electron micrographs of a 30 nm thick gold nanostar on a 15 nm thin Si3N4 substrate, STEM HAADF image (top) and EELS map integrated over the elastically scattered electrons (bottom), (b) EELS maps of three different plasmon eigenmodes at energies as indicated in the insets, raw data (left) and deconvolved data (right), (c) EEL spectra originating from three different regions 1–3 as indicated in (a), raw data (dotted lines) and deconvolved data (solid lines)

# How Dark Are Radial Breathing Modes in Plasmonic Nanodiscs?

F.-P. Schmidt, A. Losquin, F. Hofer, A. Hohenau, J. Krenn, M. Kociak




ACS Photonics 5, 861-866 (2018)

In recent years, focused electron probes have opened new paths for the spectral analysis of nanostructures with broadband optical sensitivity. Electron energy-loss spectroscopy (EELS) in a TEM/STEM provides information with nanometre spatial resolution and sub-eV spectral resolution. EEL spectroscopy allows to record the linear optical properties of nano-objects and is now the most important method to study plasmon eigenmodes in nanostructures. A related technique is cathodoluminescence (CL), but it has been used very rarely in TEM or STEM, even though it has been known for some time that it provides complementary information to EELS. In this report we show one of the first combined EELS/CL measurements to elucidate how the dark mode character of plasmonic nanodiscs is relived with increasing disk diameter.

Due to a vanishing dipole moment, radial breathing modes in small flat plasmonic nanoparticles do not couple to light. Therefore, they have to be probed with a near-field source, as in electron energy-loss spectroscopy. With increasing particle size, retardation gives rise to light coupling, enabling probing breathing modes optically or by cathodoluminescence. Here, we investigate single silver nanodisks with diameters of 150 - 500 nm by EELS and CL in an dedicated STEM* and quantify the EELS/CL ratio, which corresponds to the ratio of full to radiative damping of the breathing mode. For the investigated diameter range, we find the CL signal to increase by about 1 order of magnitude, in agreement with numerical simulations. Due to reciprocity, our findings corroborate former optical experiments and enable a quantitative understanding of the light coupling of dark plasmonic modes.

* The investigations have been performed with the VG HB501 STEM (cold-FEG, 100 kV) of the Laboratoire de Physique des Solides, CNRS and Université Paris-Sud, Orsay, France.

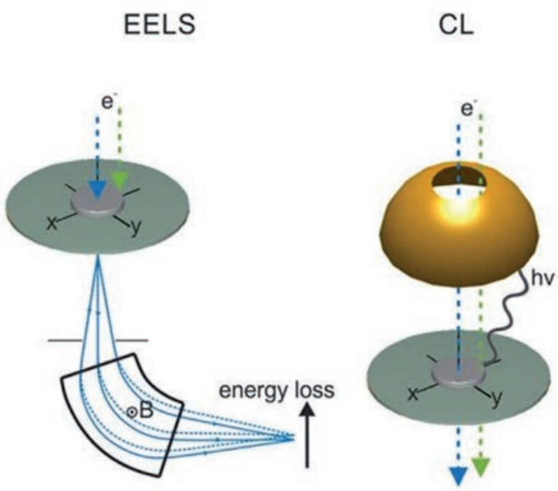



**Figure 1:** Sketches of experimental EELS and CL setups in a scanning transmission electron microscope.

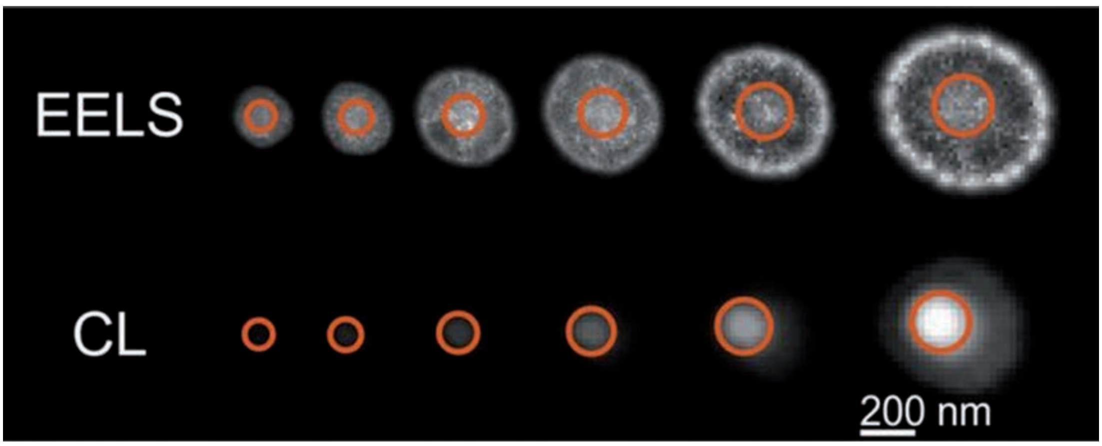



**Figure 2:** EELS and CL maps of the radial breathing mode of single silver nanodiscs. The inner area of the orange circles indicates the region with the highest radial breathing mode signal.

# A consistent path for phase determination based on TEM techniques and supporting simulations


Micron 115, 41–49 (2018)

**L. Konrad, H. Zhao, C. Gspan, J. Rehr, U. Kolb, M. Lattemann, G. Kothleitner**

The complex phase chemistry and structural diversity of industry relevant materials such as hard metals require several characterization techniques to be employed simultaneously. However, these methods often lack connection to yield a complete and consistent picture. This also applies to transmission electron microscopy (TEM), in which various characterisation methods, ranging from electron diffraction and TEM imaging to spectroscopic methods, are used.
In this paper, we describe a continuous path for quantitative TEM work, starting with the acquisition of 3D electron diffraction data – alongside classical high-resolution imaging techniques – and linking the structural characterisation with energy-loss fine-structure simulations, quantitative electron energy-loss (EEL) and energy-dispersive X-ray (EDX) spectroscopy.
Three hard metals were studied in detail: tungsten carbide (WC) as a demonstrator to connect crystallographic data obtained by electron diffraction tomography with electron energy-loss fine structures, an unknown titanium carbonitride coating on a WC substrate and a Ti3SiC2 MAX phase, acting as a reference.
The structure of the MAX phase could be directly visualised via high-resolution STEM imaging (fig. 1). The nano-laminated crystal revealed a stacking sequence common to the n = 2 system and the HAADF-images show that the TiC layers are twinned with respect to each other and separated by the Si mirror plane.
The combination of crystallographic data and the analytical methods effectively paves the way to more reliable EELS quantifications including spectral simulations with the FEFF code (fig. 2). The multiple scattering calculations not only confirmed the phases of the examined samples, but also revealed the need for modifications of the screening parameters for the hydrogenic cross-sections for EELS quantification.

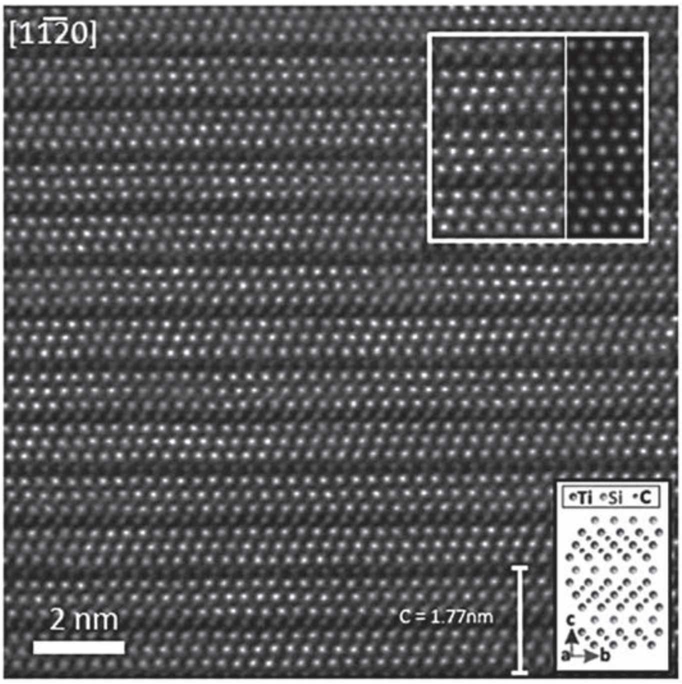



**Figure 1** STEM-HAADF image of the MAX phase Tin+1 SiCn (n=2), grown on corundum.

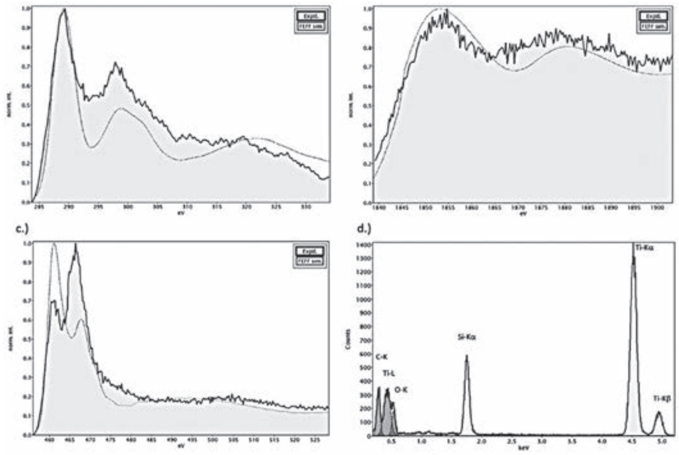



**Figure 2** Experimental EELS edges of Ti3SiC2, taken under off-axis, non channeling conditions, in comparison with FEFF-code simulation (dotted line); (a) C-K, (b) Si-K, (c) Ti-L2,3, (d) EDS spectrum.

# Modelling Electron Beam Induced Dynamics in Metallic Nanoclusters

**D. Knez, M. Schnedlitz, M. Lasserus, A. Schiffmann, W. E. Ernst, F. Hofer**


Ultramicroscopy 192, 69–79 (2018)

Aberration corrected scanning transmission electron microscopes (STEM) have proven their excellent capability of characterising nanomaterials. However, due to the high current densities, the focused electron probe can also damage the sample during the examination. This beam controlled dynamics has been known for metallic nanoparticles for a long time and affects single atoms on the surface in particular.

To better understand the underlying processes a new computational scheme to simulate beam induced dynamics of atoms in surface dominated, metallic systems was developed. This model is tested with clusters comprised of either Ni, Ag or Au. We vary their sizes, and apply different electron energies and cluster temperatures to elucidate fundamental relations between these experimental parameters and beam induced displacement probabilities.

Furthermore, we demonstrate the capability of our code to simulate beam driven dynamics by using Ag and Au clusters as demonstration systems. The clusters in question are synthesised with exceptional purity inside inert superfluid He droplets and deposited on amorphous carbon supports. The simulations of beam induced displacement and sputtering effects are compared with experimental results obtained via STEM with HAADF electrons, showing good qualitative agreement and, to some extent, even quantitative information could be extracted. The simulations can also help to control the dosage in cluster experiments to avoid damage to the studied structures.

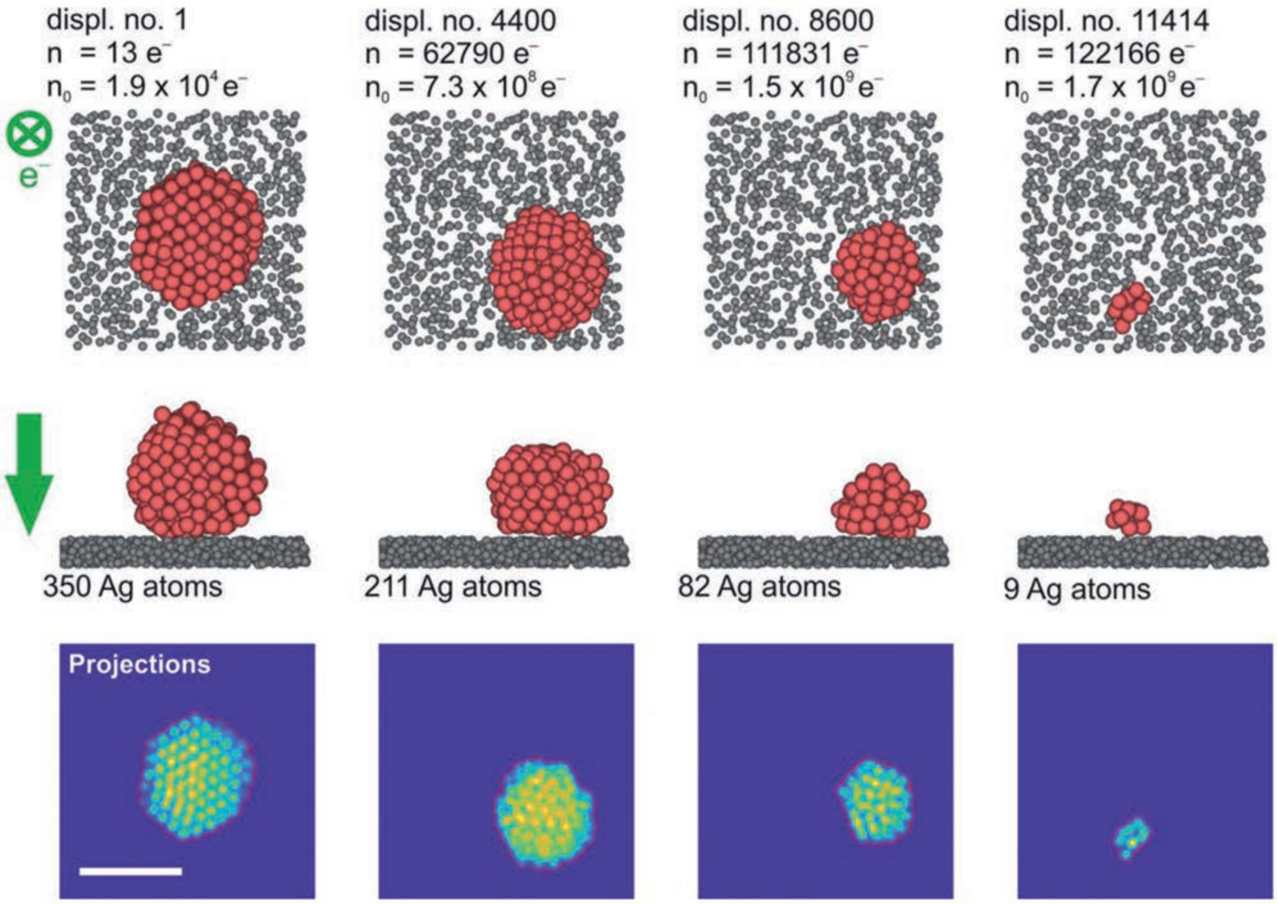



**Figure:** Transient evolution of a Ag cluster initially consisting of 350 atoms under 300 keV electron irradiation, together with the corresponding Gaussian projection images and automatically determined contours. n corresponds to the number of electron scattering events in the simulation; n0 is the corresponding number of incident electrons, scale bar is 2 nm.

Nano Letters 17,
6773-6777 (2017)

# 3D Imaging of Gap Plasmons in Vertically Coupled Nanoparticles by EELS Tomography

G. Haberfehlner, F.-P. Schmidt, G. Schaffernak, A. Hörl, A. Trügler, A. Hohenau, F. Hofer, J.R. Krenn, U. Hohenester, G. Kothleitner

Over the last decade, electron energy-loss spectroscopy (EELS) in a scanning transmission electron microscope (STEM) has been established as an ideal tool for direct observation of plasmonic fields at the nanoscale. Recently, gap plasmonics has become of great interest because it leads to an extreme light concentration in narrow gap regions of coupled noble metal nanoparticles. The fields in the gap region become strongly enhanced by coupled plasmon modes, giving rise to a myriad of novel effects. However, conventional 2D STEM-EELS is only sensitive to components of the photonic local density of states (LDOS) parallel to the electron trajectory. Therefore, it is insensitive to specific gap modes, a restriction that can only be lifted with tomographic 3D EELS imaging.

To unravel the interplay between structure and plasmonic properties of coupled nanoparticles the authors studied vertically stacked silver nanotriangle dimers separated by an insulating layer. The precise 3D geometry of the particles could be revealed by EELS tomography and the gap mode LDOS of the nanotriangle dimer can be fully imaged. Besides probing the complete mode spectrum, it is demonstrated that the tomographic approach allows disentangling the signal contributions from the two nanotriangles that superimpose in a single measurement with a fixed electron trajectory. The EELS results were compared with optical extinction spectra and simulations of optical and electron beam excitations using the boundary element method. The EELS experiments show for the first time the vertical coupling by imaging the 3D LDOS distribution of two stacked nanotriangles and allow to distinguish between the bonding and antibonding configurations.

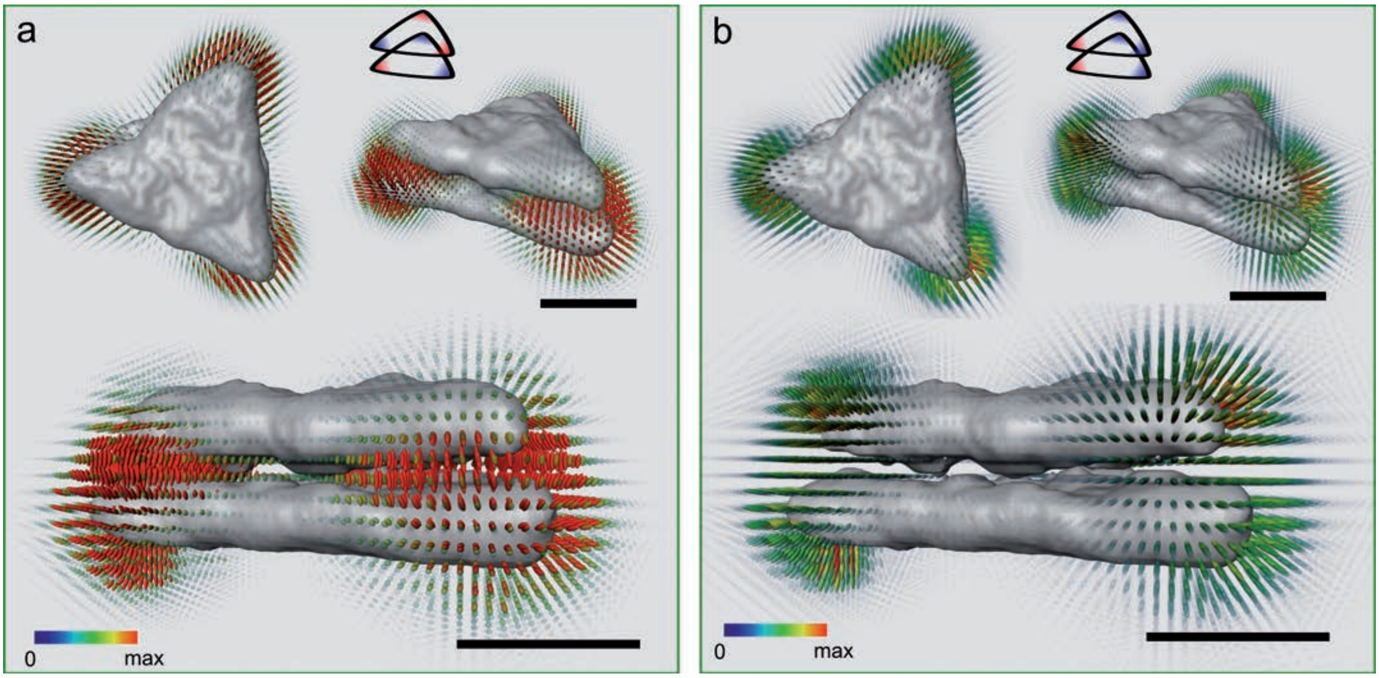



**Figure 1:** 3D reconstruction of the photonic LDOS of a silver nanoparticle dimer: (a) low-energy (bonding) mode and (b) high-energy (antibonding) mode. The orientation of the pencils indicates the direction along which the LDOS is maximal. Color, transparency, and length correspond to the magnitude of the full LDOS. Scale bars are 100 nm.

# Diffusion Defining Atomic Scale Spinodal Decomposition within Nanoprecipitates


## A. Orthacker, G. Haberfehlner, J. Tändl, M.C. Poletti, B. Sonderegger, G. Kothleitner



Nature Mater 17, 1101–1107 (2018)

Precipitates within alloys have a major impact on mechanical properties. Examples are aluminum alloys used in aircraft and space technology, where scandium and zirconium are added to improve strength, corrosion resistance and weldability. Determining the composition of nanometer-sized precipitates, and especially its evolution is a complex task, which requires atomic-resolution analysis, as well as information about the sample composition in 3D, in order to be able to separate precipitates from the matrix material.
Within the Al-alloy spherical Al3(Sc,Zr) precipitates with a core/shell structure are formed. Unexpectedly tomographic analysis of the precipitates' composition revealed higher than expected aluminum concentrations in the precipitates, along with non-neglectable concentrations of zirconium in the core and a region with the lowest aluminum concentration at the core/shell interface (Figure 1). Previously, it was assumed that scandium, which is diffusing faster than zirconium forms the Sc-enriched core of the precipitates, while later on a Zr-rich shell is formed, as no more diffusion is possible in a perfect Al3(Sc,Zr) structure.
The presence of Zr in the core along with the excess concentrations of Al indicated that this assumption does not hold. Understanding of this behavior was possible through atomic-resolution STEM images along with simulations, which both showed that with an excess of Al in the precipitates local regions with a high number of Al on Sc/Zr positions are formed. In these regions, which are visible as channel-like defects in atomic-resolution STEM images (Figure 2), diffusion within the precipitates is still possible, at least until the Sc/Zr concentration gets high enough leading to local areas of Al3(Sc,Zr), which eventually seal off the core region.

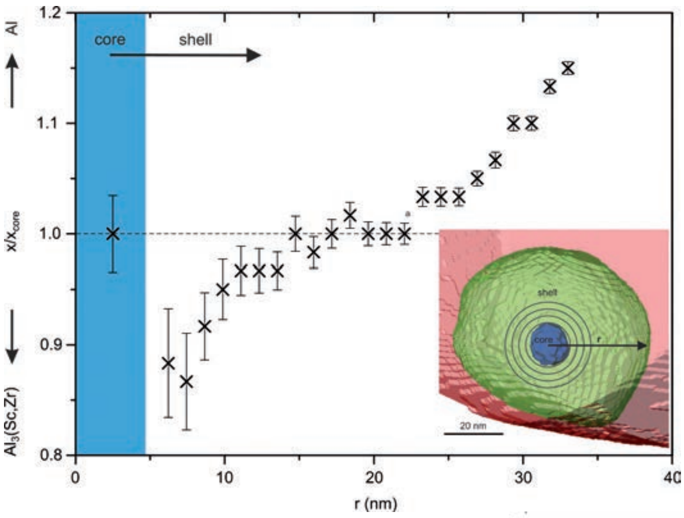



**Figure 1.** 3D reconstruction of core/shell (blue/green) particle and average relative compositional, shown as deviation of Al concentration compared to the value found for the core.

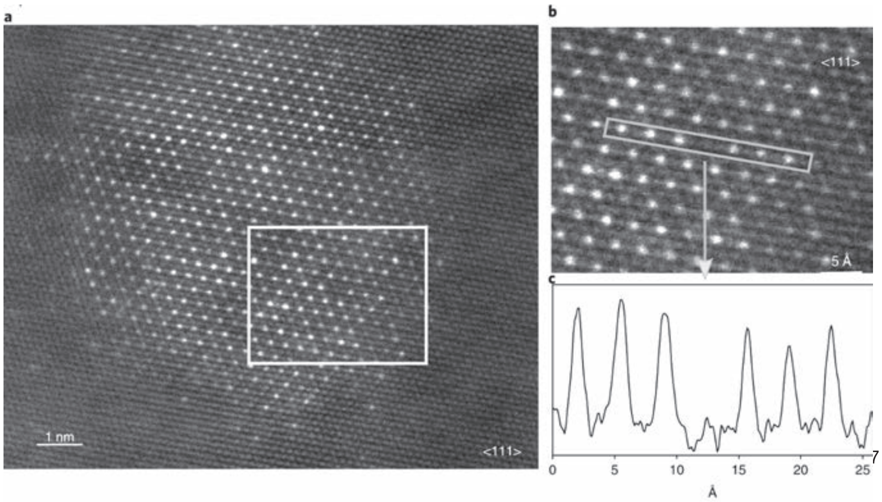



**Figure 2 (a)** Atomic-resolution HAADF image of a aged precipitate. (b) detailed view of the region indicated in (a) (c) Intensity line profile as indicated in (b). In the darker atomic columns show Sc/Zr positions are occupied by Al atoms.

# Phase Separation in Mixed Polymer Brushes on Nanoparticle Surfaces Enables the Generation of Anisotropic Nanoarchitectures


Soft Matter 14, 4551-4557 (2018)

C. Rossner, Q. Tang, M. Müller, G. Kothleitner

The preparation of inorganic nanoparticles and their targeted connection with other functional units is one key challenge in developing nanoscale devices. To use nanometer-sized building blocks in technological applications, the ability to precisely align these blocks is crucial. Therefore, current research has focused on new strategies for building precisely aligned hierarchical nanoparticle structures.

Here we report on a new experimental strategy for the development of anisotropic nanoparticle architectures. The approach is based on phase separation of binary mixed polymer brushes consisting of PMMA and PS homopolymers on gold nanoparticle surfaces leading to Janus-type structures. By equipping one of the constituent polymers in these separating mixed brushes with functional groups, site-selective incorporation of additional functional units can be achieved.

With the help of scanning transmission electron microscopy and electron energy-loss spectroscopy the phase separation on the nanoparticle surface could be visualized by direct mapping of the polymeric species (figure 1). The spectrum image maps reveal the distribution of PS (green) and PMMA (blue) around the Au-particle. Following an earlier experimental strategy (Rossner, 2014) we treated the core-shell particles with smaller Au-nanoparticles from two-phase Brust-Schiffrin synthesis. This resulted in the formation of planet-satellite nanostructures, as evidenced from the HAADF images in figure 1. In addition, by using Monte-Carlo simulations, we were able to show that the phase separation into Janus-type structures is a result of laterally mobile surface grafting points.

Our concept may in principle enable the fabrication of a variety of different anisotropic nanostructures which may find applications in nanostructures capable of undergoing directed self-assembly.

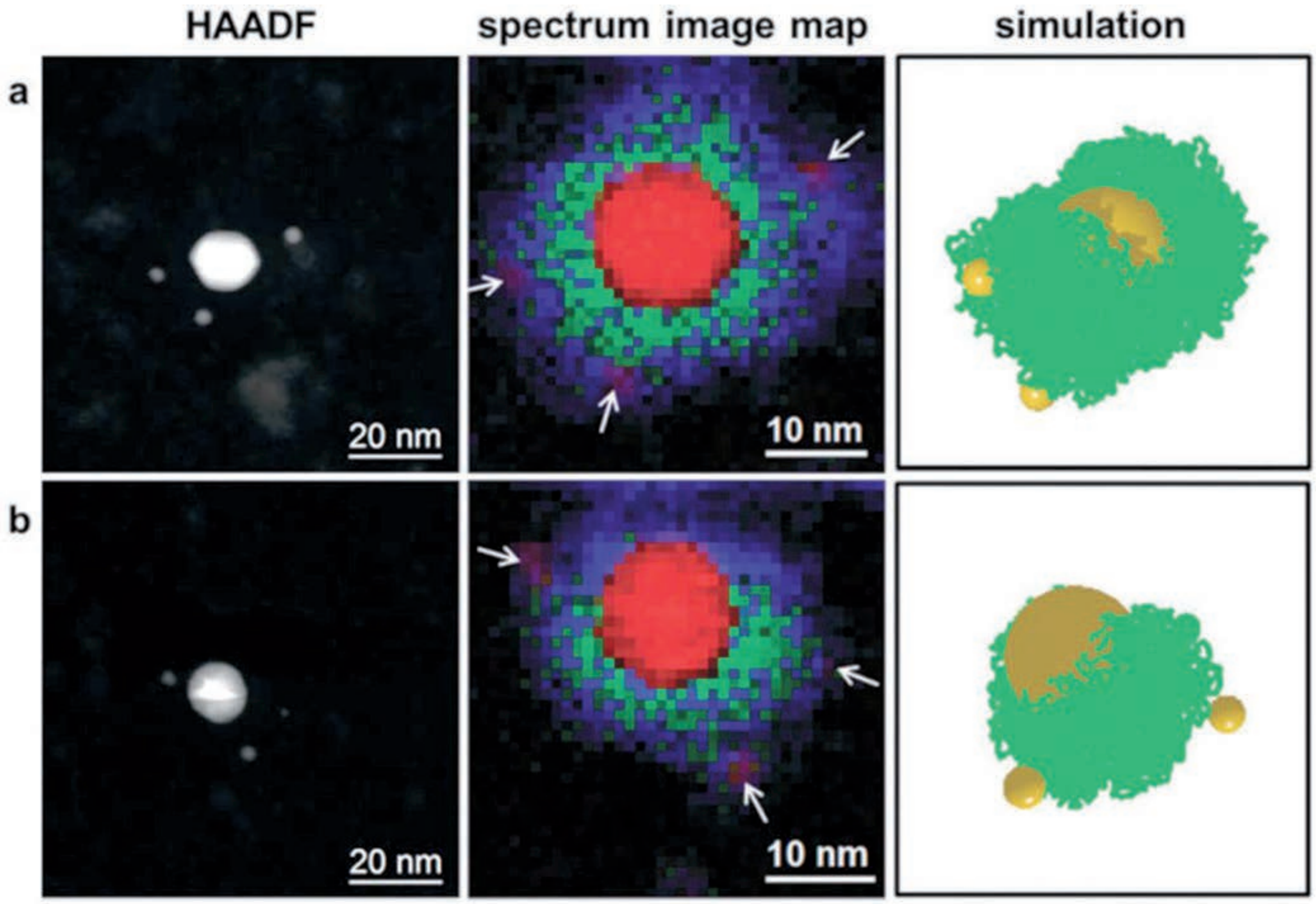



**Figure 1** STEM-HAADF images of planet-satellite structures particles (left) and corresponding spectrum image maps (middle) showing maps of gold (red), PS (green) and PMMA (blue). The satellite particles are located at the outer PS layer, isotropic (a) and anisotropic (b) particles. The right column shows the simulation snapshots with PS (green) and Au (yellow) which confirms the Au-satellite particles on the PS.

# The fracture behavior of particle modified polypropylene - 3D reconstructions and interparticle distances

**Manfred Nachtnebel, Michael Rastel, Claudia Mayrhofer, Armin Zankel, Markus Gahleitner, Peter Poelt**


Polymer 126 (2017) 65-73

Serial block-face scanning electron microscopy (SBEM) was invented in the field of live sciences, but became recently relevant in materials science. Here a scanning electron microscope (SEM) is combined with an ultramicrotome to perform automated serial cutting of thin slices of soft materials and finally imaging of the remaining block-face. For this method an environmental SEM (ESEM) is available at our institute which enables direct imaging of electrically nonconductive samples at high resolution without any coating. As a result a stack of images of the investigated sample is obtained, which subsequently can be used for a 3D reconstruction of the region of interest (ROI).
This method was used to gain information about the fracture behavior of particle modified polypropylene (PP). Therefore special precracked polymer samples initially underwent an in situ tensile test. Subsequently the ROI was cut out and stained with heavy metals. Two different types of particle modified PP specimens were investigated with SBEM: PP modified with ethylene-propylene-rubber (EPR) and linear low-density polyethylene (LLDPE). Because of the large variety of the sample appearance a semi-automated segmentation algorithm was programmed to be able to handle the recorded big datasets. With the resulting 3D reconstructions information is obtained about the size of cracks and voids and their position in respect to the EPR and LLDPE particles. Furthermore the surface-to-surface interparticle distances could be calculated. A comparison of 2D with 3D calculations of interparticle distances verifies the high overestimation when using 2D information. An introduced single quantitative parameter revealed the correlation between the formation of cracks and the interparticle distance. LLDPE modified PP showed particles debonding from the matrix and shear deformation of the particles themselves, while EPR modified PP showed the occurrence of cracks in regions of small interparticle distances.
SBEM is a powerful tool to obtain 3D information of quite large volumes of soft materials at high resolution. Therewith it was possible to prove existing theories as well as obtaining new insights into the field of fracture behavior of polymer blends.

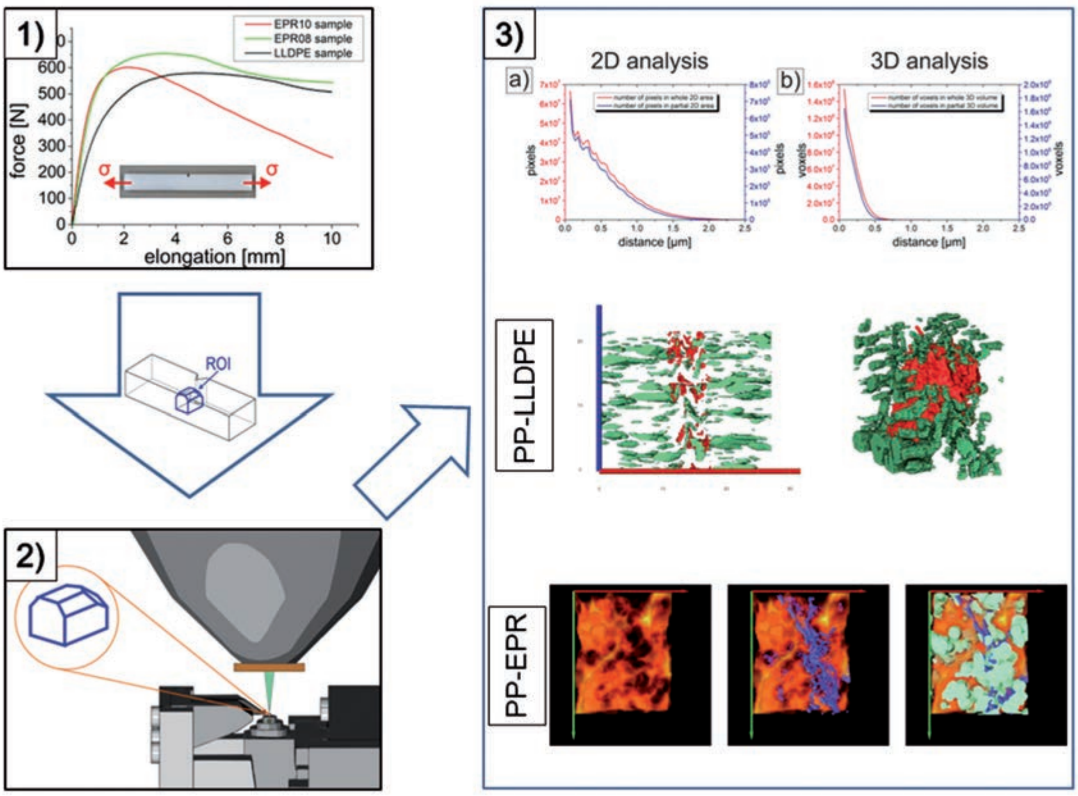



**Figure:**
Schematics of the entire investigation process with 1) the in situ tensile testing, 2) the subsequent sample preparation and the examination by SBEM and 3) some results of the obtained 3D reconstructions of different samples.

# Research Grants

Peer-reviewed research grants acquired or active from 2017 to 2018.

## FELMI Projects

**Nanolithography**
FELMI-ZFE leader: Harald Plank
Coordinator: Joachim Krenn, Institute of Physics, University of Graz
HRSM-project of the Austrian Ministry of Education, Science and Research, 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 Austrian Ministry of Education, Science and Research, 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
Production of the Future, 11. AS CHINA CAS Austrian Research Promotion Agency (FFG), Vienna, 10/2015 – 9/2017

**Combine Triple – Ion and High – Speed Atomic force Microscope for Correlative Anaysis (AIM)**
Leader: Harald Plank
Coordinator: GETec Microscopy GmbH, Vienna
Beyond Europe, Austrian Research Promotion Agency (FFG), Vienna, 08/2018 - 01/2020

**CD Laboratory for Direct – Write Fabrication of 3D Nanoprobes**
Leader: Harald Plank
Coordinator: Institute of Electron Microscopy and Nanoanalysis, TU Graz
Christian Doppler Research Association, Vienna, 03/2018 – 02/2025

**Sulfur – Aluminium Battery with Advanced Polymeric Gel Electrolytes (SALBAGE)**
Leader: Gerald Kothleitner
Coordinator: Bernhard Gollas, Institute for Chemistry and Technology of Materials, TU Graz
EC Horizon 2020, FET - Open research and innovation actions, s, 01/2019 – 12/2022, Brussels, 11/2017 – 10/2020

**„Direct – Electron – Detection" for the ASTEM Electron Microscope" (ASTEM Upgrade)**
Leader: Gerald Kothleitner
Coordinator: Institute of Electron Microscopy and Nanoanalysis, TU Graz
Zukunftsfonds Steiermark, Das Land Steiermark, 02/2018 – 01/2020

**ESTEEM 3 Enabling Science and Technology through European Electron Microscopy**
Leader: Gerald Kothleitner
Coordinator: Max-Planck-Institute for Solid State Research, Stuttgart, Germany
EC, Horizon 2020 INFRAIA, 12/2018 - 12/2022

**ELMINET Electron Microscopy Network in Graz**
Leader: Armin Zankel
Coordinator: Medical University of Graz, Graz, HRSM-project of the Austrian Ministry of Education, Science and Research, Vienna, 5/2017 - 4/2021

# ZFE Projects

**Quantitative Electron Microscopy of Hard Metals**
Leader: Gerald Kothleitner
Coordinator: ZFE Graz
Sandvik, Stockholm, Sweden, 10/2013 – 9/2017

**Structure Dependent Properties of Innovative Light Metal Systems (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 Analysis of Inner Phase Boundaries in Semiconductor Devices**
Leader: Evelin Fisslthaler
Coordinator: ZFE Graz
Austrian Research Promotion Agency (FFG), Vienna, 4/2015 – 9/2017

**Innovative Materials Characterisation**
Leader: Johannes Rattenberger
Coordinator: ZFE Graz
Austrian Cooperative Research (ACR) and Austrian Ministry of Digital and Economic Affairs, Vienna, 11/2016 – 11/2018

**Rare-earth Nickelates for Future Energy Technologies (SENTECH)**
FELMI-ZFE leader: Werner Grogger
Coordinator: Montanuniversität Leoben (MUL), Energy research e!mission 2. AS; Austrian Research Promotion Agency (FFG),
Vienna, 3/2016 – 2/2019

**Solar Cell Meets Battery: Realisation of a hybrid energy system (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

**Evaluation of Filtering systems in terms of Mimizing the Biological Risks in Interiors (AEROPORE)**
FELMI-ZFE leader: Johannes Rattenberger
Coordinator: OFI Technologie & Innovation GmbH
COIN Austrian Research Promotion Agency (FFG), Vienna, 01/2017 – 12/2020

**Microstructure of 3D Printed Metallic Components**
FELMI-ZFE leader: Mihaela Albu
Coordinator: ZFE Graz
Austrian Cooperative Research (ACR) and Austrian Ministry of Digital and Economic Affairs, Vienna, 03/2018 – 02/2020

**Dust Analysis in Interior Air**
FELMI-ZFE leader: Manfred Nachtnebel
Coordinator: HFA - Austrian Forest Products Research Society, Vienna
Austrian Cooperative Research (ACR) and Austrian Ministry of Digital and Economic Affairs, Vienna, 03/2018 – 08/2019

**Tribological Optimisation of Polymers (TRIOP)**
FELMI-ZFE leader: Ferdinand Hofer
Coordinator: V-Research GmbH, Dornbirn
Austrian Cooperative Research (ACR) and Austrian Ministry Digital and Economic Affairs, Vienna, 03/2018 – 02/2020

**Corrosion - Avoidance of Selective Corrosion of Cu-alloys and Steels.**
FELMI-ZFE leader: Hartmuth Schröttner
Coordinator: ÖGI Austrian Foundry Research Institute, Leoben
Austrian Cooperative Research (ACR) and Austrian Ministry of Digital and Economic Affairs, Vienna, 03/2018 – 02/2020

European Commission | Horizon 2020 European Union funding for Research & Innovation

FFG Forschung wirkt

Bundesministerium Bildung, Wissenschaft und Forschung

Bundesministerium Digitalisierung und Wirtschaftsstandort

# Research Projects

## Christian Doppler Laboratory: DEFINE

Ass.-Prof. Priv.-Doz. Dr. Harald Plank

On Wednesday, 25th April 2018 the **Christian Doppler Laboratory DEFINE** was officially opened under the auspices of rector Harald Kainz in the assembly hall of Graz University of Technology.
We are especially happy to host the **Christian Doppler Laboratory for the Direct Fabrication of 3D Nano-Probes** under the leadership of **Harald Plank** in the course of the upcoming years.
During the last decades, Focused Electron Beam Induced Deposition (FEBID) has attracted increasing attention in fundamental and applied research. This technology enables flexible additive manufacturing thanks to the mask-less, direct-write character for 3D fabrication on the nanoscale, which meet challenges when classical, resist based lithography methods run into their intrinsic limitations. In the course of the past ten years, Harald Plank has intensively been focusing on this topic. In 2015 he defended his Habilitation thesis where he summarised the scientific contribution to this field made at our Institute since 2008; deep insights into fundamental resolution limitations, proximity based broadening effects, high-fidelity shapes, and efficiency aspects have been gained which led to a strong performance improvement in this field, which finally paved the way for true 3D Nanofabrication.
On the pursuit of industrial applications, he teamed up with GETec Microscopy GmbH to explore next-generation nano-probe concepts for Atomic Force Microscopes (AFM), which will push their performance beyond current limitations. The full potential of the anticipated 3D nano-probes will be exploited by GETec's state-of-the-art in situ AFM product line, which is designed for the seamless integration in Scanning Electron Microscopes (SEM) and / or Focused Ion Beam (FIB) systems. By combining these individual technologies, entirely new, yet unknown capabilities will become possible.

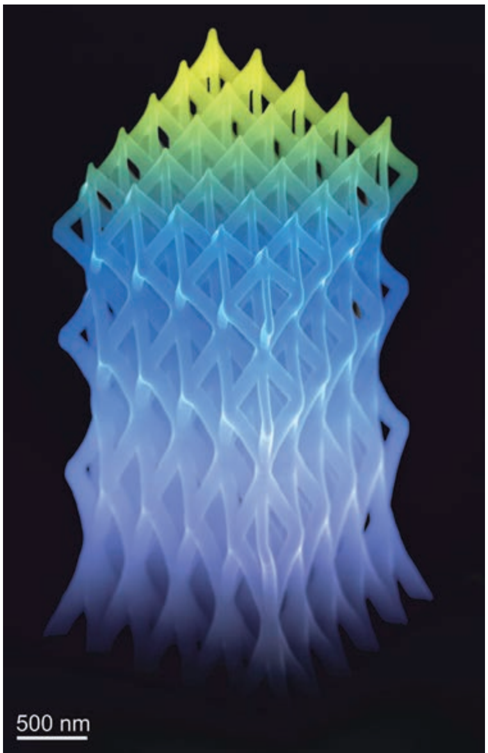
### Key Facts

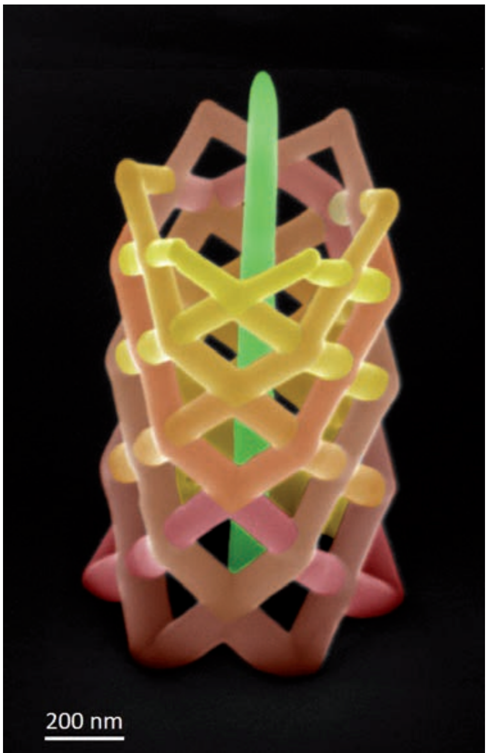
| | |
|---|---|
| **Head** | **Industrial Partner** |
| Ass.Prof. Priv.-Doz. DI Dr. Harald PLANK | GETec Microscopy GmbH |
| **Period:** | Aspern IQ Seestadt Aspern |
| 1.3.2018 – 28.2.2025 | Seestadtstraße 27 \| 1220 Vienna \| Austria |
| | www.getec-afm.com |

Christian Doppler Forschungsgesellschaft

GETec Microscopy GmbH

TU Graz



# Correlative Microscopy with RISE: One Highlight of ELMINet Graz

Harald Fitzek, Ruth Schmidt, Manfred Nachtnebel, Claudia Mayrhofer, Johannes Rattenberger, Hartmuth Schroettner, Armin Zankel

The „HRSM-Projekt ELMINet Graz“ is a cooperation between Karl-Franzens University Graz (KFU), Medical University of Graz (MedUni) and Graz University of Technology (TUG), which is financed by the Austrian Federal Ministry of Education, Science and Research. The aim of this project is the improvement of infrastructure at the three cooperating universities. Via ELMINet Graz the new system RISE (Raman Imaging and Electron Microscopy) could be established at the FELMI-ZFE, which is a combination of a Raman microscope from WITec and the scanning electron microscope (SEM) Sigma 300 VP from Zeiss. Additionally this SEM is equipped with a high performance EDXS detector (SDD technology) from Oxford (realised by the project “Innovative Materialcharakterisierung”, SP2016-002-006, part of “ACR Strategisches Projektprogramm 2016”). The combination of high resolution imaging, elemental analysis by EDXS and chemical mapping (information about chemical bonds) by Raman microscopy opens up exciting opportunities for new microscopic methods.The goal of our part in this project is to explore these opportunities as well as to establish sample preparation approaches that are suitable for both microscopic techniques. The scope of samples where the combination of SEM and Raman could be beneficial is very large as on one hand SEM always has a higher resolution than Raman and on the other hand the only material class that Raman can offer no information about are metals. Hence a broad range of samples including metal-oxides, inclusions in metals, polymers, organic-inorganic compounds, metal organic compounds, diverse particles (from cement powder to pharmaceuticals) and even exotic samples, such as a volcanic rock and a meteorite, have been investigated.

An example that nicely demonstrates the benefits of correlative SEM-Raman microscopy is shown in the figure below. The sample is a commercially available packaging foil, which comprises multiple layers of polymers, one of which contains very small particles. The different layers are clearly visible in the SEM but only by using Raman microscopy they can be identified as consisting of polyethylene (PE), polyamide (PA) and TiO2 (rutile) particles. However, while the TiO2 particles can be detected by Raman microscopy, resolving them accurately is not possible as they are too small. They can of course be resolved by SEM and EDXS. Therefore, using an EDXS analysis of Ti containing particles the size distribution of the $TiO_2$ can be measured.

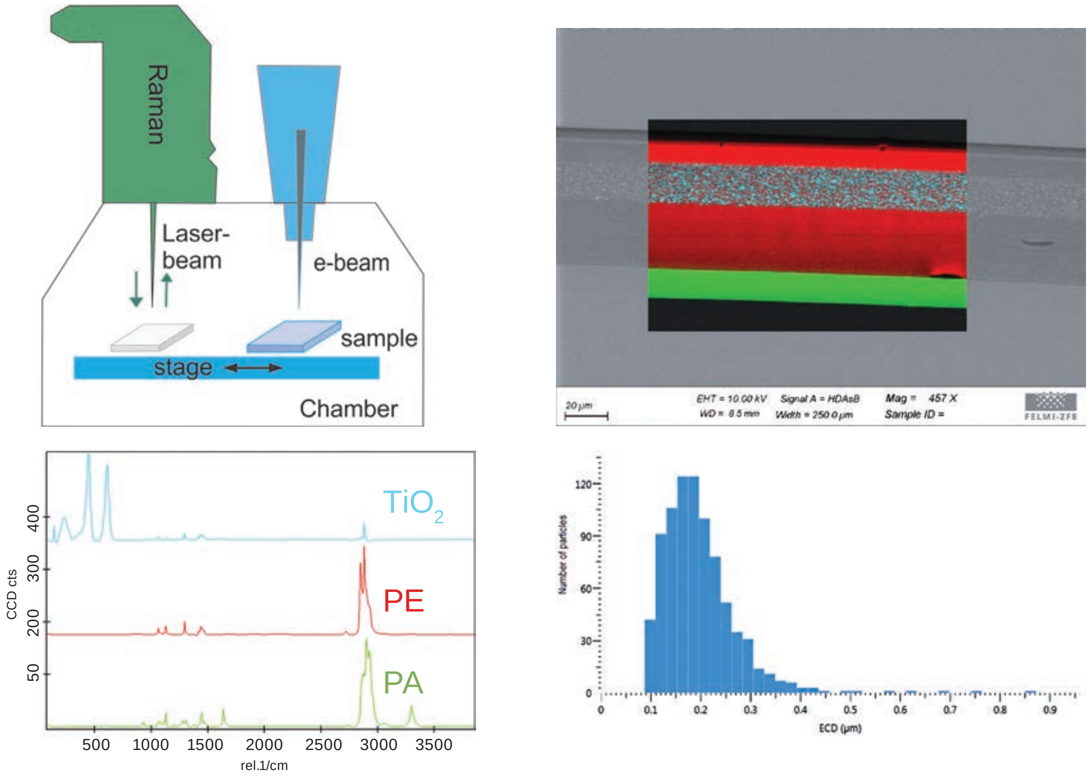



**Figure:** Top-left: Schematic drawing of the setup of the RISE system; Top-right: Correlative Raman-BSE measurement of a layered polymer system with particles; Bottom-left: Raman spectra identifying the components shown in the mapping in the top-right; Bottom-right: Size distribution of the TiO2 particles measured using a correlative EDXS analysis of Ti containing particles (ECD: equivalent circle diameter).

# Aeropore - Evaluation of Filtering Systems in Terms of Minimizing the Biological Risks in Interiors

Johannes Rattenberger, Manfred Nachtnebel, Claudia Mayrhofer, Hartmuth Schroettner

In the last decades, the number of people with allergies against at least one of the major allergens like birch, grass, ragweed pollen, or domestic cat has been steadily increasing. Nevertheless, state of the art filter tests do not consider the filter efficiency regarding their retention rate concerning these substances. Filter and filter systems are still tested with standardized mineral dust (e.g. "Arizona Road Dust") but the findings of these research methods are not representative in the context of allergens and pathogenic germs.

The FFG Coin Aufbauprojekt (capacity building) Aeropore is specifically concentrating on this topic. The Graz Centre for Electron Microscopy (ZFE) is working in close cooperation with the OFI (Österreichisches Forschungsinstitut für Chemie und Technik) in Vienna on the development of a filter testing system at pilot-plant scale. There are many different investigation methods available with which new insights can be gained regarding the allergy potential, the concentration or position of specific allergens at various sample types. A very sophisticated method is immunogold electron microscopy, which enables the location of specific proteins responsible for an allergic reaction.

In the course of this project, investigations were performed to obtain information about the presence of specific allergens at particles deposited at fine dust filters. Because of the sample size, they have to be examined in a scanning electron microscope (SEM). Unfortunately, several examinations have shown that typical TEM labelling protocols (specific sample preparation steps) are not directly transferable to scanning electron microscopy.

Therefore, the ZFE had to strike a new path by adapting these protocols to enable the labelling on the samples' surfaces. The main task was to achieve a signal (high gold particle concentration) at the exact position where the allergen is present while the background is not being labelled.

We found an iterative sample preparation process, initially for the detection of the grass pollen allergen Phl p5. To create a functioning protocol it is amongst others important to find the appropriate primary antibody concentration. Monoclonal antibodies were used because of their highly specific binding to a single class of proteins. Additionally, fixation and blocking steps were applied to fix the proteins in question on the one hand and to suppress a labelling of the background on the other hand.  In the end, this technique gives us the opportunity to accurately locate the allergenic or pathogenic germ proteins and to obtain information about the distribution of particle sizes where these proteins are present. This information will subsequently be used to calibrate and test a filter testing system, which is currently being developed by OFI.

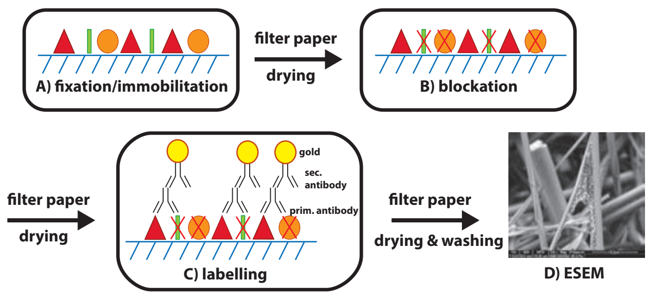



**Figure:** Labelling protocol: after fixation/immobilisation by the usage of bovine serum, blocking with glutaraldehyde (GA), labelling of the allergens by using appropriate monoclonal primary antibodies and a washing step this samples can by directly investigated in the environmental SEM (ESEM).

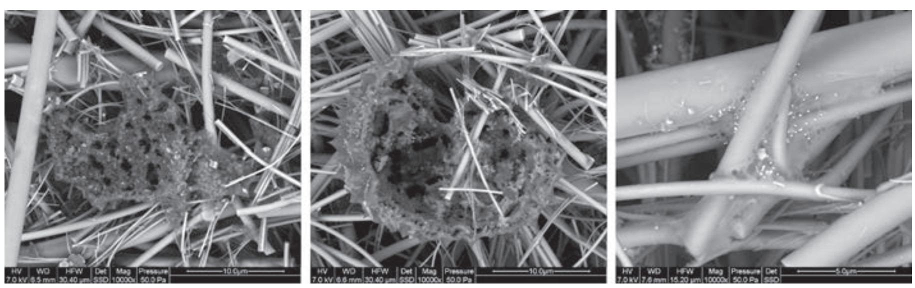


**Figure:** BSE images of particles on fine dust filters after applying the labeling protocol against Amb a1 (main Ragweed allergen) with a) the negative control (milled grass and birch pollen), b) the positive control (milled ragweed pollen) and c) particles at a used PM10 filter (arrows mark gold particle positions).

# A Closer Look on 3D Metal Print Processes

Mihaela Albu

Additive manufacturing (AM) and the 3D printing of metals denotes a relatively young, yet highly innovative and dynamic technology for the fabrication of complex metallic structures. The process essentially comes down to spreading some sort of metallic powder on a support platform, and subsequently steering a laser or electron beam over the melting particles to form the desired shapes. Light metals or alloy powders are often used, giving appropriate mechanical or electro-chemical properties along with a high degree of design freedom.

Depending on the pre-processing and the actual printing parameters, the fine-grained powders, quite frequently show different micro-structures and phase compositions than the final 3D-printed object. While particles and their respective micro-structures, for instance, can feature rather high strengths, printed devices often lack this property entirely.

To better understand the underlying processes and to optimize the metallic powder precursors as well as printing setup, the ZFE Graz has started a 2-year strategic ACR project with its partner ÖGI / Leoben and industrial partners, like Böhler and Pankl Racing.

The research programme comprises many aspects related to micro and nano-structural changes during printing. Effects such as the dissolution of amorphous zones on powders upon thermally induced recrystallizations, elemental redistributions associated with the creation of new phases and diffusion regions or precipitation formation are central topics in these studies.

To give an example: AlSi10Mg, as a light element alloy used in many print applications in aerospace technology, was investigated for its structural and chemical changes upon heat treatment (figure 1 below). Initially, the fine cellular α-Al structures are penetrated by a network of eutectic silicon consisting of highly twinned nanoparticles but also nm-sized Si(Mg)-nanoparticles evenly distributed inside the cells of α-Al. During heating, starting at about 200°C, strong coarsening of the eutectic silicon particles takes place, with a full break down of the eutectic network at 320°C. These findings and transformations clearly impact the mechanical properties of the final product.

Exhaustive expertise on the evolution and transformation of various materials systems during printing is hardly available, yet absolutely crucial for the fabrication process. In this light the project builds unique knowledge, being of great value for the metal industry and SME´s involved in specialized AM production.

## Key Facts

**Leader**

aO. Univ.-Prof. DI Dr. techn.
Gerald **KOTHLEITNER**

Institute of Electron Microscopy
and Nanoanalysis
Steyrergasse 17 I 8010 Graz I Austria

gerald.kothleitner@felmi-zfe.at

Period 1.1.2019 - 31.12.2022
Consortial Partners
14 academic partners
6 industrial partners
Project Volume: 10 Mio €

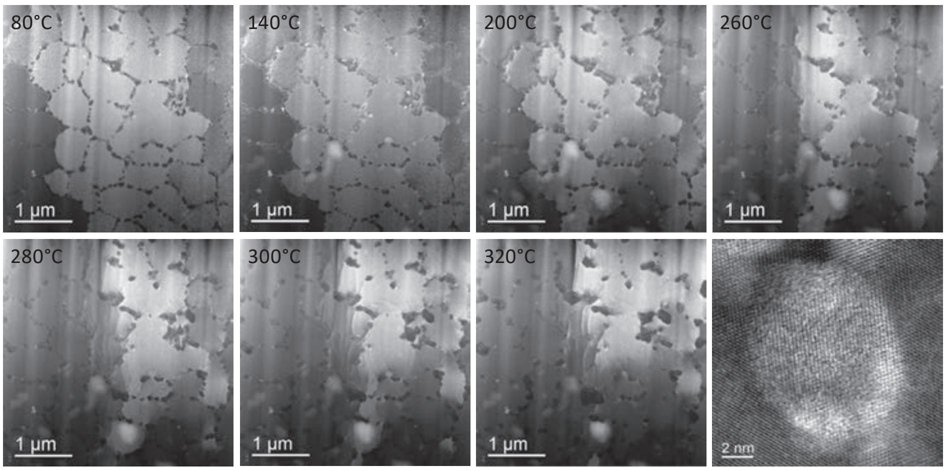



**Figure 1:** Low magnification STEM images during an in-situ heating experiment from a printed AlSi10Mg sample. Lower right corner: high resolution view on an embedded Si(Mg)-nanoparticle.

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Steyrergasse 17 I 8010 Graz I Austria

gerald.kothleitner@felmi-zfe.at

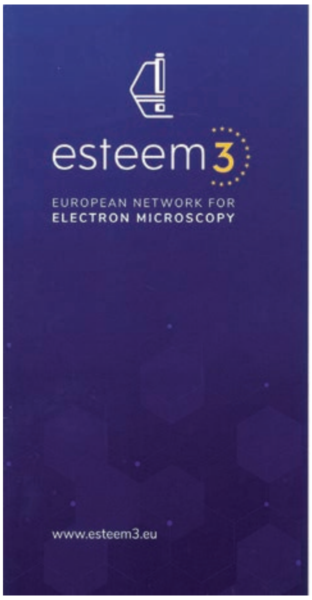
Period 1.3.2018 - 28.2.2020
Consortial Partners
ÖGI Leoben, Böhler. Pankl ??
Project Volume: XY  k€

# ESTEEM3: European TEM Network

## Gerald Kothleitner

ESTEEM3 – Enabling Science and Technology through European Electron Microscopy – is the success story of a series of EU funded projects for advanced electron microscopy. It brings together the leading European laboratories equipped with the most innovative TEM installations and thereby aims to be the key European multi-site research and user infrastructure platform for sophisticated electron microscopy characterizations.
This new research programme builds on the very successful ESTEEM platform already used by over 800 research groups from 350 academic and industrial organisations across Europe. This consortium is now established as the primary European portal for industrial and academic researchers who need access to the latest generation of TEM instrumentation, methodology or tools supported by leading TEM expertise for solving complex materials problems.

Like in ESTEEM2, the FELMI-ZFE with its unique equipment, again represents the Austrian knot and is delighted to provide access to its instrumentation. Together with prominent partners like Cambridge, Oxford, Paris, Jülich or Antwerp amongst others, Graz offers high resolution electron microscopy instrumentation to the users at the forefront of technology so that challenging characterisation problems in materials and physical sciences can be solved with unprecedented performance.
In addition, directed research programs focus on the advancement of methods for imaging and diffraction, spectroscopy, in-situ techniques and metrology, and on fostering applied research of materials. The FELMI-ZFE is having the international lead on research related to ICT (Information and Communication Technolgy) materials, being greatly synergetic with Austria´s SAL (Silicon Austria Labs) initiatives, while other topics like energy, health, and transport materials are handled by other partners.
Furthermore, activities dedicated to the dissemination of expertise, education and training in cutting-edge electron microscopy techniques, such as schools, advanced workshops and webinars, are offered to all European users from academia, research institutes and industry.




# SolaBat – Electron Microscopy for Energy Materials

**Georg Haberfehlner, Daniel Knez, Ferdinand Hofer**

The main purpose of the project was to develop a proof-of-concept new hybrid energy device, i.e. a combination of a solar cell with a storage battery integrated in the same enclosure. The principal applicant Ilie Hanzu from the Institute for Chemistry and Technology of Materials (TU Graz) managed to integrate an organic tandem solar cell with either a Li-ion battery or a Na-ion battery.

The ZFE concentrated on the detailed characterisation of the involved materials and the interfaces between these materials. Since many of the relevant materials in the hybrid devices are electron beam sensitive, special investigation strategies for high-resolution STEM had to be developed. For light elements, such as lithium, a key component in the battery part, and for polymers, which constitute the active part of the solar cell, all types of beam damage effects play a role. Thus, it was possible to enhance the EELS detection sensitivity for studying the phase separation in PTB7-Th:O-IDTBR bulk heterojunctions (fig. 1). This could only be achieved with the direct electron detection camera (K2, Gatan), which was newly installed as detector for the EELS spectrometer of the ASTEM microscope. During the project Li-containing materials like Li-Ti-oxides, Li-Mn-Fe-oxides and Li-V-oxides were analysed by means of STEM-EELS. In the course of this work, it was found that quantitative statements about the Li concentration in these oxides can only be obtained if the experiments are performed at liquid nitrogen temperature.

analysis of Li-containing battery materials e.g. for Li-Ti-oxides, Li-V-phosphates and Li-Mn-Fe-phosphates.

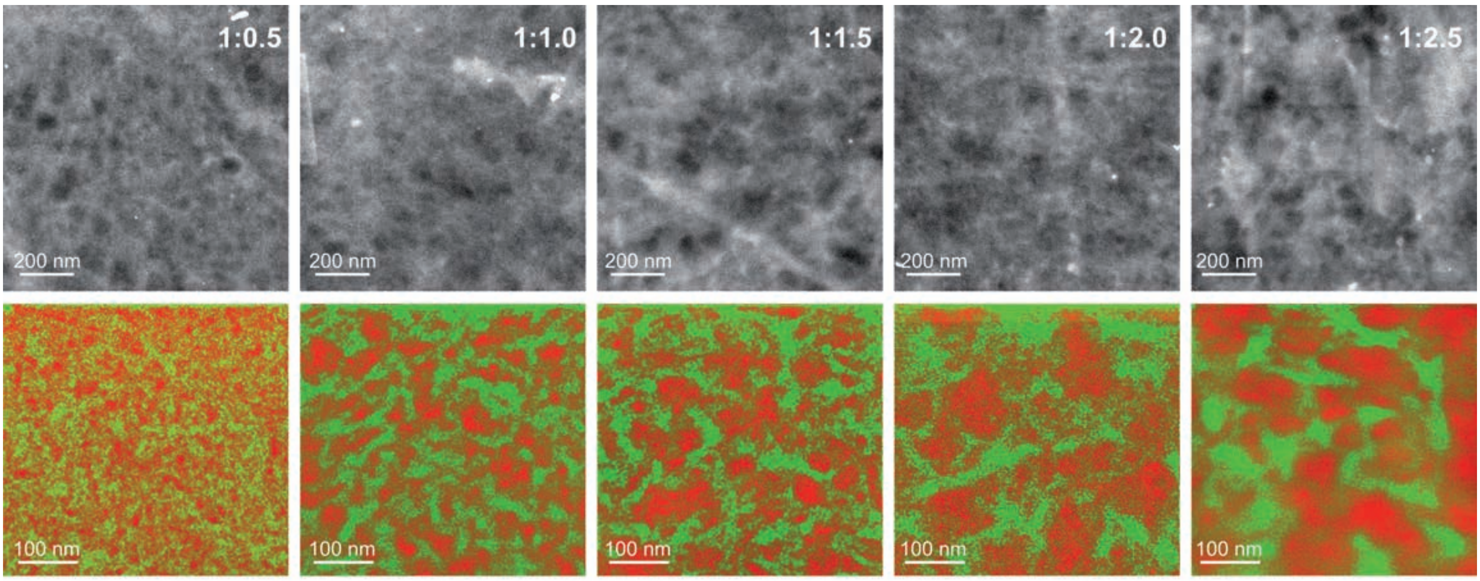



**Figure 1:** (a) STEM HAADF images and (b) sulfur-nitrogen atomic ratio maps derived (with S in green and N in red) of PTB7-Th:O-IDTBR thin films for various donor:acceptor ratios derived from EELS spectrum images.

# Guest Researcher at the Institute

**Guest Professor Helen M. Chan**

Professor Chan is the former chair of the Department of Materials Science and Engineering at Lehigh Unversity (Betlehem, USA). She was awarded a Fulbright U.S. Scholar Program grant to teach at the TU Graz in the 2016-2017 winter semester. Chan pursued her research on ceramics and gave a course about materials characterisation. Chan is the New Jersey Zinc Chaired Professor at Lehigh. She is a Fellow of the American Ceramic Society.

**Dr. Christian Rossner** joined our work group around Gerald Kothleitner focusing on analytical high resolution TEM for a year. In the course of his research project Christian concentrates on nanohybrid materials, e.g. gold-polymer nanocomposites. He gained his PhD in chemistry at the University of Göttingen, where he worked in the group for Macromolecular Chemistry headed by Philipp Vana. Supported by the Leopoldina Fellowship Programme, Christian plans to reveal the structural identity of RAFT co-polymers on nanoparticle surfaces.

**M. Sc. Javier Pablo Navarro** is a PhD student in the Magnetism in Nanostructures (MAGNA) research group at the Institute of Nanoscience of Aragon. He graduated in Physics from the University of Zaragoza and obtained the Master's Degree in Physics and Physical Technologies (2015). His focus is the nanofabrication and characterization of 2D and 3D functional magnetic nanostructures for potential applications in future spintronic devices. During his research stay at the Institute, he was involved in the growth and characterization of ferromagnetic nanostructures. In particular, he studied the chemical purification of 3D iron nanowires under in-situ heating experiments.

# New Instrumentation

## Zeiss Sigma 300: All in one: SEM & Raman & EDS = RISE + EDS

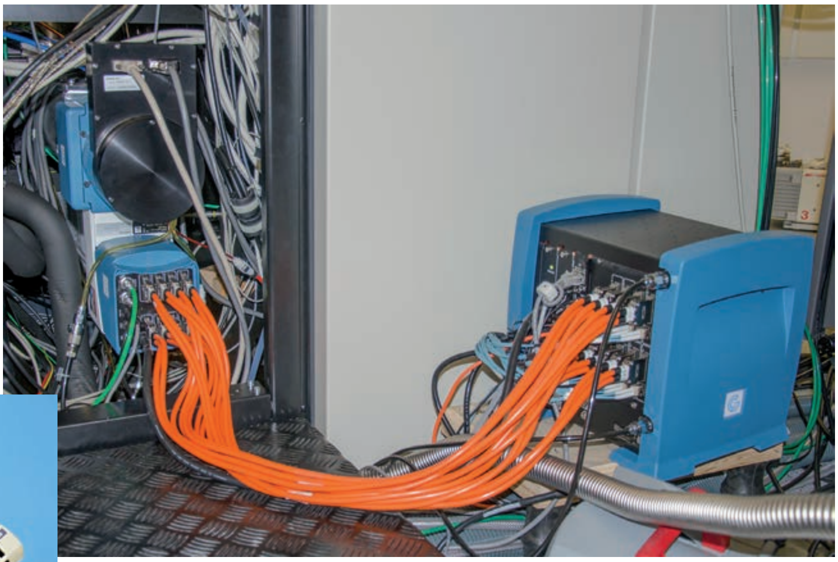
The new microscope features a fully integrated fast confocal Raman microscopy capability within the SEM chamber without any sacrifice on SEM specifications, operating independently and allowing correlated imaging between SEM (BSE-SE), EDS and Raman for structural and chemical information without complicated manipulation of the sample.
RISE stands for Raman Imaging and Scanning Electron microscopy. The seamless combination of the two techniques offers a distinct advantage when investigating samples, improves ease-of-use and accelerates experimental workflow. This combination is the first installation in central Europe.

### ELMINet Graz

The new system is a part of ELMINet Graz. ELMINet is short for Electron Microscopy Network. It is a joint initiative of three universities to unite their expertise in the field of correlative electron microscopy. The Graz-wide network enables access to new instruments thereby opening options for interdisciplinary research cooperation. The kick-off-meeting was organised as a tour to the three different facilities, starting with opening words and impulse talks and a lab tour at the Department of Cell Biology, Histology and Embryology. Lab presentations and live demonstrations took also place at Plant Sciences (University of Graz) and at our Institute. The newly installed microscope was presented as well as our microtomy.

## ASTEM Upgrade

The high-resolution microscope ASTEM was equipped with a new type of detector in the framework of the project "Direct-Electron Detection for the ASTEM microscope". This detector will be used to extend the application range of the ASTEM and to enable new investigations in the field of characterization of structured nanoscale materials.
The following goals are pursued: 1. extension with a unique electron detector (DDD-camera), 2. establishment of new imaging techniques and improvement of spectroscopic methods for radiation-sensitive materials and samples with light elements, 3. investigations of magnetic and electric fields on nanostructured materials, 4. intensification of co-operations with university and industrial partners, 5. making the institute more attractive for top European research projects.
Installation of the direct-electron detection camera K2 from Gatan Inc., USA



# Access

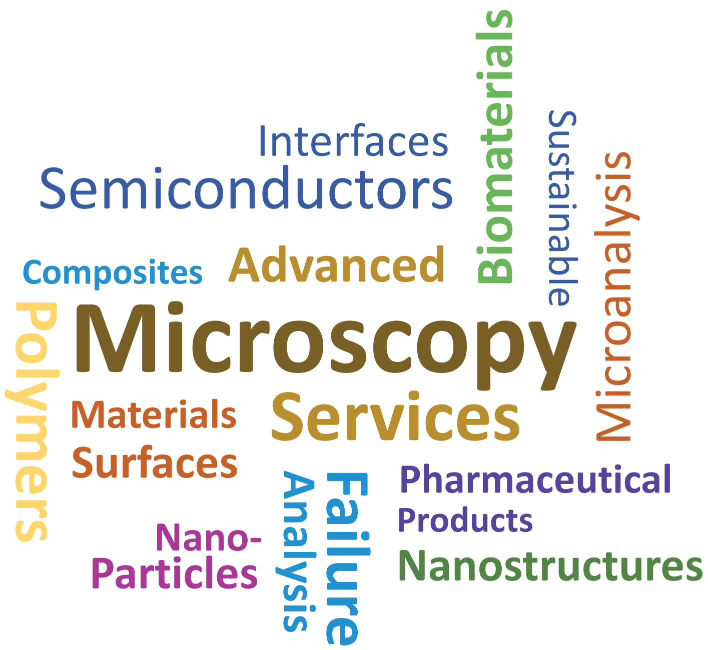
# Services to Industry and Research Institutes

The ZFE's recipe for success is based on the close cooperation with industrial companies, but also with small and medium-sized enterprises and start-ups. These cooperations strengthen our research activities and force us to constantly take on new challenges.

The ZFE supports its partners in solving problems that cannot be handled by in-house experts alone. The challenges in science, technical production processes and quality control are becoming increasingly complex. Therefore, it is no longer enough to offer routine analysis, but it is more important to develop new solutions tailored to the customer's needs.

According to our quality system we are committed to keeping up to date with latest research topics and to provide the best service to our industrial/research partners. Perhaps it is worth mentioning that in many cases the research results can also be kept confidential.

We also promote and work in collaborative R&D projects and may provide the management (planning, training, data acquisition etc.). Finally, we have access to other facilities as well as networks within the research community that is a benefit to our clients.




**ESTEEM3**

ESTEEM3 is the European infrastructure initiative in the field of high-resolution electron microscopy. The ESTEEM consortium provides access to the most powerful installations in Europe, including the ASTEM in Graz. This access applies to all scientists and companies outside of Austria. The FELMI-ZFE concentrates on nanoanalytical characterisation techniques such as EELS and EDX, electron tomography as well as studies of functional properties of nanostructures.

- **Grant-aided Partnerships**

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

- **Contract Research**

Industrial partners fund the costs of research (instrument fees, consumables and salaries for staff or scholarships). The results can be kept confidential.

- **Testing and Consultancy**

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

- **Training Courses and Know-how Transfer**

We provide training on in-house instruments for partners from industry and universities, who wish to purchase certain microscopes or use these instruments on a regularly basis and see the need to upskill their staff to become self-sufficient with their analyses (see also LLL-courses).

# Events and Workshops

## 1st EUFN Workshop in Graz

Following eleven highly successful D-A-CH FIB Workshops, the 1st EUFN (European FIB Network) Workshop was organised from July 4-5, 2017 in Graz, Austria.
The workshop presenting highlights and recent developments in the field of focused ion beam research was received with much interest and attention. More than 100 participants listened to 17 talks, 4 tutorials, 8 exhibitor talks, a podium discussion, and 15 poster presentations (organised by Harald Plank).

## Gatan Workshop

In spring 2017, FELMI-ZFE hosted a workshop dealing with new *in-situ* applications organised in cooperation with Gatan company. In the centre of interest were new *in-situ* capabilities, live demos and digital imaging for TEM (organised by Gerald Kothleitner).

## Materials Microscopy meets Companies

In 2017 we launched a new workshop series. Presentations about the advantages of electron microscopy are organised in different district capitals. Under the title of "Das Unsichtbare sichtbar machen: Wir nehmen Ihre Fragestellung unter die Lupe" we adress different topics ranging from sanning electron microscopy to transmission electron microscopy and correlative microscopy. The workshops are for free.

## Seeing with Electrons: Mit Elektronen sieht man besser

In June 2018 our head of the institute Ferdinand Hofer gave a lecture about advanced electron microscopy in the assembly hall of TU Graz. The event was organised by "Forum Technik und Gesellschaft" of the Alumni organisation of the TU Graz. The focus laid on new developments in the field of microscopy ranging from SEM to TEM and AFM and their applications in materials sceince physics and chemistry.

## Silicon Alps Spotlight 2018

The theme of this workshop was „No challenge too small: Nanoanalysis for future electronic systems“. Silicon Alps is an Austrian technology and innovation cluster for electronic based systems. The Silicon Alps Electronic Cluster is a public-private partnership that brings together Austrian players from industry, science and public authorities to develop and position the electronics and microelectronics sector with a regional focus on the locations Carinthia and Styria (organised by Stefanie Gissing).




# Academic Education

# Lectures and Laboratory Courses

To an increasing extent the scientific staff of the institute concentrates on teaching tasks in the fields of physics and advanced materials science. In the table below you will find a selection of the most important courses of the study year 2018/19. The staff members also contribute to the large experimental physics laboratories. In addition, courses are also offered for the curricula in technical chemistry, electrical engineering, chemical engineering and biotechnology.

## Winter Semester

Introduction into Physics, L 2 (Grogger)
Electron Microscopy I, L 2 (Grogger)
Materials Characterization II, L 1.33 (Hofer)
Microscopy in Biotechnology, L 2 (Hofer)
Scientific Working, SE 2 (Hofer)
Advanced 2D and 3D Nanoanalysis, L 2 (Kothleitner)
Structuring of Materials Surfaces and Functional Nanofabrication, L 2 (Plank)
Seminar on Electron Microscopy and Nanoanalysis I, SE 2 (Grogger, Hofer, Kothleitner, Plank)
Research Laboratory Microscopy and Nanoanalysis I, P 2 (Grogger, Kothleitner, Zankel)
Project Laboratory, PT 8 (Grogger, Haberfehlner, Hofer, Kothleitner, Mitsche, Plank)

## Summer Semester

Electron Microscopy II, L 2 (Grogger)
High Resolution Electron Microscopy of Materials, L 2 (Hofer)
Microscopy of Polymers, L 2 (Hofer)
Materials Chemistry, L 2.66 (Hofer with lecturers from other institutes)
Materials Characterization using Electron Microscopy, P 2 (Kothleitner)
Solid Sate Spectroscopy, L 1.33 (Kothleitner with lecturers from other institutes)
Microelectronics and Micromechanics, L 2 (Plank)
Seminar on Electron Microscopy and Nanoanalysis I, SE 2 (Grogger, Hofer, Kothleitner, Plank)
Research Laboratory Microscopy and Nanoanalysis II, P 2 (Grogger, Kothleitner, Zankel)
Exercises in Electrodynamics and Optics, PE 1 (Haberfehlner, Schmidt)
Project Laboratory, PT 8 (Grogger, Haberfehlner, Hofer, Kothleitner, Mitsche, Plank)

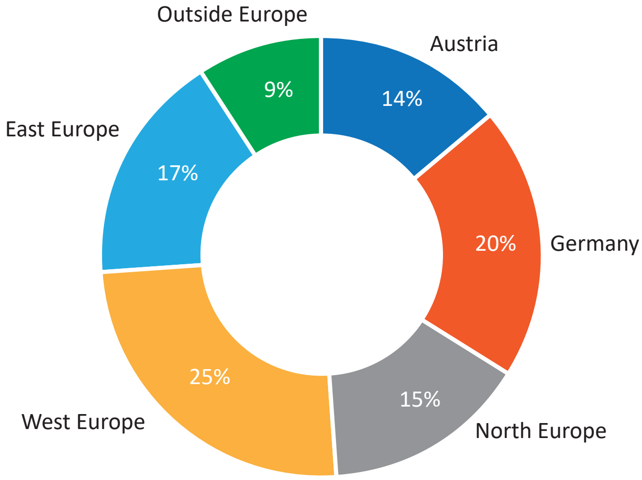
# LLL Courses

## European EELS & EFTEM School

Gerald Kothleitner, Werner Grogger

The EELS & EFTEM-School is a four days hands-on laboratory workshop taking participants step-by-step through the use of integrated FEI-Gatan energy-filtering/EELS systems (Tecnai TF20/HR-GIF and Titan/GIF Quantum). The FELMI-ZFE staff familiarises participants with the latest EELS & EFTEM equipment as well as with fundamental principles and methods. The school is organised in collaboration with Gatan company (Pleasanton, USA) every year in February. Since its introduction in 2006 the school attracted more than one 150 participants from all over the world.

Distribution of participants of the EELS & EFTEM-School:




## Problem Solving with Scanning Electron Microscopy and Spectrosocopy

Stefan Mitsche, Hartmuth Schröttner

Once a year, scientists, engineers as well as technicians profit from the three day course to solve their analytical problems. The participants learn through lecture and hands-on training how to setup and operate advanced scanning electron microscopes, including environmental and field-emission instruments. We are using the latest equipment and know-how to assure that the participants successfully record images, X-ray and Raman spectra and elemental maps (including quantitative data analysis).

*LLL = Life Long Learning course of the TU Graz*

# 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 school teachers and for students of the TU Graz, schools, universities and companies. Around 150 pupils, teachers and students from other institutions visited the Institute during the period 2017–2018.

## 2017

17 Mar. 2017, Prof. Karin KLEINSCHEK, University of Maribor, Sovenia (Hofer)

23 Mar. 2017, Engineering and Production Management, FH Joanneum Graz (Mayrhofer, Gspan)

29 Mar. 2017, Secondary School BRG Kirchengasse, Graz (Zankel, Mayrhofer)

06 Apr. 2017, Prof. Klement TOCKNER, President of the Wissenschaftsfonds (Hofer, Kothleitner)

29 Mai 2017, PLANCKS: Physics student league (Haberfehlner, Zankel, Dienstleder)

07 Jun. 2017, Prof. Dieter IMBODEN, Eidgenössische Technische Hochschule ETH Zürich (Hofer)

07 Jun. 2017, Bionik group from the FH Kärnten, Villach (Dienstleder, Fitzek, Letofsky-Papst, Pölt)

10 Oct. 2017, Dr. Erin COCKS, University of Newcastle, U.K. (Zankel, Mayrhofer)

30 Oct. 2017, Group from Plansee, Reutte (Hofer, Grogger)

02 Nov. 2017, Group from Anton Paar, Graz (Zankel, Fitzek)

09 Nov. 2017, Secondary School BRG Lichtenfels "Girls Day", Graz (Zankel, Letofsky-Papst)

27 Nov. 2017, Chemieingenieurschule, Graz (Zankel, Mayrhofer, Dienstleder, Letofsky-Papst)

28 Nov. 2017, Group from the Medical University of Graz (Zankel, Mayrhofer, Letofsky-Papst)

## 2018

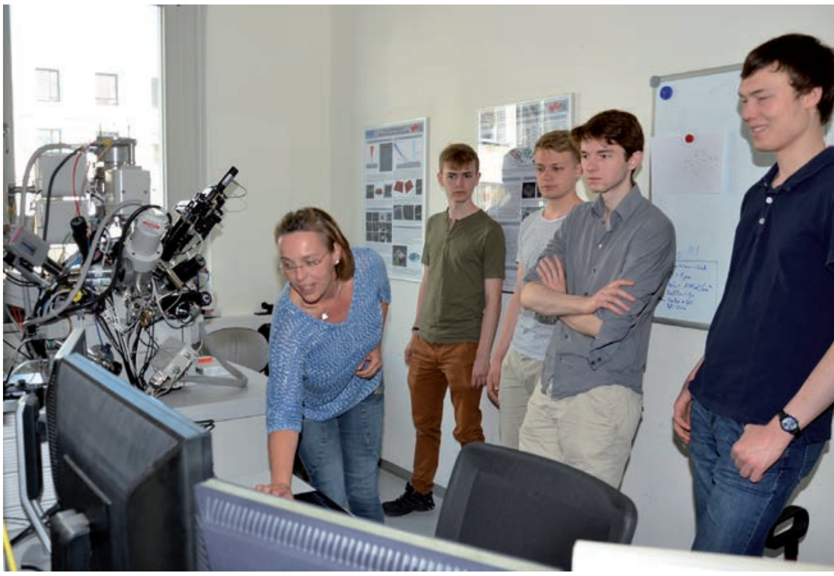
24 Jan. 2018, Group from Borealis Linz (Mayrhofer)

07 Feb 2018, Group from Borbet Austria (Schröttner)

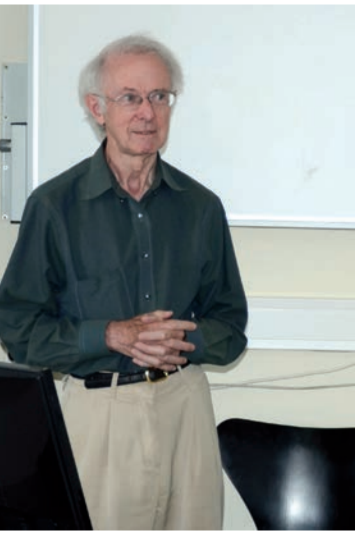
15 Mar. 2018, Group from Microinnova Graz (Schröttner, Letofsky-Papst, Wewerka, Mayrhofer)

20 Jun. 2018, Prof. M. Gael GAUTIER, Universite de Tours, INAS Centre Val de Loire, France (Pölt)

16 Oct. 2018, Prof. Volker SCHMIDT, University of Ulm, Germany (Zankel)



# Guest Speakers at the Institute



Ray Egerton,
University of Alberta, Canada

Prof. Johannes Lehmann,
Cornell University

## 2017

13 Jan. 2017, Bernd SONDEREGGER
Institute of Materials Science, Joining and Forming, TU Graz, “Thermodynamic properties of small objects within the General-Broken-Bond approach”

17 Jan. 2017, Helen CHAN
Dept. of Materials Science & Engineering, Lehigh University, USA, “Novel applications of redox reactions in metal ceramic systems”

21 Mar. 2017, Budhika MENDIS
Department of Physics, Durham University, U.K., “1s-Bloch wave simulations”

07 Apr. 2017, Jiehua LI
Chair of Casting Research, University of Leoben, “Atomistic characterization on the solute clustering and precipitation of Mg alloys”

28 Apr. 2017, Edith BUCHER
Chair of Physical Chemistry, University of Leoben, “Development of new functional ceramics for future energy technologies”

10 May 2017, Johannes LEHMANN
College of Agriculture and Life Science, Cornell University, USA, “When small is large: new paradigm for soil carbon cycles from microaggregates to global scale”

02 Jun. 2017, Andrea WANNER
Polymer Competence Center Leoben, “Characterisation of isolating resins in electrical engineering”

23 Jun. 2017, Nicolas BLANC
Eidgenössische Technische Hochschule ETH Zürich, Switzerland, “Scientific Center for Optical and Electron Microscopy – a technology platform”

20 Oct. 2017, Bernhard GOLLAS
Institute of Chemical Technology of Materials, TU Graz, “Sulfur-aluminium battery with advanced polymeric gel electrolytes i.e. SALBAGE”

31 Oct. 2017, Jason FOWLKES
Oak Ridge National Laboratory, Tennessee, USA, “Three-dimensional nano-printing using focused electron beam induced deposition: Design, simulation and experiments”

30 Nov. 2017, Richard DRUMMOND-BRYDSON
University of Leeds, School of Chemical and Process Engineering, U.K., “Prospects for high resolution analytical TEM of organic crystals”

01 Dec. 2017, Gabriele ETTENBERGER-BORNBERG
OFI, Vienna, “Characterisation of filter systems”

04 Dec. 2017, Ifat KAPLAN-ASHIRI
Weizmann Institute of Science Rehovot, Israel, “Measuring forces at the nanoscale – In situ SEM methods”

## 2018

23 Jan. 2018, Jürgen PLITZKO
Max-Planck-Institute for Biochemistry, Martinsried, Germany, “Cryo-electron microscopy and tomography: The past, the present and the future”

24 Jan. 2018, Davide TRACHIDA
BOREALIS Polyolefines, Linz, “High temperature AFM imaging and nanoindentation of polypropylene”

19 Jan. 2018, Christiane ESSL
VIF GmbH, Graz, “Experimental characterisation of thermal-runaway behavior of high capacity Li-ion batteries”

14 Feb. 2018, Kim SEJEONG
University of Technology Sydney, Australia, “Light engineering in van der Waals crystals”

16 Mar. 2018, Francisca MENDEZ-MARTIN
Department of Physical Metallurgy and Materials Testing, University of Leoben, “Atom probe tomography: physical principle, specimen preparation and applications”

13 Apr. 2018, Josip MARIC
Keyence International, Belgium, “Advances in digital microscopy and 3D profilometry”

22 Jun. 2018, Richard HUBER
University of Graz, “Joint reconstruction in spectral electron tomography”

29 Jun. 2018, Javier PABLO-NAVARRO
University of Zaragoza, Spain, “3D functional magnetic nanostructures grown by FEBID”

13 Jul. 2018, Ray EGERTON
University of Alberta, Edmonton, Canada, “Spatial resolution of low-loss electron energy-loss spectrometry”

09 Oct. 2018, Toma SUSI
Physics of Nanostructured Materials, University of Vienna, “Electron-beam manipulation of single impurity atoms in graphene”

09 Nov. 2018, Wolfgang SPRENGEL
Institute of Materials Physics, TU Graz, “Dilatometric techniques for atomic scale analysis of defects and processes in solids”

14 Dec. 2018, Markus KOCH
Institute for Experimental Physics, TU Graz, “Ultrafast photoexcitation dynamics of atoms and molecules in a quantum solvent”

# Master & Doctoral Theses at the Institute

## Finished PhD Theses

FITZEK Harald (2018), "A new simulation method for surface-enhanced Raman spectroscopy applicable to rough and highly irregular substrates", P. Pölt.

HARTLER Christian (2018), "Characterization and reliability investigations of 3D interconnection technologies", W. Grogger.

KRAXNER Johanna (2018), "Analytical TEM of organic electronics with a special focus on EDXS and geometry aspects", W. Grogger.

WINKLER Robert (2018), "Fabrication of functional and freestanding 3D nano-architectures via focused electron beam induced deposition", H. Plank.

KNEZ Daniel (2017), "Scanning transmission electron microscopy of metallic clusters and nanoalloys", F. Hofer.

NACHTNEBEL Manfred (2017), "*In-situ* experiments with polymeric materials in the environmental scanning electron microscope", P. Pölt.

## PhD Theses in Progress

KONRAD Lukas, "How multiple scattering simulations help for EELS Compositional analysis of hard metals and ceramics", G. Kothleitner.

LAMMER Judith, "Analytical high resolution STEM of cathode materials for solid state fuel cells", W. Grogger.

ORTHACKER Angelina, "Developing methods for 3D STEM to reveal atomic-scale spinodal decomposition", G. Kothleitner.

SATTELKOW Jürgen, "Direct-write fabrication of electric and thermal high resolution nanoprobes on self-sensing AFM cantilevers", H. Plank.

## Finished Master Theses

FALTHANSL-SCHEINECKER Paul (2018), "Approaching fundamental resolution limits of FEBID fabrication of gold nanostructures for Terahertz plasmonics", H. Plank.

GRASSERBAUER Jakob (2018), "*In-situ* electron backscatter diffraction: Recrystallization behaviour and microstructural evolution in Al-Mg-Si alloys during heating", P. Pölt, S. Mitsche.

ACHTSNIT Tobias (2017), "Evaluating new applications and optimizing the environmental scanning electron microscope", F. Hofer, J. Rattenberger.

FRÖCH Johannes (2017), "Mechanical properties of free-standing 3D nanostructures fabricated via focused electron beam induced deposition", H. Plank.

POSTL Andreas (2017), "Simulation of high resolution STEM images of a gold-nickel alloy and comparison with experiment", W. Grogger, C. Gspan.

RADESCHNIG Ulrich (2017), "Approaching fundamental resolution limits during focused electron beam induced gold deposition on bulk substrates", H. Plank.

RASTEL Michael (2017), "Investigation of fracture behaviour of polymers by light- and electron microscopy", P. Pölt, A. Zankel.

TRUMMER Cornelia (2017), "Preparation of TEM samples by mechanical techniques in combination with low-voltage ion milling", G. Kothleitner.

## Master Theses in Progress

FABBRO Robert, "Characterisation of thin titanium nitride layers in through silicon via technology", W. Grogger.

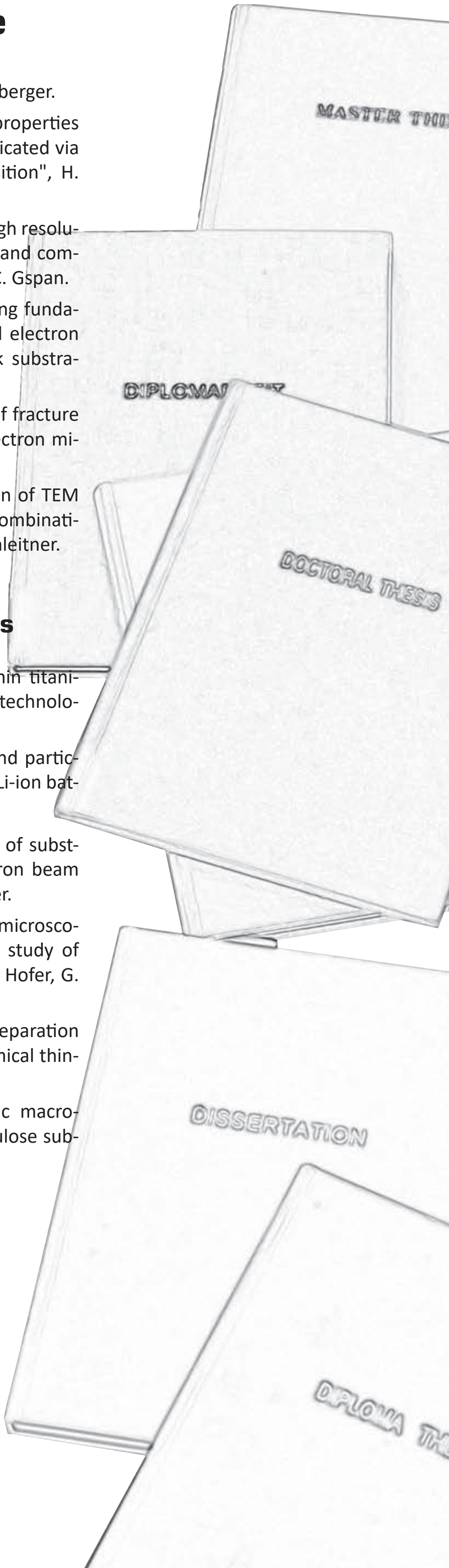
GASSER Eva, "Characterisation of gas and particles released during thermal runaway of Li-ion batteries", G. Grogger, A. Zankel.

HINUM-WAGNER Jakob, "The influence of substrate temperature during focused electron beam induced deposition", H. Plank, R. Winkler.

HJELLE Daniel, "Transmission electron microscopy - electron energy-loss spectroscopy study of lithium containing battery materials", F. Hofer, G. Haberfehlner.

OBERAIGNER Michael, "Specimen preparation for electron tomography by electrochemical thinning", G. Kothleitner, G. Haberfehlner.

ZAJKI-ZECHMEISTER Krisztina, "Dynamic macromolecule adsorption on structured cellulose substrates", H. Plank, S. Spirk.



# 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**
- Max BURIAN, PhD Thesis
- Viktor-Stavros CHRISTOLPOULOS, PhD Thesis

**Institute of Physical and Theoretical Chemistry**
- Efwita ASTRIA, PhD Thesis

**Institute of Experimental Physics**
- Olivia KLEMMER, Master Thesis
- Martin SCHNEDLITZ, Master Thesis
- Alexander SCHIFFMANN, PhD Thesis
- Roman MESSNER, Master Thesis
- Maximillian LASSERUS, Master Thesis

**Institute of Chemistry and Technology of Materials**
- Denise PRUTSCH, Master Thesis
- Sebastian HÖFLER, PhD Thesis
- Christoph BRUDL, PhD Thesis
- Christian LEYPOLD, PhD Thesis
- Bianca PREM, PhD Thesis
- Roman ZETTL, PhD Thesis
- Bernhard KRENMAYR, Master Thesis

**Institute of Chemical Engineering and Environmental Technology**
- Georg BALDAUF-SOMMERBAUER, PhD Thesis
- Klara TREUSCH, PhD Thesis
- Bernd CERMENEK, PhD Thesis
- Katharina KOCHER, Master Thesis
- Bernhard MARIUS, Master Thesis

**Institute of Process and Particle Engineering**
- Laurenz GEIHOFER, Bachelor Thesis
- Federico MARANGONI, Master Thesis
- Manuel ZETTL, PhD Thesis

**Institute of Solid State Physics**
- Martin M. FACCINELLI, PhD Thesis
- Olivia KETTNER, PhD Thesis
- Alexander DALLINGER, PhD Thesis
- Hana K. HAMPEL, Master Thesis
- Sebastian PETERKA, Master Thesis

**Institute of Materials Physics**
- Jaromir KOTZUREK, PhD Thesis
- Markus GÖSSLER, PhD Thesis
- Philipp BRUNNER, PhD Thesis

**Institute of Applied Geoscience**
- Bettina BURGSTALLER, Univ.-Ass.
- Stefanie EICHINGER, Univ.-Ass.
- Ronny BOCH, Univ.-Ass.

**Institute of Materials Science, Joining and Forming**
- Johannes TÄNDL, PhD Thesis
- Christian HOFLEHNER, PhD Thesis
- Bernhard KRENMAYR, Master Thesis

**Institute of Thermal Engineering**
- Christoph WEINLÄNDER, PhD Thesis
- Bernhard STÖCKL, PhD Thesis
- Hannes GERHARDTER, PhD Thesis
- Christian GABER, PhD Thesis
- Mario KNOLL, PhD Thesis

**Institute of Biomechanics**
- Markus GEITH, PhD Thesis

**Institute of Technology and Testing of Construction Materials**
- Claudia BALDERMANN, PhD Thesis

**Institute of Production Engineering**
- Christian HÖLLER, PhD Thesis

Institute of Chemistry, University of Graz

Institute of Biotechnology and Biochemical Engineering, Graz University of Technology

## University of Graz

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

**Institute of Mathematics**
Richard M. HUBER, Master Thesis

**Institute of Molecular Biosciences**
Barbara EICHER, PhD Thesis
Michael PACHLER, PhD Thesis

**Institute of Zoology**
Julia Baumann, Univ.-Ass.

## University of Vienna

**Institute of Biophysical Chemistry**
Denise KÖLBL, PhD Thesis

## University of Linz

**Center for Surface and Nanoanalytics**
Tia TRUGLAS, PhD Thesis

## Montan University Leoben

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

**Chair of Chemistry of Polymeric Materials**
Gisbert Riess, PhD Thesis

**Chair of Functional Materials and Materials Systems**
Christina KAINZ, Master Thesis
Tanja JÖRG, Master Thesis

## University of Innsbruck

**Institute of General, Inorganic and Theoretical Chemistry**
Michael ZOLLER, PhD Thesis

## Erich Schmidt Institute of Materials Science, Austrian Academy of Sciences

Jinming GUO, PhD Thesis

## College for Chemistry, Graz

Dominik GROSSMANN, Diploma Thesis

## Martin Luther University Halle-Wittenberg, Germany

Marta HEUSER, PhD Thesis

## Christian Albrechts University Kiel, Germany

**Institute for Materials Science**
Julian STROBEL, PhD Thesis

## University of Tübingen, Germany

**Group for Geomicrobiology**
Nikolas HAGEMANN, PhD Thesis

## University of Bremen, Germany

**Institute of Applied and Physical Chemistry**
Markus ROHDENBURG, PhD Thesis

## University of Cambridge, U.K.

**Department of Materials Science & Engineering**
Tom FURNIVAL, PhD Thesis

## University of Zaragoza, Spain

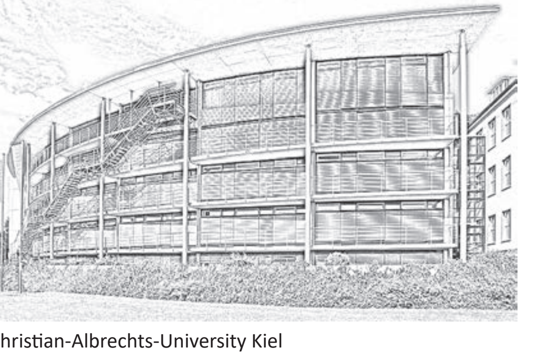
**Advanced Microscopy Laboratory**
Javier PABLO-NAVARRO, PhD Thesis

## Lehigh University, Betlehem, USA

**Department of Materials Science & Engineering**
Michael R. KRACUM, PhD Thesis

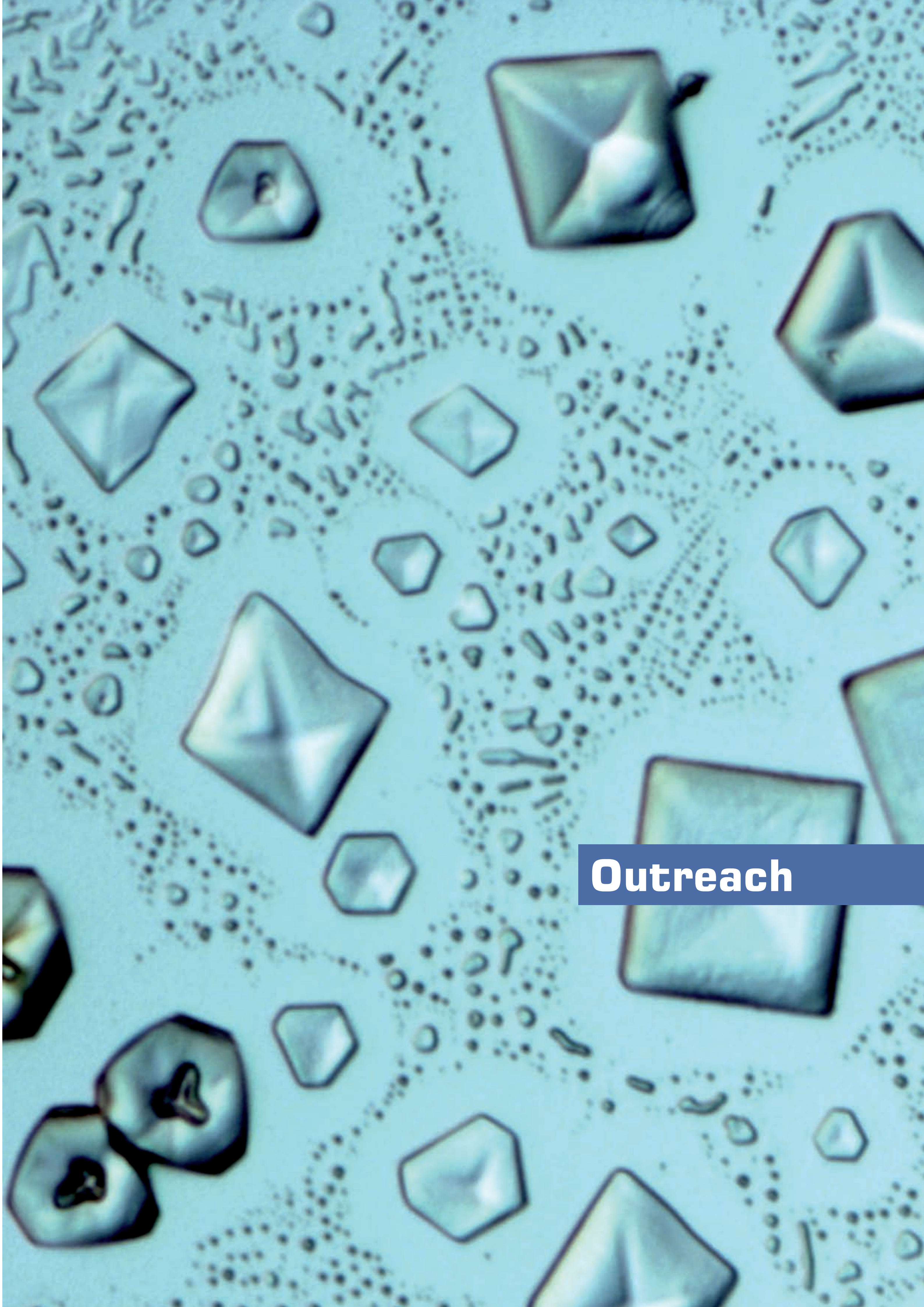
Montan University Leoben

Cambridge University



Christian-Albrechts-University Kiel

# Outreach



# The Institute in the News

## NEWS 2017

ACR Newsletter, 2 Mar. 2017, "Tanzende Atome"
DIE PRESSE/Wissen, 11 Mar. 2017, "Innere Werte von Nanoteilchen"
KLEINE ZEITUNG, 11 Mai 2017, "Analyse von Bauteilen und Werkstoffen"
WOCHE, 16 Mai 2017, "Workshop zur Materialanalyse"
DIE PRESSE/Wissen, 3 Jun. 2017, "Schau, was kommt von draußen rein"
ACR Newsletter, 6 Jun. 2017, "Gute Luft macht den Unterschied: Coin Aufbauprojekt Aeropore"
TU Graz PEOPLE, 2017-2, "E-mail from... Ireland"
DIE PRESSE/Science online, "Pollen & Elektronenmikroskopie"
derSTANDARD.at, 1 Jul. 2017, "Goldgräber auf atomarer Ebene"
ACR Newsletter, 12 Sep. 2017, "ACR series infrastructure: ASTEM" and "Exhibition from Micro to Nano:"
BIO-NANONET Newsletter, 2017-3, "Do you breathe freely? Air quality: our researchers are shifting the focus of the limelight"
ORF.at/Science, 29 Sep. 2017, "Schrauben aus Knochen"
ACR Enquete, 4 Oct. 2017, "ACR Woman Award 2017: Mit Herausforderungen mitwachsen"
KLEINE ZEITUNG, 19 Oct. 2017, "TU-Forscher betrachten Enzyme bei der Arbeit"
TU GRAZ News, 19 Oct. 2017, "Enzyme bei der Arbeit: Aufspaltung widerspenstiger Cellulose"
MEINBEZIRK.at, 13 Nov. 2017, "Materialforschung trifft Unternehmen in der Ebene"
ACR-Expertin, 28 Nov. 2017, "Evelin Fisslthaler: Auf Spurensuche"
ACR Newsletter, 30 Nov. 2017, "Pflanzenkohle als Langzeitdünger"
BIO-NANONET Newsletter, 2017-4, "Biochar in the Transmission Electron Microscope: on the Threshold of Future Fertilisers?"

## NEWS 2018

ACR-Report, 1 Jan. 2018, "Microstructures in focus"
DIE PRESSE/Wissen, 17 Feb. 2018, "Pflanzenkohle: Ein Dünger mit Zusatznutzen"
BIO-NANONET Newsletter, 2018-1, "What is house dust and how does it affect human health?"
STEIRISCHE MOBILITÄT, 1/2018, "Mikrostruktur im Fokus"
GEBÄUDE INSTALLATION, 3/2018 "Filter gegen Grippeviren und Allergene"
APA/SCIENCE, 26 Apr. 2018, "Sonden, Ortung, Papier: Drei neue Christian-Doppler-Labors an TU Graz"
TREND.at, 26 Apr. 2018, "Sonden, Ortung, Papier: Drei neue Christian-Doppler-Labors an TU Graz"
TU GRAZ News, 26 Apr. 2018, "Forschungstrio: TU Graz eröffnet zeitgleich drei CD-Labors"
KLEINE ZEITUNG, 27 Apr. 2018, "Gleich drei neue Labors an der TU"
derSTANDARD.at, 27 Apr. 2018, "Drei neue Christian-Doppler-Labors an TU Graz"
DIE PRESSE, 27 Apr. 2018, "3D-Sonden, Ortung und Verpackungen. Die TU Graz erhielt drei Forschungslabors."
ACR Newsletter, 18 Jun. 2018, "Microstructures in Focus" and "ACR series infrastructures: hhigh resolution scanning electron microscope"
BIO-NANONET Newsletter, 2018-2, "Opening: Christian Doppler Laboratory for the Direct Fabrication of 3D Nano-Probes"
DER STANDARD, 27 Jun. 2018, "Die Gefahr, die durch den Filter kam"
DIE PRESSE/WISSEN, 30 Jun. 2018, "Der Louvre aus dem 3-D-Drucker"
METALL, 7–8/2018, "Gebt dem Laser richtig Pulver: Additive Fertigung"
STANDARD, 14 Aug. 2018, "Was beim 3D-Drucken von Metallen genau passiert"
DIE PRESSE, 20 Sep. 2018, "Wie man Bettwanzen wieder los wird"
MEINBEZIRK.at, 14 Oct. 2019, "Award of Excelence 2018"
KLEINE ZEITUNG, 16 Oct. 2018, "Dieser Hartberger bekam für seine Dissertaton 3000€"
ACR Newsletter, 13 Dec. 2018, "Nanostrukturen bestimmen die Material-Qualität"

6 Lokales

**Workshop zur Materialanalyse**

Sie möchten gerne mehr über die chemische Elementverteilung wissen? Eine Beschichtung verhält sich ungewöhnlich, eine Schweißnaht bereitet Kopfzerbrechen? Diese und andere Themen werden beim kostenlosen Workshop des Zentrums für Elektronenmikroskopie Graz unter dem Motto „Materialforschung trifft Unternehmen" unter die Lupe genommen, der am Dienstag, dem 16. Mai, ab 16 Uhr im Hotel Landskron, Am Schiffertor 3

TECHNISCHE UNIVERSITÄT GRAZ

**Gleich drei neue Labors an der TU**

Gleich drei neue Christian-Doppler-Labors konnten diese Woche an der Technischen Universität Graz eröffnet werden. Karin Zojer leitet das Labor für „Stofftransport durch Papier", das sich mit der Porosität (Durchlässigkeit) von Papier beschäftigt.
Unter Leitung von Harald Plank steht das Labor für „Direkte Fabrikation von 3D-Nanosonden". Die neue Drucktechnik soll neue Sensoren ermöglichen. Unter Leitung von Klaus Witrisal steht das Labor für „Ortssensitive Elektronische Systeme". Dabei geht es um funkbasierte Ortungssysteme für Anwendungen, wo GPS-Satellitensysteme nicht verfügbar sind.
Im CD-Labor wird anwendungsorientierte Grundlagenforschung betrieben; öffentliche Hand und Industrie sind Finanziers. An der TU gibt es jetzt zehn solcher Labors.

TECHNISCHE UNIVERSITÄT GRAZ

**TU-Forscher beobachten Enzyme bei der Arbeit**

Mit dem Rasterkraftmikroskop kann die Cellulose-Verarbeitung entschlüsselt werden.

Die Presse

**3–D–Sonden, Ortung und Verpackungen**

Die TU Graz erhielt drei neue Forschungslabors.

# 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 2018, **Robert Winkler** was awarded at the ASEM Workshop in Vienna for his paper "Direct-write 3D-nanoprinting of plasmonic structures" published in ACS Applied Materials and Interfaces.

## Lecture Award

**Harald Fitzek** received the Lecture Award for young researchers at the International Conference of Advanced Vibrational Spectroscopy 2017 in Victora, Canada. His talk was entitled: "Understanding surface enhanced Raman spectroscopy using accurate nearfield-simulations of real substrates."

## Best Poster Award

The 13th Multinational Congress on Microscopy took place in Rovinj, Croatia from 24th–29th September 2017. **Daniel Knez** received the Instrumentation Session Best Poster Award for his poster: “In-situ studies of high-purity mono- and bimetallic nanostructures in experiment and theory”. The presented results came from the collaboration with the Institute of Experimental Physics (TU Graz).

## And the ACR Woman Award 2017 goes to .....

**Evelin Fisslthaler**, who has been a team member since 2009. Born in Salzburg, she earned her PhD in physics at the TU Graz. Evelin’s research efforts are concentrated on nanoanalysis of microelectronics using transmission electron microscopy. In October 2017 she was awarded with the ACR Woman Prize for leading the FFG-funded project “Quantitative analysis of internal interfaces in semiconductor devices".

## Award of Excellence

The Award of Excellence, a prize of the Austrian Ministry of Education, Science and Research honours the forty best dissertations of the past academic year. In 2018, **Robert Winkler** won the prize for his PhD thesis entitled "Fabrication of functional and freestanding 3D nano-architectures via focused electron beam induced deposition". The Award was presented in December 2018 in a festive ceremony in Vienna.

## Werner Koester Award 2017

of the German Materials Society (DGM) was awarded for the paper "Structural anisotropy in equal-channel angular extruded nickel revealed by dilatometric study of excess volume" with contributions from the Institute of Materials Physics and the FELMI-ZFE (J.A. Kotzurek, W. Sprengel, R. Würschum and S. Simic and P. Pölt).


In Anerkennung hervorragender Studienleistungen verleihe ich

Herrn Mag. art. Dipl.-Ing. Dr. techn. Robert WINKLER, BSc

den

Award of Excellence 2018

Staatspreis für die besten Dissertationen.

Bundesminister Univ.-Prof. Dr. Heinz Faßmann
Wien, am 5. Dezember 2018


The

Werner Koester Award 2017

is granted to the authors

Dr. Jaromir Anatol Kotzurek, Assoc.-Prof. Dr. Wolfgang Sprengel, Dr. Maciej Krystian, Sanja Simic, PD Dr. Peter Pölt, Dr. Anton Hohenwarter, Prof. Dr. Reinhard Pippan, Prof. Dr. Roland Würschum

of the paper

**Structural anisotropy in equal-channel angular extruded nickel revealed by dilatometric study of excess volume, IJMR 108 (2017), 163–172.**

Severe plastic deformation, as by equal-channel angular pressing (ECAP), leads to anisotropy of the ultrafine grains and excess volume due to lattice vacancies and grain-boundary expansion. In a carefully conducted dilatometric study, from the annealing induced length changes in various directions, the authors were able to characterize, in a quantitative way, the vacancy concentration, the vacancy annihilation and (the relaxation of) the grain boundary expansion. This work has shown that high precision (difference) dilatometry will be a powerful tool in future studies on structurally complex (anisotropic) microstructures.

Prof. Dr. Alexander Hartmaier
DGM-Vorsitzender

DGM-Tag
Darmstadt, September 2018

Prof. Dr. Eric Jan Mittemeijer
Editor of the International Journal of Materials Research (IJMR)

DGM | Erfahrung · Kompetenz · Wissen
Deutsche Gesellschaft für Materialkunde e.V.

# Around the World

## Cooperation with the Fritz Haber Institute Berlin

In Berlin, at the Fritz Haber Institute of the Max Planck Society (FHI) **Manfred Nachtnebel** met international colleagues focusing on ESEM. Together they wanted to find other possibilities how to use recent ESEM improvements for *in-situ* analyses. For two weeks they concentrated on the hydrogen production during heating experiments thus combining the FHI's know-how (a laser heating stage, a mass spectrometer and the possibility to use specific gas compositions) with the Graz ZFE's special equipment (UPSEM) made it possible to perform heating experiments in a more realistic scenario. For the first time, the scientists took images of catalytical processes between 700 Pa and 1200 Pa. They observed structural changes of surfaces and were able to overcome the pressure gap which was not possible with the standard system due to physical restraints.

## ACR Study Trip to Isreal

Once a year the Austrian Cooperative Research organises a study trip in order to broaden one's horizon, to discover new possibilities for innovation and to strengthen the international research network. The 2017 destination was Israel which was joined by Ferdinand Hofer and Hartmuth Schröttner. Israel is the second most innovative nation in the world, according to the World Economic Forum's Global Competitiveness Report 2016-2017. From Jerusalem to Tel Aviv the programme was full of interesting meetings and presentations: A dinner talk with the director general of the green energy association of Israel, the Hebrew University, the Porter School of Environmental Studies, Tel-Aviv University Center for Nanoscience and Nanotechnology and of course the TECHNION in Haifa!

## Sidney Calling

In September 2018 we started a long journey to Australia to visit the 19th International Microscopy Congress IMC19 in Sidney. A group of six colleagues form FELMI-ZFE presented lectures and posters and took part in the large microscopy confernce and exhibition.



## Research Stay at the Goethe Universität Frankfurt

In 2018, Ass.Prof. Priv.-Doz. DI Dr. Harald Plank went to Frankfurt am Main, Germany, for an external research stay in the frame of his tenure track position at the FELMI. Based on a long lasting collaboration with Prof. Michael Huth from the Institute of Physics at the Goethe University Frankfurt, the activities focused on thermal properties of freestanding platinum-carbon nanowires, fabricated via 3D nanoprinting. To get a more detailed understanding on nanoscale heat generation via electric currents, charge transport measurements were conducted in vacuum conditions over the wide temperature range of 20 K – 475 K. The gathered insight formed the basis for further research activities in the area of thermal 3D nano-probes, which are of central interest in the CDL DEFINE at the FELMI.

Workgroup of Prof. Michael Huth:
http://nano.physik.uni-frankfurt.de/index.html

## Core Meeting Electron Beam Induced Processing

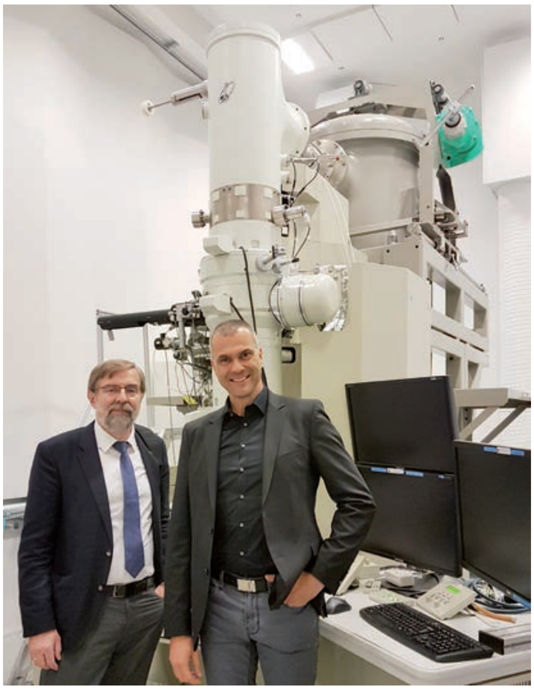
In 2018, the biannual core meeting in the field of focused electron beam induced processing (FEBIP) was held in Modena, Italy. FELMIs workgroup for Functional Nanofabrication, contributed to that meeting with 4 active talks and several co-autorships, which further strengthen FELMIs international reputation in this research field.

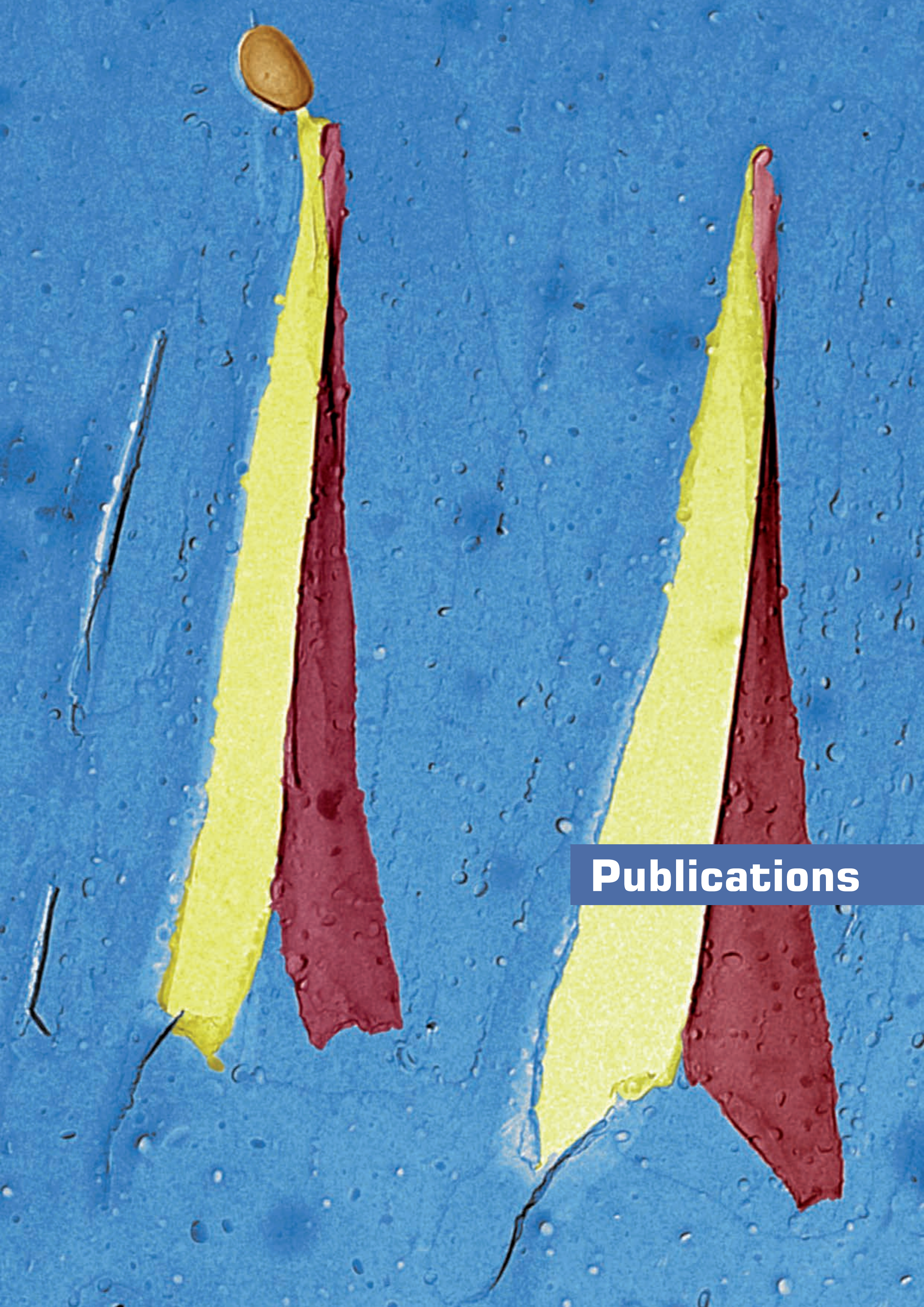
FEBIP 2018: July 10th – 13th 2018, Modena, Italy

Webpage:
http://web.nano.cnr.it/FEBIP2018-Modena/

## ACR Delegation in Finland

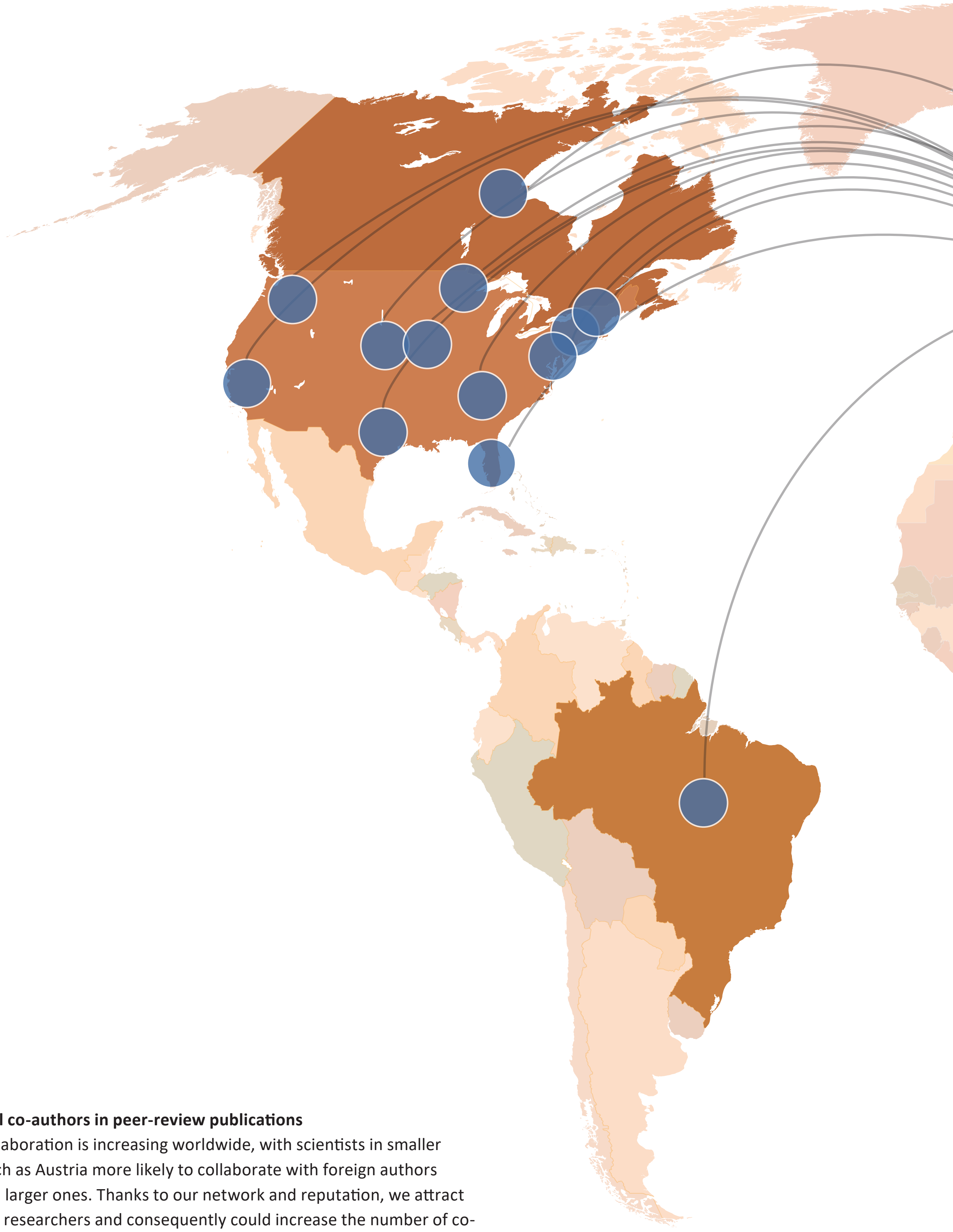
At the end of May, a 31-member ACR delegation travelled to Finland, consisting of representatives of the 18 ACR institutes, ministries, interest groups and media, to see how an "Innovation Leader" manages to get back on the fast track in research and innovation despite numerous crises. Ferdinand Hofer and Hartmuth Schröttner used the visit to revive a cooperation with Professor Esko Kauppinen from the Nanomaterials Group of the Aalto University in Espoo.



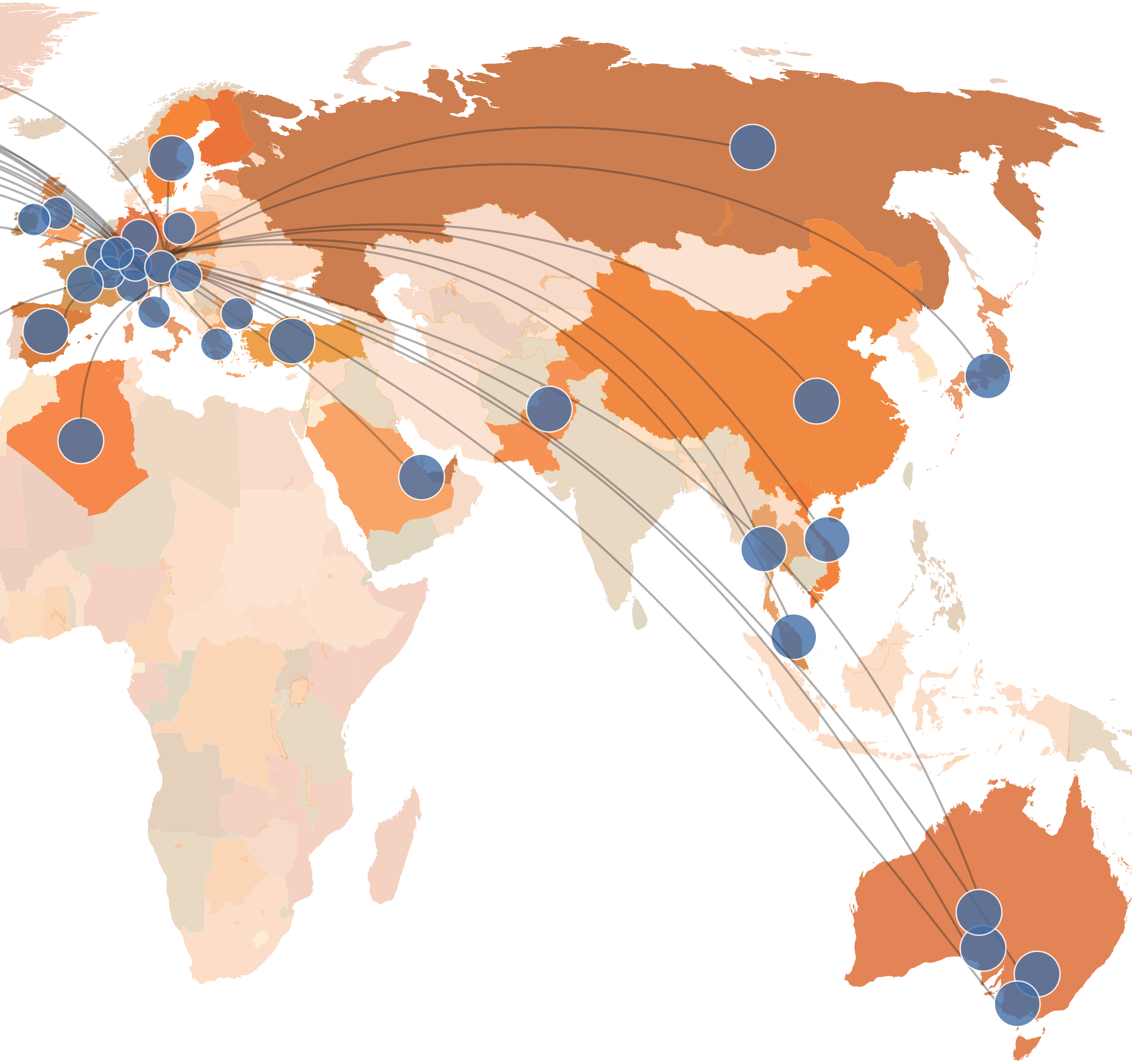


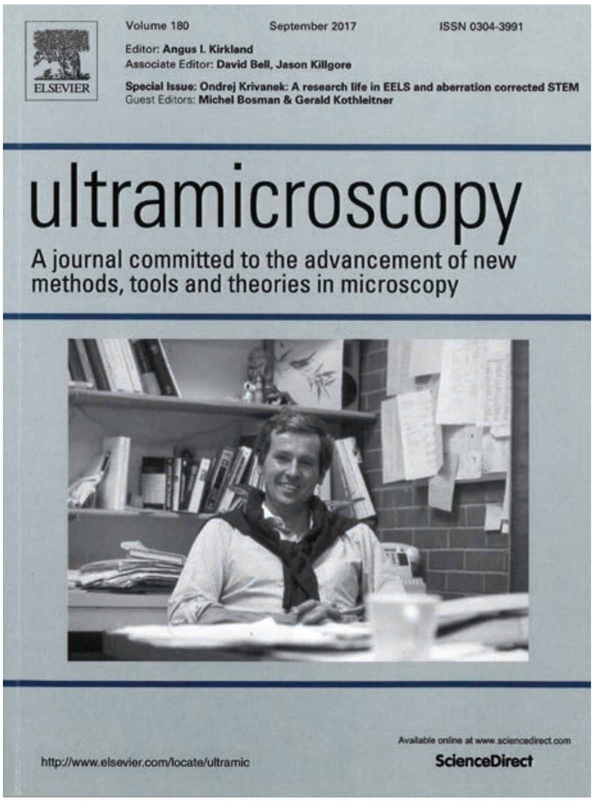
# Publications

# Publishing Activities



**International co-authors in peer-review publications**
Scientific collaboration is increasing worldwide, with scientists in smaller countries such as Austria more likely to collaborate with foreign authors than those in larger ones. Thanks to our network and reputation, we attract international researchers and consequently could increase the number of co-authors from other countries in the period 2017-2018 (see map of the world).






Gerald Kothleitner is Guest Editor for the Ultramicroscopy issue:

**Ondrej Krivanek - A research life in EELS and aberration corrected STEM**

Ondrej's tremendous achievements in electron microscopy were honored in a special issue of Ultramicroscopy featuring 23 scientific works from around the world. His developments have enabled gound-breaking experiments and have led to new insights in materials and life sciences and theoretical disciplines. We are proud that Gerald, who also serves on the editorial board of Ultramicroscopy, was given this honorable task.

*Ultramicroscopy volume 180, september 2017.*

# Publishing Activities

## Peer Reviewed Publications

### 2017


Aghazadeh Meshgi, M.; Biswas, S.; McNulty, D.; O'Dwyer, C.; Verni, A.V.; Letofsky-Papst, I.; Pölt, P.; Holmes, J.; Marschner, Ch.: "*Rapid, low temperature synthesis of germanium nanowires from oligosilylgermane precursors*", Chemistry of Materials 29 (2017) 10, 4351–4360.

Archanjo, B. S.; Mendoza, M. E.; Albu, M.; Mitchell, D. R. G.; Hagemann, N.; Mayrhofer, C.; Mai, T. L. A.; Weng, Z.; Kappler, A.; Behrens, S.; Munroe, P.; Achete, C.A.; Donne, S.; Araujo, J.R.; van Zwieten, L.; Horvat, J.; Enders, A.; Joseph, S.: "*Nanoscale analyses of the surface structure and composition of biochars extracted from field trials or after co-composting using advanced analytical electron microscopy*", Geoderma B294 (2017) 70–97.

Berger, Ch.; Bucher, E.; Gspan, Ch.; Menzel, A.; Sitte, W.: "*Impact of $SO_2$ on the oxygen exchange kinetics of the promising SOFC/SOEC air electrode material $La_{0.8}Ca_{0.2}FeO_{3-\delta}$*", Journal of the Electrochemical Society B164 (2017) 10, F3008–F3018.

Bosman, M.; Kothleitner, G.: "*Ondrej Krivanek: A research life in EELS and aberration corrected STEM*", Ultramicroscopy 180 (2017) 1–2.

Bucher, E.; Gspan, Ch.; Höschen, T.; Hofer, F.; Sitte, W.: "*Oxygen exchange kinetics of $La_{0.6}Sr_{0.4}CoO_{3-\delta}$ affected by changes of the surface composition due to chromium and silicon poisoning*", Solid State Ionics B299 (2017) 26–31.

Buchmaier, Ch.; Torvisco Gomez, A.; Pölt, P.; Wewerka, K.; Kunert, B.; Gatterer, K.; Trimmel, G.; Rath, T.: "*Nickel sulfide thin films and nanocrystals synthesized from nickel xanthate precursors*", Journal of Materials Science B52 (2017) 10898–10914.

Cvjetko, P.; Domijan, A.-M.; Vinkovic-Vrcek, I.; Tolic, S.; Peharec-Stefanic, P.; Letofsky-Papst, I.; Tkalec, M.; Balen, B.: "*Toxicity of silver ions and differently coated silver nanoparticles in Allium cepa roots*", Ecotoxicology and Environmental Safety 137 (2017) 18–28.

Cvjetko, P.; Zovko, M.; Peharec Stefanic, P.; Tkalec, M.; Domijan, A.-M.; Vinkovic Vrcek, I.; Letofsky-Papst, I.; Sikic, S.; Balen, B.: "*Phytotoxic effects of silver nanoparticles in tobacco plants*", Environmental Science and Pollution Research International B25 (2017) 6, 5590–5602.

Ebner, M.; Schennach, R.; Chien, H.-T.; Mayrhofer, C.; Zankel, A.; Friedel, B.: "*Regenerated cellulose fiber solar cell*", Flexible and Printed Electronics 2 (2017) 014002.

Egger, A.; Schrödl, N.; Gspan, Ch.; Sitte, W.: "*$La_2NiO_{4+\delta}$ as electrode material for solid oxide fuel cells and electrolyzer cells*", Solid State Ionics B299 (2017), 18–25.

Eibinger, M.; Sattelkow, J.; Ganner, T.; Plank, H.; Nidetzky, B.: "*Single-molecule study of oxidative enzymaticdeconstruction of cellulose*", Nature Communications (2017) 8:894.

Frampton, M. B.; Marquardt, D.; Letofsky-Papst, I.; Pabst, G.; Zelisko, P.: "*Analysis of trisiloxane phosphocholine bilayers*", Langmuir (2017) 4948–4953.

Fischer, J.; Mayr, M.; Spirk, S.; Reishofer, D.; Jagiello, A. L.; Schmiedt, Colson, J.; Zankel, A.; Bauer W.: "*Pulp fines: Characterization, sheet formation, and comparison to microfibrillated cellulose*", Polymers 9 (2017) 8, 366-377.

Gariboldi, E.; Bassani, P.; Albu, M.; Hofer, F.: "*Presence of silver in the strengthening particles of an Al-Cu-Mg-Si-Zr-Ti-Ag alloy during severe overaging and creep*", Acta Materialia 125 (2017) 50–57.

Rottensteiner, W.; Padure, I.; Simic, S.: "*Pollen SEM morphology in Ophrys istriensis from Istria*", Joannea/Botanik B14 (2017) 147–151.

Granitzer, P.; Rumpf, K.; Pölt, P.; Reissner, M.: "*Biocompatible Nanovehicle With Adjustable Magnetic Properties*", IEEE Transactions on Magnetics B53 (2017) 11, 2301105–5.

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Schrödl, N.; Egger, A.; Gspan, C.; Höschen, T.; Hofer, F.; Sitte, W.: "*Phase decomposition of* $La_2NiO_{4+\delta}$ *under Cr- and Si-poisoning conditions*", Solid State Ionics 322 (2018) 44–53.

Springholz, G.; Wimmer, S.; Groiss, H.; Albu, M.; Hofer, F.; Caha, O.; Kriegner, D.; Stangl, J.; Bauer, G.; Holy, V.: "*Structural disorder of natural* $Bi_mSe_n$ *superlattices grown by molecular beam epitaxy*", Physical Review Materials 2 (2018) 054202.

Stefanic, P.P.; Cvjetko, P.; Biba, R.; Domijan, A.M.; Letofsky-Papst, I.; Tkalec, M.; Sikic, S.; Cindric, M.; Balen, B., *"Physiological, ultratsructural and proteomic responses of tobacco seedlings exposed to silver nanoparticles and silver nitrate",* Chemosphere 209 (2018) 640-653.

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Winkler, R.; Lewis, B. B.; Fowlkes, J. D.; Rack, P.; Plank, H.: "*High-fidelity 3D-nanoprinting via focused electron beams: Growth fundamentals*", ACS Applied Nano Materials 1 (2018) 1014–1027.

Zankel, A.; Nachtnebel, M.; Mayrhofer, C.; Wechsler, K.: "*From electron microscopy to 3D printing: Serial block-face scanning electron microscopy of spores*", Imaging & Microscopy (2018) 38–40.

# Lectures

## 2017

Albu, M.: "*Advanced alloys and steels microstructure characterization by analytical HR-STEM*", 7th Annual Congress on Materials Research and Technology, Berlin, Germany, 20 Feb. 2017 (invited).

Grogger, W.: "*X-ray spectroscopy in scanning transmission electron microscopy*", Workshop on Transmission Electron Microscopy, Antwerp, Belgium, 22-25 Jun. 2017 (invited).

Hofer, F.: "*Direct imaging of channel constituents in beryl*", IAMNano 2017 - Int. Workshop on Advanced and *in-situ* Microscopies of Functional Nanomaterials and Devices, Singapure, 12-15 Nov. 2017 (invited).

Haberfehlner, G.: "*Electron tomography – three-dimensional atomic, elemental and field mapping*", 24th Congress of the International Union of Crystallography, Hyderabad, India, 21-28 Aug. 2017 (invited).

Hofer, F.: "*A new view on the surface of materials*", Symposium Tribo Design, Dornbirn, Austria, 26-28 Jun. 2017 (invited).

Hofer, F.: "*Electron beam induced dynamics of atoms and clusters-experiments and simulations*", Conference on Frontiers of Aberration Corrected Electron Microscopy, Kasteel Vaalsbroek, Netherlands, 30 Apr. – 4 May 2017 (invited).

Hofer, F.: *"Cryo Electron Microscopy: High resolution investigations of biological structures and macromolecules"*, Nobel-Prize Colloquium 2017 of the University of Leoben, Leoben, Austria, 14 Dec. 2017 (invited).

Kothleitner G.: "Compositional quantification of inelastic atomic resolution STEM images", Sub-nm Materials Technologies Workshop, Singapore, 16 Jan. 2017 (invited).

Kothleitner, G.: "*Chemical mapping*", QEM Quantitative Electron Microscopy 2017, Balaruc-les Bains, France, 22 May –2 Jun. 2017 (invited).

Kothleitner, G.: "*EELS and X-ray spectroscopic STEM imaging in 2D and 3D* Spectroscopy", EDGE Conference - Enhanced Data Generated by Electrons, Okinawa, Japan, 14-19 May 2017 (invited).

Kothleitner, G.: "Spectroscopic imaging in 2D and 3D", Microscopy Conference 2017, Lausanne, Switzerland, 21-25 Aug. 2017 (invited).

Kothleitner G.: "Introduction to EELS", School on Advanced Analytical Electron Microscopy, Jülich, Germany, 7 Sep. 2017 (invited).

Schmidt, F.: "*Hybrid plasmonics: From plasmon-plasmon to plasmon-exciton coupling*", EDGE Conference - Enhanced Data Generated by Electrons, Okinawa, Japan, 14 - 19 May 2017 (invited).

Rattenberger, J.: "*Universal pressure scanning electron microscopy (UPSEM) - Electron microscopy up to atmospheric pressure*", 13th Multinational Congress on Microscopy, Rovinj, Croatia, 24 - 29 Sep. 2017 (invited).

Winkler, R.: "*Direct-write fabrication of 2D and 3D plasmonic-structures via FEBID*", Symp. European FIB Network, Graz, Austria, 4 - 6 Jul. 2017 (invited).

Winkler, R.: "*3D nano printing via focused electron beams: From a concept towards stable nanofabrication*", Symp. European FIB Network, Graz, Austria, 4 - 6 Jul. 2017 (invited).

Winkler, R.: "*Research on Novel Precursor Using a Versatile Gas Injection System*", 4th Kleindiek User Meeting, Reutlingen, Germany, 25–26 Apr 2017 (invited).

Winkler, R.: "*Direct-write fabrication of electric and thermal high-resolution nanoprobes on self-sensing AFM cantilever*", 61st Int. Conf. on Electron, Ion, and Photon Beam Technology and Nanofabrication, Orlando, USA, 30 May – 4 Jun. 2017 (invited).

Albu, M.: "*Analytical microstructure characterization of advanced alloys at atomic resolution*", 19th Plansee Seminar, Reutte, Austria, 29 May – 2 Jun. 2017.

Albu, M.: "*High resolution analytical transmission electron microscopy as high-end investigation method for biochar*", European Geosciences Union General Assembly, Vienna, Austria, 23-28 Apr.

2017.

Dienstleder, M.: "*Advancement of broad and focused low energy ion milling procedures for the optimization of high resolution TEM samples*", 13th Multinational Congress on Microscopy, Rovinj, Croatia, 24-29 Sep. 2017.

Dienstleder, M.: "*Challenges of high-end specimen preparation*", 7th ASEM Workshop of the Austrian Society of Electron Microscopy, Vienna, Austria, 17-18 Apr. 2017.

Fisslthaler, E.: "*Quantitative analysis of interfaces*", 7th ASEM Workshop of the Austrian Society of Electron Microscopy, Vienna, Austria, 17-18 Apr. 2017.

Fitzek, H. M.: "*Understanding surface enhanced Raman spectroscopy using accurate nearfield-simulation of real substrates*", 9th International Conference on Advanced Vibrational Spectroscopy, Victoria, Canada, 10-15 Jun. 2017.

Haberfehlner, G.: "*Surface plasmon tomography*", 13th Multinational Congress on Microscopy, Rovinj, Croatia, 24-29 Sep. 2017.

Hofer F.: "*Laudatio für Ulrich Hohenester*", Verleihung Steirischer Forschungspreis 2017, Graz, Austria, 15 Dec. 2017.

Hofer, F.: "*Analytical scanning transmission electron microscopy at atomic resolution*", Seminar Physikalische und Theoretische Chemie, Karl-Franzens-University Graz, Austria, 31 Mar. 2017.

Knez, D.: "*In situ studies of high-purity mono- and bimetallic nanostructures in experiment and simulation*", Microscopy Conference 2017, Lausanne, Switzerland, 21-25 Aug. 2017.

Lammer, J.: "*How sample holder geometries influence the quantification of X-ray spectra*", 13th Multinational Congress on Microscopy, Rovinj, Croatia, 24-29 Sep. 2017.

Nachtnebel, M.: "*Fracture investigation of polymers by 3D reconstruction*", 7th ASEM workshop of the Austrian Society of Electron Microscopy, Vienna, Austria, 17 Apr. 2017.

Nachtnebel, M.: "*Polymer fracture - analysis by 3D reconstructions of the fracture region*", 13th Multinational Congress on Microscopy, Rovinj, Croatia, 24-29 Sep. 2017.

Orthacker, A.: "*Non-equilibrium formation of stoichiometric precipitates in multi-component alloys*", 7th ASEM Workshop of the Austrian Society of Electron Microscopy, Vienna, Austria, 17-18 Apr. 2017.

Pölt, P.: "*Polymer fracture - 3D-reconstructions as an analysis tool*", Microscopy Conference 2017, Lausanne, Switzerland, 21–25 Aug. 2017.

Rattenberger, J.: "*Scanning electron microscopy (UPSEM) to atmospheric pressure*", Microscopy Conference 2017, Lausanne, Switzerland, 21-25 Aug. 2017.

Rattenberger, J.: "*Scanning electron microscopy up to atmospheric pressure*", SCANDEM Conference, Reykjavik, Island, 5-6 Jun. 2017.

Sattelkow, J.: "*Direct-Write fabrication of electric and thermal high-resolution nanoprobes on self-sensing AFM cantilever*", 7th ASEM Workshop of the Austrian Society of Electron Microscopy, Vienna, Austria, 17-18 Apr. 2017.

Schröttner, H.: "*Charakterisierung von Werkzeugen und Beschichtungen*", TCM International Tool Consulting & Management GmbH, Austria, 1 Mar. 2017.

Schröttner, H.: "*Mikro- und Nanoanalytische Charakterisierungsmöglichkeiten von Füllstoffen und Polymeren*", MAGNIFIN Magnesiaprodukte GmbH & Co KG, Austria, 30 Mar. 2017.

Schröttner, H.: "Mikro- und Nanaoanalytik an Abgaskatalysatoren", Fresenius Kabi Austria GmbH, Austria, 29 Jun. 2017.

Schröttner, H.: "Mikroskopie-Workshop", IBIDEN Porzellanfabrik Frauenthal GmbH, Austria, 24 Aug. 2017.

Schröttner, H.: "*Neue Präparationstechniken und korrelierende Untersuchungsmethoden zur Charakterisierung von Carbonitrier-Schichten*", Lingenhöle Technologie GmbH, Austria, 11 Oct. 2017.

Schröttner, H.: "Mikroskopische *Charakterisierung von Polymeren*", Zumtobel Lighting GmbH, Austria, 10 Oct. 2017.

Schröttner, H.: "*Mikro- und Nanaoanalytik an Abgaskatalysatoren*", Remus Sebring Group, Austria, 23 Oct. 2017.

Striemitzer, R.: "*Dealing with light refraction in 3D mapping in combined Raman/SEM*", 7th ASEM workshop of the Austrian Society of Electron Microscopy, Vienna, 17-18 Apr. 2017.

Trummer, C.: "*Preparation of TEM samples by mechanical techniques and low-voltage ion milling*", 7th ASEM workshop of the Austrian Society of Electron Microscopy, Vienna, 17-18 Apr. 2017.

Winkler, R.: "*FEBID based direct-write of 2D and 3D plasmonic gold structures*", 7th ASEM Workshop of the Austrian Society of Electron Microscopy, Vienna, Austria, 17 - 18 Apr. 2017.

Zankel, A.: "*Moderne elektronenmikroskopische Methoden zur Untersuchung von polymeren Werkstoffen in Wissenschaft und Industrie*", Merseburger Elastomertage, Merseburg, Germany, 28 Sep. 2017.

## Lectures

## 2018

Albu, M.: *"Analytical scanning TEM in-situ observations of LPSO nucleation in Mg-alloys*", 15th Annual Congress on Materials Research and Technology 2018, Paris, France, 19 Feb. 2018 (invited).

Albu, M.: "*Analytical microstructure characterization at atomic resolution of advanced materials"*, 4th Global Nanotechnology Congress and Expo, Dubai, 16-18 Apr. 2018 (invited).

Albu, M.: *"Advanced ex- and in-situ high resolution TEM chracterization of alloys"*, Int. Conf. on Processing & Manufacturing of Advanced Materials 2018, Paris, France, 8-9 Jul. 2018 (invited).

Grogger, W.: *"Energy dispersive X-ray spectrometry in the TEM", Meeting on TEM in Materials Sciences*", Brno, Czech Republic , 24

Apr. 2018 (invited).

Grogger, W.: “Charakterisierung von strukturellen Defekten in der Oberflächenzone von geschliffenen Werkzeugen”, 2. Schwarzsee Talk 2018, Plaffeien, Switzerland, 5 Jun. 2018 (invited).

Grogger, W.: “EDXS in the TEM: What needs to be considered for reliable quantitative results?”, IAMNano 2017 - Int. Workshop on Advanced and *in-situ* Microscopies of Functional Nanomaterials and Device, Hamburg, Germany, 14 - 18 Oct. 2018 (invited).

Hofer, F.: *“Quantitative electron microscopy at atomic resolution”*, ELMINA - Conference on Electron Microscopy of Nanostructures, Belgrad, Serbia, 27 - 29 Aug. 2018 (invited).

Hofer, F.: *“Electron beam induced dynamics of atoms and clusters”*, IAMNano 2017 - Int. Workshop on Advanced and *in-situ* Microscopies of Functional Nanomaterials and Devices, Hamburg, Germany, 14 - 18 Oct. 2018 (invited).

Krisper, R.: “The performance of EDXS at elevated sample temperature”, *In-situ* Workshop 2018, Jülich, Germany, 6 Jun. 2018 (invited).

Mitsche, S.: *“In-situ heating EBSD investigations: Recrystallization behaviour and microstructure evolution of an Al-Si-Mg-alloy”*, Int. Conf. on Processing & Manufacturing of Advanced Materials 2018, Paris, France, 8 Jul. 2018 (invited).

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Albu, M.: *“Microstructure investigations of powders for additive manufacturing”*, Metal Additive Manufacturing Conference 2018, Vienna, Austria, 20 Nov. 2018.

Fitzek, H.: *“Recent developments in correlative SEM-Raman confocal microscopy, examples of application and perspective”*, Annual Meeting of the Hungarian Society for Microscopy, Siofok, Hungeria, 24 - 25 May 2018.

Haberfehlner, G.: *“Plasmon field tomography of coupled metallic nanoparticles”*, 19th International Microscopy Congress, Sydney, Australia, 9 - 14 Sep. 2018.

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# Book Chapters & Conference Abstracts

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## Book Chapters & Conference

# Abstracts

## 2018

### Sind das alle Beiträge?

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Mutunga, E., Rack, P.D.; Fowlkes, J.D.; Plank, H.; Winkler, R.; : “*Fine-Tuning Nanowire Shape Using 3D Focused Electron Beam Induced Deposition*”, Proc. 62nd Int. Conf. on Electron, Ion, and Photon Beam Technology, and Nanofabrication (EIPBN) (2018) p. 77, Puerto Rico, USA.

Nachtnebel, M.; Mayerhofer, C.; Pölt, P.; "Polymer fracture - 3D reconstaructionsas an analysis tool", Proc. Microscopy Conference 2017, Lausanne Switzerland (2017), pp. 298-299.

Pabel, T.; Petkov, T.; Schröttner, H.; Albu, M.; Rossmann-Perner, A.; Schumacher P.; Li, J.: “*Effect of trace elements on microstructure and material properties of an aluminium alloy*”, Proc. 73rd Worl Foundry Congress (2018) pp. 69–70, Krakow, Poland.

Padure, I.; Simic, S.; Zernig, K.; Heber, G.: “*Pollen, seed and fruit morphology of the genus Hesperis L. (Brassicaceae) in Austria and their taxonomic value.*”, Proc. 24th Int. Symp. on Biodiversity and Evolutionary Biology, German Botanical Society (2018) pp. 108–109, Klagenfurt, Austria.

Rattenberger, J.; Achtsnit, T.; Fitzek, H.; Schroettner, H.; Hofer, F.; "*Universal pressure scanning electron microscopy (USPEM)*", Proc. Microscopy Conference 2017, Lausanne Switzerland (2017), pp. 436-437.

Sattelkow, J.; Fröch, J.; Winkler, R., Schwalb, C.; Winhold, M.; Deutschinger, A.; Strunz, T.; Fantner, E.G.; Stavrov, V.; Plank, H.: “*Direct-Write Fabrication of Electric and Thermal High-Resolution Nano-Probes on Self-Sensing AFM Cantilever*”, Proc. 7th International Workshop on Focused Electron Beam Induced Processing (FEBIP) (2018) pp. 68–69, Modena, Italy.

Sattelkow,J.; Fröch, J.; Winkler, R.; Schwalb, C.; Winhold, M.; Deutschinger, A.; Strunz, T.; Fantner, E.G.; Stavrov, V.; Plank, H.: “*Direct-Write 3D Nano-Printing of Thermal High-Resolution Nano-Probes*”, Proc. 8th Advanced Electron Microscopy Workshop (ASEM) (2018)

p. 14, Vienna, Austria.

Schiffmann A.; Knez, D.; Lackner, F.; Lasserus, M.; Messner, R.; Schnedlitz, M.; Hofer, F.; Ernst, W.E.: "*Plasmon spectroscopy of metallic nanoparticles on ultra-thin h-BN films*", Proc. 68th Annual Meeting of the Austrian Physical Society (2018) p. 83, Graz, Austria.

Steyskal, E.; Pölt, P.; Albu, M.; Würschum, R.: "*In-situ Studies on the Formation and Tunable Properities of Nanoporous Metals Produced by Electrochemical Dealloying*", 68th Annual Meeting of the Austrian Physical Society (2018) p. 37, Graz, Austria.

Strobel, J.; Haberfehlner, G.; Popescu, R.; Kothleitner, G.; Kienle, L., "*Quantiative spectroscopic analysis of memristive oxide triple layers*" Proc. Microscopy Conference 2017, Lausanne Switzerland (2017), p. 85.

Trummer, C.; Winkler, R.; Plank, H.; Kothleitner, G.; Haberfehlner, G.: "*Accessing the internal morphology of nano-granular FEBID materials in 3D space by electron tomography*", Proc. 7th International Workshop on Focused Electron Beam Induced Processing (FEBID) (2018) pp. 74–75, Modena, Italy.

Trummer, C.; Winkler, R.; Plank, H.; Kothleitner, G.; Haberfehlner, G.: "*Accessing the internal morphology of nano-granular FEBID materials in 3D space by electron tomography*", 68th Annual Meeting of the Austrian Physical Society (2018) p. 111, Graz, Austria.

Trummer, C.; Winkler, R.; Plank, H.; Kothleitner, G.; Haberfehlner, G.: "*Morphological Analysis of FEBID Deposits by Electron Tomography*", Proc. 8th Advanced Electron Microscopy Workshop (ASEM) (2018) p. 20, Vienna, Austria.

Winkler, R.; Sattelkow, J.; Plank, H.; Fowlkes, J.D.; Rack, P.D.: "*3D-Nanoprinting of Functional and Freestanding Structures via Electron Beams: an Application Perspective*", Proc. 62nd Int. Conf. on Electron, Ion, and Photon Beam Technology, and Nanofabrication (EIPBN) (2018) p. 52, Puerto Rico, USA.

Winkler, R.; Sattelkow, J.; Fowlkes, J.D.; Rack, P.D.; Plank, H.: "*3D-Nanoprinting via FEBID-from Growth Fundamentals to Applications*", Proc. 7th International Workshop on Focused Electron Beam Induced Processing (FEBIP) (2018) pp. 50–51, Modena, Italy.

Winkler, R.; Sattelkow,J.; Schmidt, F.; Haselmann, U.; J.; Fowlkes, J.D.; Rack, P.D.; Plank, H.: "*3D-Nanoprinting of Functional and Freestanding Structures via Electron Beams: an Application Perspective*", Proc. 8th Advanced Electron Microscopy Workshop (ASEM) (2018) p. 9, Vienna, Austria.

Zankel, A.; Theissing, M.; Nachtnebel, M.; Mayrhofer, C.; Gahleitner, M.; Pölt, P.: "*Fracture behavior of polymer blends – Can particles, cracks and crystallinity be combined in one 3D representation?*", Proc. Polymertec (2018) p. 127, Merseburg, Germany.

Zajki-Zechmeister, K.; Schlemmer, W.; Sattelkow, J.; Spirk, S.; Plank, H.: "*Dynamic in-situ observation of protein adhesion and chemical coupling using liquid atomic force microscopy*", Proc. 68th Annual Meeting of the Austrian Physical Society (2018) p. 134, Graz, Austria.

# Poster Presentations

## 2017

Dienstleder, M.; Fisslthaler, E.; Gspan, Ch.; Kothleitner, G. "*Low energy argon ion sample preparation for HR-STEM analysis*" 7th ASEM Workshop of the Austrian Society of Electron Microscopy, Vienna, Austria, 20–21 Apr. 2017.

Fisslthaler, E.; Gspan, Ch.; Haberfehlner, G.; Grogger, W. "*Quantitative analysis of internal interfaces: Structural and quantitative analysis via high resolution STEM*", 13th Multinational Congress on Microscopy, Rovinj, Croatia, 24–29 Sep. 2017.

Fitzek, H.; Sattelkow, J.; Pölt, P.: "*Understanding surface enhanced Raman spectroscopy using accurate simulations of electric nearfields*" 7th ASEM Workshop of the Austrian Society of Electron Microscopy, Vienna, Austria, 20–21 Apr. 2017.

Fitzek, H.; Schmuck, K.; Burtscher, B.; Zajki-Zechmeister, K.; Zettl, P.; Pölt, P.: "*Student vs. spectrometer - comparing food differentiation by human taste buds and by Raman spectroscopy*" Advanced Materials Day 2017, Graz, Austria, 25 Sep. 2017.

Gspan, Ch.; Bucher, E.; Egger, A.; Schrödl, N.; Dienstleder, M.; Mitsche, S.; Sitte, W.; Grogger, W.: "*Investigation of domains in $Pr_2NiO_{4+d}$ by transmission electron microscopy*" 13th Multinational Congress on Microscopy, Rovinj, Croatia, 24–29 Sep. 2017

Haberfehlner, G.; Fisslthaler, E.; Jäger, W.; Kothleitner, G.: "*Measuring buried interface roughness by electron tomography*" 13th Multinational Congress on Microscopy, Rovinj, Croatia, 24–29 Sep. 2017.

Höfler, S. F., Hobisch, M., Haberfehlner, G., Hofer, F., Rath, T. ; Trimmel, G.: "*Indacenodithiophene-based small molecule acceptor breaking the 10% efficiency barrier in non-fullerene organic solar Cells*" 15th Austrian Photovoltaics Meeting, Vienna, Austria, 12–14 Nov. 2017.

Knez, D.; Lasserus, M. I.; Schnedlitz, M.; Fisslthaler, E.; Ernst, W. ; Hofer, F.: "*Thermal vs. beam induced dynamics in the TEM: In-situ experiments and simulation*" 13th Multinational Congress on Microscopy, Rovinj, Croatia, 24–29 Sep. 2017.

Knez, D.; Schnedlitz, M.; Lasserus, M. I.; Kothleitner, G.; Hauser, A.; Ernst, W. E.; Hofer, F.: "*In-situ studies of high-purity mono- and bimetallic nanostructures in experiment and simulation*" 7th ASEM Workshop of the Austrian Society of Electron Microscopy, Vienna, Austria, 20–21 Apr. 2017.

Knez, D.; Schnedlitz, M.; Lasserus, M. I.; Kothleitner, G.; Hauser, A.; Ernst, W. E.; Hofer, F.: "*In-situ studies of high-purity mono- and bimetallic nanostructures in experiment and simulation*" Microscopy Conference 2017, Lausanne, Switzerland, 21–25 Aug. 2017.

Knez, D.; Dienstleder, M.; Grogger, W.; Hofer, F.; Fisslthaler, E. "In situ *heating studies of diffusion barrier layers for semiconductor devices*" Microscopy Conference 2017, Lausanne, Switzerland, 21–25 Aug. 2017.

Konrad, L.; Albu, M.; Haberfehlner, G.; Hofer, F. "*Analytical microstructure characterization of advanced alloys and steels at atomic resolution*" 19th Plansee Seminar, Reutte, Austria, 29 May – 2 Jul. 2017.

Lackner, F., Messner, R., Schiffmann, A., Lasserus, M. I., Schnedlitz, M., Knez, D., Haberfehlner, G., Kothleitner, G., Hofer, F.; Ernst, W. E.: "*Gold doped helium nanodroplets: from atomic spectroscopy to localized surface plasmon resonances in deposited nanoparticles*", Conference on Quantum Fluid Clusters, Obergurgl, Austria, 7–9 Jun. 2017.

Lewis, B.B.; Mutunga, E.; Fowlkes, J.D.; Rack, P.D.; Plank, H.; Winkler, R.: "*Secondery Electron Emission during 3D Nanoscale Focused Electron Beam Induced Deposition*", Proc. 61st Int. Conf. on Electron, Ion, and Photon Beam Technology, and Nanofabrication (EIPBN), Orlando, USA, 30 May–2 Jun. 2017

Lewis, B.B.; Fowlkes, J.D.; Sang, X.; Pudasaini, P.; Mound, B.A.; Stanford, M.G.; Unoci, R.R.; Pharr, G.M.; Rack, P.D.; Winkler, R.; Plank, H.: "*In-situ Purification and Characterization of Direct-Write Nanostructures Fabricated using Electron Beam Induced Deposition*", Proc. 61st Int. Conf. on Electron, Ion, and Photon Beam Technology, and Nanofabrication (EIPBN), Orlando, USA, 30 May–2 Jun. 2017.

Mitsche, S.; Grasserbauer, J.; Pölt, P.: "*In-situ EBSD investigations of Al-Mg-Si-Alloy during heating*" 13th Multinational Congress on Microscopy, Rovinj, Croatia. 24–29 Sep. 2017.

Nachtnebel, M.; Mayrhofer, C.; Zankel, A.; Pölt, P.: "*Polymer fracture: What can the 3D reconstruction of crack regions tell us about their microscopic fracture mechanisms?*" 7th ASEM Workshop of the Austrian Society of Electron Microscopy, Vienna, Austria, 20–21 Apr. 2017.

Nachtnebel, M.; Mayrhofer, C.; Pölt, P.: "*Polymer fracture - 3D reconstructions as an analysis tool*" Microscopy Conference 2017, Lausanne, Switzerland, 21–25 Aug. 2017.

Nachtnebel, M.; Achtsnit, T.; Mertl, J.; Ettenberger, G.; Rattenberger, J.: "*Immunogold labelling of allergens at fine dust filter particles for SEM investigations*" 13th Multinational Congress on Microscopy, Rovinj, Croatia. 24–29 Sep. 2017.

Nachtnebel, M.; Pölt, P.: "*In-situ experiments in the ESEM – What can they tell us about the microstructure and behavior of polymeric microfiltration membranes?*" 13th Multinational Congress on Microscopy, Rovinj, Croatia, 24–29 Sep. 2017.

Orthacker, A.; Haberfehlner, G.; Tändl, J.; Poletti, M. C.; Sonderegger, B.; Kothleitner, G.: "*Investigation of the non-equilibrium formation of stoichiometric precipitates in multi-component aluminium alloys*", 7th ASEM Workshop of the Austrian Society of Electron Microscopy, Vienna, Austria, 20–21 Apr. 2017.

Rossner, Ch.; Glatter, O.; Vana, P.: "*The distance scaling behavior in planet-satellite nanostructures from gold nanoparticles and star polymers*", The International SAXS Symposium 2017, Graz, Austria,
26–27 Sep. 2017.

Sattelkow, J.; Fröch, J.; Plank, H.; Winkler, R.; Radeschnig, U.; Schwalb, C.; Strunz, T.; Fantner, E.J.; Stavrov, V.; Fantner, G.: "*Direct-write fabrication of electric and thermal high resolution nano-probes on self-sensing AFM cantilever*" European FIB Network, Graz, Austria. 4–5 Jul. 2017.

Schlemmer, W.; Krstulović, N.; Reishofer, D.; Zankel, A.; Trimmel, G.; Spirk, S.: "*Cold Plasma and Femtosecond Laser Treatment for the Modification of Cellulose based Thin Films*" 5th EPNOE International Polysaccharide Conference 2017, Jena, Germany, 20–24 Aug. 2017.

Wernitznig, S.; Zankel, A.; Rind, C.; Summerauer, S.; Nikolic, M. ; Sele, M.; Pölt, P.; Leitinger, G.: "*Reconstructing a looming sensitive pathway in locusts using serial block-face SEM*" 13th Multinational Congress on Microscopy, Rovinj, Croatia, 24–29 Sep. 2017.

# Poster Presentations

## 2018

Chokki, J; Darracq, G; Teychene, B; Pölt, P.; Wurtzer, S; Baron, J; Gallard, H; Joyeux, M.: "*Linking ultrafiltration membrane materials modification to filtration performances after chlorine exposure*", Euromembrane Conf. 2018, Valencia, Spain, 9–13 Jul. 2018.

Chokki, J; Darracq, G; Teychene, B; Pölt, P.; Baron, J ; Galland, H; Joyeux, M.: "*Ageing of PES/PVP membranes: Spatial characterization of degradation by IR microscopy*", Membrane Technology Conference & Exposition, West Palm Beach, Florida, USA, 12–16 Mar. 2018.

Fitzek, H. M.; Zankel, A.; Nachtnebel, M.; Schmidt, R.; Mayrhofer, C.; Schröttner, H.: "*Correlative scanning electron microscopy and Raman microscopy-Synergies*", 68th Annual Meeting of the Austrian Physical Society, Graz, Austria, 11–14 Sep. 2018.

Fitzek, Harald M.; Hinum-Wagner, J. W.; Mayrhofer, C.: "*Raman microscocpy of everyday items. What is stuff made of?*", Advanced Materials Day 2018, Graz, Austria, 21 Sep. 2018.

Knez, D.: "*Electron Beam Driven Dynamics in Experiment and Simulation*", 8th ASEM Workshop of the Austrian Society for Electron Microscopy 2018, Wien, Austria, 26–27 Apr. 2018.

Krisper, R.: "*In-situ TEM: The influence of IR radiation on EDXS at elevated sample temperatures*", 8th ASEM Workshop of the Austrian Society for Electron Microscopy, Vienna, Austria, 26–27 Apr. 2018.

Lammer, J.; Dienstleder, M.; Fisslthaler, E.; Rauch, S.; Grogger, W.: "*Oxide ceramic-electrode systems: STEM-EDXS analysis* *and heating experiments*", 68th Annual Meeting of the Austrian Physical Society, Graz, Austria, 11–14 Sep. 2018.

Lammer, J.; Dienstleder, M.; Fisslthaler, E.; Rauch, S.; Grogger, W.: "*Oxide ceramic-electrode systems: STEM-EDXS analysis and heating experiments*", ELMINA Conference on Electron Microscopy of Nanostructures, Belgrad, Serbia, 27–29 Sep. 2018.

Lammer, J.; Berger, C.; Bucher, E.; Grogger, W.: "*High resolution imaging and spectroscopy: Analysis of a n=2 Ruddlesden-Popper phase at the atomic scale*", Advanced Materials Day 2018, Graz, Austria, 21 Sep. 2018.

Moser, D.: "*Aluminium anode characterization of a novel aluminium-sulfur secondary battery*", 8th ASEM Workshop of the Austrian Society for Electron Microscopy, Vienna, Austria, 26–27 Apr. 2018.

Mutunga, E., Rack, P.D.; Fowlkes, J.D.; Plank, H.; Winkler, R.; : "*Fine-Tuning Nanowire Shape Using 3D Focused Electron Beam Induced Deposition*", Proc. 62nd Int. Conf. on Electron, Ion, and Photon Beam Technology, and Nanofabrication (EIPBN), Puerto Rico, USA, 29 May–1 Jun 2018.

Pabel, T.; Petkov, T.; Schröttner, H.; Albu, M.; Rossmann-Perner, A.; Schumacher P.; Li, J.: "*Effect of trace elements on microstructure and material properties of an aluminium alloy*", 73rd World Foundry Congress, Krakow, Poland, 23 – 27 Sept. 2018.

Padure, I.; Simic, S.; Zernig, K.; Heber, G. P.: "*seed and fruit morphology of the genus Hesperis L. (Brassicaceae) in Austria and their taxonomic value*", 24th Int. Symposium on Biodiversity and Evolutionary Biology, German Botanical Society, Klagenfurt, Austria, 19–22 Sep. 2018.

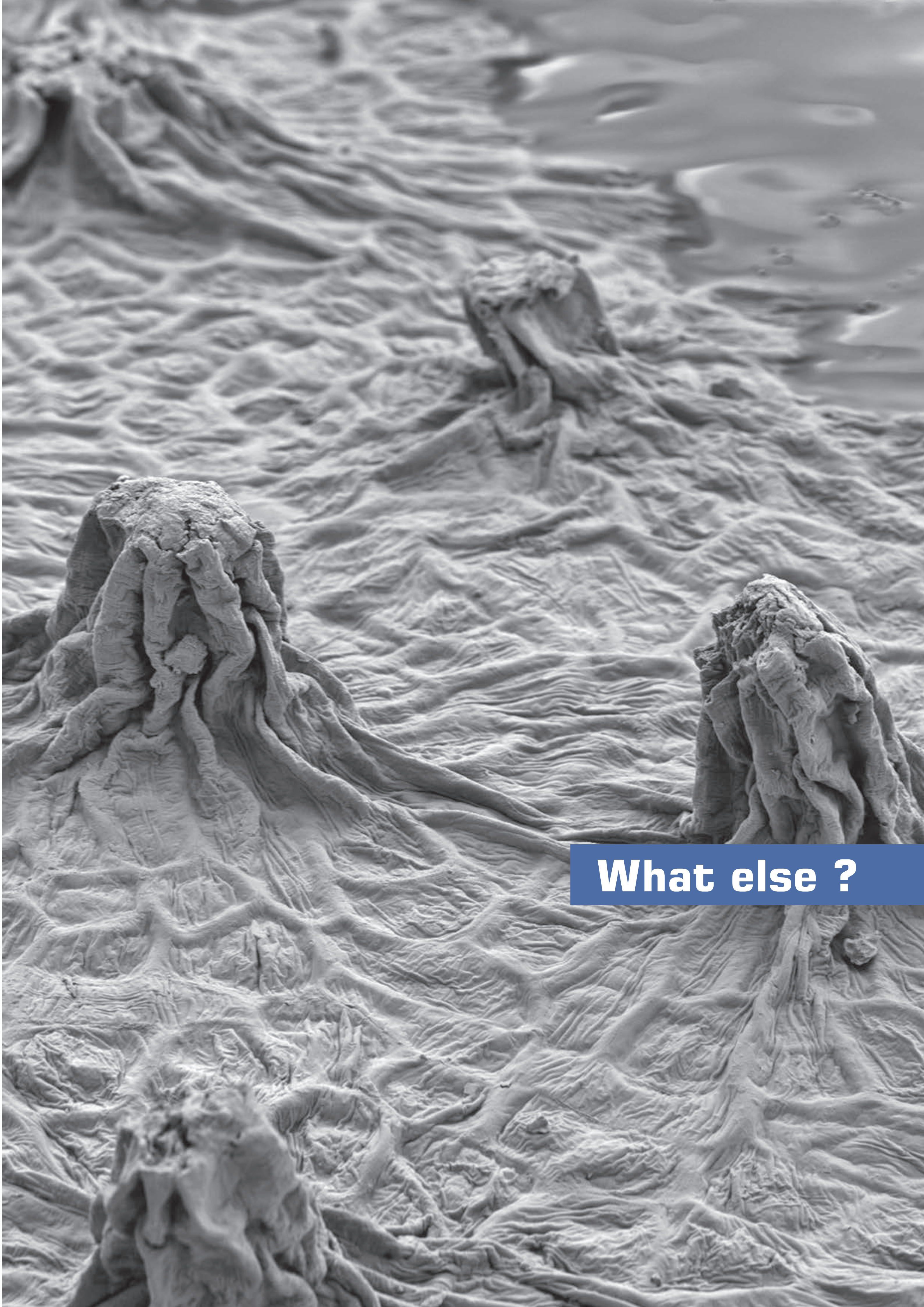
Trummer, C.: "*Tomographic analysis of FEBID structures*", 69th SCANDEM Congress 2018, Lyngby, Denmark, 25-27 Jun. 2018.

Trummer, C.; Winkler, R.; Plank, H.; Kothleitner, G.; Haberfehlner, G.: "Accessing the internal morphology of nano-granular FEBID materials in 3D space by electron tomography", 68th Annual Meeting of the Austrian Physical Society, Graz, Austria.

Trummer, C.: "*Tomographic analysis of FEBID structures*", 7th Int. Workshop on Focused Electron Beam-Induced Processing, Modena, Italy, 10–13 Jul. 2018.

Winkler, R.; Arnold, G.; Sattelkow, J.; Smith, Andrew; Plank, H.: "*A variable gas injection system for rapid precursor testing in focused electron beam induced deposition*", Conf. German Physical Society, Berlin, Germany, 11–16 Mar. 2018.

Zajki-Zechmeister, K.; Schlemmer, W.; Sattelkow, J.; Spirk, S.; Plank, H.: "*Dynamic in-situ observation of protein adhesion and chemical coupling using liquid atomic force microscopy*", 68th Annual Meeting of the Austrian Physical Society, Graz, Austria, 11–14 Sep. 2018.



# What else ?

# Organigram

## Austrian Centre for Electron Microscopy and Nanoanalysis

TU Graz

Faculty of Mathematics, Physics & Geodesy

FELMI-ZFE

ACR

Association for the Promotion of Electron Microscopy and Fine Structure Research

FELMI-ZFE

**Presidential Committee**
President: Prof. Helmut List
1. Vice President: DI Ulrich Santner
2. Vice President: Ing. Hans Höllwart

**Managing Committee**
Head: DI Ulrich Santner
Mag. Christian Knill
Prof. Horst Bischof
Dr. Jürgen Steinecker
Prof. Ferdinand Hofer

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Sabine Mitterbacher
Silke Winkler
Sabine Goger
Margit Wallner
Mira Plangger

**Techn. Laboratory**
Head:
**Prof. Ferdinand Hofer**
Ing. Albert Krisper
Gerhard Birnstingl
Markus Sittsam

**Management**
Prof. Ferdinand Hofer
Prof. Gerald Kothleitner

**Advisory Board**
Members from University and Industry

**Nanoanalysis in the TEM**
Head:
**Prof. Gerald Kothleitner**
Dr. Michaela Albu
Dr. Karin Wewerka
Dr. Georg Haberfehlner
Dr. Daniel Knez
Margit Brunegger
Martina Dienstleder
Ing. Claudia Mayrhofer
Arnela Blažević
Paul Fastian
DI Cornelia Trummer
Mag. Lukas Konrad
DI David Moser

**High Resolution Electron Microscopy**
Head:
**Prof. Werner Grogger**
Dr. Ilse Letofsky-Papst
Dr. Evelin Fisslthaler
DI Judith Lammer
DI Robert Krisper

**SEM Microanalysis & *in situ* Methods IR | Raman**
Head:
**Ing. Hartmuth Schröttner**
Dr. Stefan Mitsche
Dr. Armin Zankel
Dr. Manfred Nachtnebel
Dr. Johannes Rattenberger
DI Harald Fitzek
Sabrina Mertschnigg
Anita Rossmann-Perner
Sanja Šimić
Christian Brandl
Mag.rer.nat. Ruth Schmidt

**Functional Nanofabrication FIB | AFM**
Head:
**Dr. Harald Plank**
DI Robert Winkler
Sebastian Rauch
DI Jürgen Sattelkow
Mag. Anna Weitzer

Status: September 2018

# Masterstudents

## Master Theses in Progress


Eva Gasser


Jakob Hinum-Wagner


Daniel Hjelle


Michael Oberaigner

Robert Fabro

## Finished Master Theses

Michael Rastl


Cornelia Trummer


Andreas Postl


Tobias Achtsnit


Jakob Grasserbauer


Paul Falthansl

Johannes Fröch


Ulrich Radeschnig

Prof. Dr. Ferdinand Hofer

## Administration

Mag. Ulrike Stürzenbecher | Sabine Mitterbacher | Silke Winkler | Sabine Goger

## Media

Margit Wallner | Mag. Stefanie Gissing | Mira Plangger

## Techn. Laboratory

Albert Krisper | Gerhard Birnstingl | Markus Sittsam

## Scanning Electron Microscopy / IR / Raman

Hartmuth Schröttner | Dr. Stefan Mitsche | Dr. Armin Zankel | Dr. Johannes Rattenberger

1. Surface Analysis
2. Correlative Microscopy
3. ESEM & In-Situ-Microscopy

Dr. Manfred Nachtnebel | Dr. Harald Fitzek | Mag. Ruth Schmidt | Sanja Šimić | Sabrina Mertschnigg | Christian Brandl | Anita Rossmann-Perner

## Transmission Electron Microscopy

Prof. Dr. Gerald Kothleitner | Dr. Mihaela Albu | Dr. Karin Wewerka | Dr. Georg Haberfehlner | Dr. Daniel Knez | Dr. Lukas Konrad

## Phys./Chem. Laboratory

Ing. Claudia Mayrhofer | Martina Dienstleder | Margit Brunegger | Arnela Blacevic | Paul Fastian

## Transmission Electron Microscopy

Prof. Dr. Werner Grogger | Dr. Ilse Letofsky-Papst | Dr. Evelin Fisselthaler | DI Judith Lammer | DI Robert Krisper

## Functional Nanofabrication

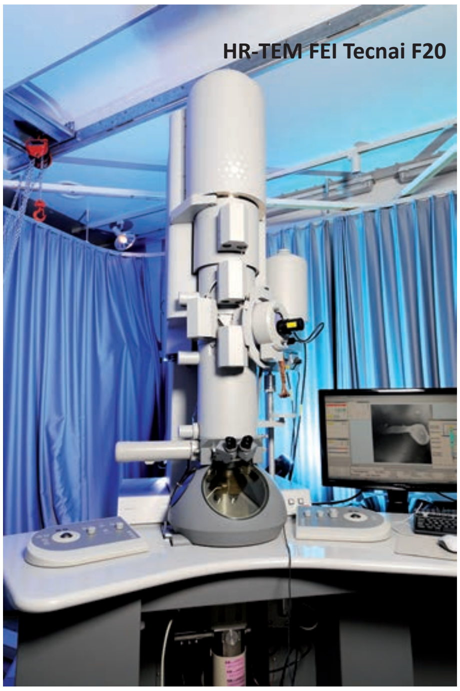
Ass. Prof.-Doz. Dr. Harald Plank | Sebastian Rauch | DI Anna Weitzer

Christian Doppler Forschungsgesellschaft

DEFINE

Dr. Robert Winkler | DI Jürgen Sattelkow


TEM Philips CM20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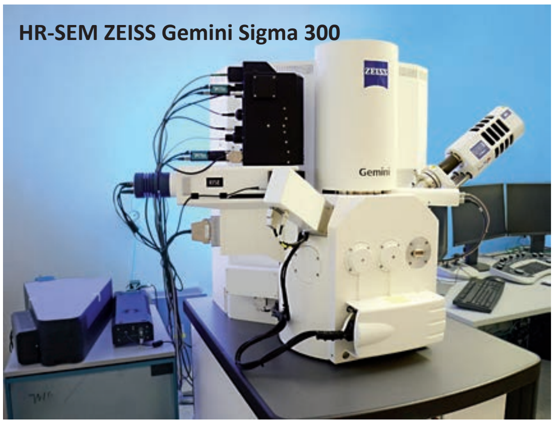
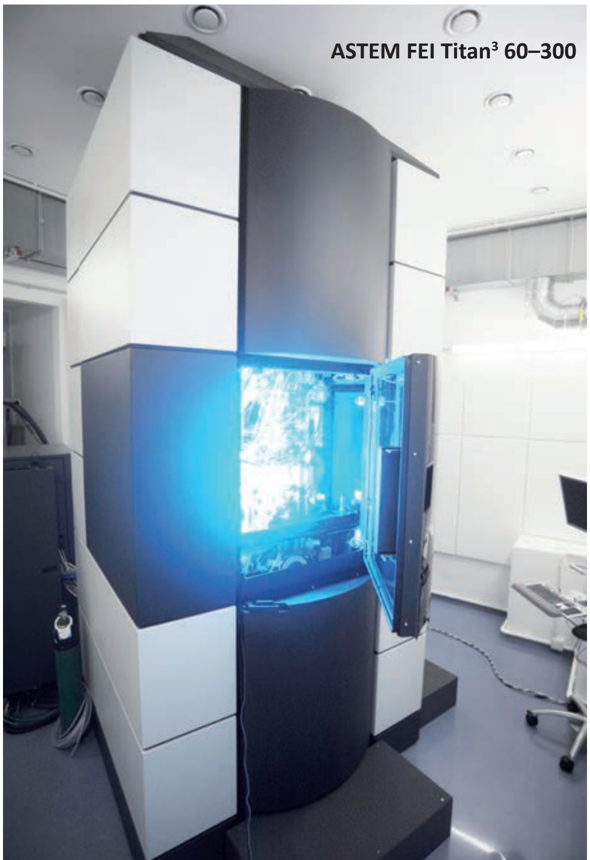

HR-SEM ZEISS Gemini Sigma 300


ESEM FEI Quanta 200


HR-SEM ZEISS Ultra 55

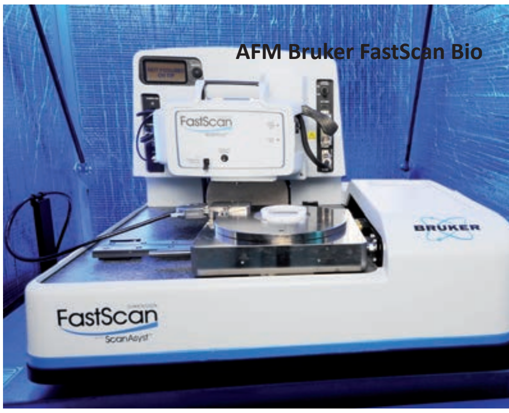

ASTEM FEI Titan$^3$ 60–300


Bio-TEM FEI Tecnai12

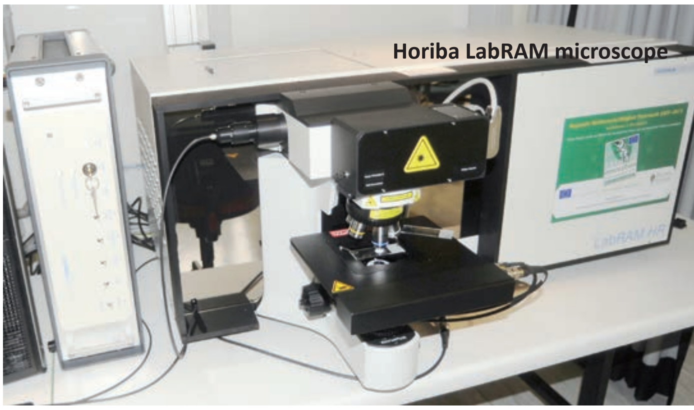

HR-SEM ZEISS Gemini DSM986

# Laboratory Facilities

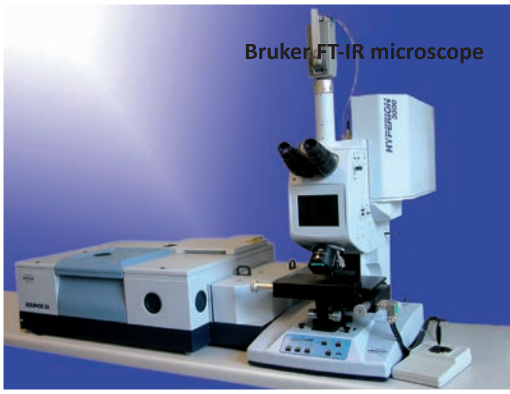
Cutting-edge in-house instrumentation is one of the keys to our success. Thanks to a broad range of different microscopes and the excellent preparation equipment our well trained staff is able to take top quality images, spectra and elemental maps. We house several scanning and transmission electron microscopes, as well as atomic force and light microscopes. The NanoMill and the Focused Ion Beam are amongst others crucial to sample preparation. In the past couple of years we invested even more in on the one hand new instrumentation (Zeiss Sigma 300 or our second FIB) and on the other in upgrades of already existing microscopes. For more information please check the pages New Instrumentation in the first chapter of this performance report!



AFM Bruker FastScan Bio


Horiba LabRAM microscope


Focused Ion Beam FEI Nova 200


Bruker FT-IR microscope

Siemens XRD D5005

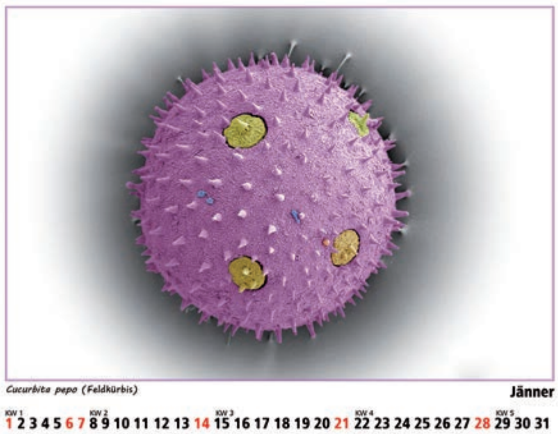

ESEM FEI Quanta 600

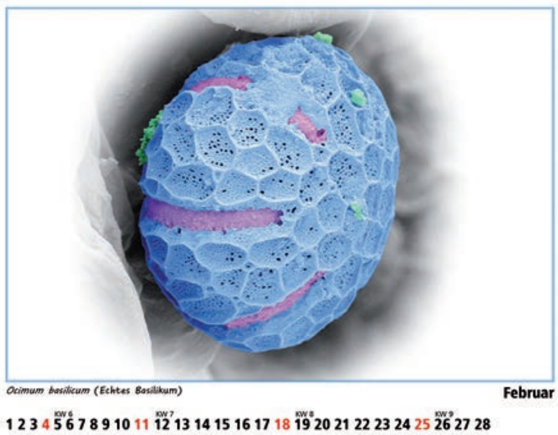

Fischione Nanomill 1040

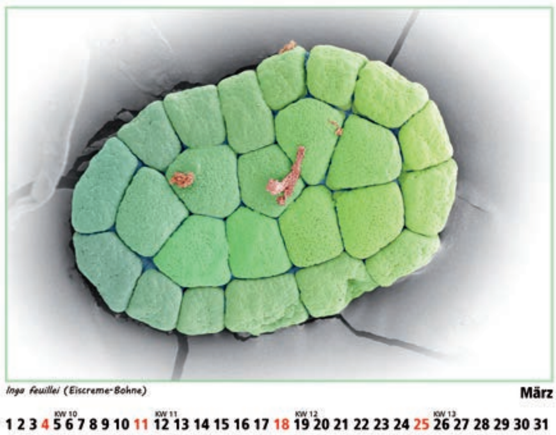
Alicona Infinite Focus G3

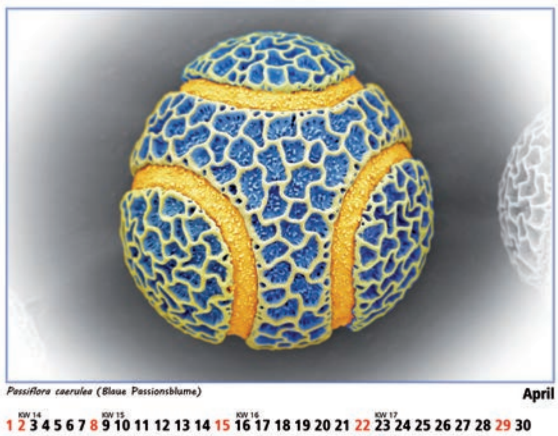
# FELMI-ZFE Calendar 2018

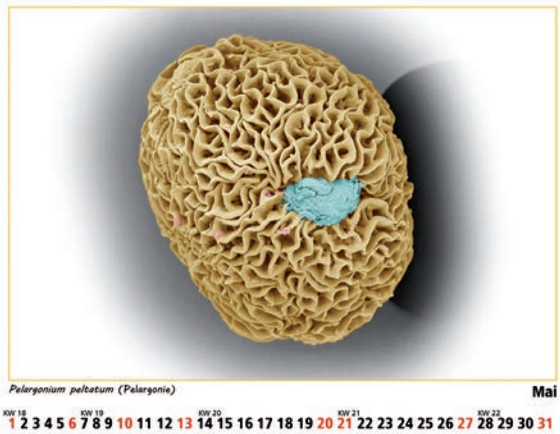

A pool of fascinating micrographs is generated via an internal image competition; the best micrographs are chosen once a year shortly before Christmas. The most interesting images are usually combined under a general theme and published in our biannual calendar. However, in 2018 we decided to focus on the beautiful world of pollen. It was tricky to find pollen which differ in their appearance.  This calendar was entirely compiled by our photographer Margit Wallner who realised the scanning microscopy images as well as the coulouring.

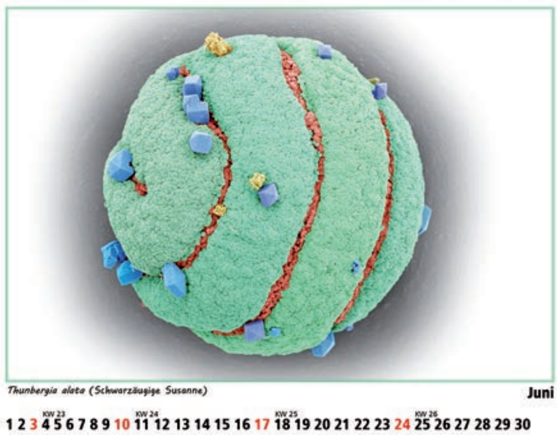


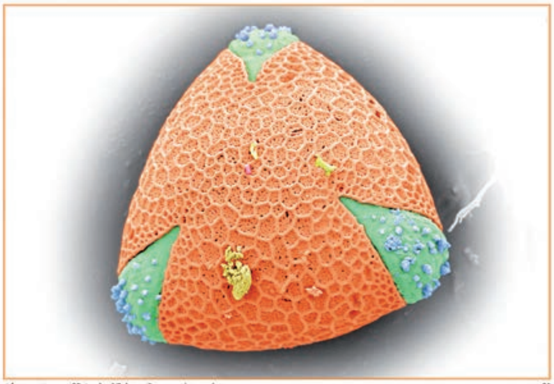



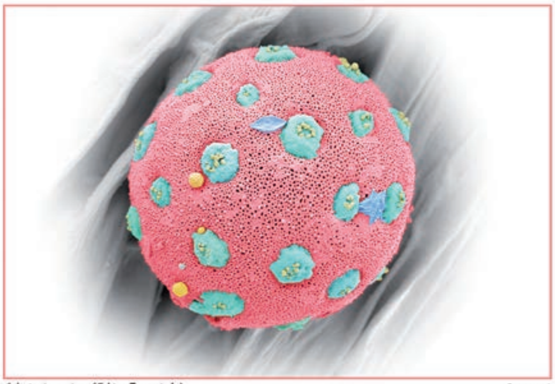





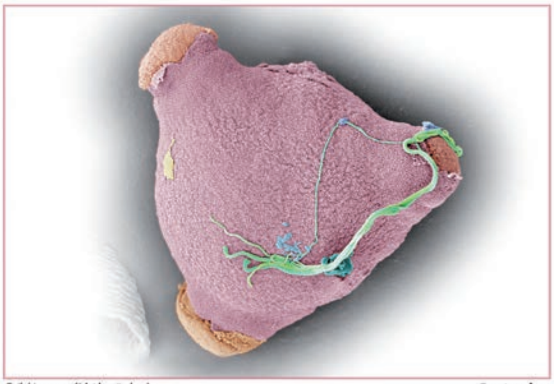
*Nasturtium officinale* (Echte Brunnenkresse) **Juli**

KW 27 KW 28 KW 29 KW 30
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31



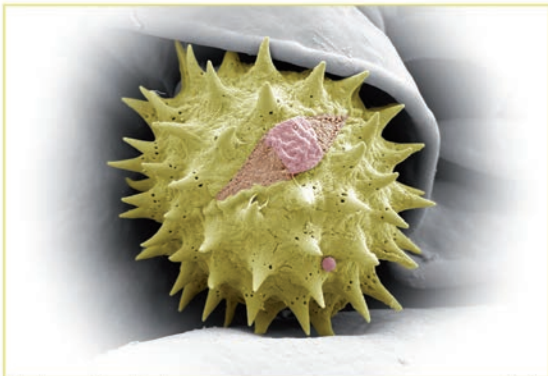
*Calystegia sepium* (Echte Zaunwinde) **August**

KW 31 KW 32 KW 33 KW 34 KW 35
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31



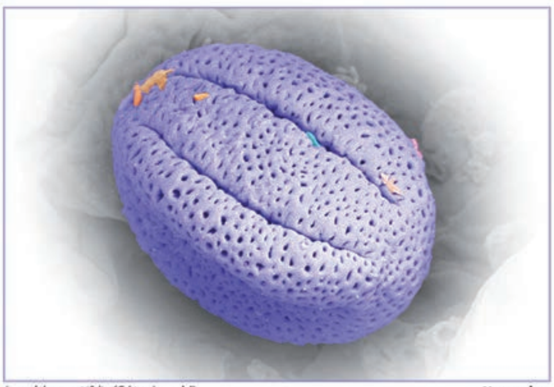
*Epilobium sp.* (Weidenröschen) **September**

KW 36 KW 37 KW 38 KW 39
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30



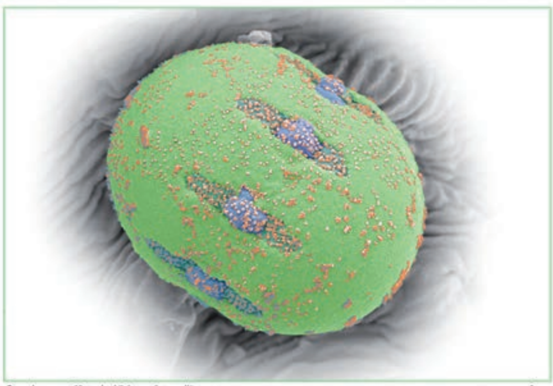
*Helianthus annuus* (Sonnenblume) **Oktober**

KW 40 KW 41 KW 42 KW 43 KW 44
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31



*Lavandula angustifolia* (Echter Lavendel) **November**

KW 45 KW 46 KW 47 KW 48
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30



*Symphytum officinale* (Echter Beinwell) **Dezember**

KW 49 KW 50 KW 51 KW 52
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31

# Peter Pölt Symposium

When one of the oldest colleagues goes into well-deserved retirement, there is a good reason to organise a farewell symposium to highlight his collaborations, research interests and professional development.

**Peter Pölt** studied Technical Physics at the TU Graz, Austria. He gained his PhD in 1980 at the Institute of Theoretical Physics at the TU Graz. Since 1980 he is working at the Institute of Electron Microscopy, Graz University of Technology, first as University assistant and subsequently as senior scientist. In the first years he was active in research and development of electrolytic capacitors in co-operation with international companies. Subsequently, he has been active in scanning electron microscopy, X-ray spectrometry, electron back-scatter diffraction, automated particle analysis in SEM, image processing, (ultra-)microhardness testing, tensile testing (polymers, fibres) and *in-situ* experiments in the ESEM. He was additionally active at the Graz Centre for Electron Microscopy and was group leader of one SEM group until 2017.

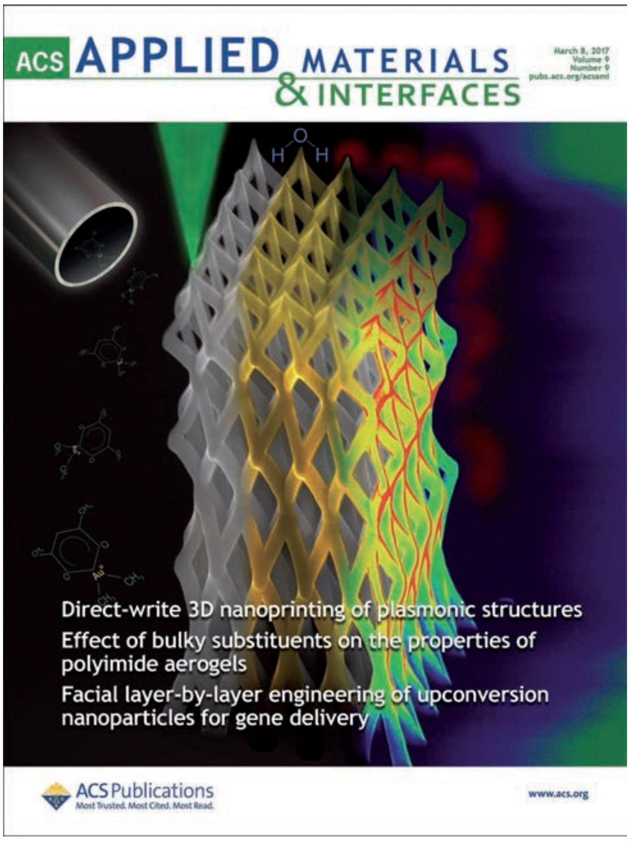
Hard work, dedication and positive attitude. These are only a few of the many precious things you've shared with us every day. Thank you and congratulations on your retirement!

# Cover Page Designed by Robert Winkler

The journal **ACS Applied Materials and Interfaces** chose Robert Winkler's contribution to decorate the cover of the March 2017 volume 9 / issue 9 and 10. ACS Applied Materials and Interfaces is a peer-reviewed scientific journal with an impact factor of 7.15 that was established in 2009 by the American Chemical Society. The journal covers amongst others advanced active and passive electronic/optical materials, colloids, biomaterials and bio-interfaces, polymers, hybrid and composite materials.

After presenting a computer aided design to fabricate complex 3D nanostructures via Focused Electron Beam Induced Deposition (FEBID) the authors demonstrated in their paper that these nanostructures are applicable for plasmonic applications. Complex freestanding architectures build out of compact and pure gold revealed strong plasmonic activity.

Read the paper: DOI: 10.1021/acsami.6b13062




# T³UG: Teens Tackle Technology 2017

Every year, around 100 girls between 16 and 18 years have the chance to experience science and technology at TU Graz during the holidays. The pupils are involved in everyday life at the institute, take part in course preparation and research activities.

In summer 2017, our trainee **Katarina Williams** 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 „T³UG Teens Tackle Technology" she gathered experience in the physical as well as chemical sample preparation and got some insight into image analysis and scanning as well as transmission electron microscopy.

FIT FRAUEN IN DIE TECHNIK STEIERMARK

# Exhibition
# Fungi: Food, Poison and Mythology

More than 1000 fungi were presented in the exhibition „Fungi: Food, Poison and Mythology“ by the Museum Wiesbaden from July 2017 to August 2018. But what does this have to do with electron microscopy? Armin Zankel realised the 3D reconstruction of five different spores by means of scanning electron microscopy. The microscopic reconstructions have been magnified and 3D printed.

**Mycology meets electron microscopy**

The German preparation experts Liselotte and Klaus Wechsler got in contact with us in order to get 3D reconstructions of spores. Since these „units of reproduction“ are very small a method using electron microscopy was chosen. Armin Zankel realized the 3D reconstruction of five different spores by means of „*in-situ* ultramicrotomy in the ESEM“ (Environmental Scanning Electron Microscope).

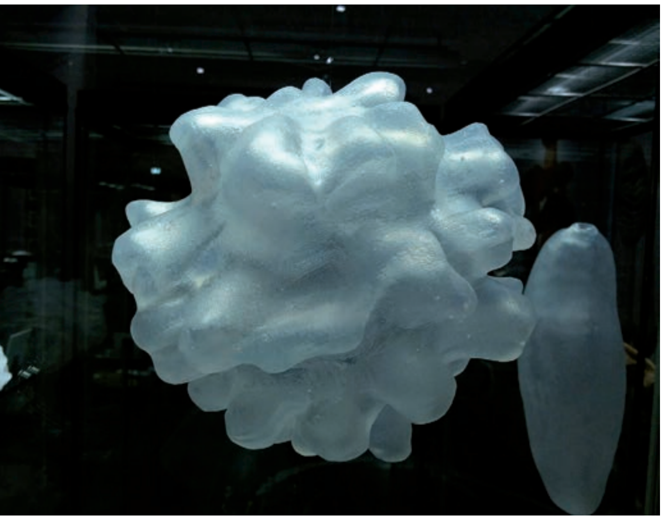
The microtome, which is mounted into the specimen chamber of the electron microscope (EM), cuts the sample slice by slice. After each slice an EM image is stored. As a result of the series of images a 3D reconstruction of a specimen can be performed. The samples in the present case were blocks of dried resin with embodied spores. As a result Klaus Wechsler produced 3D model of diameter of several decimeters. Since the spores have diameters in the range of some microns (e.g. 5 µm) and the models have a diameter in the range of decimeters this means a magnification of 20.000x.

**From the spore to the 3D model**

Embedding the spores in resin, drying of the resin, precutting of the block in the institutes‘ microtomy unit (Claudia Mayrhofer), mounting the specimens in the *in situ* ultramicrotome, alignment of the electron microscope and „slice and view“ of the samples, getting a stack of images, 3D reconstruction of the spores (Manfred Nachtnebel), exporting it in the data format for 3D printers, production of masters with the 3D printer, producing of casting mould with the help of the real 3D prints and finally producing several 3D models by filling in polymer into the mould. The steps after 3D printing were performed by the preparation expert Klaus Wechlser.

As a result several 3D representations of different scale can be found in the exhibition. Additionally some paintings on the wall of the museum are magnified images of spores performed with the scanning electron microscope ESEM in the low vacuum mode.

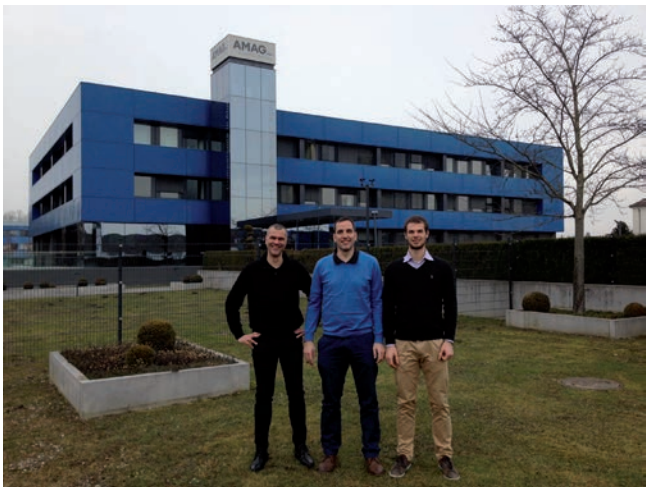
Museum Wiesbaden



# Industry & Networks

A highly motivated team and cutting-edge in-house instrumentation are simply not enough to keep up with developments happening within industrial sectors relevant to electron microscopy. To see the big picture, to foster our collaborations and to understand industrial needs better, we are member of different networks and organise workshops on the one hand and on the other hand, we try to visit companies and partners as often as possible.

The reason for a visit might be a master thesis (1) or another research project, a regular meeting combined with social activities (2), refreshing old ontacts (3) or a carefully planned trp to the south of Austria (4).





# Out of Office

## 2017

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.
Seeing each other every day means strong personal attachement and friendship. That is the reason why we have had after work parties with the prettiest cakes to celebrate birthdays and exams (1). For carneval 2017 a bunch of our guys turned into the electromagnetic spectrum of light (2). During the company outing we admired the beautiful medieval castle Riegersburg, a place blessed with a rich history and stunning views (3) and enjoyed Austrian delights and marvellous weather at the garden terrace of a *Buschenschank* (wine tavern) (4). The more active ones among us meet every Tuesday in summer to play beach volleyball (5). The last highlight of the year is always the Christmas Party, in 2017 we stayed at the Institute and decorated our rooms. We even installed a photo booth - lots of fun included! (6).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## 2018

To be continued in 2018: Carneval 2018 brought lots of funny creatures addicted to *Krapfen* (doughnuts) to our Institute (7). In spring the TU apprentices visited the Voestalpine AG in Leoben, an excursion organised by the TU works council (8). The company outing started with the visit of the ZIB (9), followed by interesting guided tours through our capital Vienna (10). 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, (11) Abschied Haingartner (12) Abschied Gspan (13)


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11



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