



PERFORMANCE REPORT 2019 | 2020









Performance Report 2019 | 2020

Institute of Electron Microscopy and Nanoanalysis Graz University of Technology

Graz Centre for Electron Microscopy ACR Austrian Cooperative Research















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Cover Image

Simulated convergent beam electron diffraction (CBED) pattern for a strontium titanate crystal with a thickness of 11 nanometers (front side) and 15 nanometers (back side), crystal orientation [001]. The electron beam is focused on the strontium atomic column in both cases (Michael Oberaigner).

Performance Report 2019-2020

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Rector of the TU Graz

The Institute of Electron Microscopy and Nanoanalysis (FELMI) is an important pillar of the Faculty of Mathematics, Physics and Geodesy, and together with the Center for Electron Microscopy (ZFE), it forms an internationally respected hub for advanced electron microscopy and nanofabrication. The broad width of new developments and the diverse applications of electron microscopy presented by the FELMI-ZFE research network in its biennial report is truly impressive.

Starting from fundamental physics, the Institute continuously advances into ever new exciting technology fields, ranging from the study of energy materials and 3D printed materials, to pharmaceutical research and life sciences, the development of completely novel nanosensors and even to the study of extra-terrestrial samples. With research deeply embedded in large national and European research contexts, an internationally highly visible recognition resulted that attracts numerous researchers to Graz for guest visits every year.

In 2020 the paths for the FELMI-ZFE have been freshly laid out. Gerald Kothleitner accepted a call by the university to become professor for electron microscopy. With him we gain an internationally renowned researcher and teacher, who will actively promote the further development of the institute.

I cordially invite you to have a look at the presented report to see for yourself, which achievements and innovative research activities have been accomplished by the colleagues from the FELMI-ZFE.



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

President of the Association

I am pleased to present the Institute's new performance report, which bears witness to the diverse and successful activities of the past two years. In this difficult time, when the pandemic situation is causing turbulence in many of the Institute's key markets, we have managed to stay on course and take new initiatives.

In June 2019, we were able to host a successful event celebrating the 60th anniversary of the Association for the Promotion of Electron Microscopy and Fine Structure Research. The Association was established in 1959 by leading Austrian industrial companies. Through all these years the Association has played a pioneering role in the development of electron microscopy in Graz and throughout Austria.

Here it is also my sad duty to announce that our long-time chairman of the board, Kommerzialrat Dipl.-Ing. Ulrich Santner, has passed away in February 2020. Ulrich Santner steered the fortunes of the Association with prudence and great commitment, for which I want to thank especially. And I would like to thank Ferdinand Hofer. He led the Institute very successfully for 21 years. And I wish his successor, newly appointed Professor of Electron Microscopy Gerald Kothleitner, all the best.

My sincere thanks go to all of the Institute's partners, to our Association committees and, above all, to the Institute's staff for their joint efforts to overcome the crisis situation. Grace to their boundless commitments, the institute doubtlessly will face a successful future.



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



Steps into the Future

The years 2019 and 2020 have brought incisive changes. Very sadly, the Chairman of the Board Ulrich Santner has passed away, necessitating organizational changes in the Association. Facing a pandemic situation, the ZFE has encountered several industrial collaborations that have been ceased by spring 2020. In addition, the retirement of the former head Ferdinand Hofer was at the door, leaving the FELMI-ZFE after many successful years. As often in such difficult situations, the stability and team spirit of an organisation shows up remarkably strong.

In the restituted board, Christian Knill took over the function of the Chairman and Stefan Rohringer further strengthened the Association, which remained in the proven hands of President Helmut List. Grace to the unbelievable cohesion of all staff members, the overall situation could be stabilized by the end of 2020. Finally, and in anticipation of Ferdinand Hofer's leave, the university in January 2019 has posted the position of a full professor for electron microscopy. Despite many renowned national and international applicants, early 2020, Gerald Kothleitner received the call by the Rector of TU Graz to become professor at the FELMI-ZFE, taking over the associated position of an institute head. Ever since, some organisational adjustments have been put on their way, while others are still ongoing. One of them being the establishment of a new work group, topically dealing with "Soft



Matter and Metals", which is headed by Dr. Ilse Letofsky-Papst.

Despite the complex circumstances, the institution was able to expand its scientific infrastructure by means of several funded research projects. That way, a new ESEM and a second FIB facility could be purchased and installed successfully. In yet another project called TIMELY, funded by the Austrian Cooperative Research and the Federal Ministry for Digital and Economic Affairs, a powerful solution for on-line access to electron microscopes was created. This infrastructure now allowed for cooperative investigations over large distances, just in time (!) to provide our research and services also throughout pandemic times.

The new institute management was also able to tackle another burning problem. Amongst the four transmission electron microscopes at the Institute, one is clearly outdated, while two others have become routine instruments and are no longer state-of-the-art. Even the ASTEM microscope, established in 2011, slowly comes into age, and despite still being Austria's most powerful electron microscope, it is not sufficient to meet the increased requirements on a European research level. Failure of this microscope would cause a total collapse of TEM research work, hitting hard also our industrial partners, who would no longer benefit from applied research nor receive services. Far-sightedly, the rectorate permitted an application to the FFG / Vienna for the funding of an ASTEM successor, titled ANGSTROEM. As this report is going to print, we have been informed that the project has been fully funded.

All the recent achievements and the flourishing of the institution would have been impossible without our hard-working colleagues at the FELMI-ZFE, who are especially thanked for their efforts. The success, however, has many names and we therefore would like to explicitly express thanks to the Rector of the Graz University of Technology, Harald Kainz, the Vice Rector of Research, Horst Bischof as well the Dean of the Faculty of Mathematics, Physics and Geodesy, Roland Würschum, for their continuous support. Our sincere gratitude also goes to Professor Helmut List, president of the Association for Electron Microscopy and Fine Structure Research.



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



Univ.-Prof. Dipl.-Ing. Dr.techn. Gerald Kothleitner Head of the Institute

The Institute

The Institute at a Glance

The research network Austrian Center for Electron Microscopy and Nanoanalysis is the leading Austrian institution in the field of advanced microscopy and nanofabrication. The Center is the result of close cooperation between two independent research institutes:





Institute of Electron Microscopy and Nanoanalysis (FELMI)

The FELMI is an institute of the Graz University of Technology and assigned to the Faculty of Mathematics, Physics and Geodesy (Dean: Univ.-Prof. Dr. Roland Würschum)

Tasks: Research and teaching in the field of microscopy, electron microscopy and nanofabrication; support of other institutes at the TU Graz and in the alliances NAWI Graz and BIOTECHMED Graz.

Having their own legal status and budget, both institutes work in close alliance to ensure efficient use of personnel and instrumental resources. The Austrian Center is organized in presently five groups, each dedicated to research topics in advanced microscopy and nanofabrication.

Graz Centre for Electron Microscopy (ZFE)

The ZFE works under the guidance of the Association for the Promotion of Electron Microscopy and Fine Structure Research, and is a member of the Austrian Cooperative Research (ACR) (President: Dipl.-Ing. Dr. Iris Filzwieser).

Tasks: Research in the field of electron microscopy and related methods; support of research institutions and industry with a focus on SMEs.



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

austrian cooperative research а cr ŢU Austrian Centre for Electron Microscopy and Nanoanalysis Functional Nanoanalysis **High Resolution** Soft Materials SEM and Metals **Microanalysis** Nanofabrication in the TEM TEM FIB and AFM in-situ Methode **IR and Raman** Hartmuth Gerald llse Harald Plank Werner Grogger Schröttner Kothleitner Letofsky-Papst Administration **Technical Laboratory** Media

Our Mission

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

Our Strengths

With a continuing tradition of excellence, the Institute bridges the gap between basic science and practical problem solving as well as industrial needs. The Institute works in close cooperation with university institutes and enterprises in Austria and Europe and increasingly worldwide. Our unique position is based on our leading-edge equipment and the expertise and ambition of our staff, thus making us the Austrian powerhouse of electron microscopy.



Quality Assurance

The Institute works under an advanced quality management system according to the rules of EN ISO 9001:2015. Our goal is to maintain and even improve the exceptional quality of our work and to optimize the organization and management. The certificate is aimed at the title "Research and teaching in the field of microstructure research and materials characterization by electron microscopy, micro- and nanoanalysis and the development of analysis and preparation methods". The Institute is certified by TÜV Austria.



Research Expert for SMEs

For 70 years, the Institute has supported numerous Austrian companies in their innovation efforts. In this way, the Institute contributes to strengthening the competitiveness of the Austrian economy with a focus on Austrian SMEs. Our research and development expertise and our network in academia and industry, strong and flexible research groups and expert knowledge of Austrian and European research funding are essential for our success.

Scientific Impact

FELMI-ZFE is facing a number of different tasks due to the fact that we are not only doing basic research but also scientific services for both university institutes and industry. Nevertheless, we could reach a high number of peer reviewed papers (116 in two years). This high number also comes from manifold collaborations with other research institutions. We are increasingly publishing in high impact journals such as Nature Materials, Nature, Nanoscale,

As expected, the number of talks at international conferences dropped sharply in 2020, but the number of peer-reviewed articles was maintained. All publications of the last two years are listed at the end of this report.



Scientific Output 2010-2020

Facts and Figures

Budget and Funding

The budget of the FELMI-ZFE is increasingly dependent on income from public research funding and contractual research with Austrian and European companies. Due to the pandemic situation, industry collaborations plummeted in the first half of 2020. However, a joint effort by all employees succeeded in reversing this trend in the second half of the year.

In the two years 2019 and 2020 we were very successful in project funding and therefore we were even able to make new investments for scientific infrastructure. We could purchase a fully equipped environmental scanning electron microscope (ESEM) (Thermo Fisher Quanta 450), a dual-beam focused ion beam microscope (FIB) (Thermo Fisher Quanta 3D) and an urgently needed streaming device (Kapsch & Cisco).



Teaching

During the reporting period, the number of courses has been kept constant, and the number of students in these courses has increased to an alltime high. The number of master's degrees has increased to ten students in two years, but only two students were able to complete their PhD studies.

Students in FELMI courses



Personnel

Over the last years, the Institute has developed into an important focal point for microscopy research in Austria with around 50 employees including PhD students (head count). The percentage of women employees is 43%. The number of PhDs and master students is continuously increasing, and this is already leading to major space problems. In 2019 and 2020, we had 17 master students and among them four female students.

Staff of December 2020	Headcount
Professors	
Senior Scientists	
Junior Scientists / PhDs	
Technical Staff	
Adminstrative Staff	
Master Students	
Apprentices	

blue = FELMI (TU) red = ZFE

Austrian Cooperative Research (ACR)

Since 1959, the Association has been a proud member of the ACR organization, which has set itself the goal of strengthening the innovative power of small and medium-sized enterprises in Austria. The ACR supports these companies as outsourced development departments that can be called upon as needed. The ACR's USP lies in its threefold bridging function: from science to industry, from leading companies to SMEs and from the international to the Austrian innovation system.

Presently, the ACR has 17 non-university cooperative research institutes with 62.2 million euro overall turnover and 708 employees. The institutes conduct applied research, development and innovation primarily for SMEs.

The Association (and the ZFE institute) cooperates directly with the following ACR institutes:

- Austrian Forest Products Research Society (HFA), Vienna
- Austrian Foundry Institute (ÖGI), Leoben
- Austrian Institute for SME Research (KMFA), Vienna
- Austrian Research Institute for Chemistry and Technology (OFI), Vienna
- Vereinigung der österreichischen Zementindustrie (VÖZ), Vienna
- V-Research, Dornbirn

The Association (ZFE) benefits greatly from the cooperation with the ACR group, for example via special research funding coming from the Federal Ministry of Digital and Economic Affairs.

The ZFE coordinates two strategic ACR projects in which other ACR institutes are also involved:

- 3D PRINT: Microstructure of 3D printed metallic components with the ÖGI
- TIMELY: Multimedia based on-line microscopy with the KMFA, ÖGI and V-Research

In 2019 and 2020, the ZFE cooperated in the following strategic projects led by other ACR institutes:

- TRIOP: Tribology of polymers with V-Research and OFI
- Dust Analysis in interior air with HFA
- KorroNet with ÖGI
- SERIFE-3D with OFI
- Chlorid corrosion with VÖZ

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



The new ACR management

ACR Director Dr. Sonja Sheikh (left) and ACR President Dipl.-Ing. Dr. Iris Filzwieser (right) (Copyright: ACR/Uwe Strasser).



National/International Impact

Every year we cooperate with about 30 university institutes and more than 100 companies, mainly located in Austria, but to an increasing extent also from other European countries.

The Institute pursued the following strategy during the last years: On the one hand, joint research projects in the ACR group and with local universities have been pushed forward, and on the other hand, the focus has been more strongly directed towards collaboration in large-scale European research projects or networks, such as the ESTEEM consortium. ESTEEM is a special cornerstone in our international relations: With this project we can open up access to our expertise to European scientists and also establish close links with the leading electron microscopy institutes. You will find more information about ESTEEM in the chapter Research Projects on page 53).

The collaborations of the Institute are presented in overview on the following pages. At the TU Graz and in the NAWI and BIOTECHMED alliance, 17 collaborations were active in the last two years, which are described in detail below.

A large proportion of our collaborations result in scientific publications in peer-reviewed journals. The co-authors of these papers are employed at numerous foreign research institutions, whose location has been marked with a red dot in the following maps. The members of the Association and the project partners are marked with blue and green dots, respectively.





Joint peer-reviewed papers with the following institutes in Austria:

Institutes from TU Graz, University of Graz and Medical University of Graz (see page 14)

Montan University Leoben

Chair of Physical Chemistry Chair of Functional Materials and Materials Systems Chair of Nonferrous Metallurgy Chair of Geology and Economic Geology

University of Innsbruck

Institute of General and Inorganic Chemistry Institute of Mechatronics Institute of Zoology

University of Linz

Institute of Semiconductor and Solid State Physics

Center for Surface and Nanoanalytics

University of Vienna

Department of Biophysical Chemistry

Vienna University of Technology, Vienna USTEM University Service Center for

Transmission Electron Microscopy

ISTA Institute of Science and Technology Austria, Klosterneuburg

V-Research, Dornbirn

- Voestalpine, Kapfenberg
- Materials Center Leoben (MCL)
- Amag Rolling, Ranshofen
- Metallwerk Plansee, Reutte

GETec, Vienna

17



Joint peer-reviewed papers with institutes in Europe and worldwide

University of Maribor, Slovenia Faculty of Mechanical Engineering Institute of Technology Materials University of Zagreb, Croatia Department of Biology Faculty of Pharmacy and Biochemistry Hungarian Academy of Sciences, Budapest, Hungary, Center for Energy Research Politecnico di Milano, Milano, Italy **Department of Mechanics** CNR Istituto di Officina dei Materiali, Trieste, Italy CNR Institute of Condensed Matter Chemistry and Technologies for Energy, Padova, Italy Goethe University, Frankfurt am Main, Germany Physical Institute University of Münster, Germany Institute of Inorganic and Analytical Chemistry Max Planck Institute for Solid State Research, Stuttgart, Germany, Physical Chemistry of Solids University of Bremen, Bremen, Germany Institute of Applied and Physical Chemistry University of Kiel, Germany Institute of Materials Science Helmholtz Center for Materials and Energy, Berlin, Germany University of Zaragoza, Spain

Institute of Nanosciences of Aragon

Aristotle University, Thessaloniki, Greece **Department of Solid State Physics** Masaryk University, Brno, Czech Republic Department of Condensed Matter Physics CEITEC Brno University of Technology, Brno, **Czech Republic** University of Poitiers, Poitiers, France Institute of Environmental Chemistry Karlstad University, Karlstad, Sweden Department of Engineering and Physics ETH Zürich, Switzerland Department of Information Technology and **Electrical Engineering** Porsche AG, Stuttgart, Germany Sabancy University, Istanbul, Turkey Integrated Manufacturing Technologies Res. University of Newcastle, Newcastle, Australia School of Information and Physical Sciences International Islamic University, Islamabad, Pakistan, Department of Physics Ural Branch Russian Academy of Sciences, Ekaterinburg, Russia University of Tennessee, Knoxville, USA Materials Science and Engineering

Oak Ridge National Laboratory, Oak Ridge, USA Center for Nanophase Materials Sciences

FELMI-ZFE Collaboration Partners in ESTEEM3

- Stuttgart Center for Electron Microscopy, Max Planck Institute for Solid State Research, Stuttgart, Germany
- Ernst-Ruska-Centre for Microscopy and Spect roscopy with Electrons, Research Center Jülich, Germany
- Centre d'Elaboration des Matériaux et d'Etudes Structurales, CEMES-CNRS, Toulouse, France
- Laboratoire de Physique des Solides, CNRS-LPS, Orsay, France
- Electron Microscopy for Materials Science (EMAT), University of Antwerp, Belgium

Department of Materials, University Oxford, U.K.

Department of Materials Science & Engineering, University of Cambridge, U.K.

- Department for Nanostructured Materials, Jožef Stefan Institute, Ljubljana, Slovenia
- Advanced Materials Laboratory, University of Zaragoza, Spain
- Structure and Chemistry of Nanomaterials, Uni versity of Cádiz, Spain
- International Centre for Electron Microscopy for Materials Science, AGH University of Science and Technology, Krakow, Poland
- Materials Analysis Laboratory, Chalmers Universi ty of Technology, Sweden
- Department of Physics, Norwegian University of Science and Technology, Trondheim, Norway
- Institute for Microelectronic and Microsystems, National Research Council, Catania, Italy

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 local and international universities and industry.

Professor Helmut List is the president of the Association which has currently 33 members mainly from Austria.

Presidential Committee

President: Prof. Dipl.-Ing. Dr-Ing.h.c. Helmut LIST 1. Vice president: Ing. Hans HÖLLWART 2. Vice president: Mag. Christian KNILL

Managing Committee

Head: Mag. Christian KNILL Vice-Head: DI Stefan ROHRINGER Financial referee: Dr. Andreas FÖSSL Representative of the Styrian Universities: O.Univ.-Prof. DI Dr. Horst BISCHOF Head of ZFE:

Ao.Univ.-Prof. DI Dr. Ferdinand HOFER

Ing. Bernd **KRAUS** Gatan GmbH

Prof. Dr. Emil LIST-KRATOCHVIL Humboldt-Universität zu Berlin

DI Dr. Rainer MINIXHOFER ams AG, Premstätten

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> Dipl.-Ing. Christian **RAINER** Omya GmbH, Gummern

DI Dr. Stefan **SCHERER** Alicona Imaging GmbH, Grambach DI Gerhard **SCHINDELBACHER** ÖGI, Leoben

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Microscopy and Fine Structure Research

Members



Our Networks



60 Years Association for Electron Mid

Back in the year 1949, inspired by a suggestion of Rector Bernhard Baule, the senate of the former "Technische Hochschule Graz" made a trend setting and far-reaching decision: In order to implement a research unit for electron microscopy ("Forschungsstelle") and its application in the natural and technical sciences, a bright scientist, Dr. Fritz Grasenick was hired and the first electron microscope was installed in Styria in 1951.

Due to the limited resources of the Austrian Universities in the fifties of the last century, Fritz Grasenick rapidly realized that the research unit had to generate additional income through industrial collaborations. The rapidly increasing interest and the rising demand in electron microscopic investigations soon made it necessary to expand both in personel and in equipment. For this purpose, Dipl.-Ing. J. Fränzel from the Ebenseer Solvaywerke brought up the idea to create a special foundation for supporting the research unit.

After careful consideration of all pros and cons and the impact of such a foundation for the universities in Styria, Professor Hanns Koren from the Provincial Government of Styria finally chaired the inaugural meeting on June 24th, 1959. Josef Krainer, the Provincial Governor of Styria, was elected the first president of the "Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung". Important industrial and public institutions soon joined the Association: Böhler, Ebenseer Solvaywerke, Plansee, Semperit, Veitscher Magnesitwerke, the Chamber of Commerce to name just a few. Furthermore, the Association on also installed an advisory board consisting of members both from academia and industry. This board should represent the Association in front of the Styrian Universities, sense new developments and implement necessary changes and infrastructure.

The foundation established the "Zentrum für Elektronenmikroskopie Graz (ZFE)" with the status of a private industrial non-profit organisation. The close connection with the "Technische Hochschule Graz" is documented by the fact that it was conducted by one director only, Dr. Fritz Grasenick, who primarily had to act as a mediator between scientific research and industrial needs.

One of the ZFE's main efforts was to steadily advance the overall scientific infrastructure while carefully coordinating between all interested parties. A special success of all these efforts was the financing of a new building via ERP credits which allowed the ZFE and the "Forschungsstelle" to move into the new building Steyrergasse 17 in 1965. Up until 1983 most of the research infrastructure was financed through the ZFE and the Association solely. Since then, electron microscopes have mostly been built through the mixed funding model, with the Association and the Graz University of Technology jointly raising funds.

Due to the close cooperation of the Association with the TU Graz, the international competitiveness in the field of electron microscopy can be secured for the FELMI-ZFE also in the future. More information about the Association is available on page 18 of this report.



croscopy and Fine Structure Research



Foundation assembly of the Association ("Verein") on June 24th,1959.

Presidents of the Verein zur Förderung der Elektronenmikroskopie



1959 – 1971 Governor of Styria Josef Krainer sen.



1971 – 1986 Governor of Styria Dr. Friedrich Niederl



1986 – 1996 Dr. Friedrich Pfohl



1996 – Prof. DI Dr.-Ing. h.c. Helmut List



10 Years Austrian Scanning Transmission Electron Microsope (ASTEM)

In 2011, after years of effort, a high-resolution transmission electron microscope with a spherical aberration corrector was installed at the Institute. This step was of extreme importance because it allowed us to perform electron microscopic studies with atomic resolution – for the first time in Austria. The microscope also contributed significantly to the international positioning of the Institute, as it gave us access to the first league of electron microscopy in Europe – to the ESTEEM consortium.

The ASTEM is a FEI Titan cubed (60-300) TEM-STEM system, which was set up by FEI Company (Eindhoven, Netherlands). It is equipped with an extremely bright electron source, a monochromator, an excellent objective lens and a variety of detectors. But the central element of the ASTEM is the spherical aberration corrector, with which electron microscopic investigations with atomic



resolution can be carried out. This was not directly possible with conventional TEMs before. The corrector is a high-tech "spectacle", which was originally developed in the 1990s by Harald Rose, Max Haider and Knut Urban in Germany.

The main application of ASTEM is in structural studies of crystalline materials with atomic resolution. Mostly bright and dark field images are recorded, which provide information about the local arrangement of the atoms in the crystal lattice. For example, the high-angle dark field detector (HAADF) allows the acquisition of images in which the contrast can be attributed to the changes in the average atomic number in the individual atomic columns.

Since 2018, we also have a differential phase contrast detector (DPC), with which we can measure local electric and magnetic fields in nanostructured samples. Furthermore, light element mapping at atomic resolution is brought to a new level ustilizing this detector.

The ASTEM is also equipped with excellent spectroscopic methods, with a 4-quadrant X-ray detector (FEI Super-X) and with an imaging filter/ spectrometer (Gatan Quantum ERS), recently upgraded with a direct electron detection camera (Gatan K2).

In order to fully exploit all the advantages of this microscope, we also had to intensively deal with new methodological developments. Thus, the quality of the sample preparation had to be significantly improved (Martina Dienstleder), completely new evaluation methods had to be introduced, and the experimental data had to be interpreted with the help of theoretical modelling. Of course, this was only possible with a large team of dedicated colleagues.

Only a few outstanding successes can be briefly mentioned here: Introduction of a new method for quantitative elemental analysis with atomic resolution (Gerald Kothleitner), further development of electron tomography in previously unknown quality (Georg Haberfehlner) (Fig. 1), improved imaging of surface plasmons by electron energy-loss spectroscopy and even cathodoluminescence (Franz Schmidt, Ferdinand Hofer) and the introduction of molecular dynamics modelling and image simulation techniques by Christian Gspan and Daniel Knez.

Within a few years, the microscope became a central hub for tackling numerous scientific problems in physics, chemistry and the materials sciences. Here again, we can only cite a few examples: Analysis of defects in semiconductor devices, secondary phases in light metal alloys and in steels, nanoparticles and metallic clusters, nanostructures in 3D-printed materials, interfaces in solar cells and battery materials, dopants in photocatalysts and porous materials (Mihaela Albu, Evelin Fisslthaler, Werner Grogger, Lukas Konrad, Johanna Kraxner, Robert Krisper, Judith Lammer, Arno Meingast, Angelina Orthacker et al.) (Fig. 2). All these efforts have led to numerous national and international research collaborations with universities and industry, culminating in 189 publications by the end of 2020 that include results from the ASTEM (see page 24).



Fig. 1 3D imaging of plasmons by EELS tomography. 3D reconstruction of the photonic LDOS of a silver nanoparticle dimer, (a) low-energy (bonding) mode and (b) high-energy (antibonding) mode (Georg Haberfehlner).



Fig. 2 EELS elemental maps of the $La_{1.9}Ba_{1.1}Fe_2O_7$ Ruddelsden-Popper phase 2^{nd} order, (a) annular dark field image with the atomically resolved elemental maps for iron, lanthanum, barium and oxygen, (b) RGB image with the corresponding EEL spectra (Judith Lammer).

Nanofabrication - From a New Instrument Towards 3D Nanoprinting

In 2003, one of the first European Dual Beam Microscopes (DBM) was installed at the FELMI-ZFE, which started a new era with respect to site specific sample preparation for transmission electron microscopy lamellas, sub-surface inspection and analytical 3D material reconstruction. Shortly after, Michael Rogers started to explore the aspect of functional nanofabrication via subtractive and additive micro- and nanofabrication via the DBMs focused ion/electron beam capabilities.

In 2007, Harald Plank joined the institute with particular focus on focused electron beam induced deposition (FEBID), which led to the dedicated workgroup Functional Nanofabrication in 2010. The research strategy initially focused on the fundamental FEBID processes, including resolution limitations, the minimization of unwanted side effects and the controlled transfer of notoriously carbon-containing FEBID materials into high quality metals. That growing toolbox then enabled the demonstration of sensors, nano-probes and MEMS/NEMS concepts.

In 2015, first at-tempts into real 3D nanofabrication were done, which matured in the following years. Based on the fundamental understanding of growth mechanisms in 3D space, an upfront design software was introduced, which allows reliable transfer of CAD models into real 3D nanoarchitectures.

The vast progress attracted the attention of industry, which led to the Christian Doppler Laboratory for Direct-Write Fabrication of 3D Nanoprobes (DEFINE) in 2018 together with the companies GE-Tec Microscopy (Vienna) and Anton Paar (Graz). Since then, this laboratory focuses on radically new concepts in the area of 3D nanoprobes for atomic force microscopy (AFM) using this emerging 3D nanotechnology, which led to a first commercially available product in 2019, while further concepts are currently in the transfer phase. The entire activities were conducted in collaboration with academic and industrial partners in Austria, Germany, US, Switzerland and Spain. So far, the activities led to multiple projects at FELMI-ZFE with a total funding of > 3 M€, produced > 60 peer reviewed articles, > 130 conference contributions (30 invited/plenary/key-note talks), 5 bachelor-, 18 master-, and 5 PhD theses. Till 2020, the workgroup was awarded by 10 scientific awards, including the prestigious Houska Award for Academic Research in 2020.

By that, FELMI-ZFE has gained world-wide leading position in the area of additive direct-write manufacturing of 3D nanostructures. The future orientation is focusing on advanced 3D nanoscale applications in research and development, based on the continuously growing capabilities of 3D nanoprinting via focused electron beams.



FEBID relies on the highly localized decomposition of surface adsorbed precursor molecules by the focused electron beam, which immobilizes the material according to the beam movement. When applying slow lateral beam velocities, small nano-deposits are growing slightly displaced, which finally enables the fabrication of freestanding 3D architectures with single features down to the sub-20 nm regime. As the beam can be controlled very precisely, complex structures can be fabricated in a single-step on many materials (vacuum and certain e-beam compatibility) and almost any surface morphology. That expands classical nanofabrication methods, which often are limited to flat surfaces, and even enables the nanofabrication on highly irregular surfaces or prefinished devices.

Correlative Microscopy

To develop a comprehensive understanding of the microstructure of a sample, it is often necessary to combine several microscopic methods or contrast techniques. A typical example is the linking of light and electron microscopy, where the advantages of light, such as color information, can be combined with the advantages of electron microscopy, such as better resolution.

A few years ago, colleagues from the institute started to develop new methods of correlative microscopy and new quantitative evaluation methods for the multimodal image data. An early example is the direct correlation of atomic force microscopy (AFM) images with transmission electron microscopy (TEM) images (Nadejda Matsko, 2012). The innovative approach led to the development of a unique cryo-AFM system that was directly integrated into the cryo chamber of an ultramicrotome. Using this method, the surface of the sample (block face) is directly scanned with the AFM tip after sectioning and the ultramicrotome cut is used for TEM examination (see Fig. 1). The combination of these methods enables the analysis of soft and frozen-hydrated materials immediately after sectioning under room temperature or cryo conditions.

But we were also able to provide decisive impulses for the correlation of SEM and AFM in cooperation with the company GETec in Vienna: Harald Plank's group installed the AFSEM system from GETec into our dual-beam focused ion beam instrument (FIB), allowing us to immediately investigate samples with high lateral resolution and accurately measure heights, distances and even materials properties.

Fig. 1 Correlative AFM and TEM microscopy investigation of tissue of Caenorhabditis elegans with the (a) cryo AFM phase (block face) and (b) TEM (ultrathin section) images (N. Matsko). In 2017, we were able to set up the first RISE system in the world, (RISE: Raman imaging and scanning electron microscopy, Zeiss and Witec, Germany) which was additionally equipped with a high-performance X-ray detector (silicon drift detector from Oxford). The combination of highresolution imaging, elemental analysis and chemical information opened up completely new possibilities for the investigation of complex structures. With this system, we have already been able to solve several interesting research questions, such as the investigation of different polymer types and composites (Ruth Schmidt, Armin Zankel), the analysis of nano-plastics in foodstuffs and tissue (Ruth Schmidt, Harald Fitzek) and, more recently, the investigation of the microstructural composition of extra-terrestrial materials such as the Chelyabinsk meteorite (Harald Fitzek, Armin Zankel) (Fig.2).



Fig. 2 Correlative microscopy study of the microstructure of the Chelyabinsk meteorite: the Raman map which is overlaid on the backscattered electron image reveals the chemical phases (Harald Fitzek).



Microscopy & Sustainability

The FELMI-ZFE is committed to making important contributions to the Sustainability Development Goals (SDG) of the United Nations. In recent years, we have increasingly focused our research on sustainable aspects and contributed to the development of energy materials and devices.

In energy research, complex material systems consisting of different chemical phases on the nanometer scale are increasingly being used. Their properties are determined not only by the local chemical composition, but also by the morphology and crystallinity of these phases and their interfaces. In all these areas, advanced electron microscopy is urgently needed to increase the efficiencies of solar cells, batteries and fuel cells. Here we present a brief summary of our activities to achieve sustainability goals:

SDG 6 Clean Water and Sanitation

Pores in microfiltration membranes

Flat sheet micromembranes are widely used for waste water treatment. However, problems with membrane degradation may occur which leads to a gradual reduction in performance. Here, Herbert Reingruber and Armin Zankel achieved a major breakthrough with the study of the 3D structure of porous polymer membranes. They developed a new in-situ method for membrane studies by using serial sectioning with an ultramicrotome directly installated in an environmental scanning electron microscope (ESEM). The method allowed the quantitative characterization of the pore structure in membranes by 3D reconstructions. Additionally, in-situ wetting and drying helped to solve several issues connected to membrane degradation.

SDG 7 Affordable and Clean Energy

Increasing the efficiency of solar cells

Classic inorganic solar cells dominate the market today, but there are numerous application fields where organic and inorganic/organic hybrid solar cells are also important. This is due to their flexibility and simpler and cheaper production. Gregor Trimmel from the Institute of Chemistry and Technology of Materials (ICTM) founded the Christian Doppler Laboratory for Nanocomposite Solar Cells with the FELMI-ZFE as partner in 2008. In this laboratory, solar cells were developed that consisted of semiconducting nanoparticles embedded in a conjugated polymer matrix. It was recognized early on that the efficiency of these cells is determined by the morphology of the phases and their interfaces, and here we were able to contribute important findings using electron microscopy methods. The methods developed, including the preparation of cross-sections of solar cells using focused ion beam microscopy (FIB) and the transmission electron microscopy (TEM) investigations of the beam sensitive nanocomposite thin films formed our basis for all further research in the field of energy materials. A typical example is the elemental nanoanalysis in fullerene-free organic tandem solar cells on page 49 of this report.

Interfaces and degradation of fuel cells

For more than 20 years, we have been cooperating with Edith Bucher and Werner Sitte from the University of Leoben on solid-state fuel cells (SOFCs), which are operated at high temperatures (> 700°C). Under these conditions elemental diffusion and chemical reactions can lead to a deterioration of long-time stability which can be explored by high resolution TEM and by EELS and EDX spectroscopy. A typical example of this research is shown on page 25.

> Fig. 1 SEM investigation with back-scattered electrons on a cross-section of a solid state fuel cell (a); image (b) reveals carbon nanotubes in a delaminated interface (Hartmuth Schröttner).



In a cooperation with Vanja Subotić and Christoph Hochenauer from the Institute of Thermal Engineering, we are investigating the microscopic structure of (SOFCs) operated at high temperatures under both degradation-free and degradation inducing operating conditions. The focus is set on the study of the morphology changes at the surfaces and at the internal interfaces between the solid phases that can occur as a consequence of diverse operating environments (Fig. 1). This in turn allows to prevent undesired changes in future SOFC runs enabling safe operation.

Structure and chemistry of battery materials

Since 2017, we have been collaborating with Ilie Hanzu from the ICTM of the TU Graz in the projects SOLABAT and DISH, whose goal is to build a new hybrid energy device, i.e. a combination of a solar cell with storage battery integrated in the same disclosure. We focused on the microscopic characterization of the materials in the Li batteries, where the containing Li is impossible to detect using X-ray spectroscopy. Using STEM microscopy in conjunction with EELS spectroscopy, we have been able to quantitatively analyze Li at the nanometer scale, but only at lower temperatures (77 K).

In the EU-funded project AMAPOLA, Gerald Kothleitner is cooperating with Bernhard Gollas from the ICTM at Graz University of Technology. In this project and in the predecessor project SALBAGE, the aim is to develop a rechargeable aluminum-sulfur battery, with novel electrolytes based on highly eutectic mixtures being used here for the first time. The cooperation partners focus on optimizing the reversible charging of the Al anode. Electron microscopic methods are used for rapid characterization of the metallic Al electrodes as well as for high-resolution measurements at the interfaces of the electrodes.

SDG 15 Life an Land

Environmental analysis

At the beginning of the zero years Peter Pölt and co-workers developed the automated particle analysis by means of SEM and X-ray spectroscopy (EDX). However, one drawback of the method was due to technical limitations: Only a few hundred particles could be measured at this time thus limiting the informative value of this method. Only with a new high end SEM equipped with a powerful X-ray spectrometer (silicon drift detector) at the FELMI-ZFE allows now the automated particle analysis with high statistical relevance. The new detector technology allows the analysis of thousands of dust particles within a relatively short time. It is therefore ideally suited to investigate substances in environmental samples about which little is yet known in terms of occurrence and behaviour in the environment (Fig.2).





ed features with chemistry data in area "Silvester_2017_LAM" : 11397 Wt%



Fig. 2 Analysis of particles on a fine dust filter used during New Year's Eve of 2017/18 in Graz. An area of 1.2 mm² was recorded in 166 stitched segments and more than 11.000 particles were identified and analysed revealing the following elements Cu, Ti, Cr, Sr, Pb, Bi, Ag, P, Mn, Zr, Nd and F (Manfred Nachtnebel).





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 highresolution analytical transmission electron microscopy. In the course of his scientific career, he carried out research at the National Center for Electron Microscopy, Berkeley.

Analytical High-Resolution Electron Microscopy

Improving EDXS and EELS Quantification for High-Resolution Applications

TEM in combination with spectroscopic methods such as EELS or EDX offers the unique opportunity to locally characterize the composition down to single atoms in crystalline materials. To achieve atomically resolved resolution, the researcher has to be precise in sample preparation, experiment execution and finally the correct data evaluation. Phenomena like electron scattering, crystal orientation, sample thickness, artefacts from preparation, as well as absorption of low-energy X-rays have to be considered. Our team developed an EELS quantification method for complex rare-earth oxides, which column-by-column determines the elemental distribution in the crystal including correction mechanisms for the above-mentioned factors. For instance, in barium lanthanum ferrate, the Ba/La ratio strongly varies within the layers, but also from atomic column to atomic column (Fig. 1). Such concentration variations might influence transport properties and magnetic properties of ceramic oxides and can be determined by this technique.

Furthermore, our team improved the EDXS quantification methods and developed a procedure to overcome thickness related issues when obtaining reference parameters especially for light elements (C, O, N, ...). This enhances the accuracy of the absorption correction in EDXS for light elements and therefore the quantification results.



Fig. 1 High-resolution EELS elemental map of barium lanthanum ferrate: La (red), Ba (green) Fe (blue). Changes in colour as well as the line profile (right) show the inhomogeneous distribution of Ba and La.

EDX(S) at Elevated Sample Temperatures

Since the development of MEMS heating holders, dynamic in-situ experiments at elevated temperatures may be complemented by X-ray



Fig. 2 Schematics of IR emission characteristics for a MEMS heater (red) and X-rays (green) in polar patterns. X-rays are emitted uniformly in space, while the surface of a MEMS emitter mainly emits in forward direction (a maximum of 100% in a.u.) and only a fraction of the maximum intensity to the side. Tilting a holder to avoid shadowing for X-rays from holder parts will lead to stronger IR irradiation exposure of the detector.

spectrometry for chemical analysis. Although the amount of IR radiation is small, the influence of IR radiation emitted from the heating device on the quality of the X-ray spectra is significant. This is why we systematically examine the influence of infrared (IR) radiation generated by MEMSbased in situ heating systems on the results and interpretation of energy-dispersive X-ray (EDX) spectra through simulation and measurement. Focal points of interest in this study are the influence of holder geometry, shadowing and orientation with respect to the different emission characteristics of IR and X-ray photons and their interaction with side-entry and multidetector systems (Fig. 2). IR photons substantially contribute to count rates, dead time, electronic noise levels, energy resolution, and detection efficiency of semiconductor detectors. At higher sample temperatures, they ultimately limit the feasibility of EDXS for elemental characterization and especially the traceability of low-Z elements.

Microstructure of Mo-Ti-Zr-C alloy processed via laser powder-bed fusion

Molybdenum, processed by laser powder-bed fusion (LPBF), is susceptible to hot cracking because segregated oxygen impurities significantly weaken grain boundaries through the formation of MOO_2 . The present study reports on the LPBF processing of the most important molybdenum alloy TZM, whose alloying elements - titanium, zirconium, and carbon - lead to particle and solid solution strengthening. TZM has demonstrated its potential for use in applications where robust high-temperature resilience is required.

Precipitates within the grains typically have a size of < 50 nm and are spherical in shape. Using EFTEM and EDX analyses, two types of spherical precipitates could be distinguished: Mo_2C and ZrO_2 . Also found were elongated particles which are molybdenum-titanium carbides (Mo,Ti)_xC_y with unspecified stoichiometry (Fig. 3). The ability to dissolve oxygen in ternary molybdenum-titanium carbide is a mechanism that assists in suppressing the segregation of oxygen and thus probably purifies the grain boundaries.



Fig. 3 (a) TEM bright field image, (b) C K jump ratio image, (c) Ti L elemental map, (d) O K elemental map of a ternary elongated molybdenum-titanium carbide with a certain amount of dissolved oxygen.

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Analytical Electron Microscopy

Advances in STEM imaging

Scanning transmission electron microscopy (STEM) denotes one of the most versatile approaches to image nanoscale structures with electrons. This comes grace to an adaptable electron optics and the possibility to freely select the scattered angles of interest. Expanding on this approach, the AEM group has started exploring new techniques that offer even more information about a specimen than before.

First, we broke new grounds with the funding and installation of a novel direct electron detector. This camera, financed by the "Zukunftsfonds Steiermark", opened up new possibilities for noise-free electron spectroscopy. Lowconcentration, light elements can now be easily detected and spectra can be processed in an artefact-free manner. Secondly, our tomography research intensively dealt with the question of 3D reconstructions of chemical gradients in specimens. For this, it has cooperated with KFU Graz mathematicians to work on improved

Fig. 1 Principle of DPC imaging by tracking and processing the shifted CBED disc with respect to the individual detector segments.

software – now freely available. Last but not least, the path was laid to enter intriguing new science with some of our international collaborators in Orsay, France. Phonons have become experimentally accessible by electron microscopes and the combination of highenergy resolution spectroscopy with powerful reconstruction algorithms holds large promise to shed light on fundamentally unsolved research questions.

Yet another route is followed by a newly implemented technique entitled differential phase contrast imaging (DPC). Tracking the exact movement of the scattered electron beam disc (Fig. 1), an image can be generated that carries extra information about the electric and magnetic fields. The potential of mapping out the magnetic field situation in a specimen is currently of particular interest; an example is shown.





Fig. 2 Elemental distribution of Fe, Ni and Cu, (a) shows the phase separation growing along the <100> crystallographic direction, (b) DPC magnetic induction map containing the micromagnetic domain structure.
DPC for studying the magnetic microstructure of a spinodally decomposed CuNiFe alloy

The relationship between the microstructure and the physical properties of an alloy is of utmost interest - the latter can change dramatically when manipulating the former.

Spinodal decompositon of an alloy denotes one way of such a manipulation. The example shown, a CuFeNi alloy, decomposes upon heat treatment into a Cu-rich and a Ni-rich phase, accompanied with a switch from paramagnetic to ferromagnetic behavior. The left image in Figure 2 reveals the elemental distribution of the elements Fe, Ni and Cu, and nicely displays the segregation into two phases growing along the <100> crystallographic directions. The right image in the panel illustrates a DPC characterization of a similar region, given as a "color-wheel" representation. There, the direction of the magnetic induction vectors is

plotted as a function of hue, and the color-wheel inlet (in the upper right of the image) reveals the relation of color and direction. Within the DPC image the micromagnetic structure is nicely visible. The direction of the magnetization vector (drawn as white arrow) is given for several domains and shows a pattern with 90° and 180° domains.

Apart from this specific application, the DPC technique offers other potential applications too. For example, it is possible to study polarizations in crystals on an atomic scale and first experiments are on the way. Medium-term, the group aims to implement detectors that allow for an even deeper analyses on a pixel-accurate level, enabling electron beam phase reconstructions.

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Fig. 2 Basic principle of 3D nanoprinting via the systematic stacking of nano-volumes $(1\rightarrow 2)$, where the lateral beam velocity is the decisive parameter.

Functional Nanofabrication

From Fundamentals Towards Applications

Focused Electron Beam Induced Processing (FEBID)

In recent years, additive direct-write manufacturing experiences significant attention in research and development due to its manifold advantages. Aside of the low demands on the surface to be modified, the possibility of 3D designs is a key element of this technology class. The fabrication down to the micron range has meanwhile technologically matured and already found its way into industrial products. While the mesoscale is currently targeted in research & development, direct-write methods for nanoscale fabrication is still a challenging task. At that scale, spatial precision in 3D space as well as predictability and reliability are very demanding and constrain the potential candidates to only few techniques. Within that small pool, focused electron beam induced deposition (FEBID) is a promising candidate, as it fulfils the requirements of "direct-write", "additive manufacturing" and "3D fabrication" at the nanoscale. For more than one decade, the workgroup "Functional Nanofabrication" at FELMI-ZFE





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

works with that technology with the vision to leverage it into the status of a true 3D nanoprinting method and to reach out for industrial relevant applications.

As shown in Figure 1, FEBID relies on the highly localized dissociation gaseous precursor molecules, which are injected in a scanning electron microscope, where they adsorb onto and diffuse along the relevant surface. As long as the area of interest is accessible for the electron beam, FEBID allows the fabrication of different materials ranging from electrically insulating, over semiconducting towards metallic and even magnetic.

Even more important, FEBID enables the fabrication of freestanding 3D nanoarchitectures with feature sizes below 20 nm. The concept is shown in Figure 2 and relies on the systematic, laterally displaced stacking of nano-volumes, which allows single step fabrication of even complex 3D nanoarchitectures.

In the past 12 years, the workgroup has focused on FEBIDs fundamentals to obtain control over the process, which paved the way towards true 3D nanoprinting. That includes spatial precision aspects as well as material related details, which

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From Fundamentals Toward Applications

enabled the demonstration of novel 3D applications, such as gas sensors or the long-lasting promise of FEBID based 3D plasmonics. Based on the new possibilities and already running projects, industry became interested.

In 2018, the "Christian Doppler Laboratory for Direct Write Fabrication of 3D Nano-Probes" (CDL-DEFINE) was originally initiated in collaboration with "GETec Microscopy" (Vienna). In April 2019, "Anton Paar GmbH" (Graz) joined the CDL and currently, both partners aim on the development of radically new 3D nano-probes concepts for application in their atomic force microscopy (AFM) business branches. Though still in progress, the CDL introduced a 3D thermistor concept for probing local surface temperatures as summarized in Figure 3. The 3D architecture consists of a 4-legged multi-pod nanostructure, which bridges to electrodes on an AFM cantilever (a). The nanoprobe itself is shown in (b) and (c) in side and top view, respectively, which relies on temperature dependent changes of its electric resistance, which allows to access highly localized surface temperatures (< 10 nm) with sub-°C resolution [1].

During the exploration of new application concepts, novel phenomena were observed as well. A key finding on that route was the deeper understanding of thermal effects during 3D-FEBID. In a collaboration with the "Oak Ridge National Laboratories" (USA), localized beam heating was identified as the origin of lacking spatial precision [2]. The findings not only shed light on the dynamic temperature evolution and its consequences but also allowed to derive a counterstrategy to maintain controlled growth in 3D space. That study has to be considered as gamechanger concerning spatial accuracy, predictability and reliability, which are essential for an industrially relevant 3D nanoprinting technology as summarized in a review article [3].

Currently, the workgroup focuses on the expansion of 3D-FEBID's capabilities from mesh-like objects, as shown in figure 3(a-c), towards closed designs. Two examples are shown in figure 3 by means of stacked but rotated diamonds (a) and a more complex 3D structure, both having wall widths of less than 100 nm. Before such possibilities can be used for real application, an upfront design-tool is needed, which also is a research aim of the workgroup. By that, the efforts at FEL-MI-ZFE not only aim on industrially relevant applications but also on the expansion of 3D-FEBID design capabilities.



Fig. 3 A novel 3D nanoprobe concept for local temperature probing in atomic force microscopy applications. While (a) shows the nanoprobe integration in an overview, (b) and (c) shows the actual 3D nano-bridge in a side and top view, respectively. Once, the sub-10 nm sharp tip is in contact with the sample, the temperature changes the electric resistance of the 3D nano-bridge, which allows to access highly localized temperatures at the nanoscale. Currently, the workgroup works on the expansion of 3D FEBID towards closed 3D designs. While (d) shows stacked, but rotated diamonds, (e) shows a complex structure, which opens up new application possibilities in the future.

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Works at the FELMI-ZFE since 1991. He is group leader in the field of scanning electron microscopy and microanalysis since 2005. He leads collaborations within the TU Graz, NAWI Graz and the EL-MINET Graz as well as with institutes of the Austrian Cooperative Research (ACR).

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SEM/IR/Raman

New Solutions for Advanced Materials Characterization

The working group focuses on the development of new preparation methods for microscopic examination methods. In a new approach, different microscope techniques are combined in a versatile wayand data analysis is advanced. The new methods are applied to industrial problems to achieve sustainable value creation and know-how for our partners.

Remote Microscopy and New Evaluation Techniques

In the TIMELY project, we have developed a new and safe method to reduce the number of customer visits to the institute and thus the corresponding CO_2 emissions on the one hand, but also to comply with the social distance rules in times of the Corona pandemic. This allows customers to participate directly in the microscopic investigations of their materials from their workplace. Find out more information in the project description on page 54. In the TIMELY project we also focus on the evaluation of large data sets acquired by correlative investigation methods, using new software approaches and machine learning tools.

Modern Alloys & Energy Materials

In the KORRONET project, we are working together with the ÖGI in Leoben on the study of corrosion processes on metallic materials, such as the corrosion of steels triggered by bacteria in the Koralm Tunnel and the prevention of selective corrosion of copper alloys [1]. Currently, numerous research collaborations are focused on the investigation of 3D printed materials, Li-ion batteries, pouch cells [2] and fuel cells [3].

Environmental Analysis & Life Science

Correlative microscopy has become indispensable for the study of all these complex materials. Our RISE (Raman imaging and scanning electron microscopy) system - the worldwide first installation - combines high resolution SEM with EDX and Raman spectroscopy. Since its setup in 2018, the system has become a main tool for materials science analysis, including the analysis of dust and powder samples. With the RISE system, so-called large area maps (LAM) recorded with EDX and Raman spectroscopy can be combined with automated particle analysis. This method is used in the two research projects "Dust Analysis in Interior Air" and "Aeropore" (Collaborations with the HFA and the OFI in Vienna), but also in collaborations in urban air quality monitoring (Fig. 2). More information can be found in the chapter about environmental analysis on page 29.

The increasing importance of analysing polymers and micro- and nanoplastics in different materials [5] and body tissues is evident due to the number of reports of the occurrence of plastic polluting our environment and affecting life. Presently, we are working on correlative SEM-Raman studies of nanoplastics in complex environments such as food and amniotic fluids (R. Schmidt).

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Fig. 2 Brass alloy CW602 Cross section, FESEM investigation combining EDX elemental mapping and electron backscatter diffraction (EBSD) measurements, (a) EBSD inverse pole figure (IPF) map of CW602 a-phase; (b) EBSD IPF map of CW602 b-phase, (c) image quality (IQ) map correlative to the field of nanoindentations,

(d) EDX elemental map Cu Ka,(e) EDX elemental map Pb Ma,(f) EDXS elemental map Zn Ka

image width: 55 μm

Correlative Micrsocopy (RISE)



Fig. 2 Correlative Microscopy
(RISE) of a dust sample:
(a) SE image of the dust particles with marked regions of interest for the correlative analysis,
(b) EDX spectra of the marked particles, (c) correlative Raman spectra of the marked particles.

Research partner for universities, COMET Centers, SMEs, industry & ACR institutes

Due to our longtime experience, materials knowhow and good networking we can act as a solution provider for your problems and as a project partner for your research studies. We offer a multi scale analysis of surfaces and materials with a wide variety of methods from the mm to nm range for your quality assurance, current problems

and future developments.

The well-connected SEM/IR/Raman team is a main pillar of the institute and acts successfully in national and international cooperations in the fields of quality-control, troubleshooting, problem solving, failure analysis, industrial and university research.

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Research Highlights

Three-Dimensional Nanothermistors for Thermal Probing



ACS Applied Materials and Interfaces, 11 (25) (2019) 22655.

J. Sattelkow, J.E. Fröch, R. Winkler, S. Hummel, C. Schwalb and H. Plank

Thermal influences play a central role in advanced materials, as they impact physical, chemical, and functional properties. Consequently, accessing their absolute values, spatial distributions and temporal evolution is of essential importance for fundamental studies as well as for industrial development. While comparably easy at macroscopic dimensions, it gets more complicated at the microrange, and becomes very challenging when entering the nanoscale. The latter, however, becomes increasingly relevant due to the still ongoing trend towards smaller structures. Within the small pool of techniques, which allow thermal probing in the sub-100 nm range, scanning thermal microscopy (SThM) is the most powerful technology to access laterally resolved temperatures for further correlation with surface morphology and related materials. However, SThM performance strongly depends on the used nanoprobes, where overall design and material properties play a decisive role. Based on that motivation in agreement with the main aims of the CDL-DEFINE in collaboration with industry, we introduced a novel 3D nanoprobe concept for thermal probing at the nanoscale. For fabrication, we used 3D nanoprinting via focused electron beam induced deposition (3D-FEBID), which allows additive, direct-write manufacturing on most materials and surface morphologies, including pre-finished device elements as used here. The path towards such thermal nanoprobes is summarized in Figure 1a we started with simulations to derive the ideal design for AFM applications (step 1), which led to a tetra-pod nanoarchitecture as shown by the blue structure, which bridges the electrodes. In step 2, we aimed on the material quality to increase both, electric properties and mechanical rigidity, which is indispensable for atomic force microscopy (AFM) operation. After full optimization, we could demonstrate full AFM compatibility with very high scans speeds up to 80 µm/s, representatively shown in Figure 1b. The applied material modification not only led to higher mechanical stiffness as needed for AFM but also led to highly predictable electric properties for thermal probing. In more detail, the nano-probes base on a thermistor principle, which uses the temperature dependent variation of electric resistance to inform about the local temperatures. For evaluation of thermal responses, we used MEMS heating chips. We could successfully demonstrate reversible, quantitative probing characteristics with sub -1 °C resolution and a very fast response rate of better than 30 ms/K as shown in Figure 1c. By that, we proved the advantages of 3D FEBID for the fabrication of novel 3D nanoprobes with currently unique capabilities.



Fig. 1 Sthermal 3D nanoprobes fabricated by 3D-FEBID. (a) shows the overall concept of the study, ranging from design aspects, over material optimization towards thermal response evaluation. After step 2, the 3D nanoprobes were found to be fully AFM compatible with high scan speeds up to about 80 μ m/s, as representatively shown in (b), (c) shows a direct comparison of the heating stage temperature (blue dotted line) together with the dynamic response of the here introduced 3D nanoprobe (green solid line), revealing fast response rates of better than 30 ms/K. The inset shows the low noise level of less than 1°C, which underlines the high performance of the here demonstrated concept.

3D Nanoprinting via Focused Electron Beams

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

In this perspective article we summarize various aspects of 3D nanoprinting using focused electron beams (3D FEBID) and give an outlook for the remaining challenges and possible fields of applications. First, the basic principles of 3D FEBID are presented, including selected examples of complex 3D-geometries (Fig. 1, top left), followed by an overview of historical developments with particular emphasis on the last three years. In the following, the status quo is discussed, starting with technical requirements such as gas injection systems and the patterning engines. Particular focus is placed on FEBIDs main process parameters by means of primary beam energies, beam currents, beam focus, patterning velocities and patterning sequences (Fig. 1, top right). In a dedicated section, simulations and calculations of 3D growth dynamics are discussed including 3D FEBID CAD software solutions for a user-friendly design of arbitrary 3D-geometries. An important chapter is the possibility to use various precursor materials for 3D FEBID in order to allow the fabrication of geometries with different functionalities such as insulating, conductive or magnetic properties. The actual status of 3D-FEBID is rounded up with the presentation of 3D-FEBID applications such as thermal nanoprobes, gas sensing concepts, magnetic applications and plasmonically active structures in 3D space (Fig. 1, bottom row). The final chapter gives the authors' perspective concerning remaining challenges of this powerful 3D technology. In more detail, ideas to optimize the instrumental setup are discussed as well as open questions concerning the growth fundamentals for a deeper insight. In addition, that chapter emphasize the need for development and testing of new precursor materials, suitable for 3D FEBID to exploit the full potential of this rising technology. Beyond that, approaches for the modification of 3D FEBID materials by post-processing procedures are evaluated. The advances in the recent years clearly demonstrates current and yet unexplored potential of this emerging technology in the fields of nanoscale probes for scanning probe microscopy, sensors, optics, and magnetics, which are finally envisioned as well. In conclusion, this perspective article gives a comprehensive stateof-the-art overview of 3D-nanoprinting via focused Eeectron beam induced deposition in terms of literature review, ongoing activities and future perspectives.



Journal of Applied Physics, 125 (2019), 210901, invited perspective article.



Fig. 1 Aspects of 3D nanoprinting via focused electron beam induced deposition. (top left) shows an example of a complex 3D geometry with curved individual wires on the nanoscale. (top right) is a comparison of PtC tetrapod structures, fabricated with special patterning strategies. Examples in the bottom row show applications that became possible thanks to the unique capabilities of 3D-FEBID: (a) 3D-nanothermistor (Sattelkow et al. 2019), gas sensor concept (Arnold et al., 2018), demonstration of plasmonic activity on 3D geometries (Winkler et al., 2017), and artificial magnetic lattices (Keller et al., 2018).



Additive Manufacturing 36 (2020) 101605.

Microstructure Evolution during in-situ Heating of AlSi₁₀Mg Alloy Powders and Additive Manufactured Parts

M. Albu, R. Krisper, J. Lammer, G. Kothleitner, J. Fiocchi, P. Bassani

Additive manufacturing or 3D printing is a manufacturing process in which components are built up additively, i.e. layer by layer, from a wide variety of materials. This process is becoming increasingly popular not only in research, but also in industrial production. In the initial boom, there was a focus on newer and newer manufacturing processes, but little attention was paid to the specifics of the microstructure of these materials. Mihaela Albu, in collaboration with colleagues from Italy, is trying to close this gap with the help of electron microscopy.

A good example for this research activities is the structural characterization of 3D printed components made of AlSi₁₀Mg, which have even better mechanical properties than the corresponding cast alloy. However, these special properties of the as-built 3D printed alloy deteriorate during conventional heat treatment.

To better understand this unwanted effect, Albu et al. investigated both the AlSi₁₀Mg alloy powders and the components printed with them using scanning transmission electron microscopy (STEM). Essentially, the microstructures typical for the very fast local cooling rates during the printing process could be found. In-situ heating experiments in the STEM revealed the local structural changes that degrade the mechanical properties during heat treatments. As expected, the overall appearance of the microstructure at low magnification remained unchanged up to 280 °C (Fig. 1). However, at higher magnifications and temperatures below 240°C we observed the crystallization of amorphous or disordered crystalline phases in the eutectic Si network (Fig. 2). At temperatures above 240°C the formation of coarse globular crystalline Si precipitates and breakup of the original Si network happened. On the other hand the Si clusters and nanoparticles evenly dispersed in the matrix, evolved mainly by changing their coherency with the matrix.

The structural changes of the 3D printed alloys found in the *in-situ* TEM measurements helped to explain the rapid deterioration of the mechanical properties during heat treatments. The results of this work provided valuable information for the manufacturers to adjust their post-processing parameters to either reach higher hardness or ductility for the final additive manufactured parts.



Fig. 1Low magnification STEM HAADF images of a FIBFlamella cut from the as-built sample, at different heating
temperatures.r

Fig. 2 High resolution STEM HAADF images of a FIB lamella cut from a powder grain containing amorphous/disordered eutectic Si phases at different heating temperatures.

Analyzing the Nanogranularity of Focused Electron Beam Induced Deposited Materials by Electron Tomography

C. Trummer, R. Winkler, H. Plank, G. Kothleitner, G. Haberfehlner

Nanogranular materials are of great interest for a variety of applications in research and industry. The physical properties of this class of materials are greatly influenced by the size of the nanoparticles, their morphology and the chemistry of the embedding matrix. With focused electron beam induced deposition (FEBID), arbitrarily shaped nanocomposite materials can be designed, where metallic, nanogranular structures are embedded in a carbonaceous matrix. By using special "postgrowth" electron-beam curing, these materials can be tuned for an improved electric-transport or mechanical behaviour.

Such optimization, however, requires a thorough understanding of the changes in the chemistry and microstructure of the composites. This can be achieved primarily with high-resolution electron microscopy, but the conventional two-dimensional imaging methods are not sufficient. Therefore, Cormelia Trummer and colleagues used scanning transmission electron tomography to obtain a comprehensive picture of the three-dimensional distribution and embedded platinum nanograins after initial fabrication and demonstrate the impact of subsequent electron-beam curing.

The electron tomography study of FEBID-based Pt-C nanopillars revealed the true nature of Pt grain shapes and sizes, as well as their spatial distribution in 3D space (Fig. 1). Previously, it was assumed that the particles are spherical and separated from each other. However, Trummer et al. find a more elliptical shape of the metal particles, which is often the result of merged nanograins. Additionally, they discovered that even as-deposited particles can contain interconnected metal networks, which reduce nanogranular characteristics such as tunnelling-based electric transport and matrix-dominated mechanical properties. Using suitable electron-beam curing doses, the physical properties can be tuned from nanogranular toward an extended interconnected metal network (Fig. 2).



Applied Nano Materials 2 (2019) 5356-5359 (2019).



Fig. 1 Schematics of the fabrication process and STEM-HAADF projection images of Pt-C nanopillars; (a) FEBID deposition; (b) STEM-HAADF image of an as-desposited pillar; (c) electron-beam curing; (d) STEM-HAADF image of the cured pillar.

Fig. 2 Tomographic reconstruction of as-deposited (a) and electron-beam cured (b) nanopillars.



Nanoscale 11 (2019) 5617.

Total Generalized Variation Regularization for Multimodal Electron Tomography

R. Huber, G. Haberfehlner, M. Holler, G. Kothleitner, K. Bredies

Electron tomography using a scanning transmission electron microscope (STEM) is a versatile tool for the investigation of nanomaterials in three dimensions down to the single atomic level. Insight into the 3D elemental and chemical make-up of a sample is provided by the spectroscopic signals originating from inelastic scattering in analytical tomography experiments. In such an experiment, tilt series of several signals such as X-ray spectra, electron energy-loss spectra, annular dark-field, or bright field data are acquired at the same time and subsequently reconstructed in three dimensions. However, the acquired data are often incomplete and suffer from noise, and generally each signal is reconstructed independently of all other signals, not taking advantage of correlation between different datasets. This severely limits both the resolution and validity of the reconstructed images.

In a collaboration between the Institute of Mathematics at University of Graz and FELMI-ZFE, Richard Huber and colleagues have shown how image quality in analytical electron tomography can be greatly improved by employing variational modeling and multi-channel regularization techniques. To achieve this aim, a coupled "Total Generalized Variation" (TGV) regularization was employed, which allows to reconstruct both hard transitions and gradual changes inside a sample, and links different signals at the level of first and higher order derivatives. This favors similar interface positions for all reconstructions, thereby improving the image quality for all data in particular for 3D elemental maps (Fig. 1).

Huber et al. demonstrated this joint multi-channel TGV reconstruction on tomographic energydispersive X-ray spectroscopy (EDXS) and highangle annular dark field (HAADF) data (Fig. 2), but the reconstruction method is generally applicable to all types of signals used in electron tomography, as well as all other types of projection-based tomography techniques.



Fig. 1 Slice through reconstruction of three EDXS elemental maps using a conventional reconstruction algorithm (SIRT) and the proposed algorithm (TGV coupled).



Fig. 2 Volume rendering of the 3D reconstruction of the HAADF signal and of the elemental maps of Yb, Al and Si.

The Impact of Swift Electrons on the Segregation of Ni-Au Nanoalloys

D. Knez, M. Schnedlitz, M. Lasserus, A.W. Hauser, W.E. Ernst, F. Hofer, G. Kothleitner

For sensitive specimens, electron irradiation in the electron microscope may cause specimen damage. To avoid this, one normally tries to keep the irradiation dose as low as possible.

In the following example, however, electron irradiation is used to specifically modify the sample. For this purpose, we used Au-Ni clusters with a diameter of less than 10 nm, which were grown fully inert in superfluid helium droplets at a temperature of 7 K. The alloy system Au-Ni is of interest because of a miscibility gap. Initially, the clusters exhibit a core-shell morphology given by the sequence of He-droplet doping in the synthesis facility. Nickel is in the center of the cluster and gold forms the shell (Fig. 1).

Upon heating above the miscibility gap directly in the electron microscope, the clusters start to form a homogeneous alloy. Under prolonged electron irradiation, however, these clusters transform to a Janus-type morphology and surprisingly, nickel and gold separate into two distinct phases (Fig. 2). This behaviour is actually not possible from a thermodynamic point of view and therefore this unexpected behaviour was investigated in more detail.

The underlying mechanisms were studied experimentally via in-situ scanning transmission electron microscopy and theoretically via atomistic simulation techniques under consideration of elastic electron interactions. The observed effects can be attributed to displacements of cluster atoms by elastic scattering with the incident electrons. The experiments reveal an exponential temperature relationship of the segregation kinetics, which can be explained by diffusive relaxation processes which follow beam-induced displacement events.

These results shed light on radiation induced phenomena using clusters as a model system and suggest new routes for the synthesis of structures in nonequilibrium configurations.



Applied Physics Letters 115 (2019) 123103.





Microelectronics Reliability 100-101 (2019) 113366.

High-resolution Cross-sectional Analysis of the Interface between SiC and SiO2 in a MOSFET Device via Atomic Resolution STEM

E. Fisslthaler, G. Haberfehlner, C. Gspan, G. Gruber, W. Grogger

The structure and chemical composition of internal interfaces in electronic devices but also dynamic processes in these delicate regions such as the formation of interlayers and defects is crucial for the properties of the final products. Therefore, the detailed study of these interfaces is of great technological importance. The most suitable method is certainly high-resolution electron microscopy (STEM) in combination with electron energy-loss spectroscopy (EELS).

In this work, we have studied the interface between silicon carbide (SiC) and silicon dioxide (SiO_2) in a STEM with atomic resolution. This interface plays a critical role in SiC-based metal oxide semiconductor field effect transistors (MOSFETs).

Samples of four-layer hexagonal silicon carbide (4H-SiC) grown with a 4° offset angle with respect to the c-axis and covered with a deposited oxide (SiO_2) were treated with a post oxidation anneal in NO-containing atmosphere. Electron transparent cross-section samples were prepared with a focused ion beam instrument (FIB) and subsequent milling with a "Nanomill".

HR-STEM investigations were carried out with our ASTEM microscope (FEI Titan3 G2 60-300) (Fig. 1) and by using the "Dual EELS" mode twodimensional "spectrum images" were recorded across the interface area. Here we explored possible intermediate states that can be only revealed due to their contributions to the energyloss near-edge structure (ELNES) of the Si-L_{2,3} ionization edge.

We could show that the nitrogen can be located reproducibly at the interface, and that it is localized on all faces of the faceted interface (Fig. 2). The quantification of the elementary EELS signal of nitrogen shows an estimated areal density of $C_N = 4 \cdot 1014 \text{ cm}^{-2}$. This corresponds approximately to 1/3 of a monolayer of atoms on the SiC surface.

In addition, closer analysis of the EELS signal reveals a transition region that is 3 nm in width, extending approximately 1 nm further into the SiO_2 than the N-peak. This phenomenon could be caused by varying oxidation states of the SiO_2 in the immediate vicinity of the interface.



Fig. 1 HR-TEM bright field image of the interface region, showing the 4° growth offset; viewing direction is [1-100].



Fig. 2 Superimposed EELS elemental maps of nitrogen (green) and the residual signal from the MLLS fit of SiC and SiO_2 (red) and resulting profile of the central region (below).

sition [nm]

Large Magnetic Gap at the Dirac Point in $Bi_2Te_3/MnBi_2Te_4$ Heterostructures

E.D.L. Rienks, S. Wimmer, J. Sanchez-Barriga, O. Caha, P.S. Mandal, J. Ruzicka, H. Steiner, V.V. Volobuev, H. Groiss, M. Albu, G. Kothleitner, G., J. Michalicka, S.A. Kahn, J. Minar, H. Ebert, G. Bauer, F. Freyse, A. Varykhalov, O. Rader, G. Springholz

Topological insulators are a new class of materials with very interesting properties including really exotic phenomena. For example, magnetically doped topological insulators enable the quantum anomalous Hall effect (QAHE) which supplies quantized edge states for lossless charge-transport.

Current research is focused on Bi_2Se_3 and Bi_2Te_3 doped with magnetic elements such as manganese, because this class of materials could be very suitable for the fabrication of spintronic devices. However, these new effects can only be measured at very low temperatures below 1 Kelvin and therefore very great efforts are made to induce these effects also at room temperature.

Rienks et al. investigated the specific properties of Mn-doped Bi₂Se₃ and Bi₂Te₃ thin films prepared by molecular beam epitaxy in a broad multimethod approach. They used angle-resolved photoemission spectroscopy (ARPES) and spinresolved ARPES at BESSY II in Berlin and additional transport and magnetic measurements and correlated these results with microstructural assembly. Here, XANES and EXAFS measurements

C

QL

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were used and especially the high-resolution STEM work by Mihaela Albu and Gerald Kothleitner provided the essential information about the crystallographic arrangement of the Bi, Te and Mn atoms in the thin film materials.

The STEM-HAADF investigations show a high structural perfection and that additional septuple layers are formed only with Mn doping, whereas the pure Bi₂Se₃ film consists only of quintuple layers (Figs. 1a and b). Atomic layer resolved distribution of the Bi, Te and Mn atoms of the Mn-doped Bi₂Te₃ film ($X_{Mn} = 10$ %) reveal that the Mn atoms are predominantly incorporated in the centre of the septuple layers and to a lesser extent in the outer layers of the septuple units (Fig. 1c).

The STEM results clearly show that Mn doping results in a self-assembled superstructure, which leads to a larger magnetic energy gap. The formation of this septuple layers explain the observed phenomena and these findings open up possibilities for further optimization of these topological materials for use in quantum computers.

> QL (Bi2Te3) QL (Bi2Te3)

> QL (Bi2Te3)

Fig. 1 STEM HAADF images of cross-sections of pure Bi_2Se_3 (a) and Mn-doped Bi_2Se_3 ($X_{Mn} = 6\%$) (b) and EDX elemental maps of Mn-doped Bi_3Te_3 (c).



b



Nature, 576 (2019) 423–428.



Macromolecular Symposia 384 (2019) 1800237.

The Combination of Electron Microscopy, Raman Microscopy and Energy Dispersive X-Ray Spectroscopy for the Investigation of Polymeric Materials

R. Schmidt, H. Fitzek, M. Nachtnebel, C. Mayrhofer, H. Schröttner, A. Zankel

Polymers play an important role in materials science, because of their advantages over other types of materials. The huge variety in terms of types, arrangements and combinations of monomers found in polymers leads to an extremely wide diversity of polymeric materials. Therefore, polymer morphology is a prevailing topic and there already exists a wide range of different microscopic techniques for polymer characterization. However, the significance of these individual methods can be greatly enhanced by combining them. This is the rapidly growing field of correlative microscopy.

In this work, the focus is laid on a novel correlative microscopy method, combining Raman microscopy, scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDXS). Three special polymeric specimens were examined with the new RISE system of the Institute (Raman imaging and scanning electron microscopy) in combination with an advanced EDXS spectrometer.

The RISE microscope is based on the "off axis" system, a setup where the axes of the Raman microscopes objective lens and the SEM's electron optical column are parallel, but not identical (Fig. 1).

It is clear that the imaging properties of the SEM - in our case a Zeiss Sigma 300 - play a decisive role: The Zeiss SEM can be operated with the variable pressure mode (VP), where nitrogen is used as imaging gas at pressures between 10 and 133 Pa. Furthermore, the low-voltage mode in the high vacuum is discussed which gives a better surface resolution and a lower beam damage. Both methods work without a conductive coating on the specimen, which is absolutely necessary for the Raman investigations.

The potential of the new combination of methods is demonstrated by the successful investigation of various polymeric multilayer systems (packaging foil) (Fig. 2) and of fillers and additives in polymer materials and in paper.



the setup of the RISE system (Zeiss & Witec)

> Fig. 2 a) SEM image of the embedded peel film (1, 2 peel arms, 3 bulge due to sealing, 4 upper regions of seal area); b) schematic of a peel film, c) Raman spectra of polymeric components according to the map (correlated colors).



Elemental Nanoanalysis of Interfacial Alumina–Aryl Fluoride Interactions in Fullerene-Free Organic Tandem Solar Cells

S.F. Hoefler, G. Haberfehlner, T. Rath, R. Canteri, M. Barozzi, F. Hofer, G. Trimmel

Organic solar cells based on fullerene now achieve efficiencies of well over 15 %. However, these cells have some important disadvantages compared to fullerene-free organic solar cells, such as functionalization, optical absorption behaviour and, above all, production costs. For this reason, Gregor Trimmel's team is focusing on the further development of fullerene-free organic solar cells. The performance of these cells is significantly influenced by the optimal material combination for the absorber layers, the electrodes and especially of the interfacial layers.

In this work, tandem solar cells were developed with fluorine-containing absorber layers consisting of the fluorinated polymer donor PTB_7 -Th and the indacenodithiophene-type small molecule acceptor O-IDTBR and $MoO_3/AI/PFN$ -Br as recombination layers.

Modern materials for organic photovoltaic cells are usually stable during the solar cell fabrication, but unexpected chemical reactions at the interfaces have to be considered in the final device, in particular, if reactive metal interlayers are employed. However, these interfacial processes

Ag

MoO,

are hardy detectable by routine solar cell analysis. Therefore, the FELMI-ZFE team concentrated on elaborated electron microscopy methods (STEM-HAADF in combination with EDX- and EELS-spectroscopies) in order to study the interface phenomena and elemental distributions with nanometer resolution.

Surprisingly, the STEM investigations revealed an unexpected phenomenon. It was found that significant amounts of fluorine (together with Al and O) are accumulated in the recombination layer region (Fig. 1) which originates very likely from alumina-aryl fluoride interactions responsible for the partial defluorination of the conjugated polymer in the absorber layer.

Further investigation and understanding of this interface phenomenon and if this reaction also happens in similar absorber materials are of utmost importance for the further development of organic electronics. This is because there is a possibility that these interfacial reactions could affect the performance of the devices and also limit their lifetime.

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Mo

С

S

PFN-Br/Al/MoO3

PTB7-Th:O-IDTBR

ZnO

ITO

Br

Ag

В

AI





Advanced Materials Interfaces 6 (2019) 1901053.

Zn

Sn

In

Ν

Research Grants

Peer-reviewed research grants acquired or active from 2019 to 2020.

FELMI Projects

Combined Triple-Ion and High Speed Atomic Force Microscope for Correlative Analysis (AIM)

Leader: Harald Plank Coordinator: GETec Microscopy GmbH, Vienna Beyond Europe, Austrian Research Promotion Agency (FFG), Vienna, 08/2018 - 05/2020

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)

FELMI-ZFE leader: Gerald Kothleitner Coordinator: Bernhard Gollas, Institute of Chemistry and Technology of Materials, TU Graz EC Horizon 2020, FET - Open research and Innovation Actions, 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 FELMI-ZFE leader: Gerald Kothleitner Coordinator: Max-Planck-Institute for Solid State Research, Stuttgart, Germany EC, Horizon 2020 INFRAIA, 12/2018 - 12/2022









Bundesministerium
 Digitalisierung und
 Wirtschaftsstandort

ELMINET - Electron Microscopy Network in Graz FELMI-ZFE leader: Armin Zankel

Coordinator: Christian Gülly, Medical University of Graz

HRSM-project of the Austrian Ministry of Education, Science and Research, Vienna, 5/2017 - 12/2021

A Marketable Polymer Based Aluminium Sulfur Battery (AMAPOLA)

FELMI-ZFE leader: Gerald Kothleitner Coordinator: Bernhard Gollas, Institute of Chemistry and Technology of Materials, TU Graz EC Horizon 2020, FET - Open Research and Innovation Actions, Brussels, 10/2020 – 09/2022

Intelligence Reliability (iRel4.0)

FELMI-ZFE leader: Werner Grogger Coordinator: Institute of Automation and Control, TU Graz EC Horizon 2020, ECSEL Call 2019_1, Brussels, 05/2020 – 04/2023

High Quality AIN Based Piezoelectric Thin Films for SAW and BAW Applications

FELMI-ZFE leader: Werner Grogger Coordinator: Silicon Austria Labs GmbH (SAL), Graz

Comet K1 Center: ASSIC Austrian Smart Systems Integration Research Center, 01/2020 – 12/2022

ZFE Projects

Rare-earth Nickelates for Future Energy Technologies (SENTECH)

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

Self-organized Proton Conducting Composites for Future Energy Technologies (PROTEC)

FELMI-ZFE leader: Werner Grogger Coordinator: Edith Bucher, University of Leoben, Energy Research elmission 5. AS Austrian Research Promotion Agency (FFG), Vienna, 93/2019 – 8/2022

Solar Cell Meets Battery: Realization of a Hybrid Energy System (SOLABAT)

FELMI-ZFE leader: Ferdinand Hofer Coordinator: Ilie Hanzu, 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 Minimizing the Biological Risks in Interiors (AEROPO-RE)

FELMI-ZFE leader: Johannes Rattenberger Coordinator: Gabriele Ettenberger-Bornberg, OFI Technologie & Innovation GmbH, Vienna COIN Austrian Research Promotion Agency (FFG), Vienna, 01/2017 – 12/2020

Microstructure of 3D Printed Metallic Components

FELMI-ZFE leader: Mihaela Albu, Gerald Kothleitner

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 (AEROPORE)

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



European Commission



Tribological Optimization 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 (KorroNet)

FELMI-ZFE leader: Hartmuth Schröttner Coordinator: Gerhard Schindelbacher, ÖGI Austrian Foundry Research Institute, Leoben Austrian Cooperative Research (ACR) and Austrian Ministry of Digital and Economic Affairs, Vienna, 03/2018 – 02/2020

Multimedia Based Online Microscopy (TIMELY)

FELMI-ZFE leader: Johannes Rattenberger, Harald Plank

Coordinator: ZFE Graz

Austrian Cooperative Research (ACR) and Austrian Ministry of Digital and Economic Affairs, Vienna, 06/2020 – 05/2022

Material and Process Optimization for Series Production of 3D Printer Components (SERIFE 3D)

FELMI-ZFE leader: Hartmuth Schröttner Coordinator: Heinz Haider, OFI Austrian Research Institute for Chemistry and Technology, Vienna Austrian Cooperative Research (ACR) and Austrian Ministry of Digital and Economic Affairs, Vienna, 07/2020 – 06/2022

Chlorid Korrosion

FELMI-ZFE leader: Hartmuth Schröttner Coordinator: VÖZ Vereinigung der Österreichischen Zementindustrie, Vienna Austrian Cooperative Research (ACR) and Austrian Ministry of Digital and Economic Affairs, Vienna, 07/2020 – 06/2022

The Sun Battery – a Hybrid System of Solar Cell and Battery (DISH)

FELMI-ZFE leader: Werner Grogger, Ferdinand Hofer

Coordinator: Ilie Hanzu, Institute of Chemistry and Technology of Materials (TU Graz) Zukunftsfonds Steiermark, Graz, 01/2021 – 12/2022

> Bundesministerium Bildung, Wissenschaft und Forschung

Research Projects

Harald Plank

Christian Doppler Laboratory: DEFINE

Key Facts

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Funding: Christian Doppler Forschungsgesellschaft, Vienna

Period 1.3.2018 - 28.2.2025

Industrial Partner:

GETec Microscopy GmbH, Vienna

www.getec-afm.com

Anton Paar, Graz, Austria www.Anton-Paar.com In March 2018, the Christian Doppler Laboratory for "Direct-Write Fabrication of 3D Nanoprobes (DEFINE)" was officially opened under the auspices of Rector Harald Kainz at Graz University of Technology. The origin of that CD Laboratory goes back to several research projects together with the Austrian company GETec Microscopy GmbH (Vienna). GETec realized the high potential of an emerging nanofabrication technology, which lies in the scientific main focus of Harald Plank.

That technology, called focused electron beam induced deposition (FEBID), enables flexible additive manufacturing thanks to the maskless, direct-write character for 3D fabrication on the nanoscale, which meet challenges when classical, resist based lithography run into their intrinsic limitations.

On the pursuit of industrial applications, Harald Plank teamed up with GETec to explore next generation nanoprobe concepts for atomic force microscopes (AFM), which will push their performance beyond current limitations. Harald Plank's team developed electrically conductive nanoprobes (Fig. 1a) for conductive AFM (C-AFM), which are meanwhile commercially available, and magnetic tips (Fig. 1c) for magnetic force microscopy (MFM), which are currently in the transfer phase for small series fabrication.

Due to 3D-FEBID's strengths, more complex 3D nano geometries become possible, which led to the introduction of an entirely new application concept for thermal probes. As shown in Figure 1e, multi-leg structures with branch diameters of less than 100 nm are used as electrical nanobridges, which changes the electric resistance with temperature. As the proposed concepts are truly unique for their applications, Anton Paar GmbH (Graz, Austria) became interested in the related possibilities, which led to the expansion of the CD Laboratory in April 2019.

Current activities include the expansion of fabrication and post-growth possibilities for further applications for the AFM product lines of the companies.



Fig. 1 3D FEBID based nanoprobe concepts; a) tilted SEM image of an all-platinum nanoprobe with apex radii below 10 nm (see TEM inset top left), intended for C-AFM; 500 nm wide morphology scans of an Au nanoparticle sample; c) SEM side view of a magnetic nanotip on top of a standard cantilever, again with apex radii in the sub-10 nm range; d) 2.5 µm wide, magnetic phase signals of a PtCo sample; e) SEM side view image of a FEBID-based 3D nano-bridge together with a top view as inset.









ESTEEM3: European TEM Network

Gerald Kothleitner

ESTEEM3 - Enabling Science and Technology through European Electron Microscopy – is an EU funded project for electron microscopy, which aims at providing access to the leading European state-of-the-art electron microscopy infrastructures. The ESTEEM consortium facilitates transnational access services of the most powerful atomic scale characterization techniques in advanced electron microscopy research to a wide range of academic and industrial research communities.

The consortium of 14 research laboratories and 6 companies builds on the previously very successful ESTEEM1 and ESTEEM2 platforms. Like in ESTEEM2, the FELMI-ZFE with its unique equipment, again represents the Austrian knot and provides access to its instrumentation and scientific manpower.

ESTEEM activities are focused on three main areas:

1. Transnational access (TA) to ESTEEM3 centers is obtained through a transparent peer review process based on merit and scientific priorities. 2. Optimum service to users is supported by Networking Activities and Joint Research Activities (JRA), which address key issues of advanced electron microscopy. The focus lies on specimen preparation, data interpretation and automation through theory and simulation, and standardization of protocols and methodologies. 3. Directed research programs also focus on applied research of materials related to information and communication technologies (ICT), energy, health and transport for the benefit of European scientists and industry.

In Transnational Access, the FELMI-ZFE supports external groups worldwide, with 125 days for specimen preparation, 160 microscope days and 240 days for data analysis in 4.5 project years. By the end of 2020, the FELMI-ZFE team has already been able to support 19 research groups.

The Institute leads the research focus on ICT materials creating an important synergy to the cooperation with Austria's SAL (Silicon Austria Labs) initiative.

Key Facts

Leader

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

Institute of Electron Microscopy and Nanoanalysis (TU Graz)

gerald.kothleitner@felmi-zfe.at Period 1.1.2019 - 31.12.2024

Coordinator: Prof. Peter van Aken, Max-Planck-Institute for Solid State Research, Stuttgart, Germany

14 academic partners & 6 industrial partners







Multimedia Based Online Microscopy – TIMELY

Key Facts

Leader

DI Dr. techn.

Johannes RATTENBERGER Graz Centre for Electron Microscopy

johannes.rattenberger@felmizfe.at

Funding: Federal Ministry of Digital and Economic Affairs

Period 1.6.2020 - 30.5.2022

Consortial Partners:

ÖGI - Leoben, Austria www.ogi.at

V-Research - Dornbirn, Austria https://www.v-research.eu/

KMU Forschung Austria - Wien, Austria https://www.kmuforschung. ac.at/

Bundesministerium Digitalisierung und Wirtschaftsstandort

austrian cooperative research

The goal of this strategic ACR project is to extend the possibilities of correlative microscopy in which several methods such as SEM, AFM, FIB, EDXS and Raman are to be combined in a unique way. In this project we could built-up a focused ion beam microscope (FIB), a FEI Quanta 3D with an integrated atomic force microscope (AFM) provided by GETec Microscopy. Therefore it is now possible to integrate FIB and AFM into our network of correlative microscopic methods (Fig. 1).

Johannes Rattenberger, Harald Plank

Furthermore, we develop new computer assisted methods to analyse the multidimensional data sets, which are obtained from the correlative measurements, with the help of machine learning. The data must be accurately aligned and subsequently the different information from different layers (images) must be cross-linked to identify phases or features in a quantitative way. All these method developments should lead to quickly available, reproducible and objective results, which cannot be generated by a human being.

These new investigation possibilities are to be made directly available to external cooperation partners via the internet (mulTIMedia basEd onLine microscopY – TIMELY). In 2020, the new multimedia online microscopy has been successfully implemented and many aspects of service research, such as direct discussions of tasks and results and joint measurements on the microscopes, could thus be shifted into virtual

> Fig. 2 Streaming platform of Kapsch with Cisco components and Webex interface which can be used for all microscopes of the Institute.

space. In the past, this was often difficult and time-consuming and in the case of more distant partners also involved longer journeys.

The core of this new service is the joint online microscopy. With the help of the portable streaming device (Kapsch AG with Cisco components, see Fig. 2) the ZFE can send the live image of the electron microscope or analysis device as a content in a video conference. Sensitive customer data is protected by end-to-end encryption and the video signal can be streamed in real time and in high resolution.





Fig. 1 Concept of the SEM-AFM-FIB-EDXS platform SAFE with the new infrastructure a dual beam FIB, Raman not included.

Direct-Electron-Detection for the ASTEM Microscope

Gerald Kothleitner

The high-resolution electron microscope ASTEM was equipped with a new type of detector in the framework of the project "ASTEM Upgrade" which was funded by the Zukunftsfonds Steiermark.

Since the installation of the ASTEM in summer 2011, the ASTEM has been equipped with a conventional "charge coupled device" (CCD) camera for taking images and spectra. The CCD camera, however, delivers only mediocre results for radiation-sensitive materials such as biological samples, polymer samples or 2D materials. This is due to several limiting factors such as readout noise, point spreading and scintillator sensitivity of the CCD cameras.

In order to extend the application range of the ASTEM to radiation-sensitive ("dose-limited") materials, a new improved detection technology had to be introduced, the so-called direct electron detection cameras (DDD, direct detection device, Fig. 1). This principle very effectively eliminates all problems of the conventional CCD cameras and allows images and spectra to be generated almost noise-free and with much better resolution. Being able to count single electrons with highest sensitivity opens a wide range of 2D and 3D investigation possibilities in the field of bio- (structure elucidation) and material sciences (light metal alloys, energy materials).

ZUKUNF ISFONDS STEIERMARK

Sensitive materials such as biological samples, complex polymer mixtures, light metal alloys and energy materials, for example, have been investigated at very low doses with a high detection limit for volatile and/or trace elements. On the other hand, *in-situ* heating experiments (imaging and EEL spectroscopy measurements) with a significant improvement in spatial and energy resolution are now possible for different functional materials as presented in Figure 2.

Key Facts

Leader

Univ.-Prof. DI Dr. Gerald Kothleitner Institute of Electron Microscopy and Nanoanalysis (TU Graz)

gerald.kothleitner@felmi-zfe.at Funding: Zukunftsfonds Steiermark, Graz

Period 1.6.2018 - 31.12.2020

Consortial Partners: Medical University of Graz Karl-Franzens-University of Graz



Fig.1: Direct electron detection camera "K2" hardware setup on the ASTEM microscope.



Fig. 2 *in-situ* heating performed for a neutron irradiated beryllium sample. HAADF images at room temperature (left) and at 520°C (right) of regions presenting rectangular gas filled (H and He) features. The EEL spectra from the marked regions at different temperatures are shown on the graph.

Academic Education

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Lectures and Laboratory Courses

Since our integration into the Faculty of Mathematics, Physics and Geodesy in 2004, we have been able to continuously expand our range of courses. Today, scientific staff members of the Institute conduct courses for more than 950 students of Graz University of Technology, but also to an increasing extent for the University of Graz within the NAWI network.

Our teaching focus is on advanced microscopy, micro- and nanoanalysis and in recent years we have also been able to set up first-class courses in the emerging field of 3D nanostructuring.

Members of staff also make important contributions to the laboratory courses in technical physics and advanced materials science. We also offer courses in chemistry, electrical engineering, biomedical engineering and biotechnology.

Winter Semester 2019/20

Lectures

Electron Microscopy 1, PHT902UF, 2 L, Grogger

Introduction into Physics for Chemical Engineering, 519.100, 2 L, Grogger

Electron Microscopy in Biotechnology, MOL.991UF, 2 L, Hofer

Electron Microscopy Imaging for Biomedical Engineering, 717.236, 1 L, Hofer

Scientific Working for PhDs, 519.011, 2 SE, Hofer Materials Characterisation II, MAS.160UF, 1.33 L,

Kothleitner

Spectroscopy, PHU.006UB, 2 L, Kothleitner with Knoll (Uni Graz)

Structuring of Materials Surfaces and Functional Nanofabrication, PHT.906UF, 2 L, Plank

Physics (ET) – Exercises, 511.202, 1 UE, Zankel, Letofsky-Papst, Rattenberger, Winkler

Seminar Electron Microscopy & Nanoanalysis 1, PHT.907UF, Grogger, Hofer, Kothleitner, Plank.

Laboratory courses

Project Laboratory, MAS.190_5UF, 8 PT, Grogger, Haberfehlner, Hofer, Kothleitner, Mitsche.

Research Laboratory Microscopy & Nanoanalysis, PHT.905UF, Grogger, Kothleitner, Zankel

Bachelor Project, PHY.120_9UF, 1 PT, Grogger, Hofer, Kothleitner, Plank

Master's Project, PHT.010_9UF, 0.5 PT, Grogger, Hofer, Kothleitner, Plank

Laboratory Course Metals and Ceramics, MAS.200UF, 6 P, Kothleitner, Mitsche

Laboratory Course Biobased Materials, MAS.400UB, 6 P, Wewerka (with Uni Graz) Chemical Laboratory for Biologists, MOL.305_1UF 4P, Mitsche, Wewerka (with ICTM, TU Graz)

Methods for IC Evaluation and Failure Analysis, 439.221 Mitsche (with IFE, TU Graz)

Summer Semester 2020

Lectures

Electron Microscopy 2, PHT904UF, 2 L, Grogger

High Resolution Electron Microscopy, MAS.366UF, 2 L, Hofer

Soft Matter Microscopy, MAS.460UF, 2 L, Hofer, Fitzek

Materials Chemistry, CHE.173UF, 2.66 L, Hofer with lecturer from ICTM

Advanced 2D and 3D Nanoanalysis, PHT.903UF, 2 L, Kothleitner

Microelectronics and Micromechanics, PHT.901UF, 2 L, Plank

Physics 2 (Electrodynamics, Optics), UNT.074UF, 2 L, Plank

Exercises in Electrodynamics and Optics, UNT.075UF, Haberfehlner, Schmidt

Seminar on Electron Microscopy and Nanoanalysis 2, PHT.908UF, W. Grogger, F. Hofer, G. Kothleitner, H. Plank.

Laboratory courses

Project Laboratory, MAS.190_5UF, 8 PT, Grogger, Haberfehlner, Hofer, Kothleitner, Mitsche.

Research Laboratory Microscopy and Nanoanalysis, PHT.905UF, Grogger, Kothleitner, Zankel

Bachelor Project, PHY.I20_9UF, 1 PT, Grogger, Hofer, Kothleitner, Plank

Master's Project, PHT.010_9UF, 0.5 PT, Grogger, Hofer, Kothleitner, Plank

Materials Laboratory, MAS.190_5UF, 4 P, Mitsche (with IMAT, TU Graz)

Practical Class in Basic Chemistry, 638.902, 4 P, Mitsche, Wewerka (and ICTM, TU Graz)

Imaging Laboratory 717.340, 2 P, Mitsche (with IMT, TU Graz).

Privatissima in winter and summer semester

Special Aspects of Transmission Electron Microscopy 1, 519.019, 2 SE, Grogger

Special Aspects of Analytical Electron Microscopy 1, 519.015, 2 SE, Hofer

New Methods in Electron Microscopy 1, 519.017, 2 SE, Kothleitner

Current Trends in Additive Manufacturing, 519.033, 2 SE, Plank





Life Long Learning Courses

European EELS & EFTEM School

Gerald Kothleitner, Werner Grogger

The EELS & EFTEM-School is a four days hands-on laboratory workshop taking participants step-bystep 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 190 participants from all over the world.

EELS Electron Energy-loss Spectroscopy EFTEM Energy-filtering TEM

Problem Solving with Scanning Electron Microscopy and Spectrosocopy

Stefan Mitsche, Hartmuth Schröttner

This three-day course teaches the fundamentals and advanced applications of analytical scanning electron microscopy. The course is aimed at scientists, engineers and technicians who are interested in solving problems in materials research and quality control.

The participants learn through lectures and hands-on training how to operate advanced scanning electron microscopes, including environmental and field-emission instruments. We use stateof-the-art facilities and know-how to ensure that participants learn to acquire first-class images and X-ray and Raman spectra as well as elemental distribution images and also to evaluate them quantitatively.



Fig. 1 Distribution of participants of the EELS & EFTEM School since 2006





Guests at the Institute

Guest Lectures



Mojca OTONIČAR, Jožef Stefan Institute, Lubljana

20 May. 2019, Mojca OTONIČAR Jožef Stefan Institute, Lubjana, "Contributions to electromechanical response in relaxor ferroelectrics from macro to nano-scale".

8 Aug. 2019, Maria Valera del ARCO Universidad Complutense de Madrid, "High Resolution Views of the Nanoworld".

8 Nov. 2019, Bernhard BICHLER Fa. Videko GmbH Hitachi, "Bericht über Videko GmbH Hitachi Vertrieb".

22 Nov. 2019, Stefan EICHINGER Applied Geosciences TU Graz, "CaCO₃ Versinterung in Tunneldrainagen – Anorganische vs. Mikrobielle Mineralablagerungen".

10 Jan. 2020, Martin NAPETSCHNIG Institut für Physik, TU Graz, "Optimisation of NTC ceramics containing iron".

24 Jan. 2020, Andre BALDERMANN Applied Geosciences TU Graz, "Die Größe macht den Unterschied - Anwendungsbeispiele der Elektronenmikroskopie in Natur und Technik".

31 Jan. 2020, Bernhard STÖCKL Wärmetechnik TU Graz, "Ammonia as Promising Fuel for Solid Oxide Fuel Cells: Experimental Analysis and Performance Evalution".

Josef ZWECK, University of Regensburg



Visiting Scientists

Petra Peharec, Department of Molecular Biology, University of Zagreb, Zagreb, Croatia

Nikolai Zimber, Institute of Applied Materials, KIT Karlsruhe Institute of Technology, Karlsruhe, Germany

Johannes Fröch, School of Physics and Advanced Materials, University of Technology, Sidney, Australia

Levi Tegg, School of Information and Physical Sciences, University of Newcastle, Australia

Paola Bassani, Institute of Condensed Matter Chemistry and Technologies for Energy, Italian National Research Council, Padova, Italy

Elisabetta Gariboldi, Institute of Mechanics, Politecnico Milano, Milano, Italy

Regina Ciancio, Istituto Officina dei Materiali, Italian National Research Council, Trieste, Italy

Anna Martin Vilardell, Department of Materials Science, Nagoya University, Meidai, Japan

Scientific guests who could not come to Graz because of COVID-19. Nevertheless, their materials and problems were successfully processed by the FELMI-ZFE staff.

Simon Hoeges, GKN Sinter Metals, Radevormwald, Germany

Julian Strobel, Institute of Materials Science, University of Kiel, Kiel, Germany

Pavel Krakhmalev, Department of Engineering and Physics, Karlstad University, Karlstad, Sweden

Jonathan Peters, Department of Physics, University of Warwick, Coventry, U.K.

Vesna Srot, Max-Planck-Institute for Solid State Research, Stuttgart, Germany

Luc Lajaunie, Department of Materials Science and Metallurgy Engineering, University of Cadiz, Spain

Eskilla Venkata Ramana, Institute for Nanostructures, Nanomodelling and Nanofabrication, University of Aveiro, Aveiro, Portugal

DIPLOMAT

Master & Doctoral Theses at the Institute

Finished PhD Theses

ORTHACKER Angelina (2019), "Developing methods for 3D STEM to reveal atomic-scale spinodal decomposition", G. Kothleitner.

KONRAD Lukas (2020), "How multiple scattering simulations help for EELS compositional analysis of hard metals and ceramics", G. Kothleitner.

PhD Theses in Progress

KRISPER Robert, "*in-situ* heating experiments in high resolution electron microscopy", W. Grogger. LAMMER Judith, "Analytical high resolution STEM of cathode materials for solid state fuel cells", W. Grogger.

MOSER David, "Electron microscopy as an essential tool in electrochemistry: Development of an novel aluminum-sulfur battery", G. Kothleitner.

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

TRUMMER Cornelia, "Analytical electron tomography of metallic materials", G. Kothleitner.

WEITZER Anna, "Expanding capabilities of focused electron beam based 3D nano-printing: From meshes towards closed 3D nano-architectures", H. Plank.

SEEWALD Lukas, "Advanced AFM nanoprobe concepts via 3D Nanoprinting", H. Plank.

OBERAIGNER Michael, "Direct imaging of electron orbitals near interfaces by STEM-EELS", G. Kothleitner.

RADLINGER Thomas, "Differential phase contrast imaging in high resolution scanning transmission electron microscopy", F. Hofer, G. Kothleitner.

REISECKER Verena, "Direct-write fabrication of 3D nano-electro-mechanical-systems (3D-NEMS)", H. Plank.

Finished Master Theses

FABBRO Robert (2019), "Characterization of thin titanium nitride layers in through silicon via technology", W. Grogger.

GASSER Eva (2019), "Characterization of gas and particles released during thermal runaway of Liion batteries", G. Grogger, A. Zankel.

HINUM-WAGNER Jakob (2019), "The influence of substrate temperature during focused electron beam induced deposition", H. Plank, R. Winkler.

HJELLE Daniel (2019), "Transmission electron microscopy - electron energy-loss spectroscopy

study of lithium containing battery materials", F. Hofer, G. Haberfehlner.

OBERAIGNER Michael (2019), "Specimen preparation for electron tomography by electrochemical thinning", G. Kothleitner, G. Haberfehlner.

ZAJKI-ZECHMEISTER Krisztina (2019), "Dynamic macromolecule adsorption on structured cellulose substrates", H. Plank, S. Spirk.

STRIEMITZER Robert (2020), "Contamination-free immersion approaches for Raman microscopy", F. Hofer, H. Fitzek.

SCHÖNHUBER Benedikt (2020), "Measurement of residual stress in cemented carbides using electron backscatter diffraction", W. Grogger, S. Mitsche.

RAUCH Nikolaus (2020), "Optimization of sensitivity factor measurements for quantitative X-ray spectrometry in the TEM", W. Grogger.

Master Theses in Progress

ŠIMIĆ Nicola, "High resolution STEM simulation of beryl crystal impurities using multislice methods", F. Hofer, D. Knez.

GURKER Jakob, "Methodic optimizations in scanning electron microscopy to improve correlative electron microscopy", W. Grogger.

FRITZ Verena, "Temperature measurements on a TEM sample using PBED", W. Grogger.

ALATRASH Anas, "Advanced preparation methods for HR-TEM samples from semiconducting materials", W. Grogger.

THEISSING Moritz, "Correlative electron microscopy – combing SEM, EDX, Raman spectroscopy and microtomy", W. Grogger.

THEISSING Moritz, *"in-situ* investigations of recrystallization processes of cold rolled aluminum alloys by EBSD", F. Hofer, S. Mitsche.

BRUGGER-HATZL Michele, "High performance 3D nanoprobes for advanced electric and magnetic atomic force microscopy", H. Plank.

ZANDONELLA Robert, "Alternative contrasting agents for biological materials for application in transmission electron microscopy", W. Grogger.

HINUM-WAGNER Jakob, "Fundamental property studies of 3D nano-heaters for AFM applications", H. Plank.

GRUBER Alexander, "3D printing of functional nano-substrates for surface enhanced Raman spectroscopy" H. Plank, H. Fitzek. DISSERTATION

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MASTER TOOD

COSTORAL THESE

Access and Knowledge Transfer

Support for 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 transnational 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.

Open to all

Grant-aided Partnerships

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 & 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 on page 55).

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.

2019

24 Apr. 2019, Tag der offenen Tür TU (Zankel).

14 Nov. 2019, Girls day, FIT, Führung Lichtenfelsgymnasium (Simic, Brunegger, Lammer).

11 Nov. 2019, Führung Borg Birkfeld (Zankel, Mayrhofer).

04 Feb. 2019, Führung Chemieakademie (Letofsky-Papst, Dienstleder, Zankel, Mayrhofer)

2020

29 Jun. 2020, Landesrätin MMag.^a Barbara Eibinger-Miedl (Kothleitner).

Urania Course

For more than 15 years, the Urania workshop "Electron Microscopy" has taken place every year in November. Under the direction of Werner Grogger, colleagues from the Institute offer an introductory course in electron microscopy and its many applications in science and technology. The participants are other scientists, students, teachers or simply inquisitive people (see below).









F. Hofer and G. Kothleitner welcome Landesrätin MMag.^a Barbara Eibinger-Miedl

Events and Workshops

9th ASEM Workshop 2019

The Institute organised the annually held ASEM workshop, which provides an open forum for discussion among young and experienced electron microscopists in Austria. More than 107 researchers from academia and industry across all career levels came to the TU Graz (from April 25th to 26th, 2019). The programme included invited and contributed talks, posters and an industry exhibition. The topics ranged from microscopy research in the life sciences to materials sciences, physics and chemistry). The event also provided a good opportunity to present the ASEM Fritz Grasenick Award to the two winners Martin Niedermeier and Jinming Guo. The ASEM Workshop was organised by a student team led by Robert Krisper.



____ program

9th ASEM Workshop Advanced Electron Microscopy



er Alm Microsov

April 25th & April 26th 2019, Graz University of Technology, BMT Lecture Hall, Stremayrgasse 16, 8010 Graz

Thurse	day, April 25" 2019
1500 7	Departmention and refreshments
12:45 6	Imming ceremony
13/00	Invited Speaker, Heiko Grotff (Iotannas Kepler, University Lius) Phase Decomposition in Metastable Ge, Sn. Pployers'
33150	Folish Lammer (FELMI-ZFE Groz) - Atimic Structure Analysis of Eq. La, Fc.O, Using High-Beachdion/STEMT
13)45	Daniel Phaler (ThermoFisher Scientific) "Developments in Multiple Ian Species Plasma FIII Technology"
1100	Altice Mores (TU Wen) "Investigation of the Honoganesity in Elemental Distributions of Superconducting No.56 Wires"
1015 0	loffee break
18/85	Fritz Grosenick Lannaha: Martin Niedermezer (University of Selfburg) "Diomizoralization of Strantisers and Barton Contributos to Deloxification in the Fre- rian"
15:15	Shotaro Otsuka (University of Vienna) "Nuclear Envelope and Pore Assembly Revealed by Correlating Live Imaging With 'B Iron Microscopy"
15.30	Wolfgang Schwinger (ZEISS) "ZEN Invidious - Machine Learning Approaches for Image Segmentation APER - The Open Organi Microscopy Platform"
15/45	Silvia Groiss (Modeca: University of Graz) When Every Broth Counts: Impaired Masociliary Clearance Mean thebal Medicin
16111	office break
16(9)	Fritz Conseniók Lauronn: Jinming Guo (Ench Schmid Institute of Materials Science "Atomic-scale Councilerization of Severely Delormed Cu-basell Nanoczystulline A Floctron Microscopy"
17,00	Andrea Steitz (110faforschung Austria) "Dust in Indoor Air - What is it Composed of ?"
17:15	Georg Raggl (HEDL) "The NeoARM - IEOL's Newest High-End Atomic Resolution Microscope"
17:50	Richard Thiber (University of Genz)

- Graphie An Efficient Reconstruction Tool for Electron Tomography
- 17:45 Fritz Grasenick Assard ceremony
 - KUM Warkberg Advant Events money Gale April - April 19-100

Fast Forward 4 You

Together with the ACStyria mobility cluster, FEL-MI-ZFE organised a workshop in the series "Fast Forward 4 You". On 25 February 2019, numerous representatives from the Styrian economy came to TU Graz to learn about the latest results from electron microscopy. Under the motto "Seeing the Invisible", the institute's staff presented new examination techniques for industrial problems and the guided tour of the institute's most important research facilities enabled an in-depth discussion about the advantages of electron microscopy.



K2 Workshop

The K2 workshop was held at the Institute together with Gatan company in February 2019. Numerous electron microscopists from Austria and neighbouring countries accepted the invitation to study the advantages of the new camera technology, the "direct electron detection" camera. For this purpose, a K2 camera from Gatan was connected to the ASTEM microscope. The camera fulfilled the high expectations and was purchased by the institute in the course of a grant from the province of Styria.



60 Years Association for the Promotion of Electron Microscopy and Fine Structure Research

Due to the initiative of some Austrian industrial companies, the Association for the Promotion of Electron Microscopy was founded in November 1959. We celebrated this special occasion with a birthday party in the auditorium of Graz University of Technology. More than eighty participants came to the event on 25 June 2019.

Rector Harald Kainz, ACR President Martin Leitl and Association President Helmut List paid tribute to the achievements of the Association. The programme also included two scientific lectures by Wayne Kaplan from the Technion in Haifa, Israel and by Josef Zweck from the University of Regensburg, Germany.



Dipl.-Ing. Martin Leitl, President of the ACR



Prof. Helmut List, President of the Association



Univ.-Prof. Harald Kainz, Rector of the TU Graz





Zentrum für Elektronenmikroskopie Graz

Technische Universität Graz Rechbauerstraße 12 Aula, 1.Stock

25. Juni 2019 16:00

ab 18:30 Buffet im "Sitzungssaal des Rektorats"

Registrieren

Anmeldeschluss 21: Juni 2019

Zentrum für Elektronen mikroskopie (ZFE) Steyrergasse ay Boto Graz, Österreich Tel. +43 (0)316 833 8320 Fax +43 (0)316 831 596

email: office@felmi-zfe.at web: www.felmi-zfe.at

60 Jahre

Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung

16:00

Univ.-Prof. DI Dr.techn. Dr. h.c Harald Kainz Rektor der Technischen Universi

DI Martin Leiti ent der Austria rch (ACR), Wien

Wissenschaftliche Vorträge Prof. Dr. Wayne Kaplan

Prof. Dr. Josef Zweck Universität Regensburg, Deutschland "Pushing the limits of differential phas the discrement increase opy"

Outreach

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C Lunghammer - TU Graz

The Institute in the News

NEWS 2019

Die Presse, Jul. 20th, 2019, "Einzeller schützen sich mit Wolframrüstung", M. Albu.

Steirische Berichte, Mar. 1st, 2019, "Spitzenforschung ohne Ende", G. Kothleitner.

TU Graz people, Mar. 1st, 2019, "Fachjury zeichnete unter 101 Einreichungen die sechs besten Abschlussarbeiten der TU Graz aus", R. Winkler.

ACR-Newsletter, Sep. 16th, 2019, "Spitzenforschung 4.0 am ZFE", G. Kothleitner.

ACR-Newsletter, 9 Oct. 2019, "ACR Kooperationspreis 2019: Neues Verfahren vernichtet Bettwanzen und deren Eier", C. Mayrhofer.

APA, Oct. 28th, 2019, "Spitzenforschung 4.0 am ZFE", G. Kothleitner.

Die Presse (online), Nov. 8th, 2019, "Neues Verfahren tötet Bettwanzen im Ei", C. Mayrhofer.

Die Presse - Wissen & Innovation, Nov. 9th, 2019, "Neues Verfahren tötet Bettwanzen im Ei", C. Mayrhofer.

TUGraz - Media Service, Nov. 13th, 2019, "TU Graz-Forschende entwickeln neuen 3D-Druck zur direkten Fertigung von Nanostrukturen", H. Plank, R. Winkler, J. Sattelkow, A. Weitzer, D. Kuhness.

APA, Nov. 13th, 2019, "Nano-Printing für Rasterkraftmikroskopie: Forscher tunen 3D Druck", H. Plank, R. Winkler, J. Sattelkow, A. Weitzer, D. Kuhness.

Studium.at (online), Nov. 13th, 2019, "Nano-Printing für Rasterkraftmikroskopie: Forscher tunen 3D-Druck", H. Plank.

nanowerk (online), Nov. 13th, 2019, "Researchers develop new 3D printing for the direct production of nanostructures", H. Plank, R. Winkler, J. Sattelkow.

Factory (online), Nov. 13th, 2019, "TU Graz: Direkte Fertigung von Nanostrukturen mit 3D-Druck", H. Plank, R. Winkler, J. Sattelkow, A. Weitzer, D. Kuhness.

Chemie.de (online), Nov. 14th, 2019, "Forscher entwickeln neuen 3D-Druck zur direkten Fertigung von Nanostrukturen", H. Plank, R. Winkler, J. Sattelkow, A. Weitzer, D. Kuhness.

Die Presse - Wissen & Innovation, Nov. 16th, 2019, "Nanostrukturen aus dem 3D-Drucker", H. Plank.

WOTECH (online), Nov. 21th, 2019, "TU Graz entwickelt neuen 3D Drucker zur direkten Fertigung von Nanostrukturen", H. Plank; R. Winkler; J. Sattelkow.

ACR-Newsletter, Dec. 5th, 2019, "Das FFE Graz kooperiert mit zwei Christian Doppler Laboratorien", H. Plank, H. Groiss.



(//www.wotech-technical-media.de/womag)

TU GRAZ ENTWICKELT NEUEN 3D-DRUCK ZUR DIREKTEN FERTIGUNG VON NANOSTRUKTUREN



Beispiele aus der dreidimensional gedruckten Nanowelt zeigen die Komplexität (a), die erreichbaren Strukturgrößen (b) als auch den Weg von Gittern zu flächigen Strukturen (c), Alle Bilder sind nachträglich eingefärbte rasterelektronenmikroskopische Aufnahmen mit einem 500 nm breiten Messbalken / Bildquelle: 80 Harald Plank, Institut für Elektronenmikroskopie und Nanoanalytik der TU Graz

Im Nanometerbereich sind komplexe, freistehende 3D-Architekturen durch die erforderliche Präzision sehr schwer herstellbar. Im Christian Doppler Labor für direkte Fabrikation von 3D-Nanosonden widmen sich Wissenschafter der TU Graz deshalb den Grundlagen des 3D-Nanoprintings und entwickeln die Technologie weiter. Die Gruppe nutzt die fokussierte Elektronenstrahlabscheidung (Focused Electron Beam Induced Deposition - FEBID), die bereits bei der Fertigung komplexer, jedoch oftmals flacher Nanostrukturen erfolgreich eingesetzt wird.

Eierkappen aufgelöst

Dass die Methode tatsächlich wirksam ist, wurde vom Zentrum für Elektronenmikroskopie (ZFE) in Graz durch eine eigens entwi-ckelte Schnellmethode überprüft und bestätigt. Die Untersuchung zeigte, dass bereits wenige Stunden nach der Behandlung bei allen Schädigungssymptome Wanzen auftraten, nach sechs Tagen waren inaktiv. sämtliche Bettwanzen Auch die sonst extrem langlebigen Wanzeneier wurden nicht ver-schont, erläutert Claudia Mayrhofer vom ZFE: "Durch die Behandlung verlieren die Wanzeneier die Endkappen, was vermutlich eine Art ,Frühgeburt' auslöst." Für das neuartige Verfahren

Das erfolgreiche Team des Christian Doppler Labors für direk te Fabrikation von 3D-Nanosonden (v.I.): Anna Weitzer, David

Kuhness, Lukas Seewald, Robert Winkler, Jurgen Satte

Jakob Hinum-Wagner und Laborleiter Harald Plank

wurden Braincon und das ZFE vom Dachverband für forschende kleine und mittlere Unternehmen, dem Austrian Cooperative Re-search (ACR), mit dem Kooperationspreis 2019 ausgezeichne





An der TU Graz, Institut für Elektronenmikrösköple und Nanoanalytik, wurde 2011 eines der weltweit leistungsfähigsten Elektronenmikrösköpe installiert, das unglaublich exakte Mess sogar von einzelnen Atomen, erlaubt, in der Hand exzellenter Förscher Voraussetzung für

Yb Si MA




NEWS 2020

Der Standard, Mar. 25th, 2020, "Der 3D-Drucker für die allerkleinsten Dinge" H. Plank.

Kurier/futurezone, Apr. 14th, 2020, "Filter saugen Viren aus der Luft", J. Rattenberger, G. Ettenberger-Bornberg, N. Novotny.

Der Standard, Apr. 29th, 2020, "Forschung schnappt nach Luft" J. Rattenberger.

TU Graz people, 75/2020-3, "Neuberufung-TU-Graz, Gerald Kothleitner".

ACR-Newsletter, Jul. 1st, 2020, "ZFE: Wie man Viren sichtbar macht", F. Hofer.

ACR Wissen-Web, Aug. 15th, 2020, "Leistungsfähige und langlebige Brennstoffzellen? Auf das richtige Material kommtes an!", J. Lammer.

TU Graz Media Service, Sep. 25th, 2020, "Houska-Preis 2020 an TU Graz-Forscher Harald Plank".

Kronen Zeitung/Forschung, Sep. 26th, 2020, "Einzigartiger 3D-Nanodruck", H. Plank.

TU Graz Media Service, Oct. 7th, 2020, "Neue Erkenntisse ebnen den Weg zu umweltfreundlichen Superkondensatoren", C. Prehal, Q. Abbas, H. Fitzek.

Kleine Zeitung/Steirer des Tages, Oct. 8th, 2020, "Ultrafeine Sitzen, preisgekrönt", H. Plank.

Die Presse, Nov. 28th, 2020, "Corona und die Hygiene im Reisebus", J. Rattenberger, G. Ettenberger-Bornberg.

ACR-Newsletter, Dec. 16th, 2020, "ZFE: Online-Mikroskopie Lösung ermöglicht Kooperation auf Distanz", A. Zankel.

ACR-Newsletter, Dec. 16th, 2020, "ZFE: SolaBat-Solarzellen trifft Batterie", G. Haberfehlner.

Ural Federal University-News, Dec. 17th, 2020, "Ural and Austrian Scientists Studied Multicomponent Nanomaterials For Bone Implants", H. Fitzek.



CHEMIE.DE



Forscher entwickeln neuen 3D-Druck zur direkten Fertigung von Nanostrukturen

14.11.2019 - Einem Team der TU Graz ist es gelungen, mithilfe der FEBID-Methode erstmals kom plexe 3D-gedruckte Nano-Bauteile ohne zusätzliche Stützstrukturen zu fertigen

bei der Fertigung komplexer, jedoch oftmals flache Nanostrukturen erfolgreich eingesetzt wird.

NEWS

Mehr Effizienz und mehr Möglichkeiten

Einzeller schützen sich mit Wolframrüstung

Mikrobiologie. Urtümliche Mikroorganismen können in den extremsten Lebensräumen gedeihen. Nun wurde erstmals gezeigt, dass sie auch Wolfram für ihren Stoffwechsel nutzen es könnte sie fit für den Weltraum machen.

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VON WOLFGANG DAURLE unter dener trifft, si

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den Zellen Neues Verfahren tötet Bettwanzen im Ei sbekämpfung. Mit einer Mischung aus Nervengift und Sauerstoffradikalen sowie speziellen Zerstäubern will ein Wiener Unternehmen die immer häufiger vorkommenden Blutsauger in allen Entwicklungsstadien bekämpfen.

WOLFGANG DAURLE en finden in Rucksä ern, in Sofas, Matra

Houskapreis-Gewinner 2020 in der Kategorie "Hochschulforschung"



Gift

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Der 3D-Drucker für die



Awards

Houska-Prize 2020

Harald Plank was awarded the prestigious Houska Prize in the University Research category on 24 September 2020. The Houska Prize is the most important private research prize in Austria, awarded annually by the B&C Private Foundation. The main prize of 150,000 euros was presented to Harald Plank by the foundation's chairman Erich Hampel in a festive ceremony in Vienna.

The researchers led by Harald Plank developed a novel 3D nanoprinting technology, the so-called FEBID technology (Focused Electron Beam Induced Deposition), for the production of complex, three-dimensional objects in the nanoscale. This new method is intended to open up new areas of application for industry that were previously not possible with conventional 3D printing processes. See more details of Harald's research results in the chapter "Research Highlights" of this Report. The results of Harald's research group were partly developed in the Christian Doppler Laboratory DEFINE and also supported by the Austrian Cooperative Research.

ACR Cooperation Award 2019

New process destroys bed bugs and their eggs

Claudia Mayrhofer received the ACR Cooperation Award for her research work, which was carried out in cooperation with the company Braincon from Vienna. Claudia Mayrhofer was able to clearly demonstrate that a new technology not only effectively kills adult bed bugs, but also their eggs and nymphs. With elaborate microscopic examinations, Claudia Mayrhofer and Manfred Nachtnebel were able to show that with the new chemical treatment the bed bug eggs lose their end caps, which presumably triggers a kind of "premature birth" of the nymphs. This leads to the death of the young bed bugs.

The award was presented by ACR President Martin Leitl at a festive ceremony in the Julius-Raab-Hall of the Austrian Chamber of Commerce in Vienna on 8 October 2019.





Graduate Student Award 2019

This special distinction was presented to **Robert Winkler** at the E-MRS conference in Warsaw Poland in recognition of his outstanding paper entitled "3D Nanoprinting with Electron Beams – Growth Characteristics, Simulations and Applications".

And the Promotion Award 2019 goes to

Robert Winkler for his PhD Thesis with the title "Fabrication of Functional and Freestanding 3D Nano-architectures via Focused Electron Beam Induced Deposition". Every year, the Forum Technology and Society at TU Graz awards prizes for socially relevant master's or doctoral theses. Out of 101 submissions, the expert jury selected Robert's research work as the best doctoral thesis of 2019.

WITec Paper Award 2020

Ruth Schmidt received the Paper Award Bronze for her paper "The Combination of Electron Microscopy, Raman Microscopy and Energy Dispersive X-Ray Spectroscopy for the Investigation of Polymeric Materials" published in Macromolecular Symposia in 2019.

Every year, the WITec Paper Award competition recognizes three exceptional peer-reviewed publications that feature results acquired with a WITec microscope. A record number of 113 publications was submitted 2020 from different fields of application such as cancer research, electrochemistry, semiconductor research, geology and microplastics research.

WKO Scholarship 2019

In 2019 Jakob **Hinum-Wagner** received a scholarship sponsored by the Styrian Chamber of Commerce for his research work "Characterization of 3D nano-heating elements for atomic force microscopy". This scholarship is awarded for economically relevant Master Thesis projects.





Around the World

Teaching at the Aalto University

Claudia Mayrhofer is our expert for ultramicrotomy and examines plastics and biological samples. Based on her experience, she was invited by Professor Herbert Sixta from the Institute of Bioproducts and Paper Technology in Otaniemi in Espoo to teach a one-week ultramicrotomy course at Aalto University (May 27 to June 1, 2019). In this course, Claudia taught the basics of preparing plastics, fibres, pulp and paper and examining these samples under the electron microscope.



Claudia Mayrhofer (second from right) with the members with Professor Sixta's group.

Fourteen Months in Trieste

In April 2019, Daniel started a postdoctoral position at the Istituto Officina del Materiali of the Italian National Research Council (CNR), which is situated at the Synchroton ELLETRA in Trieste, Italy. He was appointed to conduct research in the field of complex oxide heterostructures by using advanced transmission electron microscopy techniques. Daniel applied automated, quantitative data analysis methods and the data interpretation was supplemented by atomistic simulations techniques, such as Molecular Dynamics and Density Functional Theory, combined with Multislice simulations of high resolution TEM images. The results of his work led to several publications, including one in the well-known journal Nano Letters.



Daniel Knez with his colleagues Dr. Piu Rajak (left) and the group leader Regina Ciancio (middle).



Belgrade Calling

In September 2019, a delegation of ten Institute staff attended the 14th Multinational Congress on Microscopy in Belgrade, Serbia. The colleagues presented two invited talks, contributed lectures and posters in several sessions of this important event with more than 500 participants, who came from all over Europe.

ACR Delegation in England

In early June 2019, a 35-member ACR delegation travelled to the United Kingdom. The group consisted of senior staff from the 18 ACR institutes, ministries and representatives of Austrian media. In order to see how English research institutions work on the front lines of research and development, some important institutes and technology policy institutions were visited. Ferdinand Hofer and Hartmuth Schröttner from FELMI-ZFE took part in this study tour. The delegation visited the UK Research and Innovation Forum and the Enterprise Department at the Imperial College in London. The next day the programme included facilities in Cambridge, the Department of Engineering at the University of Cambridge and the Bradfield Centre at the Cambridge Science Park.

Research Stay at the Goethe University

In Summer 2019, Harald Plank completed a threemonth research stay at the Goethe University in Frankfurt am Main in Germany. Due to the longstanding successful cooperation with Professor Michael Huth from the Institute of Physics, the research work focused on free-standing nanostructures, which were produced using the 3D nanoprinting process partially developed in Graz. With this FEBID method (see Research Highlights), three-dimensional nano-probes can be fabricated for applications in the field of nanotechnology and for use in scanning probe microscopy. The results from Frankfurt are important for the further development of Harald's Christian Doppler Laboratory DEFINE.





Robert Winkler. Harald Plank and Jürgen Sattelkow (from left to right)

Transfer Network Center in London

Gerald Kothleitner and Ferdinand Hofer visit the high voltage electron microscope facility of the Stuttgart Center for Electron Microscopy.





Team and Organization

Organization until September 2020



Organization after October 2020

Central Services

Administration

Mag. Ulrike Stürzenbecher Sabine Mitterbacher Silke Winkler Sabine Goger

Nanoanalysis TEM

Head: Prof. Gerald Kothleitner Dr. Georg Haberfehlner Dr. Daniel Knez DI Thomas Radlinger DI Cornelia Trummer DI David Moser DI Micheal Oberaigner Margit Brunegger Martina Dienstleder Arnela Blažević Paul Fastian

IT / Media

Ing. Albert Krisper Margit Wallner Mira Plangger

High Resolution TEM

Head: Prof. Werner Grogger Dr. Evelin Fisslthaler DI Judith Lammer DI Robert Krisper Gerhard Birnstingl Markus Sittsam

Management

Prof. Gerald Kothleitner Prof. Werner Grogger Prof. Ferdinand Hofer

Soft Materials & Metallurgy TEM

Head: Dr. Ilse Letofsky-Papst Dr. Mihaela Albu Dr. Karin Wewerka Ing. Claudia Mayrhofer

SEM Microanalysis & in-situ Methods & IR | Raman Head: Ing. Hartmuth Schröttner

lead: Ing. Hartmuth Schröttn Dr. Stefan Mitsche Dr. Armin Zankel Dr. Johannes Rattenberger Dr. Harald Fitzek Mag. Ruth Schmidt Mag. Thomas Planko Sanja Šimić Sabrina Mertschnigg Christian Brandl Anita Rossmann-Perner

Functional Nanofabrication FIB | AFM

Head: Prof. Harald Plank Dr. Robert Winkler Dr. David Kuhness DI Jürgen Sattelkow Mag. Anna Weitzer DI Lukas Seewald DI Verena Reisecker Ing. Sebastian Rauch



Masterstudents





Jakob Hinum-Wagner









Michele Krisztina Brugger-Hatzl Zajki-Zechmeister





Verena Fritz







Theissing

Moritz Robert



Striemitzer



Schönhuber

Nicola Šimić

Nikolaus Rauch

Jakob Gurker

Alexander Gruber

DI Anna Weitzer

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Sabine Goger

DI Verena Reisecker







Laboratory Facilities

Due to the experimental focus of the institute, the scientific infrastructure is a crucial core element for successful research, the support of other research groups and for industrial cooperation. The Institute is pursuing the strategy of building up internationally outstanding instrumentation in order to be able to keep up well with European programmes such as ESTEEM. In the reporting period, two new scanning electron microscopes and a focused ion beam facility were introduced. The ASTEM microscope was equipped with a highly sensitive "Direct Electron Detector".

Transmission electron microscopes (TEMs)



ASTEM FEI Titan³ 60-300 G2 Atomic resolution TEM/STEM, 60-300 kV, X-FEG with monochromator, ABF, HAADF and DPC detectors, C_s-probe corrector (DCOR), Lorentz lens, Super-X four quadrant X-ray detector, imaging filter/spectrometer Quantum ERS (Gatan), fast parallel EELS & EDX mapping, direct electron detector K2 (Gatan), resolution 0.07 nm, installation 2010-2011.



Bio TEM FEI Tecnai 12

TEM 80-120 kV with LaB₆ cathode, low-dose CCD (Gatan), cryotransfer system (Gatan), Plunge freezer Leica EMPG (Leica), installation 2003-2004.



Bio-TEM FEI Tecnai F20

High-resolution TEM/STEM, 80-200 kV, Schottky-FEG, with Wien filter monochromator, HAADF detector, EDX spectrometer (EDX), imaging filter/spectrometer Quantum HR (Gatan), resolution 0.2 nm, installation 2001-2002.



TEM Philips CM20

Analytical TEM/STEM 80-200 kV, with LaB_6 cathode, with HPGe EDX-detector (Noran) and imaging filter/spectrometer (Gatan), resolution 0.24 nm, installation 1990-1991.

TEM Specimen Holders

Gatan Cryo-specimen Holder

Gatan Vacuum Transfer Holder (Model 648 double-tilt)

MEMS Heating and Biasing Stage (DENSsolution Wildfire D6)

Fischione Tomography Holder

Focused Ion Beam Microscopes (FIBs)



FIB FEI NOVA200

Dual Beam FIB-SEM, Schottky-FEG and Ga-ion source, micromanipulator (Omniprobe), various gas injection systems (Pt, Au and SiO₂ deposition), SDD X-ray detector (XFlash, Bruker), *in-situ* AFSEM (GETec), Kleindieck microtools (rotation needle, gripper, Gas injector), installation 2003-2004.



FIB FEI QUANTA 3D NEW!

Dual Beam FIB-SEM, Schottky-FEG and Ga-ion source, with low-vacuum mode, SDD X-ray spectrometer XL-30 (EDAX), gas injection system for Pt deposition, *in-situ* AFSEM (GETec), Peltier cooling stage (home built), Kleindiek microtolls, installation 2019-2020.

Scanning Electron Microscopes (SEMs)



LV-SEM Zeiss Sigma 300 RISE NEW!

Low vacuum SEM, Schottky-FEG, SDD X-ray detector (XMax 80, Oxford), confocal Raman spectrometer (RISE, Witec), installation 2018-2019.



ESEM FEI Quanta 450 NEW!

Environmental SEM (ESEM), 0.1-30 kV, Schottky-FEG, SDD X-ray spectrometer (Thermo Scientific), macro navigation camera, installation 2019.



HR-SEM Zeiss Ultra55

Analytical high resolution SEM, Schottky-FEG, 0,1-30 kV, STEM-, ESB-, AsB- and in-lens detectors, Si(Li) EDX-detector Genesis (EDAX), WDX-detector Lambda-Spec (EDAX), EBSD-detector (EDAX), installation 2006.

HR-SEM ZEISS Gemini DSM986

Analytical high resolution SEM, 0.1-30 kV, Schottky-FEG, Si(Li) EDX-spectrometer (Vantage, Noran), installation 1997-1999.



ESEM FEI Quanta 600

in-situ environmental SEM (ESEM), 0.1 -30 kV, Schottky-FEG, large specimen chamber, SDD X-ray spectrometer (EDAX), Peltier cooling and wetting stage, heating stage up to 1300°C, tensile testing stage (Kammrath & Weiss), 3View ultramicrotome (Gatan), installation 2001-2003.

Light and Scanning Probe Microscopes



AFM Bruker FastScan Bio in-situ atomic force microscope with PeakForce probes, installation 2015.



Siemens XRD D5005

X-ray diffractometer with heating stage up to 1200oC (Anton Paar), installation 2014.



Horiba LABRAM microscope Confocal Raman microscope with Olympus BX41, red, blue and green lasers and DuoScan imaging system, installation 2010.



Bruker FT-IR microscope

Infrared microscope Hyperion 3000 with Tensor 27 FT-IR spectrometer, ATR and GIR objective, FPA and MCT detectors, installation 2015.



Zeiss Axioplan

Light microscope, transmission and reflection, polarization, interference and phase contrast mode, with IcC1 camera, installation 1995.



Alicona Infinite Focus G3

Focus variation light microscope as optical 3D measurement system, for profilometry and roughness measurements, installation 2013.

Specimen Preparation Instrumentation

Fischione Nanomill 1040

Ultra low energy Ar ion mill with cryo-stage for TEM sample preparation, installation 2015.

Gatan PIPS II- Model 695

Broad Ar ion mill with cryo-stage for TEM sample preparation, installation 2018.

Allied HighTech - Multiprep

Semiautomatic grinding and polishing system for SEM and TEM preparations, installation 2011.

Gatan Illion Slope Cutter - Model 693

Broad Ar ion mill with cryo-stage for SEM sample preparation, installation 2012.

Struers Tegramin25

Semiautomatic grinding and polishing system for SEM preparation, installation 2007.

Leica EM ACE200

High vacuum carbon sputter coating system, installation 2016.

Leica EM ACE600

High vacuum metal thin film sputter coater, installation 2016.

Leica Ultracut UCT with cryochamber FCS (liquid N₂)

Leica EM UC6-NT

with Leica IC90€ Camera (NEW) and cryochamber EM FC6 (liquid $\rm N_{2})$

Leica EM Trim

The Basis of the FELMI – ZFE | Performance Report

Publications

Publishing Activities

Peer Reviewed Publications 2019

Abbas, H.; Nadeem, K.; Saeed, N.; Hassan, A.; Rahman, S.; Krenn, H.; Letofsky-Papst, I.: "*Photocatalytic activity and two-magnon behavior in nickel oxide nanoparticles with different silica concentration*", J. Appl. Phys. 125 (2019) 144305.

Abbas, Q.; Fitzek, H. M.; Schröttner, H.; Dsoke, S.; Gollas, B.: "*Immobilization of polyiodide redox species in porous carbon for battery-like electrodes in eco-friendly hybrid electrochemical capacitors*", Nanomaterials *9* (2019) 1413.

Astria, E.; Thonhofer, M. S.; Ricco, R.; Liang, W.; Angela, C.; Tarzia, A.; Alt, K.; Hagemeyer, C.; Rattenberger, J.; Schröttner, H.; Wrodnigg, T. M.; Amenitsch, H.; Huang, D.; Doonan, C.; Falcaro, P.: "*Car-bohydrates@MOFs*", Materials Horizons 6 (2019) 969.

Baldermann, C.; Baldermann, A.; Furat, O.; Krüger, M.; Nachtnebel, M.; Schroettner, H.; Juhart, J.; Schmidt, V.; Tritthart, J.: "*Mineralogical and microstructural response of hydrated cement blends to leaching*", Construction and Building Materials 229 (2019) 2697.

Berger, C.; Egger, A.; Merkle, R.; Bucher, E.; Stuhlhofer, B.; Schrödl, N.; Lammer, J.; Gspan, C.; Maier, J.; Sitte, W.: "Oxygen surface exchange kinetics of $Pr_2(Ni,Co)O_{4+\delta}$ thin-film model electrodes", J. Electrochemical Soc. 166 (2019) 14, F1088.

Berger, C.; Bucher, E.; Gspan, C.; Sitte, W. "Crystal structure, oxygen nonstoichiometry, and mass and charge transport properties of the Sr-free SOFC/SOEC air electrode material $La_{0.75} Ca_{0.25} FeO_{3-5}$ ", J. Solid State Chem. 273 (2019) 92.

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Bossu, J.; Eckhart, R.; Czibula, C.; Winter, A.; Zankel, A.; Bauer, W.: "Fine cellulosic materials produced from chemical pulp: The combined effect of morphology and rate of addition on paper properties", Nanomaterials 9 (2019) 321.

Braun, J.; Kaserer, L.; Stajkovic, J.; Leitz, K. H.; Tabernig, B.; Singer, P.; Leibenguth, P.; Gspan, C.; Kestler, H.; Leichtfried, G.: "*Molybdenum and tungsten manufactured by selective laser melting : Analysis of defect structure and solidification mechanisms*", Int. J. Refractory Metals and Hard Materials 84 (2019) 104999.

Cermenek, B.; Ranninger, J.; Feketeföldi, B.; Letofsky-Papst, I.; Kienzl, N.; Bitschnau, B.; Hacker, V.: "Novel highly active carbon supported ternary PdNiBi nanoparticles as anode catalyst for the alkaline direct ethanol fuel cell", Nano Research 12 (2019) 683.

Chokki, J.; Darracq, G.; Pölt, P.; Baron, J.; Gallard, H; Joyeux, M.; Teychene, B.: "Investigation of poly(ethersulfone)/polyvinylpyrrolidone ultrafiltration membrane degradation by contact with sodium hypochlorite through FTIR mapping and two-dimensional correlation spectroscopy", Polymer Degradation and Stability 161 (2019) 131 – 138.

Egger, A.; Perz, M.; Bucher, E.; Gspan, C.; Sitte, W.: "Effect of microstructure on the degradation of $La_{o.5}Sr_{o.4}CoO_{3-5}$ electrodes in dry and humid atmospheres", Fuel Cells 19 (2019) 458.

Falkinger, G.; Regl, K.; Mitsche, S.: *"Recrystallized cube grains in an Al–Mg–Si alloy dependent on prior cold rolling"*, Mat. Sci. Techn. 35 (2019) 1081.

FissIthaler, E.; Haberfehlner, G.; Gspan, C.; Gruber, G.; Grogger, W.: "High-resolution cross-sectional analysis of the interface between SiC and SiO₂ in a MOSFET device via atomic resolution STEM", Microelectronics Reliability (2019) 100. Gaber, C.; Demuth, M.; Prieler, R.; Schluckner, C.; Schroettner, H.; Fitzek, H.; Hochenauer, C.: *"Experimental investigation of thermochemical regeneration using oxy-fuel exhaust gases"*, Applied Energy 236 (2019) 1115.

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Gößler, M.; Albu, M.; Klinser, G.; Steyskal, E.-M.; Krenn, H.; Würschum, R.: "Magneto-ionic switching of superparamagnetism", Small 15 (2019) 1904523.

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Lectures 2019

Albu, M., "HRSTEM analytics for material science", University of Vienna, Vienna, Austria, May 10, 2019.

Albu, M., "Advanced quantitative transmission electron microscopy: materials research in several dimensions", E-MRS 2019 Spring Meeting, Nizza, France, May, 28, 2019.

Albu, M., *"Multiscale and correlative analytical electron microscopy of extra-terrestrial minerals"*, University of Vienna, Vienna, Austria, Sep. 27, 2019 (invited).

Albu, M., "*Ex- and in-situ high-resolution microstructure investigations of powders and additive manufactured parts*", LightMAT 2019: 3rd Int. Conf. on Light Materials – Science and Technology, Manchester, U.K., Nov. 6, 2019.

Albu, M., "Ex- and in-situ high-resolution microstructure investigations of powders and additive manufactured parts", Metal Additive Manufacturing Congress, Oerebroe, Sweden, Nov. 25, 2019.

FissIthaler, E., "High-resolution cross-sectional analysis of the interface between SiC and SiO₂ in a MOSFET device via atomic resolution STEM", 30th Eur. Symp. on Reliability of Electron Devices, Failure Physics and Analysis, Toulouse, France, Sep. 23, 2019.

FissIthaler, E., *"In-situ observation of precipitate growth and decomposition in AlCu4 during heat treatment via analytical STEM"*, 14th Multinational Congress on Microscopy, Belgrade, Serbia, Sep. 20, 2019. Fitzek, H., "Correlative Raman-SEM-EDX microscopy – examples of application", 10th International Conference on Advanced Vibrational Spectroscopy, Auckland, New Zealand, Jul. 12, 2019 (invited).

Fitzek, H., "Correlative Raman microscopy, SEM and EDX – fundamentals and applications", Seminar Modern Analytical Chemistry, Vienna, Austria, Nov. 22, 2019 (invited).

Fitzek, H., "Correlative Raman microscopy, SEM and EDX-examples of application and best practice", 14th Multinational Congress on Microscopy, Belgrade, Serbia, Sep. 17, 2019 (invited).

Haberfehlner, G., "Nanoscale 3D measurements by electron tomography", 7th Dresden Nanoanalysis Symposium, Dresden, Germany, Aug. 30, 2019 (invited).

Haberfehlner, G., "Applications of spectroscopic electron tomography", Microscopy Conference 2019, Berlin, Germany, Sep. 4, 2019 (invited).

Hinum-Wagner, J., "The influence of substrate temperature during focused electron induced deposition (FEBID)", Eur. Mat. Res. Soc., Warsaw, Polen, Sep., 17, 2019.

Hofer, F., "Electron beam induced dynamics in experiment and simulation", Microscopy Conference 2019, Berlin, Germany, Sep. 5, 2019.

Hofer, F., *"Analytical electron microscopy: Recent advances and a pplications"*, 14th Multinational Congress on Microscopy, Belgrade, Serbia, Sep. 19, 2019 (invited).

Knez, D., "Atoms in motion: On the impact of swift electrons on mass transport phenomena at the nanoscale", University of Venna, Vienna, Austria, Jun. 28, 2019 (invited).

Knez, D., "Atoms in motion: Electron beam induced dynamics in experiment and simulation", Fritz-Haber-Institute Berlin, Germany, Dec. 12, 2019.

Kothleitner, G., "Electron Tomography of Nanomaterials", 19th TEM-UCA, University of Cadiz, Spain, Sep. 16, 2019 (invited).

Krisper, R., "In-situ TEM microstructure study of AlSi10Mg powder for 3D-printing", Microscopy Conference 2019, Berlin, Germany, Sep. 5, 2019.

Krisper, R., "Measuring the Temperature Homogeneity Across FIB Lamellae for In-Situ TEM Experiments via Raman Scattering in Crystalline Silicon", 14th Multinational Congress on Microscopy, Belgrade, Serbia, Sep. 20, 2019.

Lammer, J., "Atomic structure analysis of $Ba_{1.1}La_{1.9}Fe_2O_2$ using highresolution STEM", 9th ASEM Workshop for Advanced Microscopy, Graz, Austria, Apr. 25th, 2019.

Lammer, L., "Atomic structure analysis of a 2nd order Ruddlesden-Popper ferrite – a high-resolution STEM study", E-MRS 2019 Spring Meeting, Nizza, France, May 29, 2019.

Moser, D., *"Develomant of new alloys for secondary aluminum batteries"*, 9th ASEM Workshop for Advanced Microscopy, Graz, Austria, Apr. 26, 2019.

Moser, D., "Electron microscopy as an essential tool in electrochemistry: Assessing the surface morphology of Al electrodes", 14th Multinational Congress on Microscopy, Belgrade, Serbia, Sep. 18, 2019. Nachtnebel, M., "Correlative and automated particle analysis by SEM, EDX and Raman: Features and limitations", 6th Particle Forum 2019, Leoben, Austria, Apr. 29, 2019.

Plank, H., "Advances in 3D nanoprinting via electrons", Nanotechnology & Materials Science 2019, Amsterdam, Netherlands, Apr. 25, 2019 (invited).

Plank, H., "Direct-write 3D nanoprinting", NanoNetwork 2019, Tjome, Norway, Jun. 12, 2019 (invited).

Plank, H., "3D nanoprinting: Advances and applications", Eur. Mat. Res. Soc., Warsaw, Polen, Sep. 17, 2019 (invited).

Rattenberger, J., "Improvements in environmental scanning electron microscopy - Universal pressure scanning electron microscopy (UPSEM)", 14th Multinational Congress on Microscopy, Belgrade, Serbia, Sep. 16, 2019.

Rattenberger, J., "Broad ion beam cross-section preparation on nitrocarburised and oxidised steel samples to correlate microstructure and tribological behaviour", 14th Multinational Congress on Microscopy, Belgrade, Serbia, Sep. 16, 2019.

Sattelkow, J., "3D nanoprobes for AFM based thermal microscopy in electron microscopes", 9th ASEM Workshop for Advanced Microscopy, Graz, Austria, Apr. 25, 2019.

Sattelkow, J., "3D nanoprobes for scanning thermal microscocpy in electron/ion beam microscopes", Eur. Mat. Res. Soc., Warsaw, Polen, Sep. 18, 2019.

Schmidt, R., "Correlative microscopy: SEM, Raman, EDX", 9th ASEM Workshop for Advanced Microscopy, Graz, Austria, Apr. 26, 2019.

Schmidt, R., "Correlative microscopy - The new system RISE combined with EDXS applied on complex materials", Microscience Microscopy Congress, Manchester, U.K., Jun. 9, 2019.

Schröttner, H., "Microbiologically influenced corrosion (MIC) of steel - A study using correlative SEM,EDX and Raman microscopy", 14th Multinational Congress on Microscopy, Belgrade, Serbia, Sep. 18, 2019.

Trummer, C., "Analytical electron tomography of metallic samples", 14th Multinational Congress on Microscopy, Belgrade, Serbia, Sep. 19, 2019.

Weitzer, A., "Controlled 3D nanoprinting of closed and semi-closed nanoarchitectures", Eur. Mat. Res. Soc. Warsaw, Polen, Sep. 17, 2019.

Wewerka, K., Haberfehlner, G., Plank H., Zankel, A., "Recent developments at FELMI-ZFE - Beyond conventional microscopy", University of Leoben, Leoben, Austria, May, 7, 2019.

Winkler, R., "Advances in direct-write 3D nanoprinting", DPG Spring Meeting 2019, Regensburg, Germany, Apr. 3, 2019 (invited).

Winkler, R., "3D nanoprinting with electron beams – Growth characteristics, simulations and applications", Eur. Mat.Res.Soc., Warsaw, Polen, Sep. 17, 2019.

Zankel, A., "Raman Imaging and scanning electron microscopy (RISE) combined with EDX - correlative microscopy for different fields of science and technology", Microscopy Conference 2019, Berlin, Germany, Sep. 4, 2019 (invited).

Zankel, A., "The combination of electron microscopy, Raman microscopy and energy dispersive X-ray spectroscopy. Examples from materials science ", 16th Confocal Raman Imaging Symposium, Ulm, Germany, Sep. 24, 2019.

Zankel, A., "Core Facility Cell Imaging and Ultrastructure Research (CIUS)", Workshop New Concepts in Light and Electron Microscopy, Vienna, Austria, Dec. 12, 2019 (invited).

Zankel, A., "*Electron tomography*", Solid State Seminar, University of Graz, Austria, Dec. 18, 2019 (invited).

Lectures 2020

Albu, M., "Correlative microscopy for the investigation of powders and additive manufactured parts", Metal Additive Manufacturing Conf. 2020, Vienna, Austria, Oct. 2, 2020.

Hofer, F., "Analytical electron microscopy: Recent advances & applications", University of Salzburg, Austria, Jan. 30, 2020 (invited).

Kothleitner, G.: *"EDX and EELS chemical analysis"*, NECEM Summer School: Electron Microscopy for Energy Materials 2020, U.K., Sep. 2, 2010 (invited).

Lammer, J., *"Quantifying ordering phenomena in lanthanum barium ferrate at the atomic scale"*, Virtual Early Career Eur. Micr. Congr. 2020, U.K., Nov. 25, 2020.

Knez, D., "Structure of periodic oxygen vacancy arrays in anatase thin films", Virtual Early Career Eur. Micr. Congr. 2020, U.K., Nov. 26, 2020.

Krisper, R., *"In-situ structural analysis of AISi10Mg for additive manufacturing – from powder to thermally treated parts"*, Virtual Early Career Eur. Micr. Congr. 2020, U.K., Nov. 24, 2020.



Poster Presentations 2019

Albu, M.; Zankel, A.; Fitzek, H. M.; Dienstleder, M.; Simic, S.; Hofer, F.; Kothleitner, G.: *"Multiscale and correlative analytical electron microscopy of extra-terrestial minerals"*, 4th Workshop German Astrobiological Society, Vienna, Austria, September 26, 2019.

Fitzek, H. M.; Napetschnig, M.; Pramberger, I.; Dejan D.; Schmutzler, S. J.; Absenger, K.; Spettel, J.; Wieland, D.; Blazevic, A.: "*Raman microscopy of inorganic materials – A stone is a stone! Is a stone?*", Advanced Materials Day 2019, Graz, Austria, October 24, 2019, Graz, Austria.

Fisslthaler, E.; Haberfehlner, G.; Grogger, W.: "Investigation of interface transition chemistry in semiconductor structures via monochromated EELS", 9th ASEM Workshop for Advanced Microscopy, Graz, Austria, April 26, 2019.

Hinum-Wagner, J.; Winkler, R.; Birnstingl, G.; Plank, H.: "*The influence of substrate temperature during focused electron beam induced deposition*", Advanced Materials Day 2019, Graz, Austria, Octtober 24, 2019.

Hinum-Wagner, J.; Winkler, R.; Birnstingl, G.; Plank, H.: "*The influence of substrate temperature during focused electron beam induced deposition (FEBID)*", 9th ASEM Workshop for Advanced Microscopy, Graz, Austria, April 25, 2019..

Hobisch, M.; Mueller, D.; Fischer, W. Johann; Zankel, A.; Bauer, W.; Eckhart, R.; Zabler, S.; Spirk, S.: "Aluminium-silicate-hydratealuminium-silicate-hydrate localization of cellulosic fines in paper sheets", 15th Mini Symposium Chemical Engineering, Leoben, Austria, April 29, 2019.

Krisper, R.; Fitzek, H. M.; Fisslthaler, E.; Grogger, W.: "Measuring the temperature homogeneity across FIB lamellae for in-situ TEM experiments via Raman scattering in crystalline silicon", 9th ASEM Workshop for Advanced Microscopy, Graz, Austria, April 24, 2019.

Kuhness, D.; Sattelkow, J.; Winkler, R.; Plank, H.: "Towards highfidelity gold nano-antennas using 3D-nanoprinting", Eur. Mater. Res. Soc. Conf., Warzaw, Poland, September 17, 2019.

Landler, A.; Baldermann, A.; Mittermayr, F.; Letofsky-Papst, I.; Steindl, F. R.; Galan, I.; Dietzel, M.: "Incorporation of metal ions (Co, Cr and Zn) during calcium-aluminium-silicate-hydrate and trioctahedral smectite formation", MinPet 2019, Graz, Austria, September 5, 2019.

Moser, D.; Steiner, S.; Gollas, B.; Kothleitner, G.: *"Electron micros-copy characterization of the aluminium anode of a novel recharge-able aluminium-sulfur battery"*, 9th ASEM Workshop for Advanced Microscopy, Graz, Austria, April 25, 2019.

Nachtnebel, M.; Steitz, A.; Munoz-Czerny, U.; Mantler, J.; Grüll, G.; Schröttner, H.: "*Correlative analysis of dust particels by SEM, EDXS and Raman*", 14th Multinational Congress on Microscopy, Belgrad, Serbia, September 16, 2019.

Planko, T.; Fitzek, H. M.; Eichinger, S.; Koraimann, G.; Schröttner, H.: "Aluminium-silicate-hydrate-aluminium-silicate-hydrate microbiologically influenced corrosion (MIC) of steel - A study using correlative SEM, EDX and Raman microscopy", Advanced Materials Day 2019, Graz, Austria, October 24, 2019.

Preissegger, V.; Baldermann, A.; Landler, A.; Steindl, F. R.; Letofsky-Papst, I.; Simic, S.; Eichinger, S.; Dietzel, M.: "Incorporation of heavy metals (Co,Cu,Zn) in synthetic calcium-aluminium-silicatehydrate (C-A-S-H) gel structures: Environmental implications for metal ion transport in aqueous media", MinPet 2019, Graz, Austria, September 5, 2019.

Prem, B.; Czibula, C.; Saf, R.; Supplit, R.; Holzner, A.; Mayrhofer, C.; Teichert, C.; Trimmel, G.: *"Influence of the blend ratio on the mechanical properties of elastomer-thermoplastics blends"*, Int. Rubber Conf., London, U.K., September 4, 2019.

Purgstaller, B.; Letofsky-Papst, I.; Götschl, K.; Mavromatis, V.; Dietzel, M.: "Nanoscale investigation of amorphous Ca-Mg carbonate by STEM", MinPet 2019, Graz, Austria, September 4, 2019.

Rattenberger, J.; Nachtnebel, M.; Führer, B.; Kirchnawy, C.; Kaufmann, L.; Hartl, C.; Schröttner, H.; Ettenberger-Bornberg, G.: *"Immunogold labeling of allergens on fine dust filters for SEM investigation"*, Microscopy Conference 2019, Berlin, Germany, September 4, 2019.

Rattenberger, J.; Fitzek, H. M.; Achtsnit, T.; Schröttner, H.; Hofer, F.: "Universal pressure scanning electron microscopy (UPSEM) - Improvements in environmental scanning electron microscopy", Microscopy Conference 2019, Berlin, Germany, September 4, 2019.

Schmidt, R.; Nachtnebel, M.; Fitzek, H. M.; Mayrhofer, C.; Schröttner, H.; Zankel, A.: "*Investigation of soft matter materials with the new system RISE combined with EDXS*", Advanced Materials Day, Graz, Austria, October 10, 2019.

Steiner, S.; Moser, D.; Kothleitner, G.; Gollas, B.: "Aluminium-silicate-hydrate morphology control of aluminium anodes in secondary aluminium batteries", 70th Ann. Meeting Int. Soc. Electrochemistry (ISE), Durban, South Africa, August 1, 2019.

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", 9th ASEM Workshop for Advanced Microscopy, Graz, Austria, April 26, 2019.

Weitzer, A.: *"From meshes towards closed 3D nano-architectures"*, 9th ASEM Workshop for Advanced Microscopy, Graz, Austria, April 26, 2019.

Zankel, A.; Fitzek, H. M.; Mayrhofer, C.; Nachtnebel, M.; Schmidt, R.; Schröttner, H.: "*The combination of electron microscopy, Raman microscopy and energy dispersive X-ray spectroscopy – new methodic possibilities for materials science*", Microscopy Conference 2019; Berlin, Germany, September 3, 2019.

Zankel, A.; Nachtnebel, M.; Mayrhofer, C.; Schröttner, H.; Wernitznig, S.; Leitinger, G.: *"Serial block face scanning electron microscopy - Big data results for life sciences and materials science"*, Microscience Microscopy Congress / EMAG 2019, Manchester, U.K., July 3, 2019.

Poster Presentations 2020

Fitzek, H.; Feigl, G.; Theissing, M.; Töfferl, M.; Bauer, P.; Planko, T.; di Vora, R.; Winkler, G. J.; Sittsam, M.: "*In situ temperautre control in Raman microscopy – Hot or Not?*" Advanced Materials Day 2020, Graz, Austria, September 28, 2020.

Knez, D.; Schnedlitz, M.; Lasserus, M.; Hauser, A. W.; Ernst, W. E.; Hofer, F.; Kothleitner, G.: "*Phase segregation in NiAu-nanoalloys induced by swift electrons*" Virtual Early Career Eur. Micr. Congr. 2020, U.K., November 25, 2020. Krisper, R.; Albu, M.; Lammer, J.; Dienstleder, M.; Fisslthaler, E.; Kothleitner, G.; Grogger, W.: "In-situ structural analysis of AlSi10Mg for additive manufacturing – from powder to thermally treated parts", Advanced Materials Day 2020, Graz, Austria, September 28, 2020.

Oberaigner, M.; Kothleitner, G.; Löffler, S.: "Orbital mapping by *STEM-EELS*", Advanced Materials Day 2020 (online), Graz, Austria, September 28, 2020.

Planko, T.; Fitzek, H. M.; Eichinger, S.; Rattenberger, J.; Koraimann, G.; Schröttner, H.: "Microbiologically influenced corrosion (MIC) of steel : A study using correlative SEM, EDX and Raman microscopy", Advanced Materials Day 2020, Graz, Austria, September 28, 2020.

Radlinger, T.; Kothleitner, G.; Hofer, F.: "*Differential phase contrast imaging in scanning transmisson electron microscopy*", Advanced Materials Day 2020, Graz, Austria, September 28, 2020.

Rauch, N.; Lammer, J.; Rauch, S.; Dienstleder, M.; Grogger, W.: "EDX quantification in the TEM: How to measure ζ-factors without knowing the sample thickness", Virtual Early Career Eur. Micr. Congr. 2020, U.K., November 25, 2020.

Schmidt, R.; Fitzek, H.; Nachtnebel, M.; Zankel, A.; Schröttner, H.: "Detecting nanoplastic particles using correlative microscopy", Advanced Materials Day 2020, Graz, Austria, September 28, 2020.

Schmidt, R.; Fitzek, H.; Nachtnebel, M.; Zankel, A.; Dienstleder, M.; Mertschnigg, S.; Schröttner, H.; Poteser, M.; Hutter, P.; Eppel, W.: "*Detecting nanoplastic particles using correlative microscopy*" WITec - 17th Confocal Raman Imaging Symp., Ulm, Germany, Oct. ober 1, 2020.

Seewald, L.; Kuhness, D.; Winkler, R.; Plank, H.: "*Expanding 3D nano printing performance by blurring the electron beam*" Advanced Materials Day 2020, Graz, Austria, September 28, 2020.

Weitzer, A.; Plank, H.: "*Expanding capabilities of focused electron beam based 3D nano printing: From meshes towards closed 3D nano architectures*", Advanced Materials Day 2020, Graz, Austria, September 28, 2020.



Other Publications & Conference Abstracts 2019

Albu, M.; Kothleitner, G.; Hofer, F.: "Advanced characterization of alloys by using ex-and in-situ high resolution Scanning Transmission Electron Microscopy", Eur. Mater. Res. Soc., (2019) Z 6.12, Warsaw, Poland.

Albu, M.: "*Ex- and in-situ microstructure investigations of powders and additive manufactured parts*", LightMAT 2019: 3rd Int. Conf. on Light Materials, (2019) p. 13-15, Manchester, U.K..

Albu, M.; Zankel, A.; Fitzek, H. M.; Dienstleder, M.; Simic, S.; Hofer, F.; Kothleitner, G.: *"Multiscale and correlative analytical electron microscopy of extra-terrestrial minerals"*, 4th German Astrobiological Society Meeting, (2019) p. 36, Vienna, Austria.

Bossu, J.; Czibula, C. V.; Winter, A.; Gindl-Altmutter, W.; Eckhart, R.; Zankel, A.; Bauer, W.: "*Combined effect of the morphology and rate of addition of fine cellulosic materials produced from chemical pulp on paper properties*", Physics Conference - PaperCon (2019) p. 247, Indianapolis, USA.

Ernst, W, E.; Hauser, A.; Lackner, F.; Knez, D.; Hofer, F.: "Nanomaterials synthesized in helium droplets", Bunsentagung 2019, p 226, Jena, Germany.

Fisslthaler, E.; Haberfehlner, G.; Gspan, C.; Gruber, G.; Grogger, W.: "High-resolution cross-sectional analysis of the interface between SiC and SiO₂ in a MOSFET device via atomic resolution STEM", ES-REF 2019, Session C 2.1, (2019), Toulouse, France.

FissIthaler, E.; Krisper, R.; Grogger, W.: "In-situ observation of precipitate growth and decomposition in AlCu₄ during heat treatment via analytical STEM", 14th Multinational Congress on Microscopy, (2019) pp. 92-94, Belgrade, Serbia.

Fisslthaler, E.; Haberfehlner, G.; Grogger, W.: "Investigation of interface transition chemistry in semiconductor structures via monochromated EELS", 9th ASEM Workshop for Advanced Microscopy, (2019) p. 43, Graz, Austria.

Fowlkes, J. D.; Mutunga, E.; Winkler, R.; Sattelkow, J.; Pakeltis, G.; Rack, P.; Belianinov, A.; Ovchinnikova, O. S.; Plank, H.:"*3D nanoprinting using electron and ion beams*", MRS Fall Meeting, (2019), p. 1023, Boston, USA.

Frank, P.; Hummel, S.; Sattelkow, J.; Winkler, R.; Andany, S.; Fantner, G.; Plank, H.; Schwalb, C.: *"In-situ correlative AFM/SEM/FIB analysis of FIB-treated samples"*, 3rd Workshop of the European Focused Ion Beam Network, (2019) pp. 34-35, Dresden, Germany.

Gößler, M.; Albu, M.; Krenn, H.; Würschum, R.: "ON- and OFFswitching of ferromagnetism in nanoporous Pd(Co)", 3rd Int. Symp. on Nanoporous Materials by Alloy Corrosion, (2019), Philadelphia, USA.

Guo, J.; Haberfehlner, G.; Duarte, M. J.; Li, L.; He, Y.; Kothleitner, G.; Dehm, G.; Pippan, R.; Zhang, Z.: *"Atomic scale characterization of severly deformed Cu-based nanocrystalline alloys via transmission electron microscopy"*, 9th ASEM Workshop for Advanced Electron Microscopy, (2019) p. 15, Graz, Austria. Haberfehlner, G.; Orthacker, A.; Trummer, C.; Kothleitner, G.: "*Nanoscale 3D measurements by electron tomography*", 7th Dresden Nanoanalysis Symposium, (2019) p. 19, Dresden, Germany.

Haselmann, U.; Haberfehlner, G.; Knez, D.; Popov, M. N.; Romaner, L.; Zhang, Z.; "*Ca segregation towards the interface in bismuth ferrite thin films*", 9th ASEM Workshop for Advanced Electron Microscopy, (2019) p. 38, Graz, Austria.

Hinum-Wagner, J. W.; Winkler, R.; Birnstingl, G.; Plank, H.: "*The influence of substrate temperature during focused electron beam induced deposition (FEBID)*", 9th ASEM Workshop for Advanced Microscopy, (2019) p. 44, Graz, Austria.

Huber, R.; Haberfehlner, G.; Holler, M.; Kothleitner, G.; Bredies, K.: "Graptor - an efficient reconstruction tool for electron tomography", 9th ASEM Workshop for Advanced Microscopy, (2019) p. 25, Graz, Austria.

Knez, D.; Schnedlitz, M.; Lasserus, M. I.; Schiffmann, A.; Ernst, W. E.; Hofer, F.; Kothleitner, G.: "On the impact of swift electrons on mass transport phenomena at the nanoscale", 9th ASEM Workshop for Advanced Microscopy, (2019) p. 47, Graz, Austria.

Knez, D.; Haberfehlner, G.; Kothleitner, G.; Hofer, F.: *"Electron beam induced dynamics in experiment and simulation"*, Microscopy Conference 2019, IM3.008, (2019) pp. 457-458, Berlin, Germany.

Knez, D.; Dražić, G.; Chaluvadi, S. K.; Origani, P.; Ciancio, R.: "Impact of oxygen vacancies on electronic properties of anatase thin films", 14th Multinational Congress on Microscopy, (2019) pp. 71-73, Belgrade, Serbia.

Kopacic, S.; Walzl, A.; Hirn, U.; Zankel, A.; Leitner, E.; Bauer, W.: "Application of natural materials in the barrier coating of packaging papers", PTS Coating Symposium, (2019) p. 45, München, Germany.

Krisper, R.; Fitzek, H. M.; Fisslthaler, E.; Grogger, W.: "Measuring the temperature homogeneity across FIB lamellae for in-situ TEM experiments via Raman scattering in crystalline silicon", 14th Multinational Congress on Microscopy, (2019) pp. 79-80, Belgrade, Serbia.

Krisper, R.; Fitzek, H. M.; FissIthaler, E.; Grogger, W. "Measuring the temperature homogeneity across FIB lamellae for in-situ TEM experiments via Raman scattering in crystalline silicon", 9th ASEM Workshop for Advanced Microscopy, (2019) p. 48, Graz, Austria.

Lammer, J.; Berger, C.; Knez, D.; Longo, P.; Kothleitner, G.; Schrödl, N.; Bucher, E.; Egger, A.; Merkle, R.; Sitte, W.; Maier, J.; Grogger, W.: "Atomic structure analysis of $Ba_{1,1}La_{1,9}Fe_2O_7$ using high-resolution STEM", 9th ASEM Workshop for Advanced Microscopy, (2019) p. 17, Graz, Austria.

Messner, R.; Knez, D.; Hofer, F.; Ernst, W. E.; Lackner, F.: "Towards new plasmonic materials: Synthesis of K and K-Au nanoparticles with helium nanodroplets", Gordon Res. Conf. on Clusters and Nanostructures, (2019) p. 76, Les Diablerets, Switzerland.

Messner, R.; Knez, D.; Hofer, F.; Ernst, W. E.; Lackner, F.: "Towards new plasmonic materials: Synthesis of K and K-Au nanoparticles with helium nanodroplets", Int. Conf. on Quantum Fluid Clusters (2019) p.65, Bad Honnef, Germany.

Moser, D.; Steiner, S.; Gollas, B.; Kothleitner, G.: *"Electron micros-copy characterization of aluminum anode of a novel rechargeable aluminium-sulfur battery"*, 9th ASEM Workshop for Advanced Microscopy, (2019) p. 43, Graz, Austria.

Mutunga, E.; Winkler, R.; Rack, P.; Plank, Harald; Fowlkes, J. D. "*The* role of electron beam induced heating during 3D nanoprinting", MRS Fall Meeting (2019) p. 2207, Boston, USA.

Nachtnebel, M.; Schmidt, R.; Fitzek, H. M.; Mayrhofer, C.; Zankel, A.; Schröttner, H.: "Correlative and automated particle analysis by SEM, EDXS and Raman: Features and limitations", 15th Mini Symposium Chemical Engineering (2019) pp. 47-50, Leoben, Austria.

Padure, I.; Simic, S.: "Pollen and nutlet surface micromorphology of Clinopodium foliosumand C. menthifolium (Lamiaceae) from Istria", Joannea / Botanik 16 (2019) p. 83.

Planko, T.; Fitzek, H. M.; Eichinger, S.; Rattenberger, J.; Schröttner, H.: "Microbiologically influenced corrosion (MIC) of steel – a study using correlative SEM, EDX and Raman microscopy", 14th Multinational Congress on Microscopy, (2019) pp. 74-76, Belgrade, Serbia.

Pongratz, G.; Subotić, V.; Schröttner, H.; Stöckl, B.; Hochenauer, C.; Anca-Couce, A.; Scharler, R.: "Investigation of SOFC operation with steam gasifier product gases as a basis for enhancing its performance", Int. Conf. on Polygeneration Strategies (ICPS), (2019) p.103, Vienna, Austria.

Radlinger, T.; Surnev, S.; Bezanaj, M. M.: "Growth and structure of molybdenum oxide layers on Pd (100)", 9th ASEM Workshop for Advanced Microscopy, (2019) p. 56, Graz, Austria.

Rattenberger, J.; Fitzek, H. M.; Achtsnit, T.; Schröttner, H.; Hofer, F.: "Universal pressure scanning electron microscopy (UPSEM) - improvements in environmental scanning electron microscopy", Microscopy Conference 2019, p. 367, Berlin, Germany.

Rattenberger, J.; Fitzek, H. M.; Schröttner, H.; Hofer, F.: "Improvements in environmental scanning electron microscopy – universal pressure scanning electron microscopy (UPSEM)", 14th Multinational Congress on Microscopy, (2019) pp. 104-106, Belgrade, Serbia.

Schmidt, R.; Nachtnebel, M.; Fitzek, H. M.; Mayrhofer, C.; Schröttner, H.; Zankel, A.: "Correlative microscopy - The new system RISE combined with EDXS applied on complex materials", 9th ASEM Workshop for Advanced Microscopy, (2019) p. 29, Graz, Austria.

Schwalb, C.; Hummel, S.; Winkler, R.; Sattelkow, J.; Pinar, F.; Hlawacek, G.; Hosemann, P.; Fantner, E.; Plank, H.: "In-situ correlative analysis of ion beam treated nanostructures by combination of AFM and FIB", MRS Fall Meeting 2019, p. 2012, Boston, USA.

Schwalb, C.; Hummel, S.; Frank, P.; Sattelkow, J.; Winkler, R.; Fantner, G.E.; Plank, H.: "*Correlative in-situ analysis by combination of AFM, SEM, and FIB*", 9th ASEM Workshop for Advanced Microscopy, (2019) p. 28, Graz, Austria.

Steitz, A.; Nachtnebel, M.; Munoz-Czerny, U.; Mantler, J.; Grüll, G.: "Dust in indoor air - What is it composed of?", 9th ASEM Workshop for Advanced Microscopy, (2019) p. 23, Graz, Austria.

Weitzer, A.; Plank, H.: "Expanding capabilities of focused electron beam based 3D nanoprinting: From meshes towards closed 3D nano-architectures", 9th ASEM Workshop for Advanced Microscopy, (2019) p. 61, Graz, Austria. Zankel, A.; Fitzek, H. M.; Mayrhofer, C.; Nachtnebel, M.; Schmidt, R.; Schröttner, H.: "*The combination of electron microscopy, Raman microscopy and energydispersive X-ray spectroscopy – new methodic possibilities for materials science*", Microscopy Conference 2019, IM1.P018, pp. 385-6, Berlin, Germany.

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Other Publications & Conference Abstracts 2020

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FELMI-ZFE Calendar 2020/21

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. In 2020, however, we decided to present some high-resolution images that have been measured with the ASTEM microscope at atomic resolution in recent years. The images were taken with the HAADF detector and show an atomic number contrast. All these images show the arrangement of atoms or of atomic columns in the crystal structure of technically relevant materials, providing a unique insight into the nanocosmos never seen before.















Out of Office

2019-2020

Life at the institute is not only about science, teaching and industry. Alongside these core activities, a number of social initiatives are also developing that help to create a productive and creative working environment. Outside the daily routine, new contacts and ideas can be developed in a relaxed atmosphere.

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. As every year, a merry circle met on "Faschings-

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dienstag" (1) and the company outing in September took us to archery and a cheese and wine tasting (2). The more active ones among us met every Tuesday in summer to play beach volleyball (3).

The last highlight of 2019 was the Christmas party (4), which took place on the roof of the old chemistry building in the Restaraunt Rooftop. We were also able to welcome our retired colleagues. In addition to a review of the successes of 2019, the prizes for the best microscopic images (5) and the publication and cooperation prize (6) were also presented.

In 2020, we managed to have a merry carnival(**7**) round in the institute just before the lockdown and that was unfortunately it for 2020.

















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