Performance Report 2007/2008

Institute for Electron Microscopy and
Fine Structure Research
Graz University of Technology

Graz Centre for Electron Microscopy

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Institut für Elektronenmikroskopie und Feinstrukturforschung, Technische Universität Graz
&
Zentrum für Elektronenmikroskopie Graz
Steyrergasse 17
A-8010 Graz, Austria

Phone ++43 316 873 8320
Fax ++43 316 811 596
Email sekretariat@felmi-zfe.at
Web http://www.felmi-zfe.at

Editors

Editor: Ferdinand Hofer
Associate Editors: Ilse Letofsky-Papst, Fatima Streussnig, Karin Wewerka

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Preface

I am pleased to introduce the fifth biannual report of the Graz Centre for Electron Microscopy (ZFE Graz). The Centre, which was established in 1959 as a result of a combined effort by leading Austrian industrial companies and Graz University of Technology, has developed into a major national research facility. The Centre is managed by the association „Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung“, and a steadily increasing number of Austrian companies have acknowledged the institute’s efforts by joining the association, which now has 30 company members.

Fifty years ago the famous speech of Richard P. Feynman marked the beginning of a revolution in miniaturisation that has powered technological developments to this day. I believe the institute’s focus on advanced microscopy of materials will support Austria’s role in this rapidly growing scientific and technological field. The institute provides Austrian researchers with an essential resource for the characterisation and manipulation of physical and biological systems down to the molecular and atomic levels. Through its leading-edge instrumentation and scientific know-how, ZFE permits Austrian, and indeed international, researchers both from companies and universities to realise their scientific ideas and to improve industrial processes and products.

In my capacity as president of the “Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung“, I wish the institute many more years of success.

Prof. Helmut LIST

Introduction

This biannual report aims to summarise the latest achievements of the institute and to showcase a selection of its research results. The facility's various collaborators include researchers from other institutes of TU Graz and increasingly also from the joint NAWI faculty. The institute collaborates with other Austrian universities and national research organisations including Joanneum Research (JR), Austrian Cooperative Research (ACR), Christian Doppler Laboratories (CD) and the Austrian Research Centers (ARC). This, together with our growing industry-based collaborations and international activities, further positions the institute as the premium national resource for microscopy and micro- and nanoanalysis. In this way, we are helping researchers from Austria and elsewhere to understand materials and biomaterials down to the sub-nanometer level, an effort that is making an important and pervasive contribution to several national research priorities.

The successful extension of scientific instrumentation in combination with the introduction of new characterisation methods (e.g. 3-D micro-analysis, *in-situ* microscopy and cryo-microscopy) has attracted the interest of the scientific community. Front covers in journals and the very first published scientific papers based on the new methods have been published in the literature. The success of combining our research with major Austrian networks (e.g. Austrian Nanoinitiative) and topical scientific projects both from Austria and the European Union are of major significance.

Following on from the successful organisation of several scientific workshops and symposia in 2006 and 2007 we have also been involved in organising the largest electron microscopy conference in Europe in 2009 and 2010, the Microscopy Conference 2009, which is scheduled for the first week of September 2009 in Graz.

FELMI-ZFE staff has again been highly successful in attracting external research funding. However, even more research support is needed in 2009 to 2011, because this will allow us to focus on a new world class electron microscope. This Austrian / Analytical Scanning Transmission Electron Microscope (ASTEM) will permit the elemental analysis of materials at atomic resolution, thus providing a unique new research tool in Austria in line with the recently proposed "frontrunner" strategy for Austrian research and developments efforts. This major step forward will enable us to develop new methodologies and advanced applications as part of a broader national research effort.

One other main activity of the last two years should also be mentioned here. We are increasingly engaged in teaching microscopy in materials and life sciences, chemistry and physics, e.g. in the new master and PhD courses launched by several faculties of the TU Graz.

Finally, we would like to thank our laboratory team for their enthusiasm and dedication since this is fundamental to our success and provides a sound basis for a bright and successful future in the European science & technology environment.

Ferdinand Hofer

Contents

1. The Institute

1.1. The Institute at a Glance

The centre carries out interdisciplinary research and teaching in materials science, physics and micro- and nanotechnology. Major fields of research include micro- and nanoanalysis of materials, devices and biological tissue. The centre brings together

- well-honed laboratory methods,
- cutting-edge experimental techniques and
- advanced technology.

The primary aim is to study fundamental scientific problems and to transfer the knowledge about advanced microscopy methods into practical collaborations with university institutes and industry (with the focus on small and medium enterprises).

1.2. History

Following installation of the first electron microscope at the former Technische Hochschule Graz (later known as TU Graz) back in 1951, the centre was founded in 1959 by scientists there under the leadership of the Styrian Provincial Governor Josef Krainer sen.. At that time it consisted of several senior research groups and staff members from the Technische Hochschule Graz, which together formed the nucleus of today's "Austrian Centre for Electron Microscopy and Nanoanalysis". Dr. Fritz Grasenick was head of the centre until 1981. Many researchers at the centre have gained a high level of scientific know-how which has allowed them to go on to hold important positions both in education and industry.

1.3. Research Objectives

For both the development and application of advanced technologies it is becoming increasingly important to characterise the structure of materials and functional devices on a micro/nanoscopic scale. Whether the interests are in diagnostic techniques for product development or applied materials research, understanding the micro/nanostructure and its relationship to the performance of the material is critical.

- The institute is one of the leading facilities in Europe for microscopic characterisation of materials. With several types of microscopes, it offers a comprehensive array of advanced imaging and spectroscopy techniques for studying technologically relevant materials and associated problems.
- The institute aims to take advantage of the synergies that emerge from the various fields of research interests, from sophisticated experimental tools and from fundamental research and application of these techniques in cooperation with companies.
- Consequently, the institute endeavours to improve microscopy preparation and characterisation techniques and/or to develop new techniques, especially in the field of materials and bio sciences.
- These techniques are intensively used to characterise all kinds of materials, providing efficient answers and solutions to materials science problems.

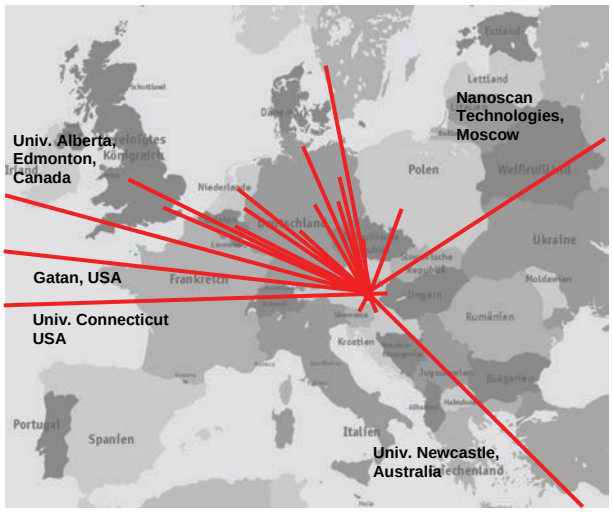
1.4. Organisation

The institute is organised in research groups focused on specific aspects of microscopy or important material classes:

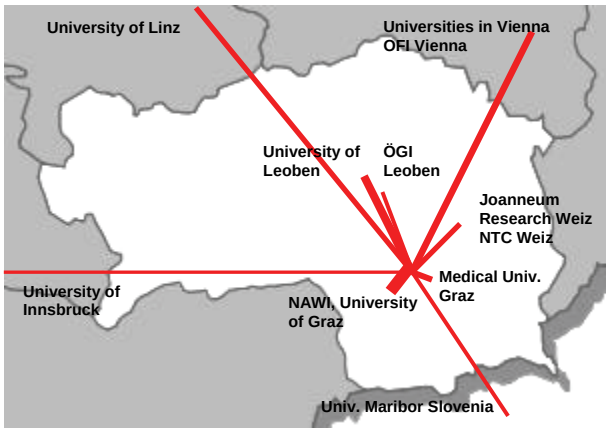
- **Peter Pölt**, Scanning electron microscopy and related physical methods
- **Hartmuth Schröttner**, Analytical scanning electron microscopy
- **Gerald Kotheitner**, Analytical transmission electron microscopy and specimen preparation
- **Werner Grogger**, Analytical high resolution electron microscopy
- **Elisabeth Ingolic**, Microscopy of polymers and biological tissue
- **Peter Wilhelm**, FTIR and Raman microspectrometry

1.5. Collaborations

During 2007 and 2008 the institute cooperated with around 40 university institutes and 105 companies (mainly in Austria but also in other European countries and overseas). Probably, it is worthwhile to mention that two main directions evolved in this period: Firstly, the rapidly developing cooperation network in the NAWI faculty of the TU Graz and the KFU Graz boosted our scientific activities.



Scientific network of the institute with ongoing international collaborations.



Scientific collaborations with local universities and research institutes

Secondly, applied research could be significantly extended due to the growing research network within the Austrian Cooperative Research (ACR).
Our broad activities in technology transfer are also manifested by more than 300 visitors from other research groups and companies per year.
During this period 77 graduate and PhD students (mainly from TU Graz) benefited from the scientific and technical support of the institute.

1.6. New Research Tools and Developments

The institute houses a significant proportion of the electron microscopes available at TU Graz. We have been successful in continuously upgrading the instrumentation, but also in improving the education of our scientists and microscope operators in order to provide state-of-the-art investigations both for university institutes and companies.

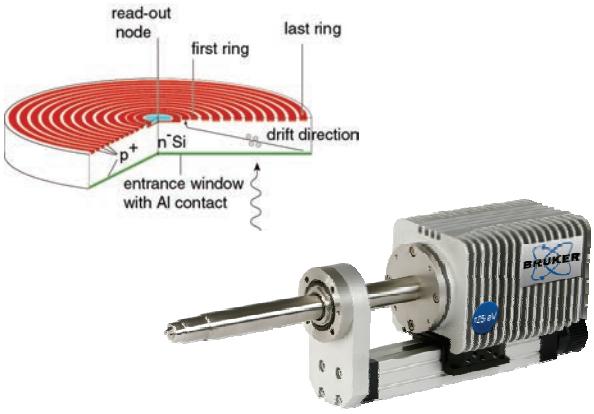
Our laboratory now includes one analytical high-resolution electron microscope, two analytical transmission electron microscopes, two environmental scanning electron microscopes, two high resolution scanning electron microscopes, one atomic force microscope as well as FTIR, Raman and advanced light microscopes. Associated techniques include energy-dispersive x-ray analysis (EDX), wave length dispersive x-ray analysis, elemental mapping, electron energy-loss spectrometry, energy-filtering microscopy, low-dose imaging, electron diffraction and many other special techniques (for details see Chapter 5).

The following section provides a brief description of the main instruments introduced over the last two years:

- **X-ray Spectrometers**

In 2008 we had to replace outdated x-ray detectors for several scanning electron microscopes.
First, it was necessary to purchase a standard **energy-dispersive x-ray spectrometer** (EDAX)

for the environmental SEM (FEI ESEM Quanta 200).
Second, a **silicon drift detector** (SDD) was installed on the focused ion beam instrument (NANOLAB Nova 200). This liquid nitrogen free XFlash® detector (Buker AXS) permits much faster and more efficient data collection, and shows better light element performance than conventional Si(Li) detectors. One further advantage is that it is also capable of handling extreme count rates which is a prerequisite for further improvement of the 3D elemental mapping method which was recently developed in the institute.



The silicon drift detector for the focused ion beam microscope (XFlash® Bruker AXS).

Third, the high resolution scanning electron microscope (Zeiss Ultra 55) was equipped with a new **wavelength dispersive x-ray spectrometer** (WDXS).This LambdaSpec instrument delivered by EDAX (USA) is a parallel beam x-ray spectrometer designed for improved resolution and better detection sensitivities compared to an EDXS spectrometer. The LambdaSpec will not only improve the quantitative microanalysis of light elements but also the detection of trace elements in materials.
With the purchase of the new WDX spectrometer, the Zeiss Ultra 55 enables now the simultaneous collection of x-rays (chemistry) and electron backscattered diffraction (EBSD) data (crystallography), allowing direct correlation between the elemental content and microstructural aspects of the material being studied.

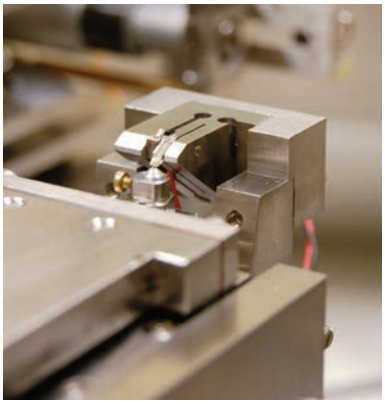
The column of the Zeiss Ultra 55, now equipped with an EDXS-, WDXS- and EBSD-spectrometer.

The x-ray spectrometers were launched by the "Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung". The purchase of the WDXS-spectrometer was partly funded by the „Wirtschaftskammer Steiermark".

- **3-D ultramicrotomy in the environmental scanning electron microscope (ESEM)**

The group of Peter Pölt performed ground breaking work by introducing the serial block face imaging in the environmental SEM (ESEM). After an intensive collaboration on the automatisation of the instrument (3VIEW™ with Gatan Company (USA) we have been the first to introduce this revolutionary 3-D method into materials science applications. A specially designed ultramicrotome operates *in situ* in the specimen chamber of a variable pressure field emission SEM, and allows

automated acquisition of 3-D ultrastructures by sequentially imaging the freshly cut, resin embedded block face. The 3VIEW™ system has been already successfully applied to the study of polymers, composites, paper and biomaterials (e.g. plant tissue).



Ultramicrotome in the 3View® system.

The 3View™ system could be installed during 2006 and was finally purchased by the "Verein zur Förderung der Elektronenmikroskopie und Feistrukturforschung", with a substantial contribution from the joint NAWI faculty of the TU Graz and the KFU Graz.

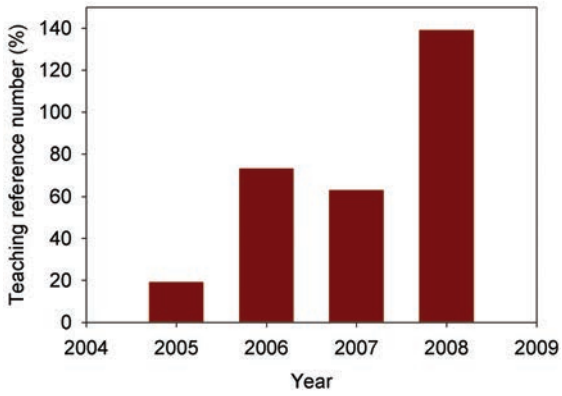
1.7. Quality Management

Meanwhile the institute works under a highly developed quality management system according to the rules of ISO 9001:2000. The aim is to maintain the high quality of our work and investigation results and improve organisation and management structures. In accordance with these aims we focused the system on the development of long-term relationships with our cooperation partners.

TÜV CERT
EN ISO 9001:2000
Zertifikat Nr. 20 100 42000725
www.tuv.at

Following the successful audit performed by TÜV Austria, the institute was awarded the EN ISO 9001:2000 certificate. It is valid until May 2009 and covers "Research and teaching in the field of microstructure research and materials characterisation by electron microscopy, micro- and nanoanalysis and the development of analysis and preparation methods". The QM system is guided by Mag. Ulrike Stürzenbecher.

We perform regular staff questionnaires and appraisal interviews and have also introduced measures to manage and document the maintenance of the microscopes and infrastructure.

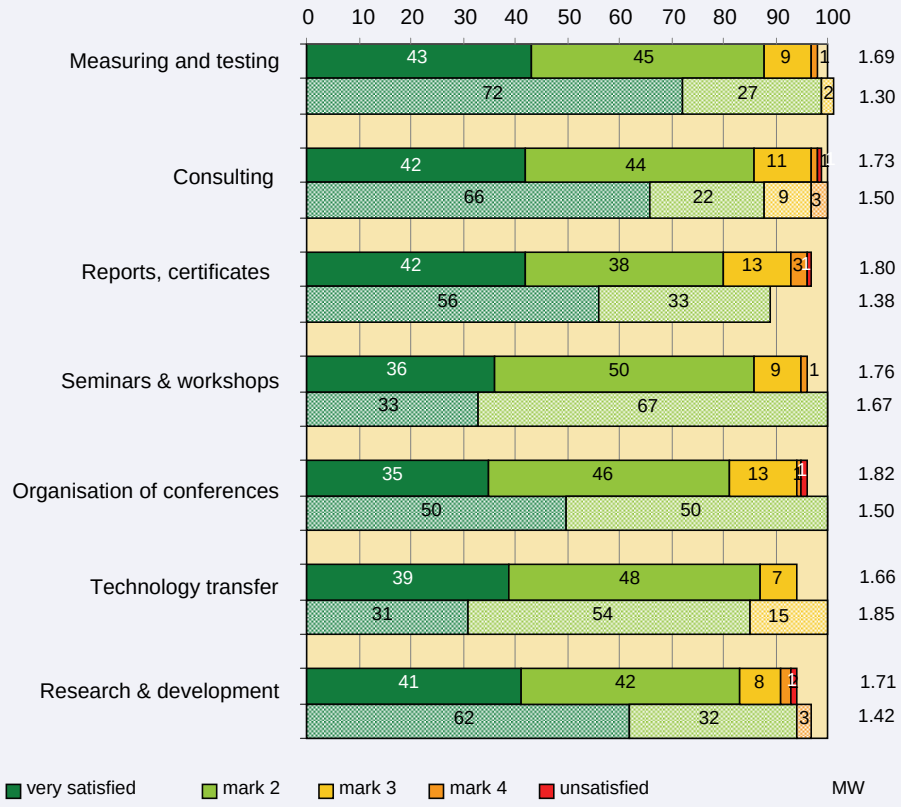


Relative increase of the teaching reference number of staff members in university courses.

Question: Consider the last collaboration with the ZFE Graz!

How satified have you been with the collaboration with the ZFE Graz? Give a mark between 1 and 5: *1= very satified, 5=unsatified. (in percent)*

upper row: average of all ACR-institutes, lower row: ZFE Graz



Investigation of the institute's role in the Austrian innovation process: The results are compared with the average values of ACR institutes. The sample was taken from 84 collaboration partners of the institute (taken from the IFES study of 2007).

1.8. The People in the Laboratory

Staff

The broad diversity of research projects and partnerships lead to a steady growth of employees and PhD students.

New coworkers in the institute:

- Dr. Nadejda Borisovna MATSKO joined the institute on 19 February 2007. She is working on cryo methods for biological and polymer science. Dr. Matzko works in the group of Prof. Grogger.
- Dipl.-Ing. Dr. Harald PLANK joined the institute on 1 August 2007 and works in Prof. Kothleitner´s group on the growth of functional nanostructures.
- Dipl.-Ing. Herbert REINGRUBER joined the institute as a doctoral student on 17 November 2008 and works in Dr. Pölt´s group.
- Dipl.-Ing. Dr. Thomas HABER joined the institute on 1 September 2008 as a post doc on the NILaustria project (Prof. Grogger).
- Dipl.-Ing. Wernfried HAAS joined the institute as a doctoral student on 1 October 2008 in the CD-Laboratory and works in Prof. Grogger's group.
- Toni UUSIMÄKI will join the institute on 1 January 2009 as a doctoral student. He will work in the CopPeR project of Prof. Kothleitner.
- Stefanie FLADISCHER joined the institute on 31 March 2008 for her diploma thesis and works in the group of Dr. Grogger.
- Sebastian RAUCH will join the institute on 1 January 2009 and will work in the physical sample preparation laboratory.
- Sabrina MERTSCHNIGG completed her apprenticeship with commendation on 4 March 2008. Since then she has been employed in Ing. Schröttner´s group.
- Daniel SCHREINER joined the institute as a chemical laboratory apprentice on 1 September 2008 and now works in the group of Prof. Kothleitner.

The institute team at the "Kleeblatt Lauf" 2008:
Front row: Katharina Riegler, Sanja Simic, Claudia Mayrhofer, Meltem Sezen. Back row: Hartmuth Schröttner, Stefan Mitsche, Julian Wagner, Thomas Haber.

Coworkers, who left the institute

- Dr. Elena TCHERNYCHOVA left the institute on 28 February 2007. Dr. Tchernychova worked in Prof. Grogger´s group since May 2005 and is now with the Josef-Stefan-Institute in Ljubljana.
- MMag. Dr. Werner RECHBERGER left the institute on 30 June 2007 to take up a position at Xenon Architectural Lighting in Graz, Austria.
- Dr. Mag. Miroslava SCHAFFER left the institute in June 2008. She worked as a doctoral student in Ing. Schröttner's group.
- Dr. Katharina RIEGLER left the institute on 4 April 2008 the institute for her maternity leave. She worked in Prof. Kothleitner´s group.
- Amtsdirektor Ing. Albert BRUNEGGER retired on 1 August 2008. Ing. Brunegger had been a member of the institute since 1971 and worked in the physical sample preparation laboratory.
- Dr. Angelika REICHMANN left on 27 October 2008 the institute for her maternity leave. She worked in Dr. Pölt´s group.
- Dr. Maria Belegratis left the institute on 31 December 2008 to take up a position at Joanneum Research Weiz, Austria. She worked in Dr. Ingolic´s group since January 2007.

In memoriam

- Oberrat Dipl.-Ing. Dr.techn. Rupert ESSL

Rupert Essl died at the age of 69 years on January 15th, 2008. He is regarded as one of the key scientists in the institute. After finishing an apprenticeship for electrical engineering Rupert Essl started to study physics at the Technische Hochschule in Graz. He gained a thorough scientific education in technical physics, which he then consolidated as a research assistent of Professor Rudolf Gebauer in the Institute for Experimental Physics. In 1979 he joined the Institute for Electron Microscopy. He started to develop new preparation methods for electron microscopy. During the course of his work he later concentrated on the characterisation of special electrolyte capacitors and started a fruitful cooperation with the company Philips. Rupert Essl retired in November 2000.

Awards

K.F.J. Heinrich Young Scientist Award for Prof. Dr. Gerald Kothleitner: The Microbeam Analysis Society of America awarded Prof. Kothleitner with this prestigious distinction at the conference "Microscopy and Microanalysis 2007 in Fort Lauderdale (USA) in August 2007.

Paul Kotula (right) awards Gerald Kothleitner (left) at the M&M Conference in Fort Lauderdale (USA).

- **"Cooperation Award"** of the Austrian Cooperative Research (ACR) for Ingenieurbüro Steiner (Spielfeld) and ZFE Graz in the "Haus der Forschung", Vienna, October, 15th 2007.

ACR-Cooperation Award for IB Steiner (Spielfeld) and the ZFE Graz; from left to right: Martin Leitl, Ferdinand Hofer, Elisabeth Ingolic, Herbert Eichler, Gottfried Steiner.

- **CARBONIUM 2007** of the Austrian Cooperative Research (ACR) for the ZFE Graz for strengthening the cooperation within the ACR-organisation in the "Haus der Forschung", Vienna, April, 6th 2008.

Martin Leitl (ACR President) awards the representative of the ZFE Graz (Ferdinand Hofer, left) with the CARBONIUM 2007.

- **"Cooperation Award"** of the Austrian Cooperative Research (ACR) for PLASMAIT GmbH (Lebring) and ZFE Graz in the "Haus der Forschung", Vienna, November 3rd, 2008.

ACR-Cooperation Award for PLASMAIT GmbH (Lebring) and the ZFE Graz; from left to right: Ferdinand Hofer, Heinz Schmidt, Martin Leitl, Herbert Kraiger, Hartmuth Schröttner, Primoz Eiselt.

Organisation of scientific conferences

The institute organised several symposia and meetings during the last two years:

2nd FIB-Workshop "Focused Ion Beams in Rese-arch, Science and Technology"
Organised by Michael Rogers (Graz) and Siegfried Menzel at the TU Graz from July 2nd to 3rd, 2007; in cooperation with the Austrian Society for Electron Microscopy (ASEM), Deutsche Gesellschaft für Elektronen-mikroskopie (DGE), Swiss Society for Optics and Microscopy (SSOM) and Deutsche Gesellschaft für Materialkunde (DGM).

90 scientists from 6 nations participated at the 2nd FIB-Workshop at the Graz University of Technology

17th European Symposium on Polymer Spectroscopy (ESOPS 17)
Organized by Peter Wilhelm (Graz), September 9th – 12th, 2007 at Seggau Castle, Leibnitz, Austria.

ESOPS is held every two years to review the latest research and development in the spectroscopic characterisation and analysis of polymer systems. More than 100 scientists and students participated in ESOPS 17, coming from 27 nations. The scientific contributions came from all fields of

spectroscopy (infrared, NIR, Raman, fluorescence, NMR, mass spectroscopy, electrical and mechanical spectroscopy). The scope of the meeting ranged from theoretical and fundamental aspects to recent advances and novel developments in characterisation and analysis of polymers.

Rector Prof. Hans Sünkel opens the ESOPS Symposium at Seggauberg, Austria.

Participants at the ESOPS-Conference 2007; in front: chief organiser Peter Wilhelm.

Workshop “Advanced Microscopy”
Organized by FELMI-ZFE at the TU Graz, November 30th, 2007, Graz, Austria.

The workshop concentrated on the introduction of the high resolution scanning electron microscope ZEISS Ultra 55. New aspects of surface microscopy in materials science applications were discussed by 64 participants.

Prof. Helmut List opens the Workshop on Advanced Microscopy at the TU Graz.

Ongoing renovation of the institute

Starting in 2005 the “Verein zur Förderung der Elektronenmikroskopie” pushed the renovation of the infrastructure of the institute. These activities will continue until 2010 and are strongly supported by the TU Graz. In 2007 emphasis was put on the planning and construction of the chemistry laboratory and the exchange of electrical installation. Security issues could be completely solved by the help of the TU Graz.
In the beginning of 2009 we will start with the renovation of the microtome lab and the extension of phase III of the microscopy centre in the basement of Steyrergasse 17.

Opening of the microscopy centre in the basement of the building Steyrergasse 17 (August 2008); from right: KR Dipl.-Ing. Ulrich Santner, Dipl.-Ing. Gerhard Kelz, Ing. Wolfgang Marth.

Chemical laboratory before and after the renovation in 2008.

The new central server of the institute

During 2008 the institute established a central server based on HP DL30 units. The storage area network (with 4 TByte) consists of a mail server, a file server and a backup server, which is supported by a 5 TByte Tape Library. The 1 GBit network of the institute is connected via three Cisco 48-port and one Cisco 24-port switches and hosts about 70 desktop computers and 4 network printers. The institute's network is protected by a Fortigate 200 Firewall.

Gerhard Windisch checks the new central server.

Activities in the scientific community

- F. Hofer is member of the editorial board of MICRON, board member of the Austrian Cooperative Research (ACR) and the Austrian Society for Electron Microscopy (ASEM) and co-chairman of the supervisory board of ANTON PAAR company in Graz.
- G. Kothleitner is board member of the working group “EFTEM & EELS” of the electron microscopy societies of Austria, Germany and Switzerland.
- F. Hofer was member of the international advisory board of the Autumn School for “Electron Microscopy and Advanced Materials” at Humboldt University, Berlin, Germany (until 2007).
- W. Grogger is member of the Curricular Commission for *Nanophysics* in the NAWI faculty.

- F. Hofer is member of the Curricular Commission for *Doctoral Studies* and for the Master Curriculum *Advanced Materials Science* at the TU Graz.
- F. Hofer is speaker of the Field of Expertise "Advanced Materials Science" of the TU Graz.
- F. Hofer was member of the International Advisory Board of the European Microscopy Conference 2008, Aachen Germany.
- F. Hofer is member of the IFSM International Advisory Board of the 17th International Microscopy Conference, Rio de Janeiro, Brazil (2010).

- Staff members were active as referees for scientific institutions, funding organisations, the promotion of scientists at other universities as well as for a number of scientific journals including:

Ultramicroscopy
Microscopy & Microanalysis
Micron
J. Microscopy
J. Electron Microscopy
Mikrochimica Acta
Chemical Monthly
J.Phys. D.
Chemistry of Materials
Spectrochimica Acta B
Philosophical Magazine A
Vacuum
Applied Physics Letters
Phys. Rev. Letters
Solid State Chemistry
Macromolecular Symposia
ACS Nano
Science
Phys. Rev. B
Scanning
……

The institute in the news

BAUMAGAZIN, 1/2007, p.28-29, „Ultra-hochfeste Betone"

ACR-Newsletter 02/2007, „Neuland in drei Dimensionen"

KLEINE ZEITUNG, August 4th, 2007, p.13: „Steirer des Tages: Dr. G. Kothleitner"

ACR-Newsletter 03/2007, „Erster deutschsprachiger Forscher ausgezeichnet"

DER STANDARD, September, 26th, 2007, p.20, „Der Nanoanalytiker"

KLEINE ZEITUNG UNI, October 2007, p. 22: "Reise in den Mikrokosmos"

STEIRISCHE BERICHTE, 5/2007, p. 26–27, „Reise in die Welt des Mikrokosmos"

KLEINE ZEITUNG, November 25th, 2007, p. 68-69: „Im Auge der Motte"

TUG Print, Ausgabe 24, 4/2007: „Landkarten im Nano-Bereich", p. 9

ACR-Newsletter 04/2007, „Oberflächenmikroskopie von Werkstoffen"

REPORT PLUS, 1/2008, vol.6, p.143, „Nanoanalytik von Werkstoffen und Biomaterialien"

FORSCHUNGSJOURNAL TU Graz, WS07/08, p. 13: „Nanometeraufgelöste Abbildungen optoelektronischer Materialeigenschaften"

Business Magazine REPORT, 4/2008, p. 15,„Nanoanalytik für die Halbleitertechnologie"

UNTERNEHMER, UIK-Austria, April 2008, p. 27, „Sandstrahlen auf atomarem Niveau"

ACR-Newsletter 01/2008, „Reise in die Welt des Mikrokosmos"

UNTERNEHMER, UIK-Austria, April 2008, p. 37, „Mit Elektronen sieht man besser!“

TÜV-News, June 5th, 2008, p.15, „Aus der Nähe besehen“

KLEINE ZEITUNG UNI, June 2008, p.40, „Holzfresser unterm Mikroskop“

Annual Report 2007/08 of the Akademisches Gymnasium Graz: „Lehrausgang ins Institut für Elektronenmikroskopie”

CHEMIE REPORT, June 2008, “Nanocomposites für flexible Solarzellen”, p. 45

WIRTSCHAFTSBLATT, October 27th, 2008, p.6, „Mit Elektronen sieht man besser”

DIE PRESSE, November 5th, 2008, p.11: „Technologie: Aus Pferdemist wird Energie”

STEIRISCHE WIRTSCHAFT, November, 14th, 2008, No.37, vol.7, p.10: „Ausgezeichnetes Zusammenspiel“

ACR-Newsletter, 02/2008, „Modern Polymer Spectroscopy“

UNTERNEHMER, 05/2008, „Innovation: Die Kraft der Kleinen“, UIK-Austria, p.21, „Mit Elektronen sieht man besser”

1.9. Science Meets Art?

The technical art of microscopy provokes common human experience in quite a similar way as the images of distant galaxies provided by huge telescopes. Since Robert Hooke's *Micrographia* was printed in London in 1665, scientists realized the hidden beauty of micro- and meanwile also nanoworlds.

People are also increasingely interested in these phantastic landscapes full of stunning details and surprising symmetries. Therefore the institute's microscopists pick the best images among the thousands of images which are collected every year and publish them via two main routes.

- „Reise in die Welt des Mikroskosmos"

The portfolio of the most spectacular micrographs was also successfully shown at the Raiffeisenhof in Graz from October 4th to 30th, 2007 and in the gallery of the TUEV Austria in Vienna from May 20th to June 30th, 2008. The exhibitions have been organised by Margit Wallner. More than two hundred participants came to each of the vernisages demonstrating the great public interest.

KR Renate Römer opens the image exhibition in the TUEV Austria Forum in Vienna; from left: Dr. Johann Jäger (ACR), Dr. Ferdinand Hofer (ZFE Graz), KR Renate Römer (Vice-President WKO Österreich), Dr. Hugo Eberhardt (Director TUEV Austria).

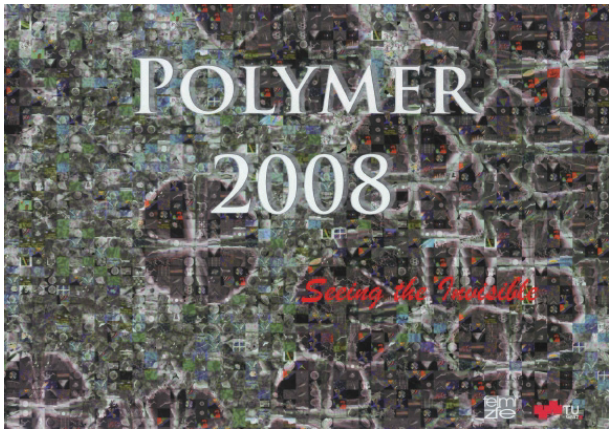
KR Dipl.-Ing. Ulrich Santner opens the ZFE image exhibition in the Raiffeisenhof[galerie] in Graz.

Image exhibition at the Raiffeisenhof[galerie] in Graz

Image exhibition at the TUEV Austria Forum

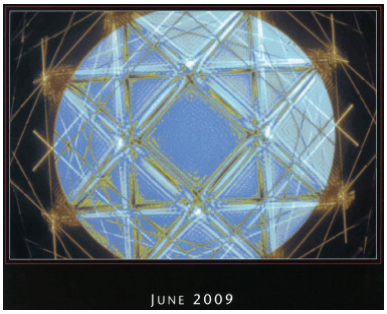
- FELMI-ZFE Calendar 2008

Based on an internal "image competition" the best micrographs are chosen once a year and published in the biannual institute's calendar. The calendar of 2008 highlights micrographs from the world of polymer materials.



- 2009 FEI Calendar

Two images recorded by Christian Gspan and Michael Rogers and colorised by Margit Wallner have been selected for the 2009 FEI Calendar of FEI Company (Eindhoven, The Netherlands).



1.10. Plans for the Future

Looking to the future, the centre will try to keep the position as a leading Austrian research institution. The next three years plan is already in preparation and hopefully will increase the international visibility of the instititue and the reputation of its home university.

The main focus is on fully exploiting existing instrumentation and introducing new leading edge equipment. We will be looking to introduce the following instruments during 2010:

- Analytical Scanning Transmission Electron Microscope (ASTEM)
- Confocal Raman microscope
- 3-D atomic force microscope with cryo-ultra-microtome
- Replacement of out-dated preparation equip-ment

In cooperation with TU Graz we will continue to develop the third extension of the new microscopy centre in the basement of the building at Steyrergasse 17.

The institute will continue to attract international scientific congresses and workshops to Styria in the coming years:

MC 2009 Graz

- *„Microsopy Conference 2009"*
  Dreiländertagung für Elektronenmikroskopie & 9th Multinational Conference on Microscopy"

31 August – 4 September 2009, in Graz

The conference will be organised by the Austrian Society for Electron Microscopy (ASEM), the Deutsche Gesellschaft für Elektronenmikroskopie (DGE) and the Swiss Society for Optics and Microscopy (SSOM). Since it is held in co-operation with the 9th Multinational Conference on Microscopy, we expect around 900 scientists mostly from the nine organising countries (Austria, Germany, Switzerland, Czech Republic, Italy, Croatia, Hungary, Slovenia, Serbia).

www.microscopy09.tugraz.at

1.11. Acknowledgements

The experimental work in electron microscopy requires not only highly motivated collaborators, but also significant financial support. Without the help of many institutions it would not be possible to maintain and to provide the high level of the instrumentation, the many cooperations and the quality of the results presented in this performance report.

We are especially grateful to university officials at TU Graz:

Rector O.Univ.-Prof. Dr. Hans SÜNKEL
Dean O.Univ.-Prof. Dr. Robert TICHY
Vicerector O.Univ.-Prof. Dr. Harald KAINZ

We are also grateful to all the coworkers of the central administration of the TU Graz, who are always open for questions and suggestions from the institute.

Financial support of our work was granted by many subsidizing organisations. These are in particular:

- Austrian Industrial Research Promotion Fund (FFG), Vienna
- Austrian Science Fund (FWF), Vienna
- Styrian Business Promotion Agency (SFG), Graz
- Land Steiermark, Graz
- Wirtschaftskammer Steiermark, Graz
- Bundesministerium für Wirtschaft und Arbeit, Wien
- Nanotechnologie Netzwerk Steiermark
- Faculty of Natural Sciences of the TU Graz and KFU Graz

Last but not least we must particularly thank

Professor Dipl.-Ing.Dr.h.c. Helmut LIST
Komm.Rat Dipl.-Ing. Ulrich SANTNER
Komm.Rat Dipl.-Ing. Dr. Gerhard KATZENBERGER

They spend much of their valuable time supporting our work in the "Verein zur Förderung der Elektronenmikroskopie".

2. Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung

2.1. The Organisation

The industrial associates' organisation was established in 1959 to support the institute and to facilitate greater interaction between industrial and academic scientists.
On the one hand the "Verein" supported the institute in terms of improvement of instrumentation thus enabling cutting-edge instrumentation which was always important because of the limited ressources of the university. On the other hand it allowed maintaining a high-skilled and well trained permanent staff in the Centre for Electron Microscopy Graz (ZFE).
The program is designed to provide industry with useful results from established and emerging new microscopy techniques and to keep the in-house specialists in industry in touch with the latest developments in the field.
Policies and procedures of the non-profit organisation are established by a steering committee consisting of academic and industrial scientists. Since 1995 the "Verein" is headed by Professor Dipl.-Ing. Helmut LIST (AVL Graz) and presently the "Verein" has 30 members mainly from Austria. Since the general business meeting on June 20th, 2008 the administrative body for the next six years is given as follows:

Presidential Committee:

President:	Prof. Dipl.-Ing. Dr-Ing.h.c. Helmut LIST
1. Vice president:	Komm. Rat Dipl.-Ing. Ulrich SANTNER
2. Vice president:	Komm. Rat DDipl.-Ing. Dr. Gerhard KATZENBERGER (until 2007)
	Mag. Christian KNILL (since 2008)

Managing Committee:

Head:	Komm. Rat Dipl.-Ing. Ulrich SANTNER
Financial referee:	DDr. Wilfried SCHÖNAUER
Representative of the Styrian Universities:	O.Univ.-Prof. Dr. Hartmut KAHLERT (until 2007)
	O.Univ.-Prof. Dipl.-Ing. Dr. Franz STELZER (since 2008)
Head of ZFE Graz:	Ao.Univ.-Prof. Dipl.-Ing. Dr. Ferdinand HOFER

Accounting Controller:

1. Controller:	Dr. Hermann PUCHER
2. Controller:	Mag. Petra SCHACHNER

Advisory Board:

Univ.-Prof. Dipl.-Ing. Dr. Horst CERJAK, Graz University of Technology
Univ.-Prof. Dipl.-Ing. Dr. Helmut CLEMENS, University of Leoben
Dipl.-Ing. Dr. Markus GAHLEITNER, Borealis AG, Linz
Univ.-Prof. Dr. Georg HOINKES, Karl-Franzens University of Graz
Dipl.-Ing. Dr. Armin HOLZNER, Semperit Technische Produkte, Wimpassing
Univ.-Prof. Dr. Hartmuth KAHLERT, Graz University of Technology (until 2007)
Min.Rat Dipl.-Ing. Dr. Stefan KOLARSKY, Bundesministerium für Bildung, Wissenschaft, Vienna
Univ.-Prof. Dr. Klaus LEDERER, University of Leoben (until 2008)

Univ.-Prof. Dipl.-Ing.Dr. Emil LIST, Graz University of Technology (since 2008)
Mag. Edmund MÜLLER, Joanneum Research, Graz
Univ.-Prof. Dr. Maria-Anna PABST, Medical University of Graz
Prof. Dr. Bernhard PELZL, Joanneum Research, Graz
Univ.-Prof. Dipl.-Ing. Dr. Wolfgang PRYBIL, Graz University of Technology (since 2008)
Univ.-Prof. Dipl.-Ing. Dr. Klaus REICHMANN, Graz University of Technology (since 2008)
Dipl.-Ing. Christian RAINER, Omya GmbH, Gummern
Univ.-Prof. Dipl.-Ing. Dr. Franz STELZER, Graz University of Technology
Vizerektor Dipl.-Ing. Dr. Johann THEURL, Graz University of Technology (until 2007)

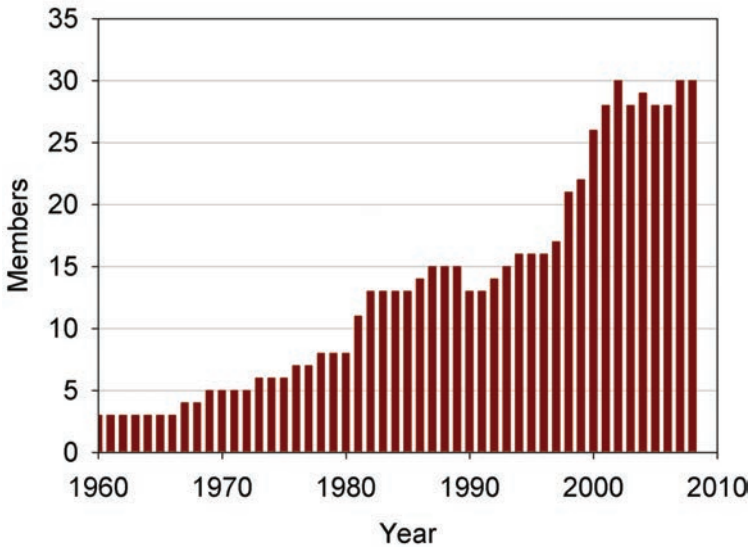
Members of the "Verein zur Förderung der Elektronenmikroskopie"

- Honorary Members: Hofrat Dr. Herwig HORN
  Univ.-Prof. Dipl.-Ing. Dr.techn. Wolfgang GEYMAYER

- Company Members:

Alicona Imaging GmbH, Grambach	Montanuniversität, Leoben
Austria Micro Systems AG, Unterpremstätten	OMYA AG Oftringen, Schweiz
Austria Technologie & Systemtechnik, Leoben	OMYA GmbH, Gummern
AVL List GmbH, Graz	Plansee AG, Reutte
Vishay Bccomponents Austria GmbH, Klagenfurt	Porzellanfabrik Frauental GmbH, Frauental
Böhler Edelstahl GmbH & Co KG, Kapfenberg	Semperit Techn. Produkte AG Holding, Wimpassing
Borealis GmbH, Linz	Surface Specialties Austria GmbH, Graz
Brigl & Bergmeister Papierfabrik GmbH, Niklasdorf	Papierfabrik Wattens GmbH, Wattens

EPCOS OHG, Deutschlandsberg	Technische Universität, Graz
Chemson Polymer Additive AG, Arnodstein	Constantia/Teich AG, Weinburg
Ingenieurbüro für Kunststofftechnik, Spielberg	Treibacher Auermet GmbH, Treibach-Althofen
Industriellenvereinigung, Graz	Karl-Franzens-Universität, Graz
Fritz-Haber-Institut der MPG, Berlin	Voith Paper Service GmbH, Wimpassing
Joanneum Research, Graz	GATAN, München
Wirtschaftskammer Steiermark, Graz	SEZ AG, Villach



Development of company members of the Verein zur Förderung der Elektronenmikroskopie

2.2. Austrian Cooperative Research (ACR)

The ZFE Graz is a member of the "Austrian Cooperative Research" (ACR) organisation. Since its foundation in 1954, ACR has offered specialised research and technology competences especially for the benefit of small and medium sized enterprises. ACR stimulates and enables innovation within trade and industry, thus improving the competitiveness of the Austrian economy.

The strengths of ACR-members:

- Close contact to small and medium enterprises
- Provision of cost-intensive scientific and technical infrastructure
- Intermediary between universities and SME
- Great flexibility of the work potential
- Cost-transparency and output-control/success governance
- Research and technology competence
- Experience of technology transfer
- Outsourced research and development department of several branches

Currently, ACR has 17 full members. In 2007, they had a total of 555 full time equivalent employees and produced a turnover of 54.7 million Euro out of which 86, 3% with SME.

The ZFE Graz cooperates with the following ACR institutes:
Österreichisches Gießerei Institut (ÖGI, Leoben),
Österreichisches Forschungsinstitut für Chemie und Technik (OFI, Wien)
Bautechnische Versuchs- und Forschungsanstalt (BVFS, Salzburg)
Forschungsinstitut der Vereinigung der Österreichischen Zementindustrie (VÖZFI, Wien)
Bautechnisches Institut (BTI, Linz).

ACR
AUSTRIAN COOPERATIVE RESEARCH
KOOPERATION MIT KOMPETENZ

Austrian Cooperative Research
Haus der Forschung
Sensengasse 1
1090 Wien
www.acr.at

3. Institute Representatives and Staff

Head of Institute

HOFER Ferdinand, Dipl.-Ing. Dr.techn., ao.Univ.-Prof.
KOTHLEITNER Gerald, Dipl.-Ing. Dr.techn. ao.Univ.-Prof.

Scientific Staff

ALBU Mihaela, Dipl.-Ing. Dr.techn.
BELEGRATIS Maria, Dr.rer.nat. ** (till December 2008)
BRUNEGGER Albert, Ing. (till August 2008)
CHERNEV Boril, Mag. Dr.rer.nat. *
GRANITZER Petra, Mag. Dr. ** (since June 2008)
GROGGER Werner, Dipl.-Ing. Dr.techn. ao.Univ.-Prof.
HABER Thomas, Dipl.-Ing. Dr.techn. ** (since September 2008)
INGOLIC Elisabeth, Dr.phil.
LETOFSKY-PAPST Ilse, Dipl.-Ing. Dr.mont.
MATSKO Nadejda, Dr. * (since February 2007)
MITSCHE Stefan, Dipl.-Ing. Dr.techn.
PLANK Harald, Dipl.-Ing. Dr.techn. (since August 2007)
PÖLT Peter, Dipl.-Ing. Dr.techn.
REICHMANN Angelika, Dipl.-Ing. Dr.techn. * (maternity leave since October 2008)
SCHAFFER Bernhard, Dipl.-Ing. Dr.techn. (till June 2008)
SCHRÖTTNER Hartmuth, Ing.
TCHERNYCHOVA Elena, Dr.** (till March 2007)
WAGNER Julian, Dipl.-Ing. Dr.techn.
WEWERKA Karin, Dipl.-Ing. Dr.techn. (since October 2008)
WILHELM Peter, Dr.phil.
ZEDLACHER Harald, Dipl.-Ing. * (till December 2008)

PhD students

GSPAN Christian, Dipl.-Ing. **
HAAS Wernfried, Dipl.-Ing. (since November 2008) **
RATTENBERGER Johannes Dipl.-Ing. *
RECHBERGER Werner, Mag.Mag. * (till June 2007)
REINGRUBER Herbert, Dipl.-Ing. ** (since November 2008)
RIEGLER Katharina Dipl.-Ing. * (till May 2008)
SCHAFFER Miroslava Mag. * (till July 2008)
SEZEN Meltem **
UUSIMÄKKI Toni ** (since January 2009)
ZANKEL Armin, Dipl.-Ing. *

Diploma thesis student

FLADISCHER Stefanie (since April 2008)

General Staff

BAHR Peter, Microscope operator
BIRNSTINGL Gerhard, Mechanic
BRANDL Christian, Microscope operator *
BRUNEGGER Margit, Chem.lab-assistant *
CZAPEK Wolfgang, Mechanic *
DIENSTLEDER Martina, Chem.lab-assistant *
ELIS Christof, Microscope operator

FREUND Angela, Cleaner *
GOGER Sabine, Secretariat *
GUSMAGG Anneliese, Secretariat *
KRANZELBINDER Elke, Cleaner
LUNELLI Martin, Chem.lab-apprentice
MAYRHOFER Claudia, Ing., Chem.lab assistant *
MERTSCHNIGG Sabrina, Chem.lab assistant * (since July 2008)
PALLER Manuel, Chem.lab assistant *
RAUCH Sebastian, Chem.lab assistant (since January 2009)
ROßMANN Anita, Chem.lab assistant *
SIMIC Sanja, Microscope Operator *
SITTSAM Markus, Mech. apprentice
STOISER Gernot, Image lab-apprentice
STREUßNIG Fatima, Secretariat *
STÜRZENBECHER Ulrike, Mag, Quality manager & Controlling *
WALLNER Margit, Design & image lab
WINDISCH Gerhard, Design, PC- & LAN-Admin.

* ZFE staff, ** supported by projects

Guest scientists

HÄUSSLER, Dietrich, University of Kiel, Kiel, Germany, May 19-24, 2007
KOTULA Paul, Sandia National Laboratory, Albuquerque, USA, Nov. 10-19, 2007
ROSSMANN Niko, University of Maribor, Faculty of Mechanical Engineering, Maribor, Slovenia, October-December 2007
KEAST Vicki J., School of Mathematical and Physica Sciences, University of Newcastle, Australia, January 2008


Mihaela Albu

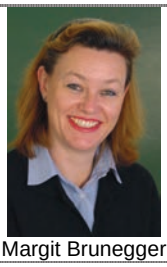

Peter Bahr


Maria Belegratis


Gerhard Birnstingl


Christian Brandl


Albert Brunegger


Margit Brunegger

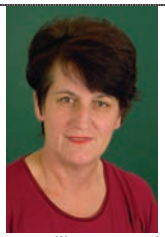

Boril Chernev


Wolfgang Czapek


Martina Dienstleder


Christof Elis


Stefanie Fladischer

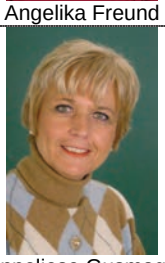

Angelika Freund


Sabine Goger


Petra Granitzer


Werner Grogger


Christian Gspan


Anneliese Gusmagg

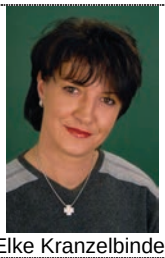

Wernfried Haas


Thomas Haber


Ferdinand Hofer


Elisabeth Ingolic


Gerald Kothleitner


Elke Kranzelbinder


Ilse Letofsky-Papst


Martin Lunelli


Nadejda Matsko


Claudia Mayrhofer


Sabrina Mertschnigg


Stefan Mitsche


Manuel Paller


Harald Plank


Peter Pölt

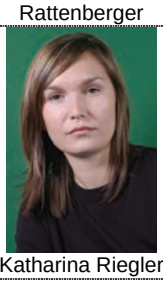

Johannes Rattenberger


Sebastian Rauch

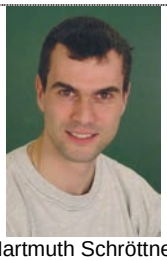
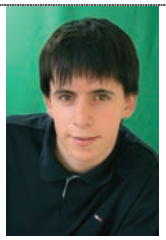

Werner Rechberger


Angelika Reichmann


Herbert Reingruber


Katharina Riegler


Anita Rossmann

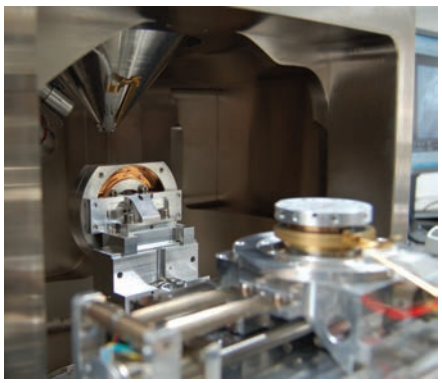
 Bernhard Schaffer	 Miroslava Schaffer	 Hartmuth Schröttner	 Emanuel Seidl	 Meltem Sezen
 Sanja Simic	 Markus Sittsam	 Gernot Stoiser	 Fatima Streussnig	 Ulrike Stürzenbecher
 Elena Tchernychova	 Toni Uusimäkki	 Julian Wagner	 Margit Wallner	 Karin Wewerka
 Peter Wilhelm	 Gerhard Windisch	 Armin Zankel	 Harald Zedlacher	

4. Laboratory Facilities

Scanning electron microscopes (SEM)

- High resolution SEM: ZEISS ULTRA 55
  0.1- 30kV, field emission source, with EsB detector, in-lens SE detector, STEM detector and EDAX Genesis EDX-system, parallel WDX-spectrometer Lambda-Spec (EDAX).

Johannes Rattenberger and Hartmuth Schröttner working at the HR scanning electron microscope Ultra 55 (Zeiss, Oberkochen).



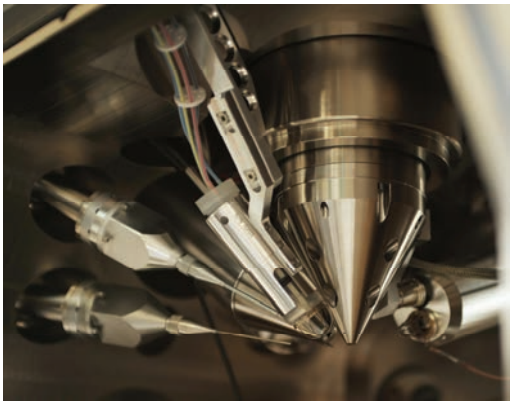
The specimen stage of the Zeiss Ultra 55.

- Analytical high resolution SEM: LEO Gemini DSM986
  0.1-30 kV, field emission gun, with EDX detector Noran Voyager 3105A, with TSL EBSD detector, micro hardness tester (Anton Paar) and cryogenic specimen transfer system (developed at FELMI-ZFE).
- Environmental Scanning Electron Microscope (ESEM): FEI Quanta 600 equipped with Noran Vantage EDX system, heating stage (up to 1500°C), Peltier cooling-stage and tensile testing stage.
- Environmental SEM (ESEM) FEI Quanta 200, W-cathode, EDAX SaphireEDX-system.
- 3View™ ultramicrotome (Gatan) for the ESEM Quanta 600.

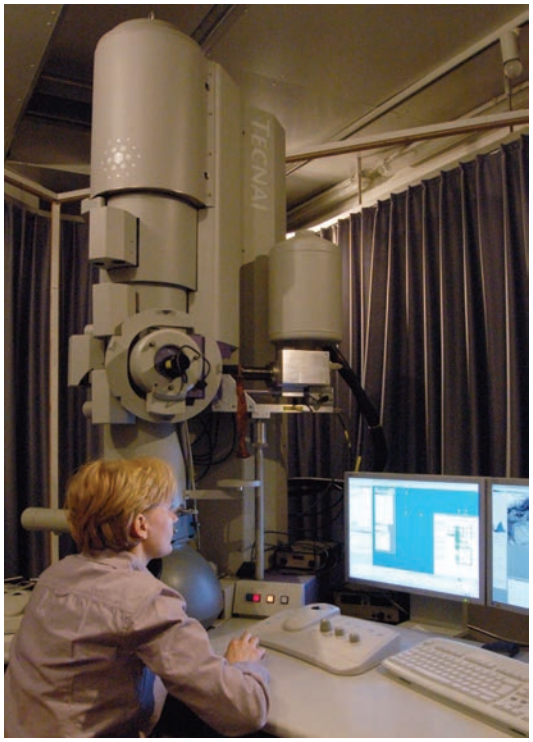
Focused ion beam instrument (FIB)

- FEI Nova™ 200 NanoLab: DualBeam™ FIB/SEM equipped with Omniprobe™ manipulator, various gas injection systems (Pt deposition, I_2 enhanced metal etch, XeF_2 insulator enhanced etch, TEOS-oxide deposition), XFlash 4010 X-ray spectrometer (Bruker AXS), direct ion detector (CDEM).

Focused ion beam microscope FEI Nanolab NOVA 200.



Specimen stage of the focused ion beam microscope FEI Nanolab NOVA 200.



Mihaela Albu operates the transmission electron microscope Tecnai F20 (FEI Company).

Transmission electron microscopes (TEM)

- Analytical high resolution TEM: FEI TECNAI F20
  200kV, field emission gun, supertwin objective lens, STEM (0.2 nm probe) with HAADF detector, with Wien filter monochromator, with EDX Si(Li) light element detector (EDAX) and high resolution Gatan imaging filter (HR-GIF, Gatan) with 2kx2k CCD camera (Gatan), magnetic field compensation system.
- Analytical TEM: Philips CM20
  200kV, LaB_6 cathode, twin lens, STEM with SE detector and Gatan BF/DF detector, EDX detector (HPGe, Noran) and Gatan Imaging Filter (including a 1kx1k CCD camera).
- Analytical TEM: FEI Tecnai 12: 120 kV, LaB_6 cathode, twin lens, and EDX Si(Li) detector with ultrathin window (EDAX), 1kX1k CCD camera, low-dose CCD camera.
- Specimen holders: Philips double tilt holder, Gatan cryo-transfer and cooling holder,
  Gatan double tilt cooling holder for analytical work, low background holders, rotation and heating holders.

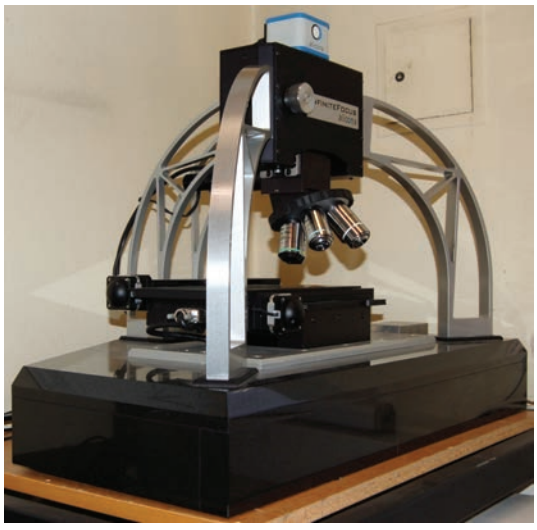
Atomic force microscope (AFM)

- Atomic force microscope VEECO Dimension 3100 with EFM and KPFM modes in a glove-box “Unilab MBraun 20”

Atomic force microscope VEECO Dimension in the glove box.

Light microscopes

- FT-IR microscopes:
  1) Bruker Equinox 55 spectrometer with Hyperion 3000 microscope, ATR objective (Ge crystal), grazing angle objective, MIRacle single reflection horizontal ATR unit (diamond and Ge crystal), Sadtler "KnowItAll" spectral libraries and search software.
  2) Spectra-Tech Advanced Analytical Microscope with ATR objective (ZnSe and Ge crystals), attached to a Bomem MB series spectrometer.
- Raman microscope: Renishaw system 2000, with Leica DMLM research microscope, dual laser system: diode laser (782 nm, 25 mW) and HeNe laser (633 nm, 17 mW), holographic notch filters, CCD detector, motorized xyz stage for mapping and confocal experiments, Raman imaging, electrochemical cell, hot-cold stage
- Light microscope: Zeiss Axioplan for observation with transmitted and reflected light with bright field, dark field, polarization, interference contrast (DIC), phase contrast and a Polaroid DMC Digital Microscope Camera.
- Advanced light microscope: "Alicona Infinite Focus" for 3D topography (Alicona, Grambach)



Advanced light microscope "Alicona Infinite Focus"

- Stereo light microscope Leica M Z6 for sample- preparation
- Light microscope METAVAR (Reichert)

Electron microscopical preparation equipment

- Diamond saw (Well)
- Diamond saw ISOMET 1000 (Buehler)
- Minimet polisher (Buehler)
- Ultrasonic disc cutter (Gatan)
- Dimple grinder (Gatan)
- Tripod polisher (Southbay Technology)
- Polisher Labopol
- Electrolytic thinning device (Struers TenuPol 5)
- Taper apparatus Leica EM TRIM
- Ultramicrotome OMU3 (Reichert-Leica)
- Ultramicrotome Ultracut UCT with EM FCS for low temperature sectioning (Leica)
- Microtome Supercut 2050 for light microscopy
- Ultramicrotome Ultracut E (Reichert-Leica)
- Sawing microtome (Leitz)
- Low angle ion milling apparatus (developed at FELMI-ZFE), equipped with low energy ion guns (Technoorg Linda)
- Ion milling and polishing system PIPS with digital zoom camera(Gatan)
- Cryo-plunging system (ZFE development)
- Cryo-preparation system EPA 101 with quadrupole mass spectrometer QMG311 (ZFE development)
- Evaporation and sputtering apparatus (ZFE development)
- Preparation system EPA 101 (ZFE development)
- Electron beam evaporators (Leybold, Balzers)
- Plasma cleaner based on EPA 101 and GEA (ZFE development)
- Plasma cleaner Fischione Model 1020
- Computer network with 95 computers, 4 switches (PC, Unix)
- Canon color laser printer and high quality printers for photographs

5. Academic Education

5.1. Lectures and Laboratory Courses

We offer modern and flexible courses including Master and Doctoral theses. The training of co-workers and students provided by involvement in research plays a crucial role. The FELMI staff offers the following courses in physics, chemistry, biotechnology and materials science at Graz University of Technology.

FELMI courses for masters and doctoral students of Technical Physics (TU Graz):

- 519.001 **Electron Microscopy in Solid State Physics I**, 2 VO, WS (Werner Grogger)

Analytical scanning electron microscopy, introduction to electron optics: electron sources, lenses, detectors, electron beam - specimen interactions, contrast formation, image quality and resolution limitations, image processing and – analysis, electron beam-microanalysis: EDX- and WDX-spectrometers, qualitative and quantitative analysis, special imaging techniques.

- 519.002 **Electron Microscopy in Solid State Physics II**, 2 VO, SS (Werner Grogger)

Analytical transmission electron microscopy: 1. introduction, basic crystallography, 2. electron scattering and diffraction, 3. diffraction patterns, 4. kinematical theory of electron diffraction, image contrast, 5. perfect and real crystal, 6. convergent beam electron diffraction 7. X-ray spectrometry, electron energy loss spectrometry, 8. specimen preparation.

- 519.007 **Electron Microscopy in Materials Science**, 2 VO, WS (Gerald Kothleitner)

Basic concepts of electron microscopy in scanning (SEM) and transmission (TEM) operation, specimen preparation techniques for electron microscopy, introduction to conventional imaging modes in the TEM: electron diffraction (ED), dark-field imaging (DF), high-resolution electron microscopy (HREM), spectroscopic techniques for the micro- and nanoanalytical characterization of solid state samples: x-ray spectroscopy (EDXS, WDXS) and electron energy-loss spectroscopy (EELS); analytical imaging techniques with energy-loss spectrometers (EFTEM): electron optics of energy-filter systems, image contents, image interpretation, resolution and detection limits, practical application examples (with particular focus on steels, alloys and composites).

Training of participants of the LLL-SEM school at the scanning electron microscope FEI Quanta 200.

- 519.008 **Materials Characterization by Electron Microscopy**, 2 PR, SS (Gerald Kothleitner)

Application of electron microscopical methods for the characterization of modern materials; the course is divided into two main parts: 1. microregion analysis: scanning electron microscopy (SEM) and energy-dispersive x-ray spectrometry (EDXS); specimen preparation; working with the microscope (recording of SE- and BSE-images); working with the EDX-spectrometer; qualitative and quantitative elemental analysis. 2. nanoregion analysis: transmission electron microscopy (TEM), energy-dispersive x-ray spectrometry (EDXS) and energy-loss spectrometry (EELS); specimen preparation; working with the microscope (recording of TEM-images and electron diffractions); working with EDX and EELS spectrometer; qualitative and quantitative elemental analysis.

- 519.013 **Analytical Electron Microscopy**, 2 VO, WS (F. Hofer)

Physical basis and types of electron microscopes (SEM and TEM) and typical examples for problem solving with microscopy; electron microscopical preparations; introduction to image formation; electron diffraction and basics of high resolution microscopy (HREM); micro- and nanoanalytical methods in EM: X-ray spectrometry (EDXS and WDXS); electron energy-loss spectrometry (EELS); scanning tunneling and atomic force microscopy; application examples: microstructure of inorganic and organic materials (steel, alloys, ceramics, polymers, composites), integrated circuits, heterogeneous catalysts, minerals, biomaterials and fine structure of biological tissue.

- 519.015 **Structure Analysis by High Resolution Electron Microscopy**, 2 VO, SS (F. Hofer)

Basics of electron diffraction, the design of the high resolution electron microscope, experimental conditions, convergent beam electron diffraction, imaging of crystalline and amorphous materials, simulation of high resolution images, applications in the field of nanostructured materials, Ceramics, semiconducting devices, e.g. defects, internal interfaces, nanoparticles and surfaces

- 519.005 **Colloquium Micro- and Nanoanalysis**, 2 WK, WS (F. Hofer, G. Kothleitner)
- 519.006 **Colloquium Micro- and Nanoanalysis**, 2 WK, WS (W. Grogger, F. Hofer)

Students, teachers and invited lecturers present or report on scientific work related to the research fields of the institute. The students present a lecture prepared under the supervision of a teacher, the student is also obliged to attend the other lectures of this term.

- 519.015 **Special Aspects of Analytical Electron Microscopy I**, 2 WK, WS (F. Hofer)
- 519.016 **Special Aspects of Analytical Electron Microscopy II**, 2 WK, WS (F. Hofer)

Scientific discussions related to current topics and master or doctoral students; instructions for scientific working and writing.

- 519.017 **New Methods in Electron Microscopy I**, 2 WK, WS (G. Kothleitner)
- 519.018 **New Methods in Electron Microscopy II**, 2 WK, SS (G. Kothleitner)

Scientific discussions related to current topics and master or doctoral students, instructions for scientific working and writing.

- 519.019 **Special Aspects of Transmission Electron Microscopy I**, 2K, WS (W. Grogger)
- 519.020 **Special Aspects of Transmission Electron Microscopy I**, 2K, WS (W. Grogger)

Scientific discussions related to current topics and master or doctoral students, instructions for scientific working and writing.

FELMI staff contributing to courses of other institutes at the TU Graz:

- 511.121 **Advanced Laboratory Excercises**, 5 PR, WS and SS (J. Wagner and teachers from the Institute of Experimental Physics)

The students attending this course have to solve advanced experimental problems in groups of two. The topics are optics, interferometry, spectrography, physics of lasers, solid state physics and surface physics. Students work with some more sophisticated equipment than in the basic labs.

- 513.119 **Experimal Laboratory Excercices**, 6 PR, WS and SS (W. Grogger, G. Kothleitner and teachers from the Institute for Solid State Physics)

The students attending this course have to solve advanced experimental problems in groups of two. The topics are optics, interferometry, spectrography, physics of lasers, solid state physics and surface physics. Students work with more sophisticated equipment than in the basic labs.

- 513.120 **Solid State Physics Laboratory Excercises**, 5 PR, SS (P. Pölt and teachers from the Institute of Solid State Physics)

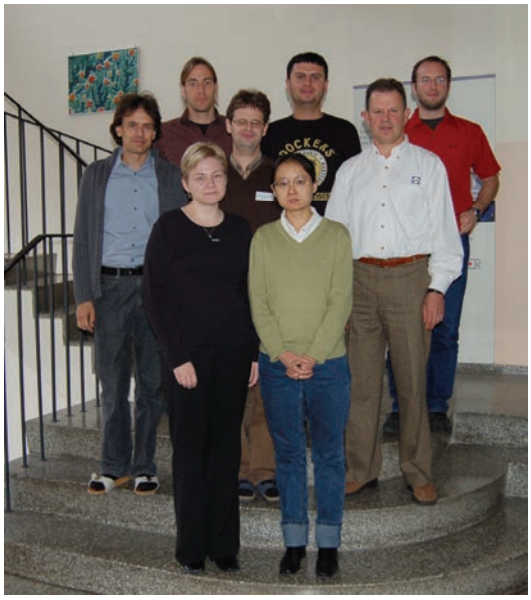
- 645.905 **Basics of Applied Analytical Chemistry VT**, 3 PR, WS, (P. Pölt and other

teachers from the Institute of Analytical Chemistry)

Scanning electron microscopy and microbeam analysis of solids in the field of chemical engineering, working with the microscope, qualitative and quantitative microanalysis using energy-dispersive x-ray spectrometry.

- 653.005 **Microscopy**, 2 PR, WS (G. Kothleitner and G. Gübitz, Institute of Environmental Biotechnology)

Schematic despription of magnification in microscopy, determination of size of microorganisms and quantitative counting, specific staining methods in microbiological identification identification and metabolism, fluorescence- and ultraviolet-microscopy, phase-contrast and polarization microscopy, principles of electron microscopy and demonstration of biological electron microscopy.



Speakers and participants of the International EELS & EFTEM School, FELMI-ZFE, November 2007.

LIFE LONG LEARNING

LLL course:
Problem Solving with Scanning Electron Microscopy and X-ray Microanalysis
(S. Mitsche, P. Pölt, A. Reichmann, H. Schröttner)

September, 16th – 18th, 2008

The course benefits scientists, engineers and technicians by helping them to solve their analytical problems. The course is a concentrated three day hands-on laboratory workshop taking participants step-by-step through the use of advanced scanning electron microscopy. Participants will be introduced to the important principles and methods in scanning electron by qualified staff members of the institute.
The course will familiarise them with the latest equipment and will cover the fundamental principles and methods critical to obtaining meaningful images, spectra and elemental maps. The methods are applicable to fields ranging from materials research (steels, ceramics, semiconductors, polymers, etc.) to biological research. An important aspect of the course is the practical use of the microscope. Several advanced microscopes are available and the participants are invited to bring their own samples and are given the opportunity to analyse them themselves with the help of the advisor.

LLL course:
GIF-School (EELS and EFTEM Course)
(G. Kothleitner, W. Grogger, B. Schaffer, F. Hofer)

February, 28th – March 2nd, 2007
November, 7th – 9th, 2007

The FELMI EELS & EFTEM course is a concentrated, three day hands-on laboratory workshop that will take participants step-by-step through the use of an integrated FEI energy-filtering / EELS system (CM20 - GIF, TF20 GIF). Participants are introduced to the important fundamental principles and methods in EELS and EFTEM acquisition and analysis by qualified staff

members. Basic familiarity with transmission electron microscopy is beneficial but no previous background in TEM analytical techniques is required. This course will familiarise participants with the latest EELS & EFTEM equipment and will teach the fundamental principles and methods critical to obtaining meaningful EELS spectra and energy-filtered images or elemental maps. The techniques are applicable to fields ranging from biological to materials research. The participants benefit from tightly coupled lectures and discussions with some of the top experts in the field and willl gain hands-on experience in various techniques. The participants learn about all important aspects of EELS/EFTEM acquisition and analysis and they will leave the workshop knowing how to apply and compare different methods.

5.2. Presentations and Lab Tours

Presentations and tours of the institute including lectures and demonstrations have also been organised for groups of physics and chemistry teachers and for students from TU Graz, schools and local universities. Around 200 pupils, teachers and students from other institutions visited the institute during the period 2007-08:

TOURS 2007

- January 18th, 2007 for students of the „Chemieingenieur-Schule“ in Graz (G. Kothleitner)
- March 27th, 2007 “Electron Microscopy” seminar for physics teachers from Styria (W. Grogger)
- July 3rd, 2007 for participants of the FIB workshop
- July 17th, 2007 for employees of ELLETRA Trieste (P. Wilhelm)
- September 20th, 2007 for the Institute for Polymer Sciences, University of Linz (A. Zankel)
- October 12th, 2007 for students of the „Chemieingenieur-Schule“ in Graz (G. Kothleitner, H. Schröttner, M. Dienstleder)
- November 14th, 2007 for employees of IPZ, TU Graz (H. Schröttner, J. Wagner)
- November 15th-22nd, 2007 Urania Seminar “Electron Microscopy” (W. Grogger, A. Bruneg-ger, E. Ingolic, P. Pölt)
- November 20th, 2007 “Electron Microscopy” Seminar for physics teachers from Styria (W. Grogger)
- December 12th, 2007 for pupils from the Akademisches Gymnasium Graz (E. Ingolic)

TOURS 2008

- February 6th, 2008 for pupils from the “Kepler Gymnasium” in Graz (S. Mitsche)
- March 25th, 2008 for the application team of ALICONA Imaging GmbH, Graz (H. Schröttner)
- April 11th, 2008 for Infineon Technologies, Villach and for SEZ Villach (G. Kothleitner)
- May 27th, 2008 for employees of other ACR-institutes (F. Hofer)
- September 17th, 2008 for a delegation of materials science professors from Bulgaria (in cooperation with the “Pädagogische Hoch-schule” in Graz (F. Hofer)
- October 8th, 2008 for an Ukrainian delegation (Austrian Ukrainian Science Day) (G. Kothleitner)
- November 12th, 2008 for pupils of the Gymnasium Kirchengasse in Graz (A. Zankel, E. Ingolic)
- November 20th-27th, 2008, Urania Seminar “Electron Microscopy” (W. Grogger, E. Ingolic, P. Pölt, M. Belegratis).

6. Diploma and Doctoral Theses

6.1. Doctoral Theses and Diploma Theses at the FELMI

- Finished habilitation thesis:
  Dipl.-Ing. Dr. Peter Pölt, "Particles and grains – the SEM as an ideal analysis tool"

- Finished doctoral theses:
  Dipl.-Ing. Andreas DITTMANN, "Micros-copical characterization of tribological systems"
  Dipl.-Ing. Michael ROGERS, "Preparation and electron microscopical charac-terisation of nanoparticles and functional nanostructures"
  MMag. Werner RECHBERGER, "High resolution scanning transmission electron microscopy in physics and materials science"
  Mag. Miroslava SCHAFFER, "Development of 3D Elemental Analysis using Focused Ion Beam Microscopy and Energy Dispersive X-ray Spectrometry"
  Dipl.-Ing. Katharina RIEGLER, "Optimizing Elemental Detection Sensitivity in Electron Energy-loss Spectroscopy by Multiple Linear Least Squares Fitting"

- Finished master theses:
  Dipl.-Ing. Franz SCHRANK, Master Thesis for Advanced Studies
  Dipl.-Ing. Michael ROGERS, Master Thesis for Advanced Studies

- Doctoral theses in progress:
  Dipl.-Ing. Christian GSPAN, "Electron micros-copical investigation of La(Sr,Co)O_3 perow-skites"
  Dipl.-Ing. Wernfried HAAS, "Morphology con-trol of high-performance polymer solar cells"
  Dipl.-Ing. Johannes RATTENBERGER, "Mic-roscopical characterization of materials surfaces"
  Dipl.-Ing. Herbert REINGRUBER, "In-situ experiments in a scanning electron microscope"
  Mag. Meltem SEZEN, „Investigation and modification of polymer structures by SEM and FIB"
  Dipl.-Ing. Armin ZANKEL, „*In-situ* experiments in the environmental scanning electron microscope"

- Master thesis in progress:
  Stefanie Fladischer, „Quantitative x-ray spectroscopy of thin films in transmission electron microscopy"

6.2. Doctoral Theses and Diploma Theses in other University Institutes

Graz University of Technology

Faculty for Technical Mathematics and Technical Physics

- Institute of Solid State Physics
  Dipl.-Ing. Lisbeth KAPPEL, Doctoral thesis
  Dipl.-Ing. Birgit JAHN, Doctoral thesis
  Dipl.-Ing. Piet REUTER, Doctoral thesis
  Dipl.-Ing. Evelin FISSLTHALER, Doctoral thesis
  Dipl.-Ing. Alexander BLÜMEL, Doctoral thesis
  Dipl.-Ing. Peter PACHER, Doctoral thesis
  Karlof MARGUC, Diploma thesis
- Institute of Experimental Physics
  Andreas APFOLTER, Diploma thesis
  Herbert REINGRUBER, Diploma thesis
- Institute for Materials Physics
  Dipl.-Ing. Martin SAGMEISTER, Doctoral thesis

Faculty of Chemistry, Engineering and Bio-technology

- Institute of Chemistry and Technology of Materials
  Andrea BALDUCCI, Diploma thesis
  Dipl.-Ing. Nicolaus S. HOCHGATTERER, Doctoral thesis
  Dipl.-Ing. Markus THALER, Doctoral thesis
  Mag. Marta PAWLAK, Doctoral thesis
  Dipl.-Ing. Peter RAIMANN, Doctoral thesis

Dipl.-Ing. Martin TSCHERNER, Doctoral thesis

- Institute of Analytical Chemistry and Food Chemistry
  Stefan KOREN, Diploma thesis
  Dipl.-Ing. Günter MISTELBERGER, Doctoral thesis
- Institute of Organic Chemistry
  Georg SCHITTER, Diploma thesis and Doctoral thesis
- Institute of Ressource Efficient and Sustainable Systems
  Dipl.-Ing. Thomas BRUNNER, Doctoral Thesis
  Dipl.-Ing. Markus JÖLLER, Doctoral Thesis
- Institute of Chemical Engineering and Environmental Technology
  Munazza MOSHIN, Doctoral thesis
- Institute for Biochemistry
  Mag. Tibor CZABANY, Doctoral thesis
  Ing. Miroslava SPANOVA, Doctoral thesis
  Dipl.-Ing. Andrea WAGNER, Doctoral thesis
- Institute of Biotechnology
  M.Sc. Malene THOMSEN, Doctoral thesis
- Institute of Paper, Pulp and Fibre Technology
  Dipl.-Ing. Ulrich HIRN, Doctoral thesis
  Christine VOURA, Doctoral thesis
- Institute of Environmental Biotechnology
  Dipl.-Ing. Anita EBERL, Doctoral Thesis
- Institute of Process and Particle Engineering
  Claire JEANQUARTIER, Diploma Thesis
  Dipl.-Ing. Stefan RADL, Doctoral Thesis
  Dipl.-Ing. Heidrun WÖLFLER, Doctoral Thesis

Faculty of Informatics

- Institute of Applied Information Processing and Communications
  Dipl.-Ing. Jörn-Marc SCHMIDT, Doctoral thesis

Faculty of Mechanical Engineering and Economics

- Institute of Material Science, Welding and Forming
  Bernhard FÜHRER, Diploma thesis
  Klaus KERSCHBAUMER, Diploma thesis
  Rene RADIS, Diploma thesis
  Denijel BURZIC, Doctoral thesis
  Dipl.-Ing. Wolfgang ERNST, Doctoral thesis
  Dipl.-Ing. Peter MAYR, Doctoral thesis
  Mag. Francisca MENDEZ MARTIN, Doctoral thesis
  Dipl.-Ing. Saeid SABERI, Doctoral thesis
  Dipl.-Ing. Andreas SCHWEIGER, Doctoral thesis
  Dipl.-Ing. Thomas WEINBERGER, Doctoral thesis
- Institute of Thermal Engineering
  Dipl.-Ing. Bernhard GATTERNIG, Doctoral Thesis

Karl-Franzens-University of Graz

- Institute of Zoology
  Elke McCULLOUGH, Diploma thesis
  Mag. Tobias PFINGSTL, Doctoral thesis
- Institute of Pharmaceutical Sciences
  Sabine ZEISMANN, Diploma thesis
- Institute for Physics
  Mag. Kashif NADEEM, Doctoral thesis
  Mag. Andreas TRÜGLER, Doctoral thesis
- Institute of Pharmaceutical Technology
  Mag. Karin WERNIG, Doctoral thesis

Medical University of Graz

- Department of Preventive and Operative Dentistry
  Helga PEINSITH, Diploma thesis
- Institute of Pathophysiology and Immunology
  Bernhard SVEJDA, Diploma thesis
  Victor AGUIRIANO MOSER, Diploma thesis

University of Leoben

- Department of General, Analytical and Physical Chemistry
  Stefan HOLZLEITNER, Diploma thesis
- Department of Physical Metallurgy and Materials Testing
  Dipl.-Ing. Robert FRANZ, Doctoral thesis
- Institute for Materials Science and Testing of Plastics
  Ermei WANG, Diploma thesis
- Polymer Competence Center
  Dipl.-Ing. Ute DASCHL, Doctoral thesis

University of Vienna

- Institute for Material Physics
  Dipl.-Ing. Clemens MANGLER, Doctoral thesis

University of Innsbruck

- Institute for Mineralogy and Petrography
  Michael WEISSENBACHER, Diploma thesis

Martin Luther University Halle, Germany

Dipl.-Ing. Michael NASE, Doctoral thesis
Dipl.-Ing. Hans Joachim RADUSCH, Doctoral thesis

University of Maribor, Slovenia

Iztok SVAB, Diploma Thesis
Saska LIPOVSEK, Diploma Thesis

Peter Pölt after his habilitation lecture in December 2007.

Teaching electron microscopy for chemistry students (G. Kothleitner at the FEI Tecnai F20).

7. Presentations at the Institute

January 26th, 2007:
Prof. Dr. Barbara HINTERSTOISSER (Department für Materialwissenschaften und Prozesstechnik, BOKU Wien) „Modifiziertes Holz“

February 9th, 2007: **Helmut GNAEGI**
(Diatome AG, Biel, Switzerland) „Anwendung der Ultramikrotomie zur Probenpräparation von Werkstoffen für TEM, REM und AFM“

February 12th, 2007: **Dipl.-Ing. Thomas RIEDL**
(Leibniz Institute for Solid State and Materials Research, IFW Dresden, Germany) “Strain and Mn Valency of Thin $La_{0.7}Sr_{0.3}MnO_3$ Films on $SrTiO_3$ Substrate”

March 23rd, 2007: **Dr.-Ing. René ANDROSCH**
(Zentrum für Ingenieurwissenschaften, Martin-Luther-Universität Halle-Wittenberg, Germany) „Struktur, Morphologie und Reorganisation von Nanokristallen in isotaktischen Polypropylen“

May 14th, 2007: **Prof. Dr. Ray EGERTON**
(University of Alberta, Edmonton, Canada) “Techniques and applications of electron energy-loss spectroscopy”

May 15th, 2007: **Dr. Esko KAUPPINEN**
(University of Technology, Helsinki, Finland) ”Nanobuds – a novel carbon nanomaterial”

June 22nd, 2007 **DI Dr. Alexandra LOIDL**
(Technologieverwertung, TU Graz) „Patente und Schutzrechte – Vorgangsweisen an der TU Graz“

September 18th, 2007: **Dr. Bruno ACHARD**
(JEOL Europe SAS, Paris, France) „New developments in high resolution electron microscopy“

October 17th, 2007: **Prof. Dr. Emil BURZO**
(Babes-Bolyai University Cluj Napoca, Romania) “Rare-earth intermetallic compounds: basic aspects and technical applications”

November 14th, 2007: **Dr. Paul G. KOTULA**
(Sandia National Laboratories, Albuquerque, USA) “Advances in Spectrum Imaging Techniques”

November 23rd, 2007:
Prof. Dr. Friedrich SCHÄFFLER
(Institute of Semiconductor Physics, University of Linz, Austria) “Self-Organized Nanostructures”

November 30th, 2007: **Dr. Peter GNAUK**
(Carl Zeiss NTS, Oberkochen, Germany) „Hochauflösende Rasterelektronenmikroskopie mit dem Zeiss Ultra 55“

December 14th, 2007:
Dr. Christof MITTERBAUER
(FEI Company, Eindhoven, The Netherlands) „Neue Entwicklungen in der Transmissionselektronenmikroskopie“

December 20th, 2007: **Dr. Ondrej L. KRIVANEK**
(NION Company, Seattle, USA) „Applications of Aberration-Corrected STEM“

January 11th, 2008: **Dr. Petra GRANITZER**
(Institute of Physics, Karl-Franzens-Universität Graz, Austria) “Porous silicon as matrix für ferromagnetic nanostructures“

January 15th, 2008: **Dr. Peter van AKEN**
(Max-Planck-Institut für Metallforschung, Stuttgart, Germany) “New developments in electron energy-loss spectroscopy in materials science“

January 29th, 2008: **Dr. Vicki J. KEAST**
(School of Mathematical and Physical Sciences, University of Newcastle, Australia) “Low-loss EELS: What it can do for you”

February 6th, 2008: **Dr. Bernd DIPPEL**
(Perkin Elmer LAS, Vienna, Austria) “Eine neue Grenzerfahrung – höchste Ortsauflösung mit ATR Imaging“

February 6th, 2008: **Pierre DELMONT**
(Perkin Elmer LAS, Vienna, Austria) “The simplicity of Raman – Raman imaging applications – the new Perkin Elmer Raman Product Line”

March 14th, 2008: **Georg MICHENTHALER**

(Institut für empirische Sozialforschung, Vienna, Austria) „Ergebnisse der Mitarbeiter- und Kundenbefragung Sommer 2007“

March 28th, 2008: **Mag. Oliver HUBER**
(Alicona Imaging, Grambach, Austria) ”Infinite Focus Messgerät – Funktionsweise und Anwendungsgebiete”

April 18th, 2008: **Dr. Harald DITLBACHER**
(Institute of Physics, Karl-Franzens-University Graz, Austria) „Oberflächen- bzw. Partikel-Plasmonen und EELS“

June 6th, 2008: **Prof. Dr. Otto GLATTER**
(Institute of Chemistry, Karl-Franzens-University Graz, Austria) „Nanostrukturierte Emulsionen als Carrier Systeme für funktionelle Moleküle und als Basis für transfer Studien“

June 18th, 2008: **Dr. Anton EFIMOV**
(Nanoscan Technology, Moscow, Russia)
“Advanced AFM and combined optical systems”

June 20th, 2008: **DI Dr. Georg JAKOPIC**
(Institut für Nanostrukturierte Materialien und Photonik, Joanneum Research, Weiz, Austria) „Impedanz-Spektroskopie an organischen Halbleitern“

September 19th, 2008: **Dr. Maria BIEL**
(Krakow, Poland) “Microstructure and Properties of Surface Treated Titanium Biomaterials”

November 20th, 2008: **Prof. Dr. Paul MIDGLEY**
(Department of Materials Science and Metallurgy, University of Cambridge, Great Britain) “3D Nanoscale Imaging and Analysis by Electron Tomography”

Dr. Ondrej Krivanek (Nion, Seattle) speaks in the institute seminar.

8. Publications of Institute Staff

8.1. Publications 2007 (peer reviewed)

Schaffer, M., Wagner, J., Schaffer, B., Schmied, M., Mulders, H.
"Automated three-dimensional X-ray analysis using a dual-beam FIB", Ultramicroscopy 107 (2007) 587-597.

Zankel, A., Ingolic, E., Pölt, P.
"New in situ investigation methods of polymers in the environmental SEM (ESEM)", Scanning 29 (2007) 53-54.

Schaffer, M., Wagner, J.
"Block lift-out sample preparation for 3-D experiments in a dual-beam focused ion beam microscope", Microchimica Acta (2007) 1-6.

Thomsen, M., Pölt, P., Nidetzky, B.
"Development of a microfluidic immobilised enzyme reactor", Chemical Communications (2007) 2527-2529.

Mitsche, S., Pölt, P., Sommitsch, C.
"Recrystallization behaviour of the nickel-based alloy 80A during hot forming", Journal of Microscopy 227, 3 (2007) 267-274.

Schaffer, M., Wagner, J., Schröttner, H., Schmied, M.
"Automated X-ray elemental analysis in three dimensions using a dual-beam focused ion beam system", Praktische Metallographie 44, 5 (2007) 248-250.

Zankel, A., Pölt, P., Gahleitner, M., Ingolic, E., Grein, C.
"Tensile tests of polymers at low temperatures in the environmental scanning electron microscope: An improved cooling platform", Scanning 29 (2007) 261-269.

Tschaikner, A., Ingolic, E., Stoyneva, M. P., Gärtner, G.
"Autosporulation in the soil alga Coelastrella terrestris", Phytologia Balcanica 13, 1 (2007) 29-34.

Wolf, C., Lederer, K., Pfragner, R., Schauenstein, K., Ingolic, E., Siegl, V.
"Biocompatibility of ultra-high molecular weight polyethylene (UHMW-PE) stabilized with α-tocopherol used for joint endoprosthesis assessed in vitro", Journal of Materials Science / Materials in medicine 18, 6 (2007) 1247-1252.

Rumpf, K., Granitzer, P., Pölt, P., Reichmann, A., Hofmayer, M., Krenn, H.
"Characterisation of a ferromagnetic porous silicon-based Ni/Si nanocomposite with a novel strong high-field anisotropy", Physica / E 37, 1-2 (2007) 270-273.

Nase, M., Zankel, A., Langer, B., Baumann, H.-J., Grellmann, W.
"Characterization of the peel behaviour of polyethylene/polybutene-1 peel-systems using peel tests and the in situ environmental scanning electron microscopy", Scanning 29 (2007) 50-51.

Rumpf, K., Granitzer, P., Pölt, P., Reichmann, A., Krenn, H.
"Fabrication and optical properties of a self-organized ferrromagnetic Ni/Si-nanocomposite", Journal of Magnetism and Magnetic Materials 316 (2007) 114-117.

Wriessnegger, T., Gübitz, G., Leitner, E., Ingolic, E., Cregg, J., de la Cruz, B. J., Daum, G.
"Lipid composition of peroxisomes from the yeast Pichia pastoris grown on different carbon sources", Biochimica et Biophysica Acta 1771 (2007) 455-461.

Granitzer, P., Rumpf, K., Pölt, P., Reichmann, A., Hofmayer, M., Krenn, H.
"Magnetization of self-organized Ni-nanowires with peculiar magnetic anisotropy", Journal of Magnetism and Magnetic Materials 316 (2007) 302-305.

Sonderegger, B., Mitsche, S., Cerjak, H.-H.

"Martensite laths in creep resistant martensitic 9-12% Cr steels - Calculation and measurement of misorientations", Materials Characterization (2007) 874-882.

Stubenrauch, K., Fritz, G., Ingolic, E., Grogger, W., Glatter, O., Stelzer, F., Trimmel, G.
"Microphase separation study of amphiphilic ROMP block copolymers by SAXS and TEM", Macromolecules 40 (2007) 4592-4600.

Moskalewicz, T., Schaffer, B., Manescu, A., Rustichelli, F., Czyrska-Filemonowicz,, A.
"Microstructure characterisation and stress analysis of the $Ti_{48}Al_2Ag$ coating on the near-α titanium alloy", Surface & Coatings Technology 201 (2007) 7635-7640.

Brossmann, U., Sagmeister, M., Pölt, P., Kothleitner, G., Letofsky-Papst, I., Würschum, R.
"Microwave plasma synthesis of nano-crystalline YSZ", Physica Status Solidi / Rapid research letters (2007) 107-109.

Balog, M., Keckes, J., Hofer, F., Galusek, D., Huang, J.-L., Sajgalik, P.
"Nano/micro-hardness and fracture resistance of Si_3N_4/SiC composites with up to 13 wt% of SiC nano-particles", Journal of the European Ceramic Society 27, 5 (2007) 2145-2153.

Tschaikner, A., Ingolic, E., Holzinger, E., Gärtner, G.
"Phycobionts of some species of Evernia and Ramalina", Herzogia 20 (2007) 53-60.

Granitzer, P., Rumpf, K., Pölt, P., Reichmann, A., Krenn, H.
"Quasi-regular self-organized porous silicon channels metallized with Ni-structures of strong anisotropy", Journal of Magnetism and Magnetic Materials (2007) e838 - e840 .

Granitzer, P., Rumpf, K., Pölt, P., Reichmann, A., Krenn, H.
"Self-assembled mesoporous silicon in the crossover between irregular and regular arrangement applicable ", Physica / E 38 1-2 (2007) 205-210.

Wenzl, F., Fian, A., Pölt, P., Rudorfer, A., Leising, G.
"Thin film morphology and ion interaction behaviour of functional polymers for iontronic applications", Electrochimica Acta 52 (2007) 6229-6239.

Buchgraber, C. H., Svagera, R., Ebel, M., Schröttner, H., Kern, W.
"UV-assisted surface modification of polybutadiene with phosphorus-containing groups", Macro-molecular Chemistry and Physics 208 (2007) 1159-1167.

Matsylitskaya, V.A., Brunkahl, O., Kothelitner, G., Bock, W., Kolbesen B.O.
"Annealing of evapoarted and sputtered niobium films in oxygen and nitrogen rich atmospheres by rapid thermal processing (RTP)", phys.stat.sol. (c) 4 (6) (2007) 1802-1816.

Tschaikner, A., Ingolic, E., Gärtner, G.
"Observations in a new isolate of Coelastrella terrestris (REISIGL) HEGEWALD & HANAGATA (Chlorophyta, Scenedesmaceae) from alpine soil (Tyrol, Austria)", Phyton 46 (2007) 237-245.

Publications 2007 (conference papers, proceedings, other publications)

Lazar, P., Redinger, J., Poldloucky, R., Rashkova, B., Dehm, G., Kothleitner, G., Sturm, S., Kutschej, K., Mitterer, C., Scheu, C.
"A combined ab-initio and experimental study of N-K, Ti-$L_{2,3}$, and V-$L_{2,3}$ electron energy-loss near edge structures for TiN and VN films", 14th Conference on Solid State Analysis, Friedbacher, G., ed., Abstract Book, Vienna (2007) KV9.

Grogger, W., Schaffer, B., Gspan, C., Rechberger, W., Kothleitner, G., Hofer, F.
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"Chemical control of the local charge carrier concentration in organic thin film transistors", 8th International Symposium on Functional Pi-Electron Systems", Graz, Abstracts (2008) T- 45.

Albu, M., Mendez Martin, F., Kothleitner, G.
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“High-temperature oxidation of steel in the ESEM with subsequent scale characterisation by Raman microscopy”, European Microscopy Conference EMC 2008, vol.1, M. Luysberg, K. Tillmann, T. Weirich (eds.), Springer-Verlag Berlin Heidelberg (2008) pp. 611-612.

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“In situ testing of plastics with the ESEM - chances for the design of new investigation methods”, 13th International Conference Polymeric Materials, in “Polymeric Materials-13”, Verlag Universität Halle-Wittenberg (2008) PI-39.

Pölt, P., Reingruber, H., Zankel, A., Elis, C.
“In-situ experiments on soft materials in the environmental SEM - reliable results or merely damage?”, European Microscopy Conference EMC 2008, vol.2, S. Richter, A. Schwedt (eds.), Springer-Verlag Berlin Heidelberg (2008) pp. 779-780.

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"Silicon/metal hybrid nanosystem with tailored magnetic properties in two characteristic field regions", ECS Transactions, The Electrochemical Society, 16, 3 (2008) 83-89.

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"Tensile tests and ultramicrotomy - the environmental scanning electron microscope as a versatile tool for the investigation of the fracture behaviour of polymers ", Book of Abstracts PPS-24, 24th Annual Meeting of the Polymer Processing Society, 24 (2008) p. II.377.

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"The (environmental) scanning electron microscope - a valuable tool for polymer characterisation?", 13th International Conference Polymeric Materials, in "Polymeric Materials-13", Verlag Universität Halle-Wittenberg (2008) A40.

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"Ultramicroscopy in the ESEM, a versatile method for the 3D reconstructures in botany", Austrian Society of Plant Biology, 17 (2008) 139-140.

Zankel, A.; Kraus, B.; Schaffer, M.; Pölt, P.; Ingolic, E.; Schröttner, H.
"Ultramicrotomy in the ESEM, a versatile method for the 3D reconstruction of specimens", Microscience-2008, The Royal Microscopy Society (2008) F1.1, p. 4.

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"Performance of functional textiles visualized by ESEM and assessed by physiological tests", 8th AUTEX Conference 2008, Italy, CD-ROM, Session 13.

Grogger, W., Hofer, F., Kothleitner, G., Schaffer, B.
„Nanoanalysis of materials by means of EELS spectrum imaging in a transmission electron microscope", 10th Annual Conference „YUCOMAT 2008", The Book of Abstracts, Uskoković, D.P. (Ed.), Institute of Technical Sciences, Belgrade (2008) p. 42.

Hofer, F.
"Nanoanalytik für die Halbleitertechnologie", Report (+) Plus / Innovationsreport (2008) p. 15.

Schiller, M.; Huisman, H.; Pelzl, B.; Klaess, P.; Wilhelm, P.; Chernev, B. S.
"Chalk as a filler - effect on weather behavior", Kunststoffe International 98, 5 (2008) 68-71.

Schiller, M., Huisman, H., Pelzl, B., Klaess, P., Wilhelm, P., Chernev, B. S.

„Kreide als Füllstoff - Einfluss auf die Bewitterung“, Kunststoffe (München) / Deutsche Ausgabe 98, 5 (2008) 102-105.

Schiller, M., Pelzl, B., Lavrencic, R., Woschitz, J., Schröttner, H., Schwarz, S.

„Umwelteinflüsse auf die Bewitterung (4) – Untersuchungen an europäischen Fensterprofilen“, GAK - Gummi, Fasern, Kunststoffe 61, 2 (2008) 88-102.

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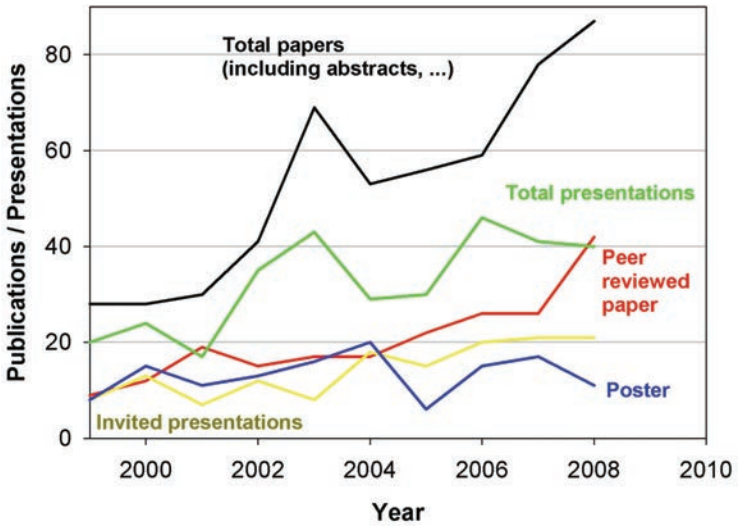


Development of publications and presentations of the FEMI-ZFE staff.

9. Oral Presentations of Institute Staff

9.1. Oral Presentations 2007

Grogger, W. (invited)
"High energy resolution EFTEM series in the low-loss regime", International Workshop on New Trends in Electron Microscopy, Ringberg Castle, Tegernsee, Germany, March 7th, 2007.

Hofer, F. (invited)
"Low-loss EELS with monochromated electrons", International Workshop on New Trends in Electron Microscopy, Ringberg Castle, Tegernsee, Germany, March 7th, 2007.

Grogger, W. (invited)
"Advanced nanoanalysis in the transmission electron microscope", Holiday course: "Probing the Nanoworld-Microscopies, Scattering and Spectroscopies of the Solid State", Research Centre Jülich, Germany, March 14th, 2007.

Pölt, P.
"EBSD - some experimental and some fundamental problems", 14th Conference and Workshop on Electron Backscatter Diffraction, New Lanark, Scotland, March 26th, 2007.

Zankel, A.
"New in situ investigation methods of polymers in the environmental SEM (ESEM)", The 18th Annual International Scientific Meeting on Scanning Microscopies, Monterey, USA, April 10th, 2007.

Zankel, A.
"Characterization of the peel behavior of polyethylene/polybutene-1 peel-systems using peel tests and in situ environmental scanning electron microscopy", The 18th Annual International Scientific Meeting on Scanning Microscopies, Monterey, April 10th, 2007.

Hofer, F.
"Elektronenmikroskopie als Werkzeug der Nanotechnologie", Workshop "Die Welt des Kleinen", Graz, Austria, May 7th, 2007.

Wagner, J. (invited)
"Dual beam FIB and analytical electron microscopy", Nanoforum, University of Linz, Austria, May 17th 2007.

Hofer, F. (invited)
"New developments in analytical electron microscopy", 4th Irsee Symposium on Selective Oxidation Catalysis, Kloster Irsee, Germany, June 7th, 2007.

Chernev, B.S.
"FTIR multichannel imaging. contrast improvement by image analysis", ICAVS, Korfu, Greece, June 10th, 2007.

Hofer, F. (invited)
„Elektronenmikroskopische Analysen in der Nanoanalytik", NSI Symposium, Techcenter Linz, Austria, June 11th, 2007.

Hofer, F. (invited)
„Anwendungen der Energiefilterungs-TEM in den Materialwissenschaften", CRISP Inauguration, CAESAR, Bonn, Germany, June 14th, 2007.

Rogers, M.
"Recent advances in FIB specimen preparation and nanostructuring", 8th Multinational Congress on Microscopy, Prague, Czech Republic, June 17th, 2007.

Albu, M.
"Quantitative compositional analysis of secondary carbides in a 9Cr-1Mo steel", 8th Multinational Congress on Microscopy, Prague, Czech Republic, June 17th, 2007.

Riegler, K.
"Detector characterization and optimization of detection limits in EELS studied on natural ruby", 8th Multinational Congress on Microscopy, Prague, Czech Republic, June 17th, 2007.

Pölt, P. (invited)
"The ESEM - a microanalytical tool and a microlab", 8th Multinational Congress on Microscopy, Prague, Czech Republic, June 17th, 2007.

Kothleitner, G. (invited)
"Recent developments in HR-EELS and EFTEM", 8th Multinational Congress on Microscopy, Prague, Czech Republic, June 18th, 2007.

Grogger, W. (invited)
"Nanoanalysis in materials science using spectrum imaging methods", 8th Multinational Congress on Microscopy, Prague, Czech Republic, June 18th, 2007.

Schröttner, H. (invited)
„Feinstrukturuntersuchungen an Polymeren", Seminar, AGORA Business Center München-Laim, Germany, June 20th, 2007.

Zankel, A. (invited)
"Environmental scanning electron microscopy (ESEM) - a versatile method for the characterisation and for in situ investigations of polymers", 11th Conference "Deformation und Bruchverhalten von Kunststoffen", Merseburg, Germany, June 20th, 2007.

Wagner, J.
"Device modification: challenges and solutions", FIB Workshop 2007, Graz, Austria, July 2nd, 2007.

Schaffer, M.
„Energiedispersive Röntenspektroskopie im Dual-beam Focused Ion Beam Mikroskop - Mikroanalytik in drei Dimensionen", FIB Workshop 2007, Graz, Austria, July 2nd, 2007.

Schaffer, B.
"Spectrum-Imaging im analytischen TEM - aktuelle Entwicklungen und Anwendungen", 14th Conference „Festkörperanalytik", Wien, Austria, July 16th, 2007.

Kothleitner, G. (invited)
„Automated EFTEM spectrum image acquisition: overcoming EELS dynamic range problems", Microscopy and Microanalysis 2007, Fort Lauderdale, Florida, USA, August 3rd, 2007.

Sezen, M.
"Ion beam irradiation damage on poly(3-hexylthiophene) (P3HT) based organic optoelectronic devices: can cryo-FIB help?", Microscopy and Microanalysis 2007, Fort Lauderdale, Florida, USA, August 05th, 2007.

Hofer, F.
"Cooperation between Austrian Cooperative Research and the Slovak Academy of Sciences in the field of advanced materials", Forschung Austria Workshop, Alpbach, Tirol, Austria, August 22nd, 2007.

Schaffer, B.
"Applied nanoanalysis by electron energy-loss spectrum imaging methods", Microscopy Conference MC2007, Saarbrücken, Germany, September 2nd, 2007.

Matsko, N.
"Correlative ultrastructural analysis of biological and polymer materials using complementary sets of AFM and TEM Images", Microscopy Conference MC2007, Saarbrücken, Germany, September 2nd, 2007.

Hofer F.
"Low-loss EELS measurements with monochromated electrons", Microscopy Conference MC2007, Saarbrücken, Germany, September 2nd, 2007.

Kothleitner G. (invited)
"Analytical electron microscopy: an independable tool for modern materials science", 5th Conference New Research Trends in Material Science ARM-5, Sibiu, Romania, September 5th, 2007.

Kothleitner, G. (invited)
"Local structure and bonding determined by EELS", ELCRYST 2007, Aachen, Germany, September 16th, 2007.

Wagner, J.
„Anwendungen und Möglichkeiten der Elektronenmikroskopie in der Mikro- und Nanoanalytik", Hochauflösende Oberflächenanalytik im Nanometerbereich, Wien, Austria, September 19th, 2007.

Hofer, F.
„Nanoanalytik in der Halbleitertechnologie", Infineon Villach, Austria, September 20th, 2007.

Hofer F. (invited)
"Electron microscopy I – methods and applications" and "Electron microscopy II – methods and applications", Autumn School on Materials Science and Electron Microscopy 2007, Humboldt University of Berlin, October 9th, 2007.

Wagner, J. (invited)
"FIB tomography and elemental analysis", Workshop on tomography in materials science using TEM and FIB, Berlin, October 11th, 2007.

Grogger, W. (invited)
"Chemical mapping on the nanometer length scale", Materials Research Society Fall Conference 2007, Boston, USA, November 25th, 2007.

Pölt, P.
„Polymere - Zugversuche im ESEM", Meeting of Working Group "Korrosion von Polymerwerkstoffen", Darmstadt, Germany, November 29th, 2007.

Schröttner, H.
„Oberflächenmikroskopie von Werkstoffen mit dem Hochauflösungs-Rasterelektronemikroskop ZEISS Ultra55", Workshop on Advanced Microscopy, Graz, Austria, November 30th, 2007.

Hofer, F. (invited)
"Energy-filtered transmission electron microscopy in materials science", Symposium on Microstructural Characterisation down to the Atomic Scale, Leoben, December 5th, 2007.

Hofer, F. (invited)
"EELS and EFTEM instrumentation and new applications in materials science", Winter School ESTEEM, Universite Paris-Sud, Orsay, France, December 10th, 2007.

Schröttner, H.
"Electron microscopy: application to semiconductor materials and device characterization", NXP Semiconductors, Gratwein, Austria, December 11th, 2007.

9.2. Oral Presentations 2008

Hofer, F. (invited)
"Über den Nutzen der Elektronenenergieverlustspektroskopie in der biomedizinischen Forschung" Symposium Professor Maria-Anna Pabst, Medical University of Graz, Austria, January 28th, 2008.

Sezen, M.
„Nanoanalysis and nanostructuring for organic optoelectronic devices", ISOTEC Seminar, Mariazell, Austria, January 17th, 2008.

Matsko, N.
"Correlative ultrastructural analysis of organic polymer materials using AFM and TEM investigations" ISOTEC Seminar, Mariazell, Austria, January 18th, 2008.

Gspan, C.
"Crystal structure of $La_{0.4}Sr_{0.6}CoO_{2.71}$ investigated by TEM and XRD", Seminar Institute of Physical Chemistry, University of Leoben, Austria, January 29th, 2008

Kothleitner, G. (invited)
"EELS-EFTEM of steels", Workshop on EELS/EFTEM of Steel, Wuhan, China, March 3rd - 8th, 2008.

Sezen, M.
"FIB / SEM and conjugated polymers: how compatible?" Materials Research Society Spring Meeting 2008, San Francisco, USA, March 24th, 2008.

Kothleitner G. (invited)
"Introduction to energy-filtering TEM", GATAN, Pleasanton, USA, April 15th, 2008.

Kothleitner G. (invited)
"EFTEM Applications", GATAN, Pleasanton, USA, April 16th, 2008.

Chernev, B.S.
"Vibrational spectroscopic imaging methods for investigation of pharmaceutical products", International Graz Workshop for Pharmaceutical Engineering, Graz, Austria, May 15th, 2008.

Hofer, F.
"Reise in die Welt des Mikrokosmos", Exhibition "Reise in die Welt des Mikroskosmos", TÜV Vienna, Austria, May 20th, 2008.

Wilhelm, P. (invited)
„Ein Bild sagt mehr als 1000 Spektren. IR-Bildgebung in der Praxis der Materialcharakterisierung", Institut für Polymerforschung Dresden, Dresden, Germany, May 22nd, 2008.

Grogger W. (invited)
"Advanced Nanoanalysis in TEM", Laboratory for Electron Microscopy, AGH University of Science and Technology, Krakow, Poland, June 3rd, 2008.

Hofer, F. (invited)
"Low-loss EELS with monochromated electrons", XIII International Conference on Electron Microscopy, Zakopane, Krakow, Poland, June 08th, 2008.

Pölt, P. (invited)
"Tensile tests and ultra-microtomy - The environmental scanning electron microscope as a versatile tool for the investigation of the fracture behaviour of polymers", Polymer Processing Society, 24th Annual Meeting, Salerno, Italy, June 15th, 2008.

Schröttner, H.
"In situ ultramicrotomy in the ESEM – A versatile method for life sciences and materials science", Microscience 2008, London, U.K., June 23rd, 2008.

Hofer, F. (invited)
"Electron energy-loss spectroscopy with a monochromated TEM", MICROSCIENCE 2008, London, U.K., June 23rd, 2008.

Hofer, F. (invited)
"Interface phenomena studied with a monochromated transmission electron microscope", Institute of Physics Symposium "Modelling and Characterisation of Interfaces", London, U.K., June 27th, 2008.

Dienstleder, M.
"A novel method for precipitates preparation using extraction replica combined with focused ion beam techniques", FIB Workshop, Luzern, Switzerland, July 3rd, 2008.

Plank, H.
„Process parameters for particle induced deposition", FIB Workshop, Luzern, Switzerland, July 4th, 2008.

Schröttner, H.
„SEM-Applikationsbeispiele für keramische Materialien", EPCOS, Deutschlandsberg, Austria, July 10th, 2008.

Wagner, J.
„Reconstruction of δ-phase in superalloy by 3-D EDXS in a dual-beam FIB“ Microscopy & Microanalysis 2008, Albuquerque, New Mexico, USA, August 4th, 2008.

Schaffer, B. (invited)
“Hyperspectral imaging in TEM: new ways of information extraction and display”, Microscopy & Microanalysis 2008, Albuquerque, New Mexico, USA, August 5th, 2008.

Kothleitner, G.
“Low-loss EELS measurements on an oxide multilayer system using monochrome electrons”, European Microscopy Conference 2008, Aachen, Germany, September 1st, 2008.

Pölt, P.
“In-situ experiments on soft materials in the environmental SEM - Reliable results or merely damage?” European Microscopy Conference 2008, Aachen, Germany, September 1st, 2008.

Dienstleder, M.
“A novel method for precipitates preparation using replica extraction combined with focused ion beam techniques”, European Microscopy Conference 2008, Aachen, Germany, September 4th, 2008.

Hofer, F. (invited)
“Recent developments of electron microscopy in organic materials research”, Symposium "Advanced Electron Microscopy", Eindhoven University of Technology, The Netherlands, September 08th, 2008.

Hofer, F. (invited)
“Nanoanalysis of materials by means of EELS spectrum imaging in a transmission electron microscope”, 10th Annual Conference YUCOMAT 2008, Herceg Novi, Montenegro, September 11th, 2008.

Granitzer, P.
“Magnetite nanoparticles embedded in biodegradable porous silicon”, JEMS 2008, Dublin, September 14th, 2008.

Grogger, W. (invited)
“Introduction into transmission electron microscopy”, Workshop on Electron Microscopy, University of Maribor, Slovenia, September 23rd, 2008.

Pölt, P.
“The (environmental) scanning electron microscope - a valuable tool for polymer characterisation”, 13th International Conference on Properties, Processing, Modification and Application of Polymer Materials 2008, Halle/Saale, Germany, September 24th, 2008.

Albu, M.
“Compositional characterisation of secondary carbides and complex nitrides in chromium”, Materials Science and Technology Conference 2008, Pittsburgh, USA, October 05th, 2008.

Granitzer, P.
“Mesoporous silicon as base material for quasi-regular arrays of nanomagnets”, NanoSmat, Barcelona, Spain, October 5th, 2008.

Hofer, F. (invited)
“Nanoanalysis of materials by EELS and EFTEM spectrum imaging”, EELS-EFTEM Workshop CAESAR, Bonn, Germany, October 10th, 2008.

Granitzer, P. (invited)
“Porous silicon/Fe_3O_4-nanoparticle composite and its magnetic behaviour”, ECS Porous Semiconductors, Honolulu, USA, October 12th, 2008.

Granitzer, P.
“Novel morphology dependent ferromagnetic behaviour of mesoporous silicon”, ECS Porous Semiconductors, Honolulu, USA, October 13th, 2008.

Kothleitner, G. (invited)

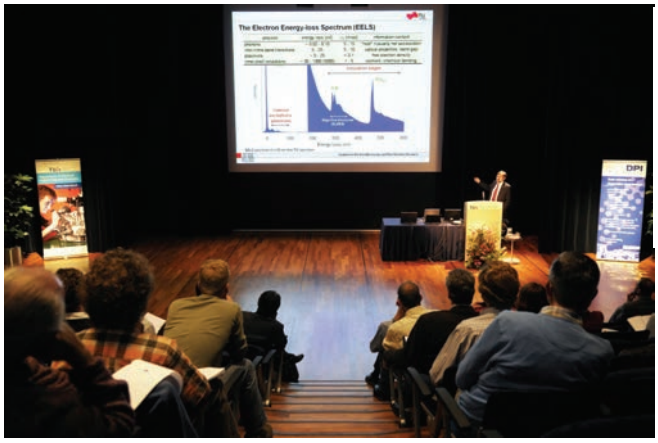
"EELS for bandgap mapping and tomography", Warsaw University of Technology, Department of Materials Science and Engineering, Warsaw, Poland, November 7th, 2008.

Hofer, F. (invited)
"Fundamentals of electron energy-loss spectroscopy", 1st Asian EFTEM Workshop, Singapore, November 10th, 2008.

Hofer, F. (invited)
"Applications of energy-filtering TEM", 1st Asian EFTEM Workshop, Singapore, November 11th, 2008.

Plank, H. (invited)
"Seeing the invisible – beyond microscopy", Fusion Biotechnology Research Center, Korean Research Institute of Chemical Technology, Daejeon, South Korea, November 27th, 2008.

Plank, H. (invited)
"Seeing the invisible", Fusion Biotechnology Research Center, Korean Research Institute of Chemical Technology, Daejeon, South Korea, November 28th, 2008.



Presentation of a lecture at the Eindhoven University of Technology, September 2008

Hartmuth Schröttner talks at the Microscience conference in London, June 2008

10. Poster Presentations by Institute Staff

10.1. Poster Presentations 2007

Mitsche, S.
"Combined EBSD, FIB and TEM investigation of the crack behaviour of the nickel based alloy 80A", 14th Conference on Electron Backscatter Diffraction, Glasgow, Scotland, March 26th, 2007.

Pölt, P.
"EBSD – some experimental and some fundamental problems", 14th Conference on Electron Backscatter Diffraction, Glasgow, Scotland, March 26th, 2007 .

Reichmann, A.
"Dynamic studies of hot corrosion of steel in the ESEM", European Microbeam Analysis Society (EMAS) Conference 2007, Antwerp, Belgium, May 6th, 2007.

Pölt, P.
"Conjugated polymers - degradation by electron and ion irradiation in a SEM / FIB", European Microbeam Analysis Society Conference (EMAS) 2007, Antwerp, Belgium, May 6th, 2007.

Mitsche, S.
"Crack behaviour of the nickel based alloy 80A during hot forming", European Microbeam Analysis Society (EMAS) 2007, Antwerp, Belgium, May 6th, 2007.

Schaffer, M.
"Automated 3D X-ray spectrometry in a dual beam - Focoused Ion Beam", European Microbeam Analysis Society Conference (EMAS) 2007, Antwerp, Belgium, May 6th, 2007.

Sezen, M.
"Modification of organic optoelectronic devices with a FIB/SEM dual beam system", Dreiländer-Workshop FIB 2007, Graz, Austria, July 2nd, 2007.

Rogers, M.
„In-situ TEM Probenpräparation von verspannten Proben mittels Sandwich Technik", Dreiländer-Workshop 2007, Graz, July 2nd, 2007.

Sezen, M.
"Organic optoelectronic device fabrication and modification by dual beam FIB", ISOTEC Meeting, Reinischkogel, Austria, July 13th, 2007.

Rattenberger, J.
"Time dependent study of ion- and electron currents under water vapour conditions", 14th Conference on "Festkörperanalytik", Wien, Austria, July 16th, 2007.

Wagner, J.
"Time resolved measurements of ion- and electron currents in an ESEM", Microscopy Conference 2007, Saarbrücken, Germany, September 2nd, 2007.

Gspan, C.
"Comparison between stoichiometric and nonstoichiometric LaSrCo-oxides by analytical TEM", Microscopy Conference 2007, Saarbrücken, Germany, September 2nd, 2007.

Belegratis, M. R.
"Multilayer foils: electron microscopy and infrared spectroscopy", 17th European Symposium on Polymer Spectroscopy (ESOPS), Seggauberg, Austria, September 9th, 2007.

Zankel, A.
"A comprehensive study of the morphology and the ion interaction behaviour of organic mixed ionic - electronic conductors", 17th European Symposium on Polymer Spectroscopy (ESOPS), Seggauberg, Austria, September 9th, 2007.

Albu, M.

"EELS quantification of secondary carbides in steels", New Research Trends in Material Science ARM-5, Sibiu, Rumania, September 5th 2007

Zankel, A.

"Estimation of beam damage of polymers caused by in situ investigations in the ESEM using IR spectroscopy", 17th European Symposium on Polymer Spectroscopy (ESOPS), Seggauberg, Austria, September 9th, 2007.

Chernev, B. S.

"Principal compositional analysis of secondary carbides in a 9Cr-1Mo steel", 17th European Symposium on Polymer Spectroscopy (ESOPS), Seggauberg, Austria, September 9th, 2007.

10.2. Poster Presentations 2008

Sezen, M.

"Organic optoelectronic device fabrication and modification by dual beam FIB", ISOTEC Meeting, Mariazell, Austria, January 17th, 2008.

Zankel, A.

"Ultramicrotomy in the ESEM – a new method for the visualization of 3-D strcutures in botany", 17th Meeting of the Austrian Society of Plant Biology, Stainz, Austria, May 22nd, 2008.

Wilhelm, P.

"FTIR-spectroscopy and imaging of heterogeneous polymeric systems", 1st Austrian-Slovenian Polymer Meeting (ASPM), Graz, Austria, March 26th , 2008.

Chernev, B. S.

"Vibrational spectroscopic imaging methods for investigation of pharmaceutical products", International Graz Workshop for Pharmaceutical Engineering, Graz, Austria, May 15th, 2008.

Rattenberger, J.

"Total scattering cross section of N_2 and effective beam gas path length measurements in a low vacuum scanning electron microscope", Microscience 2008, London, U.K., June 23rd, 2008.

Rattenberger, J.

"Experimental determination of the total scattering cross section of water vapour and of the effective beam gas path length in a low vacuum scanning electron microscope", European Microscopy Conference EMC 2008, Aachen, Germany, September 1st, 2008.

Plank, H.

"The influence of beam defocus on volume growth rates for electron beam induced platinum deposition", European Microscopy Conference EMC 2008, Aachen, Germany, September 2nd, 2008.

Mitsche, S.

"Characterisation of the subgrain structure of the aluminium alloy AA6082 after homogenization and hot forming by EBSD", European Microscopy Conference EMC 2008, Aachen, Germany, September 3rd, 2008.

Albu, M.

"EELS quantification of complex nitrides in a 12 % Cr steel", European Microscopy Conference 2008, Aachen, Germany, September 1st, 2008.

Granitzer, P.

"Tailored magnetic anisotropy by specific precipitation of ferromagnetic metals into silicon matrices", Joint European Magnetic Symposia (JEMS) 2008, Dublin, Ireland, September 14th, 2008.

Zankel, A.

"In situ testing of plastics with the ESEM - chances for the design of new investigation methods", 13rd International Conference on Properties, Processing, Modification and Application of Polymer Materials, Halle/Saale, Germany, September 24th, 2008.

11. Further Education for Staff Members

2007

January 2007
- M. Albu, “Communication Skills 1”, TU Graz
- M. Paller, W. Rossmann, W. Czapek, „Defibrillator-Schulung, TU Graz

February 2007
- U. Stürzenbecher, “Microsoft Project”, WIFI Graz
- U. Stürzenbecher, “EU-Projektmanagement”, TU Graz

March 2007
- B. Schaffer, „Umgang mit Konflikten“, TU Graz

April 2007
- G. Stoiser, „Photoshop CS2-Einführung“, TU Graz

May 2007
- U. Stürzenbecher, „EU-Projektmanagement 4“, TU Graz

June 2007
- B. Schaffer, „Teamentwicklung“, TU Graz

September 2007
- U. Stürzenbecher “EU-Projekte beantragen und durchführen”, TU Graz

October 2007
- M. Brunegger, „Ausbildung Sicherheitsvertrauensperson“, WIFI Graz

December 2007
- B. Schaffer, „Bildanalyse mit Image J-Software“, Medical University Graz

2008

January 2008
- S. Goger, U. Stürzenbecher, G. Stoiser, F. Streussnig, M. Wallner, „Outlook“, WIFI Graz
- U. Stürzenbecher, „Finanzielles Management geförderter Projekte“, TU Graz

February 2008
- B. Chernev, „Multivariate Analyse von spektroskopischen Daten“, CAMO, Innsbruck

April 2008
- A. Gusmagg, „Outlook“, WIFI Graz
- S. Mertschnigg, M. Paller, A. Rossmann, Struers Workshop „Materialographie 08“, Wien
- U. Stürzenbecher, „Personalverrechnung“, Akademie Wirtschaftstreuhänder Graz

May 2008
- M. Wallner „Webportal TU“, TU Graz

September 2008
- G. Birnstingl, “Brandschutzausbildung”, TU Graz
- U. Stürzenbecher, “Ausbildung Qualitätsauditor”, TÜV Graz

October 2008
- U. Stürzenbecher, „Management von Projektenden 7.RP“, FFG Wien

November 2008
- G. Stoiser, „Windows Workstation Administrator“, TU Graz
- M. Wallner, “Corel Draw Advanced Kurs”, WIFI Graz
- C. Gspan, “ACT-Workshop”, Dresden

December 2008
- F. Streussnig “Prüfungsmanagement”, TU Graz
- G. Stoiser „Administration Linux System“, TU Graz
- W. Grogger, G. Kothleitner, "Administration von Kongressen mit CATS", TU Graz.

12. Research – Microscopy Gives New Insight

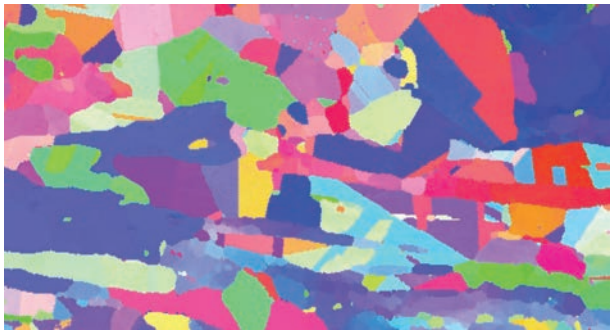
The institute's main research activities are devoted to developing new microscopic characterisation methods. These methods are used for studying the microstructure of all kinds of solids, materials and biological samples, e.g. alloys, steels, metals, ceramics, composites, minerals, polymers, nano-particles, clusters and biological tissue.
New developments were partially driven by our industry and academic partners resulting from their demands for advanced materials characterisation.
In 2007 and 2008 research funds were allocated by the FFG, the FWF, the FP7 of the EU, the Austrian Nanoinitiative and the government of Styria (see p.72).

12.1. Microscopy in Materials Research

In the past two years, FELMI-ZFE has established accessories and add-ons for analytical electron microscopy, which greatly extended the range of methods and materials for sophisticated materials characterisation. Particular progress in the area of micro- and nanoanalysis was made on the following types of materials and methods:

- Microanalysis in the SEM

The scanning electron microscope (SEM) enables the full characterisation of both bulk specimens and thin sections. The surface topography, the chemical composition of the surface and the crystallographic structure of a specimen can be determined (p.94). Recent studies have shown the particular advantages of electron backscatter diffraction (EBSD) for investigating the grain structure of superalloys and aluminium alloys after homogenisation and hot forming (see p.92).
The environmental SEM (ESEM) of the FELMI-ZFE was not only used for materials analysis, however also as a kind of micro-reactor. A variety of processes can be controlled, while simultaneously documenting the changes of the specimen with high magnification and great depth of focus on video. The fracture behaviour of both polymers and textile fibres is investigated using a tensile stage mounted on the specimen chamber of the ESEM (see p.88). Another research effort of the research group of Peter Pölt concentrates on the *in-situ* study of hot corrosion of materials, e.g. steels and alloys (see p.90). Additionally, the research group of Hartmuth Schröttner studies the physical principles of imaging processes with new detectors in scanning electron microscopy.



EBSD map of crystal grains in a duplex steel; colors indicate different crystal orientations; image length = 10 µm.

- Nanoanalysis in the TEM

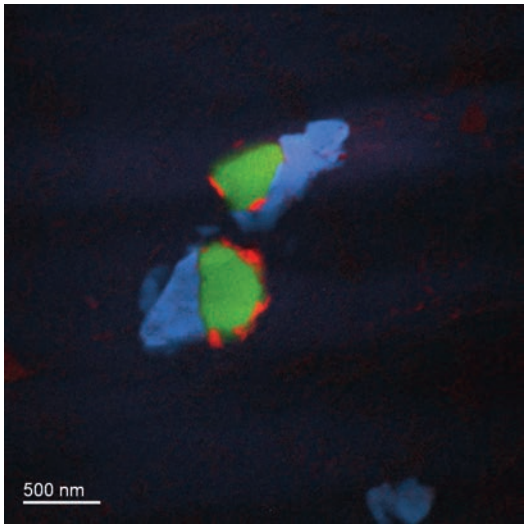
The ability of modern transmission electron microscopes to produce images at near atomic resolution has led to a tremendous progress in materials science. If the TEM is combined with analytical techniques such as electron energy-loss spectroscopy (EELS) and energy-filtering TEM, it is now possible to record elemental distribution maps at nanometre resolution.
- EFTEM spectrum imaging could be further improved and is now a powerful method for the advanced nanoanalysis of all kinds of materials (see p.78). Furthermore, progress was achieved on new spectrum imaging methods based on STEM imaging e.g. hyperspectral imaging of electron diffraction data (research groups of Gerald Kothleitner and Werner Grogger).
- EELS and EFTEM are particularly useful for studying precipitation or segregation at grain boundaries in materials such as steels, alloys and ceramics. Particular progress was achieved on the

quantitative analysis of complex nitrides in steels (see p.84). The studies are perfomed in collaboration with the Institute for Materials Science, Welding and Forming, TU Graz.

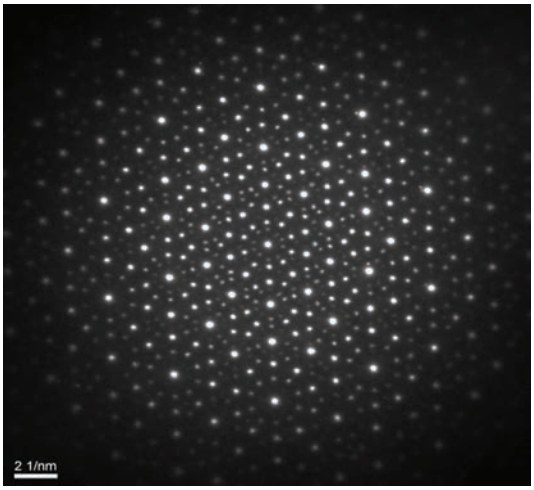
- EFTEM studies of semiconductor device materials, interconnects, insulators and device packaging materials are gaining increasing importance due to the ever shrinking size of today's devices. The research group of Gerald Kothleitner concentrates on the characterisation of semiconductor devices which have been prepared with a novel copper deposition process based on the use of non-aqueous solvents. This process is developed in the EU funded project "CopPeR" (FP7) to overcome the limitations of currently applied interconnect formation processes enabling device scaling beyond the 32 nm technology node (www.copper-project.eu).

STEM-HAADF image of a cross-section of a semi-conductor recorded with the FEI Tecnai F20.

- Our unique equipment was essential for many successful investigations: The employment of a Wien filter monochromator, incorporated in the illumination system of our field-emission TEM, improves the system energy resolution down to 0.2 eV. Valence electron and core-loss spectroscopy benefited directly from this improved energy resolution, opening up new possibilities for more accurate measurement of band gaps and optical properties via the dielectric function (see pp.66, 82).



EFTEM elemental distribution map of precipitates in a 9% chromium steel; red = vanadium, green = manganese, blue = chromium.



Electron diffraction pattern of a quasicrystalline phase in a melt-spun Al-Mn-Be alloy (sample courtesy, F. Zupanič, University of Maribor, Slovenia).

12.2. Electron Microscopy in Energy Research

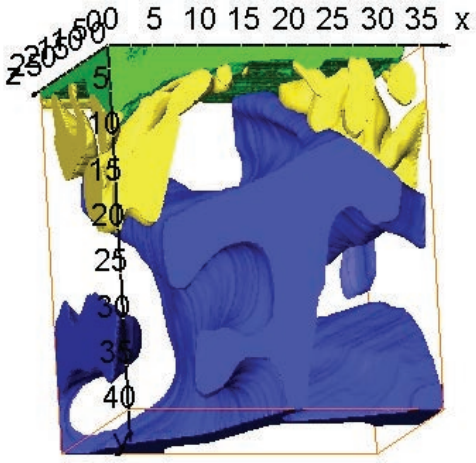
Electron microscopy is making a vital contribution to understanding the structure, property and function of energy relevant materials, such as energy-efficient solar cells, fuel cells and light emitting devices. In order to enhance the institute's position in this important field, we have started a research project with the Christian Doppler Laboratory for Nanocomposite Solar Cells (TU Graz). In this project we will control the morphology in organic nanocomposite solar cells consisting of a conjugated polymer and semiconducting nano-particles, which is crucial to improve their efficiencies and lifetime.

12.3. Three-dimensional Materials Science

Advances in microscopy techniques and software are breathing new life into the way we look at materials, allowing us to visualise nano- and microstructures in three-dimensional space. This enables us to directly see the 3-D relationships between different materials, as well as being able to determine the size and distribution of nanoparticles or pores. It is now an acknowledged and well known fact that microscopy techniques can deliver 3-D images with the benefit of site specific information. The institute, therefore, started developing new 3-D microscopy techniques several years ago. Four main directions are presently followed:

- 3-D elemental mapping

Julian Wagner, Miroslava Schaffer and Mario Schmied developed the method of 3-D elemental mapping by combining a focused ion beam instrument (FIB) with energy-dispersive x-ray spectrometry (EDXS). The FIB-EDXS method is especially powerful for the 3-D investigation of inorganic materials, such as secondary phases in steels, alloys, casting materials, hard metals, ceramics and composites (see p. 86).
The method has been fully automated and applied to solve several materials science problems. The project was supported by FEI Company, Eindhoven).



3D-elemental map of a casting alloy, presented as an RGB-image; green = silicon, blue = aluminium, yellow = iron; in µm)
(sample courtesy Österr. Gießerei-Institut, Leoben)

- 3-D microstructures of biomaterials

The internal structure of specimens can be elucidated by serial sectioning with an ultra-microtome directly installed in the environmental SEM (ESEM) and simultaneous image recording with backscattered electrons (Gatan 3VIEW™ system). This method permits high resolution tomography of soft materials, polymers and biological samples. Serial sectioning in an ESEM provides unique information about the phase distribution in organic matter. Peter Pölt and Armin Zankel were the first to apply this new method in materials science (see pp.69, 88). The method has been automated in collaboration with Bernd Kraus of Gatan company (Pleasanton, USA).

- 3-D AFM in a cryo-ultramicrotome

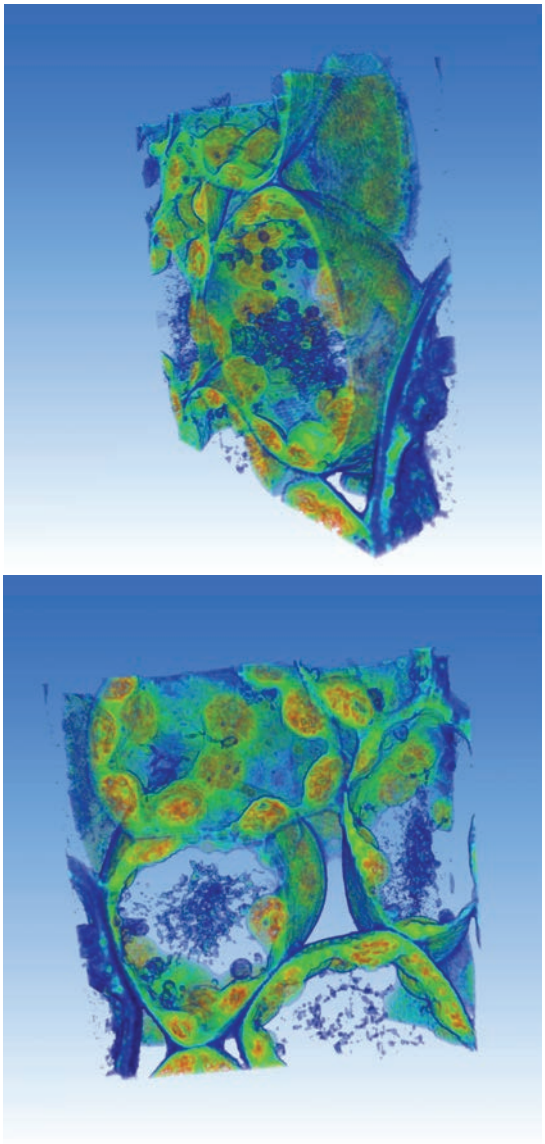
A new research project concentrates on the development of a new method using a cryogenic atomic force microscope (AFM) directly mounted in the cryogenic chamber of an ultramicrotome. This combination of methods will allow scanning immediately after sectioning, so that structural changes can be avoided as the whole structure will be stabilised by cold. Direct observation of the block surface structure of the sample by cryo-AFM will provide information about the native structure

of the bulk polymer which is not chemically or mechanically modified during sample preparation or observation. This new method will be developed in cooperation with Nanoscan Technology, Moscow, Russia. The research project developed by Nadejda Matsko and Werner Grogger is supported by the FFG (Vienna).

- Nanotomography

Electron tomography is a means by which the three-dimensional structure can be reconstructed from a series of images or projections taken at regular tilt intervals. In transmission electron microscopy these projections allow the three-dimensional structure to be determined with nanometre resolution and with remarkable accuracy. The method has already been used successfully in the biological sciences for decades. In materials science and engineering, however, electron tomography has only been introduced over the past few years. As the structures designed and grown for modern devices become ever smaller, an increasing need is arising to examine materials in all three dimensions to gain a full picture of the device structures. Consequently, nanotomogrophy is now introduced in the institute for applications in semiconductor research within the project Copper (FP7-project by Gerald Kothleitner).

- The 3-D methods are extended by the light microscope *InfiniteFocus* of Alicona Imaging, which combines the small depth of focus of an optical system with vertical scanning to provide topographical and colour information from the variation of focus.



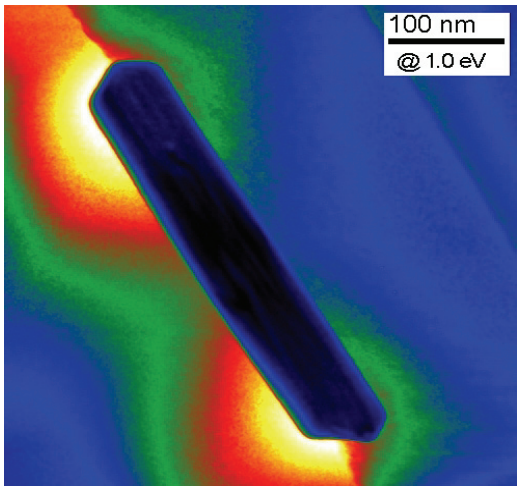
3-D reconstruction of cells of a bell flower, which has been serially sectioned with the 3View™ system in the ESEM (reconstruction by Bernd Kraus, Gatan).

12.4. Nanotechnology

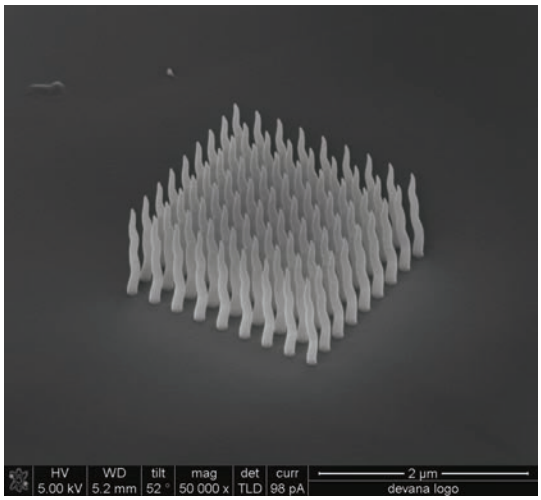
Nanostructures and nanostructured materials have attracted steadily growing interest due to new properties that are complementary or superior to those of bulk materials. The fundamental study of phenomena that occur in these materials has already evolved into a new field of research that is often referred to as nanoscience. The institute concentrates its efforts in two directions:

- Fundamental properties of nanostructures

The transmission electron microscope is an ideal tool for mapping physical properties of individual nanostructures at the sub-nm level. One research focus of the institute lies in the development of advanced EFTEM and EELS spectrum imaging techniques. In combination with the instrumental advantages provided by the monochromated TEM/STEM system (FEI Tecnai F20) it has been possible to image surface plasmons on Au and Ag nanoparticles with excellent energy resolution. For example we have been the first to demonstrate that energy-filtered TEM can be efficiently used to image surface plasmons of Au nanoparticles. These studies were performed in collaboration with the Institute of Physics of the University of Graz (Ulrich Hohenester and Joachim Krenn) and the group of David McComb at the Imperial College in London, U.K..



Distribution of surface plasmons around an Au nanorod, recorded with the monochromated FEI Tecnai F20.



Pt nanowires prepared in a focused ion beam microscope (FIB) using the electron beam.

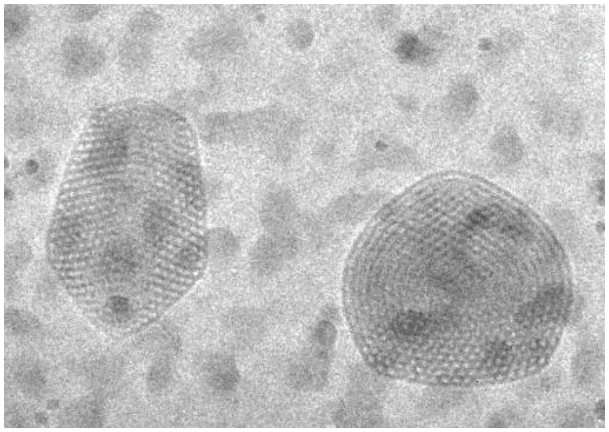
- Nanoprototyping: Nanoscale structures and devices can be fabricated in the focused ion beam (FIB) microscope using either electrons or ions. This instrument allows writing directly into a substrate by etching, or material can be laid down via a vapour deposition process. The nano-prototypes can be used to study the properties of photonic arrays and other devices (see p.74).

The FIB-method is especially useful for analysing and structuring of organic electronics such as light emitting devices (OLEDs) and organic field effect transistors (OFETs) (see pp.96, 98). These studies are performed in collaboration with the Nanotec Center Weiz and the Institute for Nanostructured Materials and Photonics (Joanneum Research, Graz).

12.5. Soft Matter Characterisation

„Soft matter“, i.e. polymers, hybrid coatings, biomedical materials and nanocomposites form an important research area nowadays with many important discoveries, developments and industrial applications. Electron microscopy is virtually the only technique by which the structure of soft matter can be resolved (see p.102). Our main research

goal in this area is to understand the organisation or (self-) assembly of molecules and nano-structures to control the properties and performance of materials and devices. The challenge in soft matter microscopy is thus not only to image molecular states of the materials but also to analyse their 3-D structural arrangement, since both are essential in soft matter science.
The research group of Elisabeth Ingolic developed unique knowledge in studying the morphology of technical polymers, the phase distribution in polymers, distribution of fillers, secondary phases and interfaces in polymers and metal-polymer composites. These investigations are mostly performed by means of transmission electron microscopy of specimens, which have been prepared via ultramicrotomy. Biological tissue and medical materials are investigated in collaboration with institutes from the Medical University of Graz and of the Karl-Franzens University of Graz (see p.104).



Oil in water emulsion investigated in the cryo-TEM (image length = 450 nm).

Cryo-methods in transmission electron microscopy (TEM) have been proven to be powerful tools in the characterisation of soft matter nanostructures. By rapid cooling (=cryo-fixation) molecular movements are stabilised and the structure is preserved and observed in its natural state. Consequently, we started with the introduction of cryo-transmission electron microscopy for studying polymeric and biological emulsions. First results show the high quality of the TEM images (see p.106). This project is supported by the Goverment of Styria (Mag.[a] Kristina Edlinger-Ploder) and the NAWI faculty of the Karl-Franzens University and the TU Graz.
All these investigations are supported by serial sectioning 3-D imaging in the environmental SEM (ESEM) which will be later supplemented by nanotomography in the TEM. *In-situ* experiments in the ESEM give new insight into the fracture behaviour of both polymers and textile fibres. This work aims at providing a controlled microstructure or morphology for an optimal relationship between structure and properties.
Electron microscopy of soft matter is embedded in a sophisticated characterisation environment providing light microscopy with vibrational spectroscopy (infrared and Raman) which enables the chemical characterisation of samples or domains as small as 10 µm (Infrared) or 1 µm (Raman). The research group of Peter Wilhelm applies these methods to analyse polymers (identification, defects, impurities, stress and density), rubbers, paper, inorganic and biological materials. Results are obtained by spectral interpretation (band allocation to functional groups, comparison with reference spectra) or imaging (2D mapping of functional groups).

12.6. Research Projects

- „Hochauflösungs-Rasterelektronenmikroskop“ supported by Steirische Wirtschaftsförderungs GmbH (SFG) im Auftrag des Landes Steiermark, May 1st, 2005 – March 31st, 2007.
- PROKIS-Project “Kompetenzaufbau und Innovation ZFE Graz” arranged by ACR and supported by Federal Ministry for Economic Affairs and Labour (BMWA), Vienna, May 1st, 2007 – December 31st, 2009.
- “Nanoanalysis and Nanostructuring for Organic Optoelectronic Devices” Werner Grogger, funded within the ISOTEC- Project (FWF and Austrian Nanotechnology Initiative, Vienna), March 1st, 2005 – February 28th, 2009.
- „Device modification - Mikroskopische Modifizierung von Halbleiterbauelementen”, Julian Wagner; project funded by the FFG

(Österreichische Forschungsförderungsgesellschaft mbH; Basisprogramme), Vienna, July 1st, 2006 – October, 31st, 2008.

- "Kryo-Transmissionselektronenmikroskopie von Kunststoffen und Biomaterialien", Ferdinand Hofer, project funded by Land Steiermark, (Abteilung Wissenschaft und Forschung), Graz, October 1st, 2007 – November 30th, 2008.
- „Copper Interconnects for Advanced Performance and Reliability", Gerald Kothleitner, project funded by the Seventh Framework Programme – STREP, European Commission, January 1st, 2008 – June 30th, 2010.
- "Mikrobereichsanalytik mit hoher Nachweissensitivität", Stefan Mitsche, project supported by the FFG (Österreichische Forschungsförderungsgesellschaft mbH, Basisprogramme), Vienna; July 1st, 2008 – June 30th, 2010.
- „3D-Mikroskopie von Polymeren und Biomaterialien", Nadejda Matsko, funded by the FFG (Österreichische Forschungsförderungsgesellschaft mbH, Basisprogramme), Vienna; November 1st, 2008 – October 31st, 2010.
- „Christian Doppler Labor für Nanokomposit-Solarzellen", project supported by the Christian-Doppler Forschungsgesellschaft mbH, Vienna, July 1st, 2008 – June 30th, 2010.
- "NILaustria – Nanoimprint Lithography in Austria", Werner Grogger, project funded by the Austrian Nanoinitiative (FFG & BMVIT), Vienna, October 1st, 2008 – September 30th, 2011.
- "Grundlagen der Verarbeitung und Charakterisierung von PET Microblends", coordinator Teufelberger GmbH, Elisbeth Ingolic, supported by FFG (Österreichische Forschungsförderungsgesellschaft mbH, Vienna) January 1st, 2008 – December 31st, 2010.
- "Ir/Reguläre magnetische Nanodrähte in porösem Silizium", supported by FWF, Vienna, Project P18593, 1.1.2006 - 1.2.2008, in cooperation with Prof. Heinz Krenn, University of Graz.
- "Austrian Scanning Transmission Electron Microscope – ASTEM", Ferdinand Hofer, project funded by the FFG (Österreichische Forschungsförderungsgesellschaft mbH, Vienna) within the programme "COIN – Aufbau", project start in 2009.

13. Abstracts of Scientific Main Results 2007-2008

With the next pages we try to give an impression of the many activities that have characterised our research during the last two years. Some contributions have been included in revised form from conference proceedings; others have been extracted from already published papers in scientific journals.

The Influence of Beam Defocus on Volume Growth Rates for Electron Beam Induced Platinum Deposition

H. Plank, C. Gspan, M. Dienstleder, G. Kothleitner and F. Hofer

Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology &
Graz Centre for Electron Microscopy, Steyrergasse 17, 8010 Graz

Electron beam induced deposition (EBID) is a technique which enables the controlled deposition of conducting (e.g. W, Pt) or insulating (tetraethylorthosilicate,) materials on the nanoscale. EBID has attracted considerable attention in recent years as an interesting alternative to ion beam induced deposition (IBID) due to its sputter-free character, the absence of ion implantation, and the reduced thermal stress [1] which is of particular importance for soft matter samples or critical applications like polymers or nanoscale devices.
EBID processes utilize a gaseous pre-cursor which is brought into the microscope chamber leading to adsorption-, diffusion- and desorption-processes on the sample surface. The interaction with the e^- beam leads to the dissociation of the pre-cursor molecules into volatile and non-volatile products. The former is pumped away while the latter remains at the samples surface and allows therefore a defined 3-dimensional deposition via a controlled beam movement as shown in Fig. 1a by a ~ 10 nm high Pt test structure. One drawback of EBID, however, is the low volume growth rate (nm^3 / sec) compared to IBID, mainly originating from the much lower secondary electron yield per incident particle (electrons respectively ions) which is one main dissociating electron species.
To push EBID towards its intrinsic limits the single elements of such deposits (as shown in Fig.1b) must be investigated in more detail to improve the understanding of the growth processes, which allows then for further improvements [2,3]. In this work the enhancement of the volume growth rate for single platinum (Pt) rods by a factor of 2.5 is demonstrated via a systematically introduced defocus during deposition. Fig. 1c shows such a single element in more detail and reveals the two intrinsic morphologies: i) the conical shape on top of the nano-rods with an end radius around 5 nm in ideal cases and ii) the conical shaft shape with almost vertical side walls (showing minimal diameters around 45 nm). When a beam defocus is introduced during the deposition the deposits become thicker in diameter and smaller in height which can be seen in Fig.2a while the nanostructure remains identical as shown in Fig. 2b and c by transmission electron microscopy images. Deriving the deposited volumes of the nano-rods as a function of the introduced defocus reveals a significant increase of the volume growth rate (VGR; nm^3/sec) by a factor of ~ 2.5 which can be seen in Fig. 3a. The reason for this increase can be explained via the temporal VGR evolution as shown in Fig. 3b. For in-focus conditions (black curve) a clear maximum within the first few seconds can be found followed by a strong decrease. The introduction of a defocus delays this maximum and expands the time period for enhanced VGR's which can be seen in Fig. 3b by the red and blue curve (compare to legend). These findings, however, imply a dependency between total growth time and ideal defocus which is shown in Fig. 3c for the used deposition parameters (30 keV, 150 pA).
Beside the practical aspect of this enhancement, it is also possible to study very early growth stages, different growth regimes (conical, cylindrical), and their transition on a larger time scale which helps to understand the growth mechanism for such deposits.

[1] R.M. Langford, P.M. Nellen, J. Gierak, Y. Fu, MRS Bulletin 32 (2007) 417.
[2] S.J. Randolph, J.D. Fowlkes and P.D. Rack, Crit. Rev. Solid State Mat. Sci. 31 (2006) 55.
[3] D. Beaulieu, Y. Ding, Z.L. Wang and W.J. Lackey, J. Vac. Sci. Techn. B 23 (2005) 2151.
[4] D.A. Smith, J.D. Fowlkes and P.D. Rack, Nanotechnology 18 (2007) 265308.

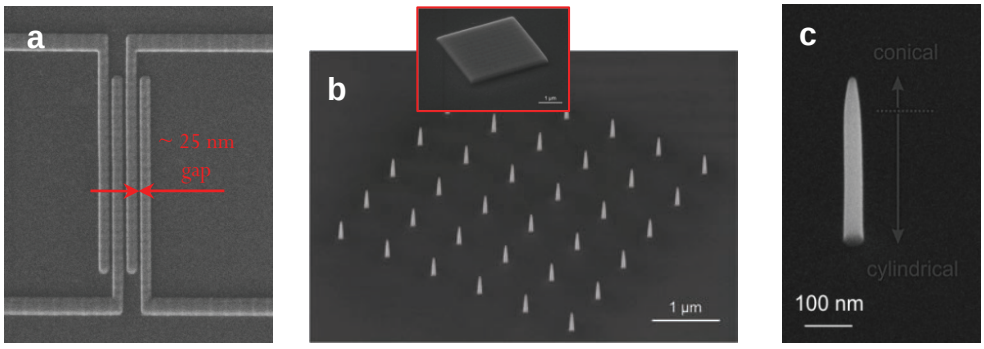


Fig. 1 Scanning electron microscopy images of electron beam induced deposition of defined Pt nanostructures (a), single elements (b) of larger deposits (inset), and intrinsic morphologies of a single Pt nano-rod (c).

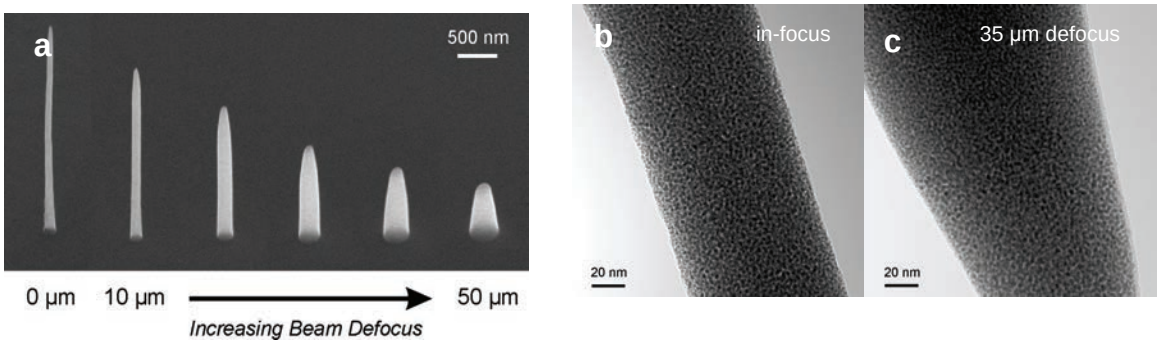


Fig. 2 Influence of beam defocus during deposition on the morphology of Pt single nano-rods (a, scanning electron microscopy images). Structural composition of Pt nano-rods for in-focus (b) and 35 µm defocus (c) conditions (transmission electron microscopy images).

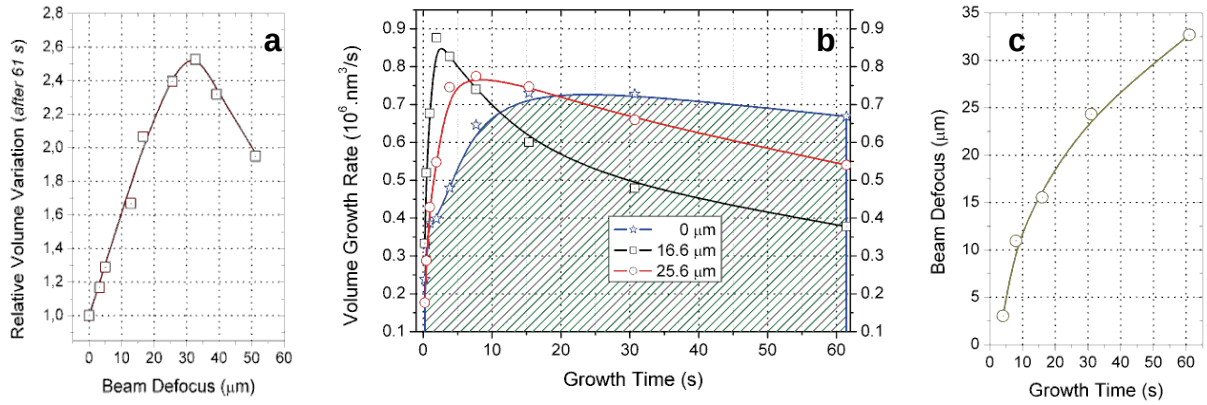


Fig. 3 Tilted SEM images of a defocus series with in-focus conditions at the left and increasing beam defocus (2nd to 6th nanorod) for 150 pA @ 30 keV.

Low-loss EELS Measurements with Monochromated Electrons

G. Kothleitner, W. Grogger, F. Hofer, B. Schaffer

Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology and
Graz Centre for Electron Microscopy, Steyrergasse 17, A-8010 Graz, Austria

The characterization of nanostructured devices and functional materials at a nanometer scale is paramount for the understanding of the physical or chemical properties. The significant enhancement of the energy resolution in the new generation of commercially available monochromated transmission electron microscopes makes it possible to obtain high quality electron energy-loss spectra. These spectra can be measured with an energy resolution in the range of 0.1-0.2 eV by means of nanometer-sized electron probes. In particular, one can obtain improved information not only about the chemical composition of a sample, but also about chemical bonding [1] and optical properties [2]. The advent of aberration correctors [3] incorporated into the illumination and/or imaging system of (S)TEMs will further improve the performance, thus leading to major advances in materials research.

For the experiments presented here a FEI Tecnai F20 microscope was used (200 kV, Schottky emitter). The system is equipped with a pre-specimen monochromator (Wien filter) [4], a high resolution imaging filter (Gatan) [5], a highly stabilized high voltage supply and a 2kx2k CCD camera. The scanning probe was operated with a DigiScan system (Gatan).

It is well known, that valence EELS can benefit from an improved energy resolution, opening up new possibilities for a more accurate measurement of bandgaps and optical properties via the dielectric function. However, several obstacles have to be considered: A critical step in the analysis of valence EELS data is the removal of the zero-loss peak, whose high-loss tail extends into the interesting region between 0-5 eV [6]. Here, a monochromator provides a clear advantage for two reasons: First, it shortens the tail by reducing the peak width, and secondly the peak shape becomes symmetric, facilitating the study of the band-gap region considerably, even with narrow band-gap semiconductors. On the other hand, some physical restrictions may limit the accuracy of band structure investigations of confined volumes (nano-objects): Delocalization of inelastic scattering events [7] and the excitation of Čerenkov radiation limit low-loss EELS for many semiconductors and insulators especially in the band gap region [8].

In order to demonstrate the spatial resolution which is presently achievable with a monochromated Tecnai F20, we report experiments with ZnO nanobelts [9]. Spectra were recorded with a STEM-probe of 5 nm yielding about 1 nA current and an energy resolution of 0.2 eV; the acquisition time for each spectrum was 1 sec with a dispersion of 0.05 eV/channel. Fig.1 shows the high-angle annular dark-field image of a ZnO nanobelt together with monochrome EELS spectra, extracted from two different positions in the line-scan perpendicular to the belt. The bandgap width can be accurately measured (3.3 eV), inelastic scattering delocalization is clearly visible far from the ZnO particle (Fig.1b, position 6) and the EELS-spectrum recorded in the centre (position 16) exhibits small Čerenkov losses in the bandgap region. Similar experiments on individual Au nanoprisms are in progress to investigate the surface plasmon modes.

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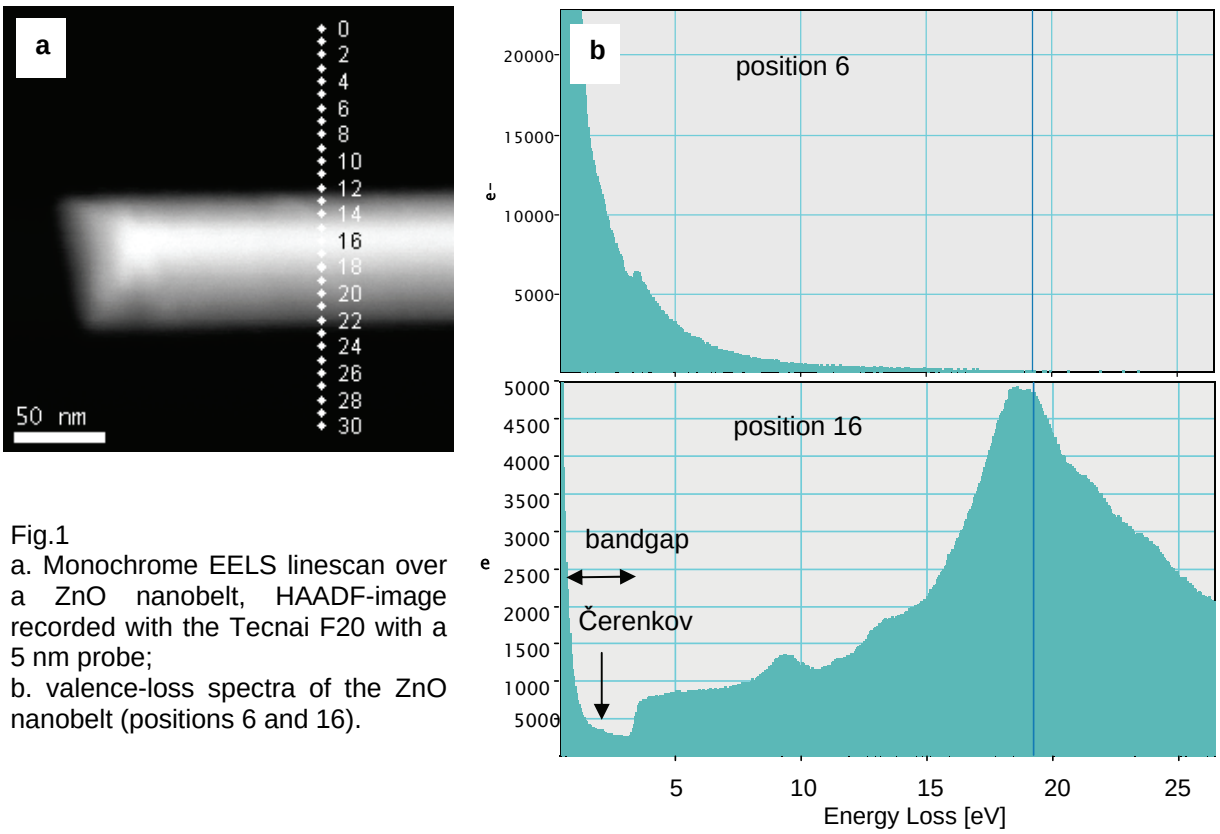

Fig.1
a. Monochrome EELS linescan over a ZnO nanobelt, HAADF-image recorded with the Tecnai F20 with a 5 nm probe;
b. valence-loss spectra of the ZnO nanobelt (positions 6 and 16).

Automated EFTEM Spectrum Image Acquisition: Overcoming EELS Dynamic Range Problems

G. Kothleitner and B. Schaffer

Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology &
Graz Centre for Electron Microscopy, Steyrergasse 17, A-8010 Graz, Austria

EELS spectrum imaging, i.e. the collection of EELS data along the spatial and the energy-loss dimension has progressed to the point where sophisticated spectrum image data processing like thickness deconvolution [1], Kramers-Kronig analysis, MLS-fitting for quantitative compositional image analysis [2,3] or automated elemental occurrence mapping [3] now can almost be carried out routinely. This became possible with the advent of combined spectral aberration-, energy- and spatial-drift correction schemes, carried out simultaneously, giving access to high-quality data [4].

However, depending on the data acquisition mode (STEM or EFTEM), experimental difficulties remain, originating primarily from the large intensity differences within an EELS data set. Although the dynamic range of modern electron detectors has been increased compared to the first generation sensors, with high-end CCDs digitizing electron counts up to 16 bit and more, the huge signal of the zero-loss peak compared to the several orders of magnitude weaker intensities in the core-loss region still requires the operator to adjust experimental conditions, preventing the acquisition of both components into one data set under identical electron optical conditions.

For EFTEM spectrum imaging this problem can be overcome with an approach that encompasses an automated, dynamic change of image acquisition conditions, realized as a DigitalMicrograph program. In brief, it comprises the input of some standard parameters such as slit width, energy range and energy step-size, plus some extra factors, like the targeted intensities, the intended exposure times for a single image acquisition and the maximum number of frames for a particular energy-loss. Other switches are used to prefer multiple frames over longer exposure times for instance. Multiple frames can be corrected for sample drift prior to summation, reducing drift-caused image blur. Energy drift and spectral aberration correction can be applied as additional post-processing steps (Fig 1a,b).
A data set acquired by this approach is shown in Fig. 1, taken from a multilayered oxide system, fabricated by pulsed laser PVD (a). The elements contained therein give rise to various edges from 50eV up to 2.2 keV, not yet visible in the uncorrected, unscaled spectrum (b).

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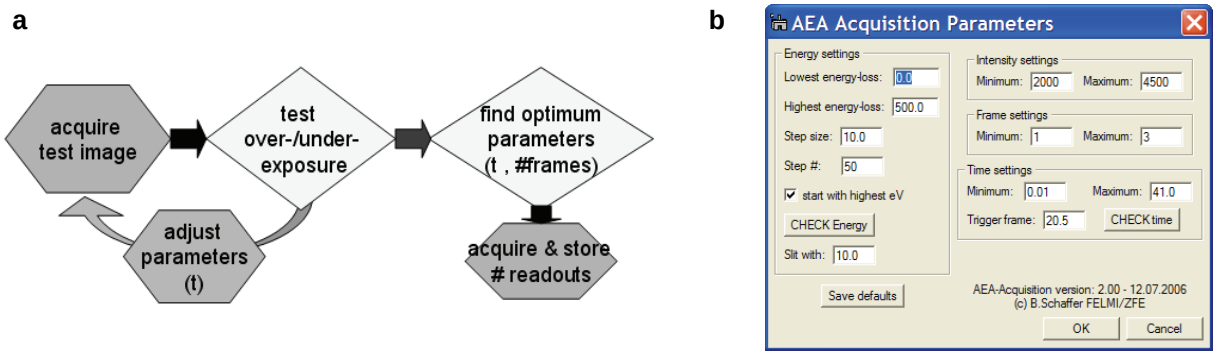

Fig.1. Simplified flow diagram (a) and user interface (b) of an EFTEM spectrum image acquisition process, which automatically adjusts for dynamic range differences in the EELS spectrum.

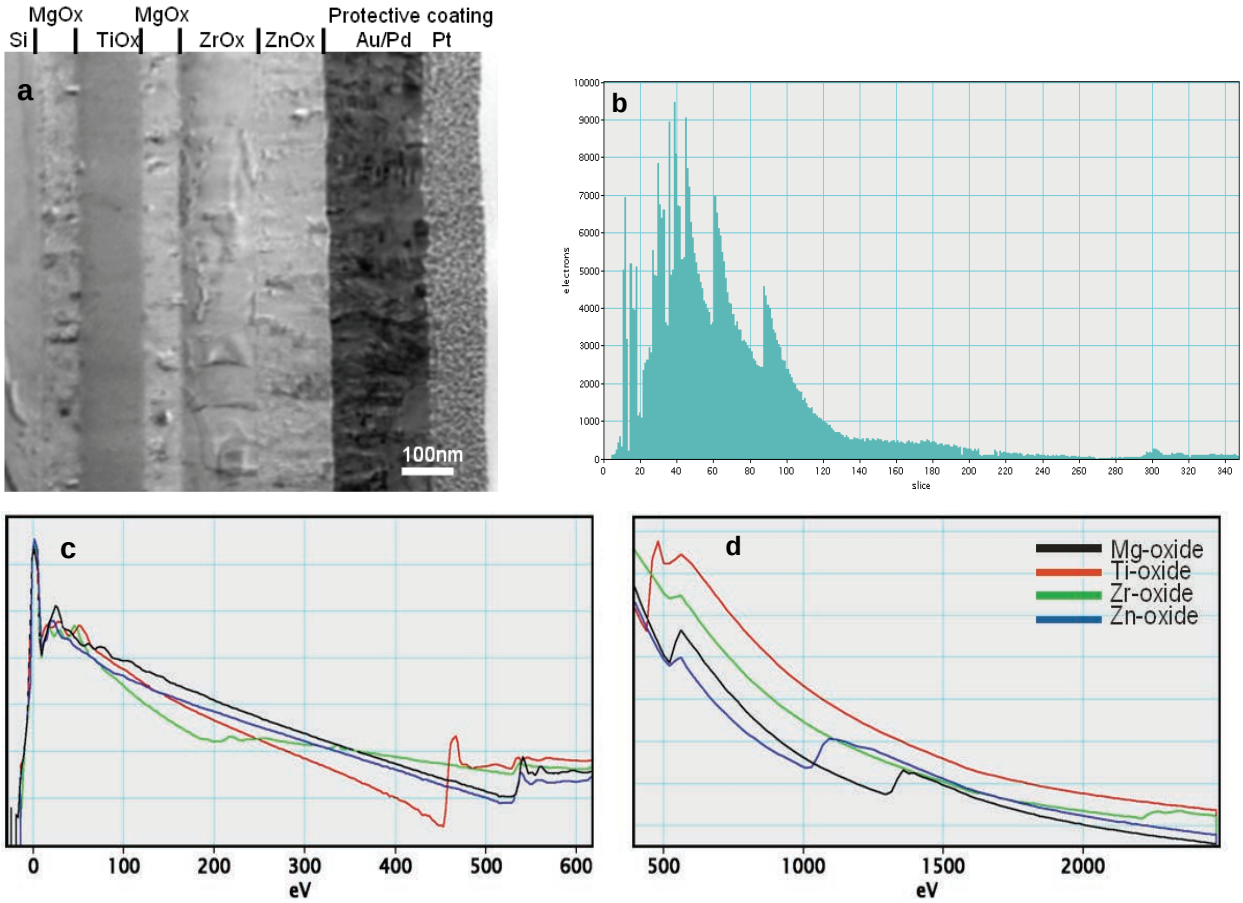

Fig.2. a) Oxide multilayer stack and b) example of an unscaled, raw EFTEM SI spectrum, reflecting different exposure times and number of image summations at different energies; c) and d) logarithmic representations of corrected spectral data spanning from the zero-loss peak to Zr-$L_{2,3}$ at 2.2 keV.

Applied Nanoanalysis by Electron Energy-Loss Spectrum Imaging Methods

B. Schaffer, W. Grogger, G. Kothleitner and F. Hofer

Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology and Graz Centre for Electron Microscopy, Steyrergasse 17, A-8010 Graz, Austria

Spectroscopy methods available in a modern transmission electron microscope (TEM) are indispensable tools whenever chemical information has to be known on a nanometre length scale. Together with the imaging capability of the TEM, the spectral information gained by energy dispersive X-ray spectrometry (EDXS) or electron energy-loss spectrometry (EELS) can be used to quickly and reliably identify chemical phases in a quantitative way. However, TEM images combined with spectroscopic “point” analysis of only a few small specimen areas can sometimes miss important information, for example in heterogeneous samples with small precipitates sparsely distributed all over. Energy-filtering TEM (EFTEM) can be used to rapidly map chemical phases with high spatial resolution (<nm) over large field of views (>µm). Such maps are usually calculated from two or three images. Thus, on the other hand, the spectral information is quite limited, and interpretation of image contrast in such maps may be hampered by overlapping of EELS features, especially if few is known about the specimen beforehand.

During the last decade, with the advent of advanced instrumentation and more powerful computers, analytics has started to thread spatial and spectroscopic data as a whole rather than as two individual results complementing each other. The concept of such a combined spectrum-image data was first introduced for EELS data by Jeanguillaume et al. in 1989 [1]. Besides the intuitive way of acquiring a spectrum-image by acquisition of spectral data point by point in a scanning TEM (STEM SI), there exists the alternative way of collecting a series of EFTEM images at successive energy-loss values (EFTEM SI) as first described by Lavergne et al. in 1992 [2].
In the meanwhile, advances in EELS and EFTEM spectrum imaging, i.e. the collection of data along the spatial and the energy-loss dimensions has progressed to the point, where sophisticated spectrum image data processing now can be carried out routinely. For EFTEM spectrum imaging this became possible with new acquisition and correction algorithms that are necessary for measuring a reliable data set as well as for interpreting the data correctly and accurately (adaptive acquisition, spatial drift correction, simulatneous energy-drift, non-isochromaticity and spatial drift correction) [3,4,5].

In this work we will present the current state of spectrum-imaging application. We will show both approaches and highlight their differences in acquisition, data quality, and some practical aspects. Latest developments in EFTEM-SI data acquisition, correction and evaluation will be presented [6].

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The authors would like to thank Achim Trampert, Paul Drude Institute for Solid State Electronics Berlin, for providing the GaN/AlN sample.

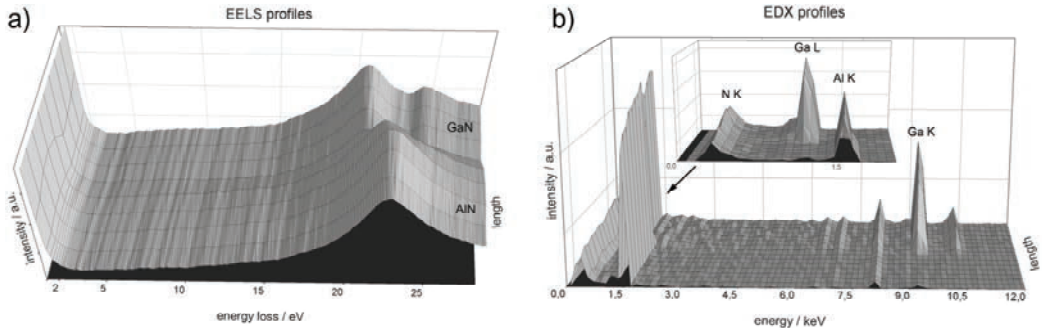

Fig. 1 Line scan across the interface of a GaN / AlN multilayer sample extracted from simultaneous EELS/EDX STEM SI: a) EELS low-loss spectra; b) EDX signal

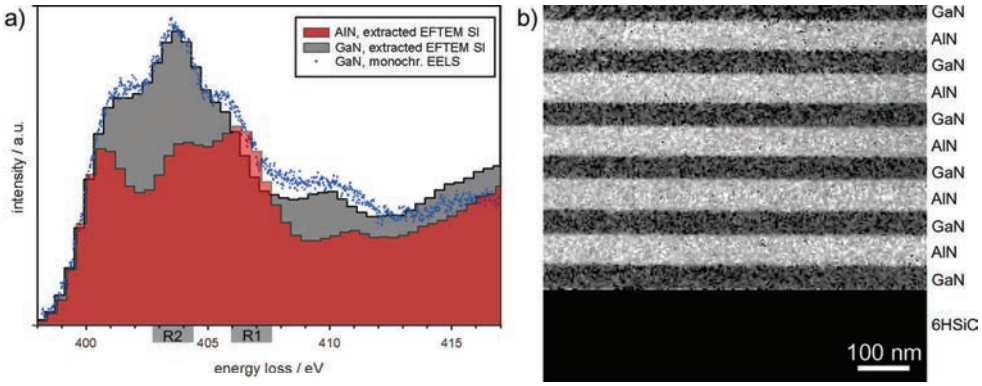

Fig. 2 a) ELNES of the N-K edge of GaN and AlN extracted from EFTEM SI. GaN data acquired in monochromated spectroscopy mode is added for comparison; b) N-K ELNES jump ratio image extracted from EFTEM SI. The energy ranges of the jump ratio image R1/R2 are indicated in a).

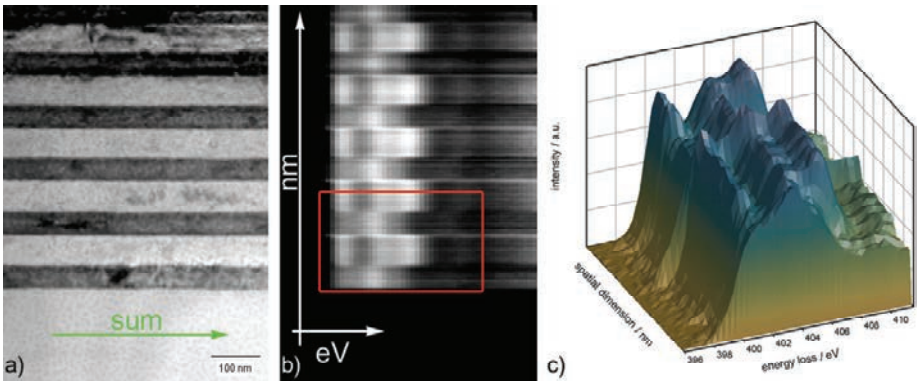

Fig. 3 EFTEM SI data at the N-K edge of GaN/AlN: a) Bright-field image showing the sample; b) EFTEM SI averaged along spatial x-direction - green arrow in a) - and plotted energy vs. spatial y-direction; c) 3D display of N-K ELNES across four layers – red area in b).

Detector Characterisation and Optimisation of Detection Limits in EELS

K. Riegler[1], G. Kothleitner[1,2]

[1] Graz Centre for Electron Microscopy
[2] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology, Steyrergasse 17, 8010 Graz, Austria

Electron energy-loss spectrometry (EELS) is an efficient microanalytical technique for gaining chemical and structural information of thin samples with high trace element sensitivity. Although in principle possible, detecting single atoms [1] is not trivial. Estimating elemental detection limits or the minimum number of detectable atoms accurately requires a) reliable signal extraction schemes, as the signal is often superimposed on a large background or overlaps with another edge, b) a good estimation on the statistical uncertainty associated with background removal, c) accurately known ionisation cross-sections and d) a good understanding of the noise introduced by the spectrometer's detector.

In this work systematic studies have been performed to evaluate the best possible way for signal extraction and optimising the signal-to-noise ratio. Natural ruby was chosen as a complex sample, featuring overlapping edges, containing 0.4 at% Cr (Cr-$L_{2,3}$ edge at 575 eV) on top of the alumina background (background plus O-K edge at 532 eV).

A comparison between SNR simulations carried out via conventional background subtraction [2] and SNR simulations, done with multiple linear least squares fit techniques (MLS) [3] has shown that the MLS fit method gains higher SNR values – regardless of the amount of Poisson noise – due to the fact that wider fitting windows can be used [figure 1 and 2). Furthermore, MLS simulations have proven that an optimum position with respect to the SNR and the χ^2 (a measure of the goodness of the fit) exists for every size of the fitting window. These theoretical findings were compared with measured spectra and it could be shown that the quantification using experimental cross-sections via MLS fit routines yields quite accurate results (fig. 3), when cross-checked with LA-MS and WDX analyses.

Predicting accurate SNRs requires also the understanding of the behaviour of the detector. The detector (Gatan UltraScan 2kx2k) was characterised by determining the modulation transfer function (MTF) and the detection quantum efficiency (DQE) of the system. Experimentally, the MTF was measured using the edge method [4]. The measured MTF in combination with power spectra of acquired noise images as a function of incident intensity and the knowledge of the conversion efficiency of the system enables to derive the DQE of the system (figure 4). The maximum DQE for this system is about 0.4 varying only slowly with dose.
Having optimised the fundamental steps necessary to detect small concentrations we could derive a reliable figure for the attainable detection limits for different microscopes.

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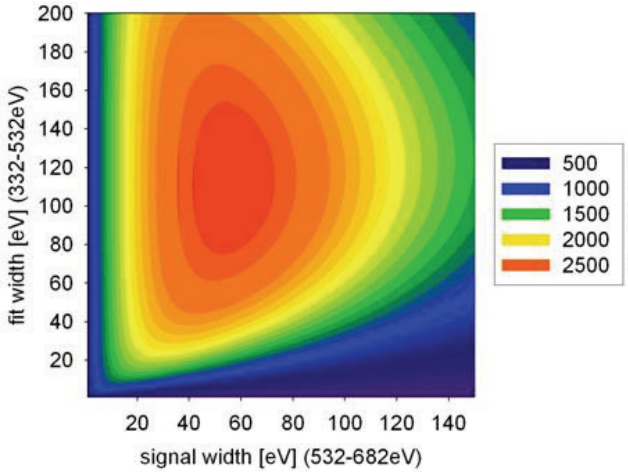

Fig. 1 Optimization of the conventional calculated SNR plotted as function of the size of the fitting window and the signal window.

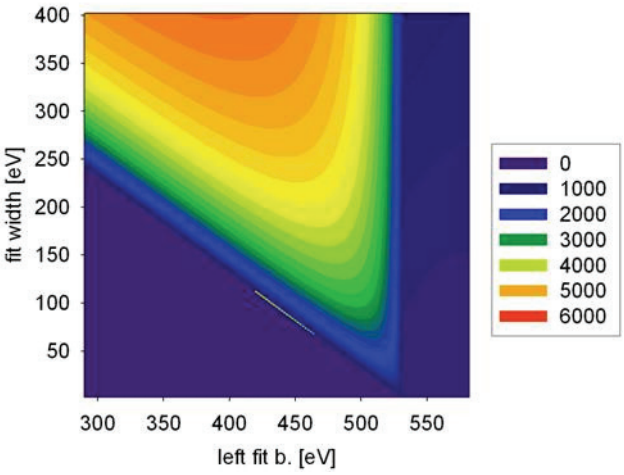

Fig. 2 Optimization of the SNR calculated with the MLS method plotted as function of the size and position of the fitting window.

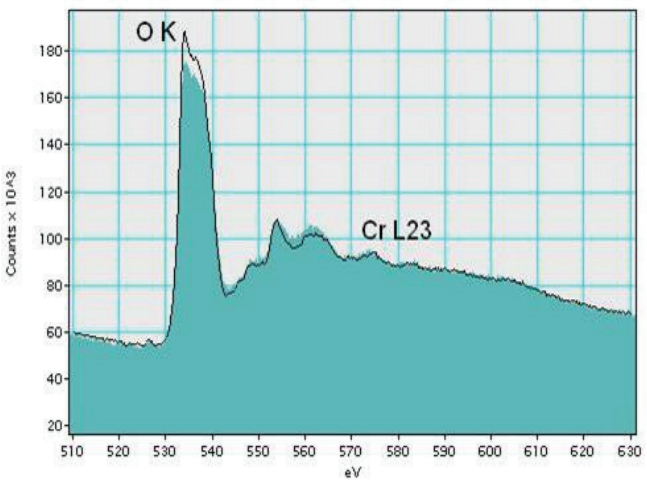

Fig. 3 EEL spectrum of ruby (filled area) with the appropriate MLS Fit (black line). Cr 0.4at%

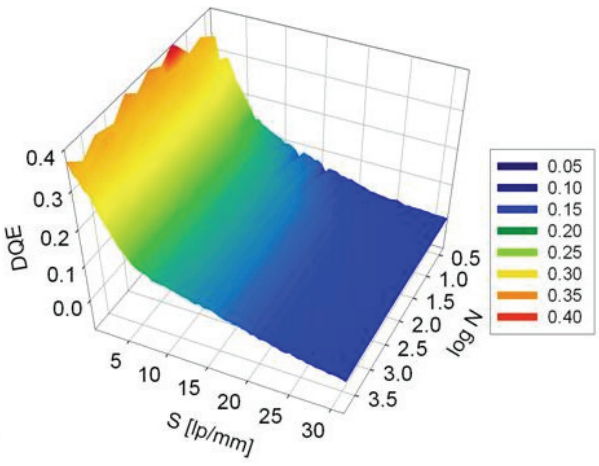

Fig. 4 Frequency-dependent DQE (Binning 1, 200kV) of the Gatan UltraScan.

EELS Quantification of Complex Nitrides in a 12 % Cr Steel

M. Albu[1], F. Méndez Martin [2] and G. Kothleitner [1]

[1] Institute for Electron Microscopyand Fine Structure Research, Graz University of Technology
Graz Centre for Electron Miroscopy, Steyrergasse 17, A-8010 Graz, Austria
[2] Institute for Materials Science, Welding and Forming, Graz University of Technology, 8010 Graz, Austria

In 9-12 % Cr steels, nitrides (MX, M_2X and modified Z-phase ((Cr, V, Nb, Fe) N)) are of special interest because of their different contribution to the creep strength of the material. The changes in the chemical composition and their crystallography were investigated using transmission electron microscopy (TEM).

The different phases have been identified by using the energy filtered TEM (EFTEM) technique [1] and the corresponding bivariate scatter diagrams of chromium and vanadium jump ratios (Fig. 1a-b). Their elemental composition has been established both with electron energy loss spectroscopy (EELS) and energy dispersive X-ray spectroscopy (EDX). Nevertheless the light elements are not easily quantifiable with EDX, for which reason EELS was involved. Furthermore, the energy-loss near edge structure (ELNES) of the nitrogen ionisation K edge has been used to differentiate between different metastabile nitride phases [2].

Analysis was carried out on a total of 60 thin particles from three differently treated samples: as-received (after tempering at 780 °C), thermally aged and creep loaded at 600 °C for 24 639 h at 115 MPa. The specimens were prepared by extracting the precipitates from the matrix into an amorphous carbon film.
In the EEL spectra, edges coming from Nb-$M_{4,5}$, Nb-$M_{2,3}$), N-K, V-$L_{2,3}$, Cr-$L_{2,3}$ and Fe-$L_{2,3}$ were recognised in addition to amorphous carbon from the extraction film. Since the edges of Nb, N, V and Cr are energetically too closely spaced and a conventional edge intensity extraction is not possible, the multiple linear least squares (MLS) fit deconvolution has been employed, fitting suitable references to all overlapping edges. Such an MLS fit together with the references used is shown in Fig. 2 for a modified Z-phase particle. Computing the relative fit weights and integrating the references over an energy range of 100 eV, allows calculation of the atomic percentages of each element quite accurately, provided experimentally determined cross-sections [3, 4] are available, as in this case.

The mean elemental concentrations of precipitates under study i.e. the M_2X, MX and modified Z-phase shows a good agreement comparing with the outcomes of the thermodynamic model implemented in the software package MatCalc (Table I) [5].

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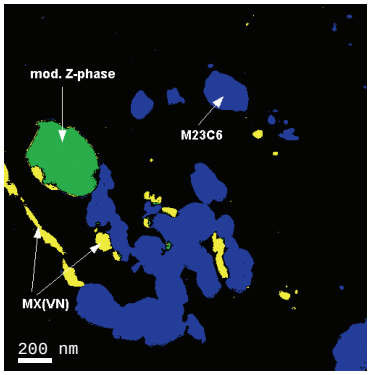

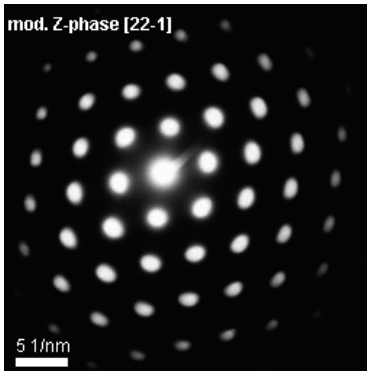

Fig.1 Phase diagram of a representative position from a creep loaded sample and the respective diffraction pattern from the modified Z-phase – green particle. $M_{23}C_6$ – blue and MX (VN) – yellow were identified as well.

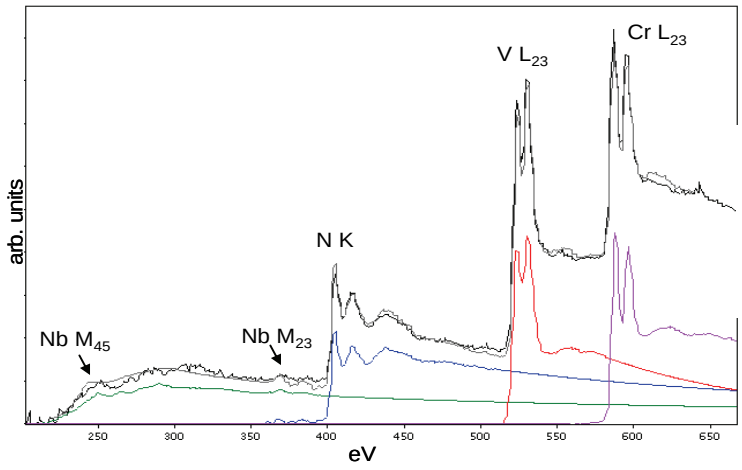

Fig. 2 MLS fit using Nb, N, V and Cr - references for a mod. Z-phase particle; the experimental spectrum is represented in black and the MLS fit in grey.

Table I. Mean concentrations and their standard deviations from EELS measurements of M_2X, MX and modified Z-phase particles compared with the MatCalc simulations.

EELS	Cr (at.%)	V (at.%)	Nb (at.%)	Fe (at.%)	N (at.%)
M_2X	49.8 ± 3.4	17.2 ± 2.1	-	2.6 ± 1.7	30.4 ± 4.2
MX	11.6 ± 4	40.0 ± 4.5	6.9 ± 2.7	1.7 ± 1.1	41.2 ± 4.8
mod. Z-phase	31.4 ± 4.2	30.1 ± 2.4	3.1 ± 1.6	3.5 ± 1.2	32.3 ± 3.4

Simulations	Cr (at.%)	V (at.%)	Nb (at.%)	Fe (at.%)	N (at.%)
M_2X	50.69	14.75	1.04	0.09	32.87
MX	0.05	47.39	5.40	0.02	46.61
mod. Z-phase	31.31	31.20	3.45	3.30	30.70

Dual-Beam FIB Application of 3D EDXS for Superalloy δ-phase Characterization

J. Wagner [1], M. Schaffer [1] H. Schroettner [1], S. Mitsche [1], I. Letofsky-Papst [1], Ch. Stotter [2], and Ch. Sommitsch [2,3]

[1] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology and Graz Centre for Electron Microscopy, Steyrergasse 17, A-8010 Graz, Austria
[2] Christian Doppler Laboratory of Materials Modelling and Simulation, University of Leoben, Franz-Josef-Strasse 18, A-8700 Leoben, Austria
[3] Chair of Metal Forming, University of Leoben, Franz-Josef-Strasse 18, A-8700 Leoben, Austria

Optimizing the functionality of materials often depends on a precise control of the size, shape, crystal structure and composition of the material being synthesized. But in the age of micro- and nanotechnology it can be a significant challenge to characterize solids in an appropriate way. Therefore many sophisticated analysing methods were established and combined in the past [1, 2]. Automated serial sectioning in combination with physical analytics achieved by using a script-able Dual Beam -Focused Ion Beam system (DB-FIB) equipped with an energy dispersive X-ray spectrometer (EDXS) is only one of the possible characterization opportunities mentioned above [3].
In this work we present an excellent example how powerful EDXS-mapping in three dimensions combined with the advantages of the block lift out technique [4] provides a better understanding of material properties. Today, there are several commonly used alloys like nickel-based superalloys, which consumes up to 30% of the weight in advanced aerospace engines. Some examples of alloys in use are Udimet 720, Waspaloy and Alloy 718, the latter is the most widely used due to its relatively low costs and good formability. Recently, a new nickel-based alloy was developed (Allvac 718 Plus^{TM}) by the company ATI Allvac. More details of the extended mechanical properties of this alloy can be found at Bergstrom [5]. Its properties are attributed to the combined effects of chemistry, heat treatment and microstructure. Especially for hot forged gas turbine disks, the influence of δ-phase is important. However, it is well known that the start of δ-phase precipitation strongly depends on the experimental conditions. Thus, an understanding of the phase stability with time and temperature is essential in order to tailor the microstructure of forged turbine blades for high temperature applications.
Among several investigative tools and techniques like electron back scatter diffraction (EBSD), back scatter electron imaging (BSE) and transmission electron microscopy (figure 1) the 3D micro-structural characterization was carried out on the FEI Nanolab Nova200 dualbeam focused ion beam (DB-FIB - FEI company, Eindhoven, The Netherlands) equipped with an energy dispersive Si(Li) X-ray detector (10 mm²) system from EDAX (Mahwah, USA) using the Genesis software version 4.52. Final data visualization was performed using the Amira 3.1 software (Mercury Computer Systems SA). The serial–sectioning thickness was selected to be 500 nm. Due to the lack of an EBSD system in the DB-FIB additional ion beam imaging giving a good channelling contrast was performed and therefore rotating, translating and tilting the sample between the cross-section milling position and the SEM imaging position was required. Figure 2 depicts the 3D models, reconstructed from the experiment. It can be seen in Figure 2b that the plate-like δ-phase appears at both, grain boundaries and twins. δ-phase that appear needle-like have a very small angle to the cross section. Hence, a complete plate-like reconstruction is not possible. The FIB-SEM investigations confirm the plate-like shape of the δ-phase, which agrees well with previous 2D experiments and data from literature.

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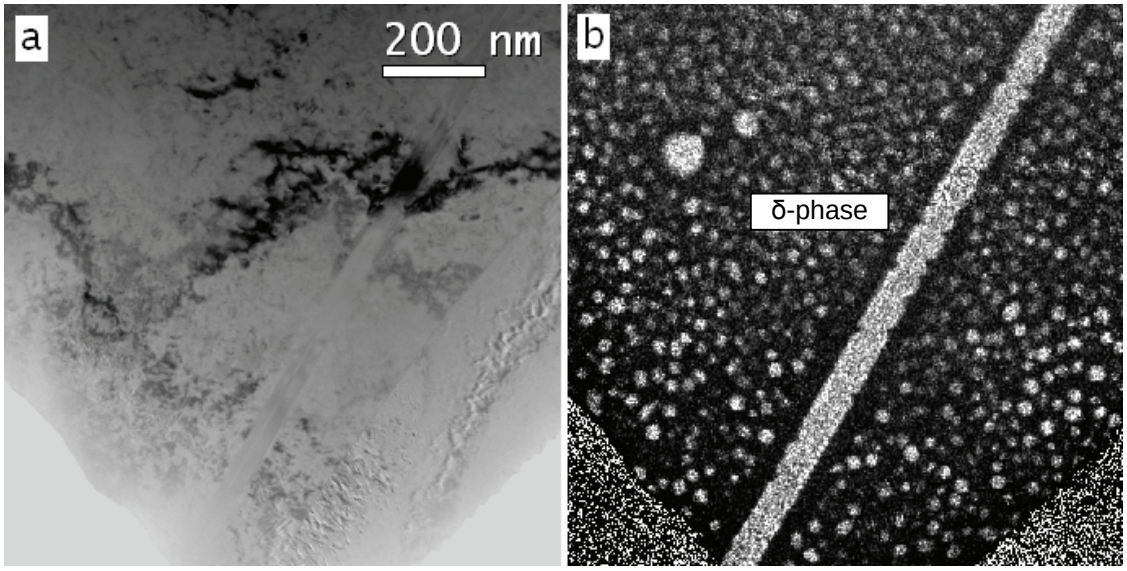

Fig. 1a) TEM bright field image of Allvac 718 Plus™, b) EFTEM Ni-$M_{2,3}$ jump ratio image.

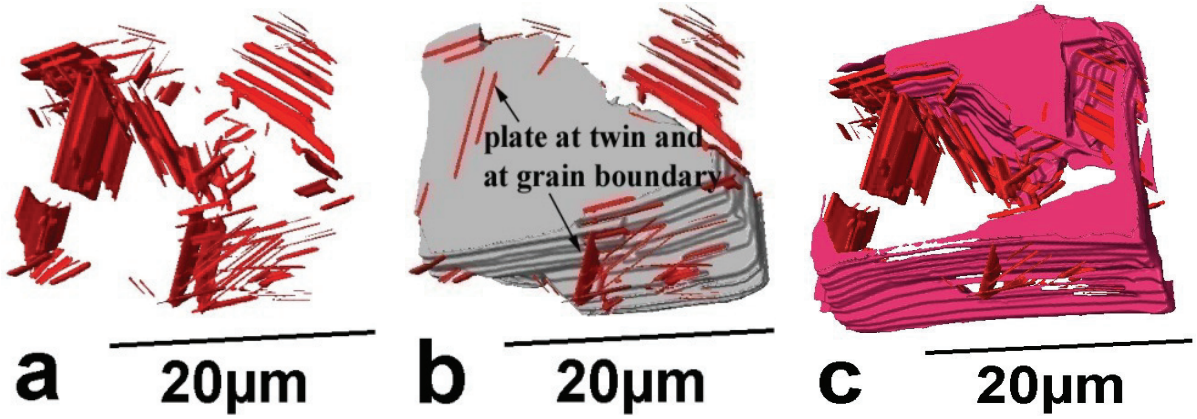

Fig. 2a) Reconstruction of the δ-phase, b) twin-reconstruction with the δ-phase and c) surrounding grain with the δ-phase.

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In-situ Experiments on Soft Materials in the Environmental SEM - Reliable Results or Merely Damage?

P. Pölt[1], H. Reingruber[2], A. Zankel[1] and C. Elis[1]

[1] Institute for Electron Microscopy, Graz University of Technology, Graz Centre for Electron Microscopy, Steyrergasse 17, A-8010 Graz, Austria
[2] Institute of Experimental Physics, Graz University of Technology, Petersgasse 16, A-8010 Graz, Austria

The conventional high vacuum scanning electron microscope is an instrument which is mainly used for the analysis of solid materials. In contrast, the environmental scanning electron microscope (ESEM) is also ideally suited for the operation and control of a great variety of dynamic experiments. Since the type of gas in the specimen chamber, the relative humidity in case of water vapour as gas, the gas pressure and the specimen temperature can be varied over a broad range, an ESEM forms a sort of micro reactor, where the wetting, melting, recrystallization, corrosion of materials can be investigated. Additionally, no coating of non-conductive materials is necessary to prevent charging. All this seems to make the ESEM an excellent tool for the investigation of soft materials and also their behaviour in a wet environment.

But several shortcomings make these experiments much more difficult and the results less reliable than one would predict beforehand. Firstly, soft materials are mainly carbonaceous and therefore give notoriously poor contrast. In the low vacuum the contrast decreases additionally with increasing pressure. Although this could be compensated for by an increase in the probe current, a concurrent increase in the irradiation damage makes this very often impossible [1]. But in many cases a thin coating of the material with e.g. chromium or gold suffices to substantially reduce the damage.

Moreover, the presence of water can strongly increase the amount of the irradiation damage due to the formation of highly mobile and reactive free radicals [2, 3]. It can also change the wetting behaviour of the material. Figure 1 shows that both the wetting of a material and its drying-up can be affected by the electron irradiation.

But the figures 2 and 3 prove that despite all these shortcomings the ESEM can be a very valuable tool for in-situ experiments of soft materials. A new and exciting application is automated ultra microtomy in the ESEM and the 3D-representation of the internal structure of materials [4]. Other applications are for example the fracture behaviour of textile fibres in dependence on the relative humidity in the specimen chamber or the imaging of the transport of fluids through porous media [5].

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Financial support by the NAWI-Faculty of the TU Graz and the KFU Graz is gratefully acknowledged.

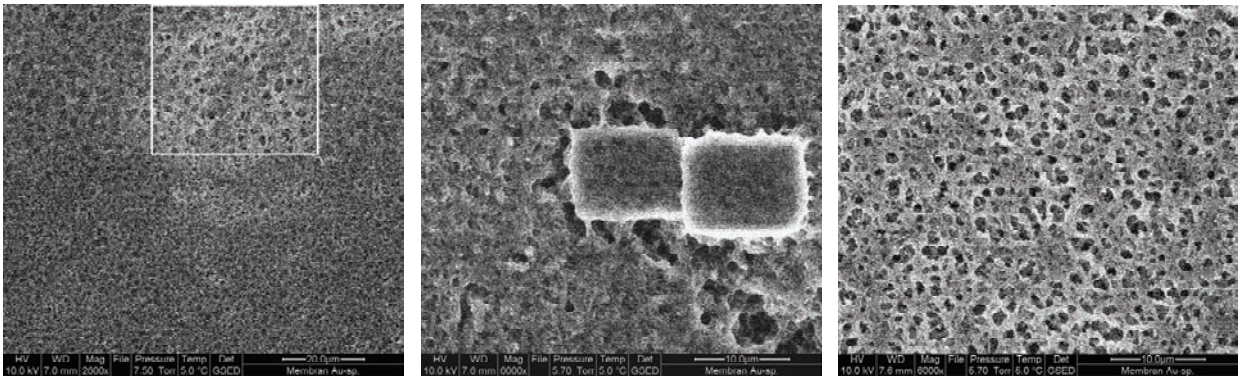

Fig. 1 Wetting and drying-up of a cellulose nitrate membrane (10 nm Au-coating). Left: Before recording the image, mainly the marked area had been irradiated. Contrary to the surrounding, many of the pores in this area are not filled / fully filled with water (image width: 100 µm). Centre and right: Drying-up is delayed in the irradiated areas (centre) compared to other areas (right). In the irradiated areas strong damage is visible (image width: 42 µm).

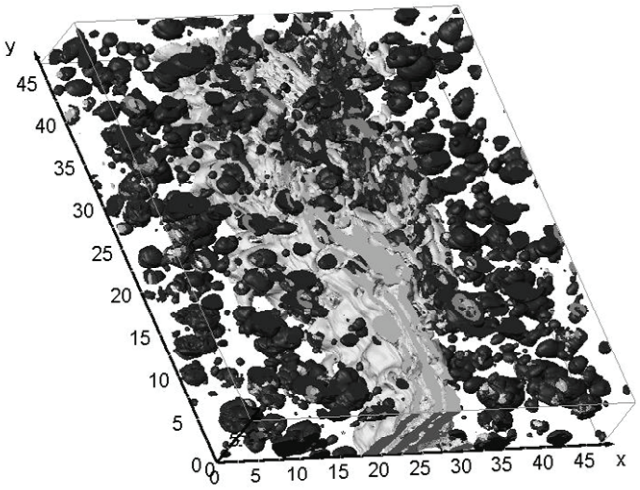

Fig.2 3D-representation of EPR (ethylene propylene rubber) modified iPP (isotactic polypropylene) after a tensile test, stopped at 25% yield and stained with RuO_4 (160 slices, slice thickness: 100 nm); black: EPR particles; grey: cracks.

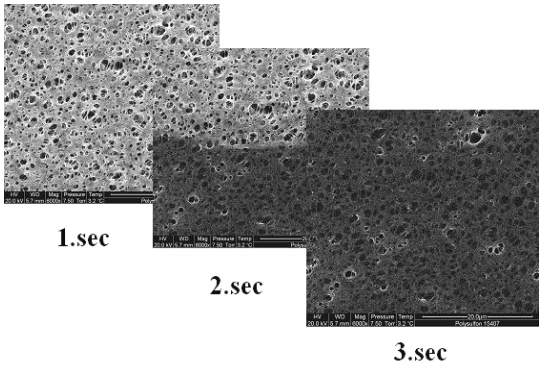

Fig.3 Wetting of a polyethersulfon membrane (nominal pore size: 450 nm) with water in dependence on time (width of the images: 45 µm). Some of the big pores remain partially filled / unfilled. The water was provided by condensation at a Peltier cooling stage.

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High-temperature Oxidation of Steel in the ESEM with Subsequent Scale Characterisation by Raman Microscopy

A. Reichmann, P. Pölt, C. Brandl, B. Chernev and P. Wilhelm

Institute for Electron Microscopy, Graz University of Technology and Graz Centre for Electron Microscopy, Steyrergasse 17, A-8010 Graz, Austria

Stainless steels used as materials for technical processes at elevated temperatures are protected against high temperature corrosion by the formation of a Cr-rich oxide layer [1]. It is very important that this protective scale is developed very fast at the beginning of the technical process, so that a chemical attack of the steel, which is exposed to different gas atmospheres, is prevented [2].
The ESEM (Environmental Scanning Electron Microscope) equipped with a heating stage enables the direct observation of changes of the morphological structure during heating with high magnification and high depth of focus. It is a great advantage of an ESEM is that the studies can be carried out with different gases. These gases are used for amplifying the secondary electrons generated from the metal substrate, balancing the negative charge on the surface and they can react with the sample itself [3].
After the in situ investigation of the high temperature oxidation the composition of the oxide scales has to be characterised. Raman Microscopy is a good complementary technique to SEM/EDX to identify thin layers of oxide corrosion products on metal surfaces [4].

In our work we investigated the high temperature oxidation of the austhenitic steel 353MA, used for heat exchanger tubes. “Figure 1” shows the scale formation of the polished steel in the ESEM with a heating rate of 2 K/min till 973K in air at a pressure of 133Pa. At about 623 K the first grains appeared at the polished surface. They grew along lines because of remaining scratches from the polishing procedure before. At the end of the experiment the whole surface was covered by a dense oxide layer interrupted by the original grain boundaries, which got visible as deep groves. “Figure 2” shows a BSE image of the created scale after the high temperature study. The oxidation products were analysed by Raman Microscopy and consist mainly of Cr_2O_3 and spinels. The grains, which formed lines, contain Fe_2O_3, too.
In conclusion we can state that the ESEM is a proper instrument for the investigation of the early stages of scale formation of stainless steels during high temperature oxidation. The identification of different phases could be improved by the development of a BSE detector for high temperature experiments.

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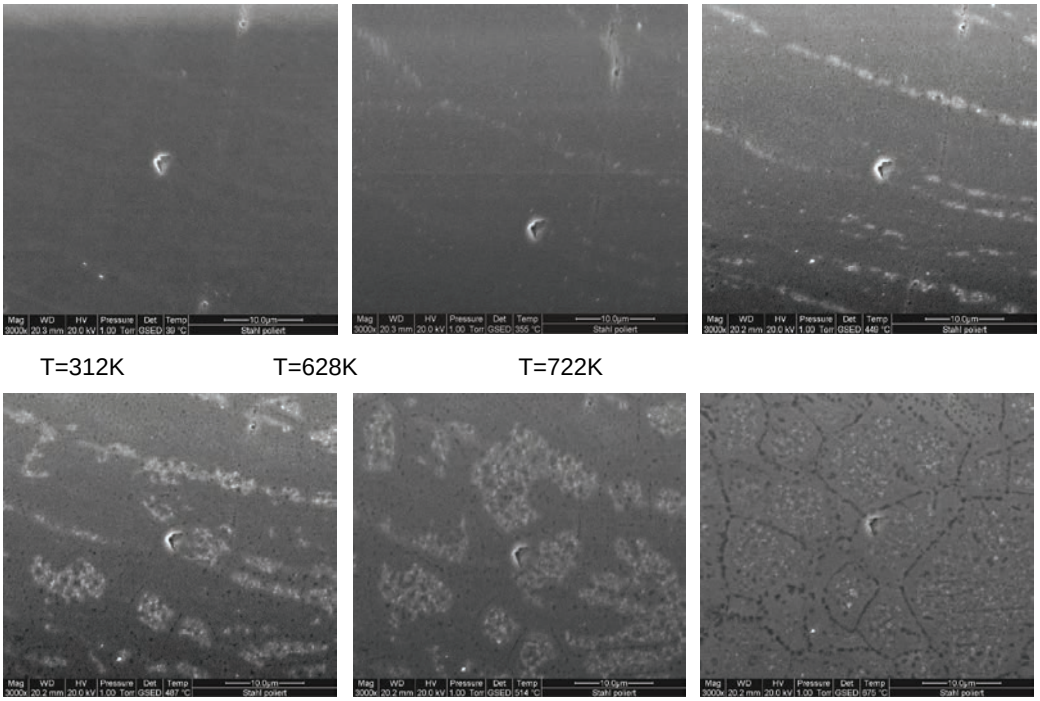

Fig. 1 Observation of scale formation during heating in the ESEM

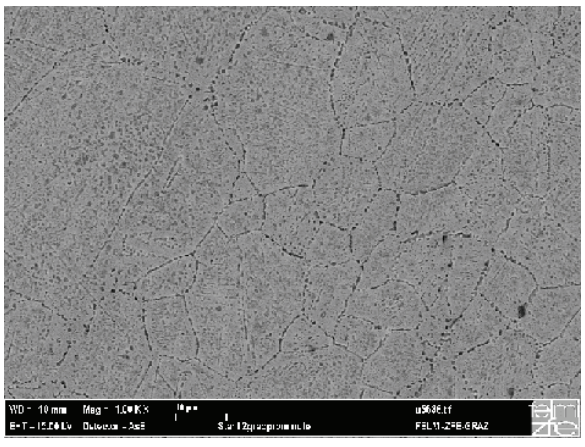

Fig. 2 BSE image of the scale

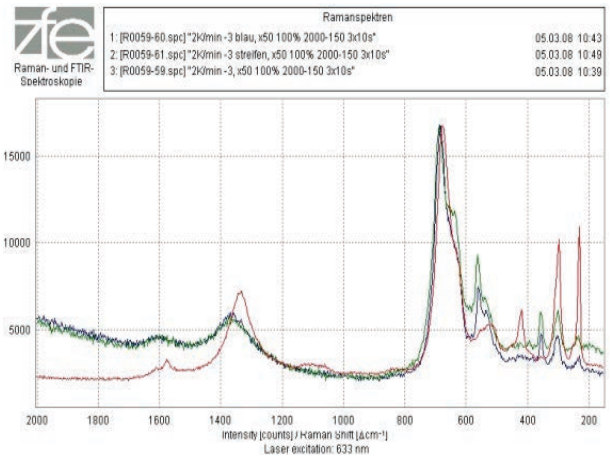

Fig. 3 Raman spectra of the scale

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Characterisation of the Subgrain Structure of the Aluminium Alloy AA6082 after Homogenization and Hot Forming by EBSD

S. Mitsche[1], P. Sherstnev[2], C. Sommitsch[2] and M. Hacksteiner[3]

[1] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology and Graz Centre for Electron Microscopy, Steyrergasse 17, A-8010 Graz, Austria
[2] Institute of Metal Forming, University of Leoben, Franz-Josef-Strasse 18, A-8700 Leoben, Austria
[3] AMAG Rolling GmbH, Postfach 32, A-5282 Ranshofen

The properties of a hot rolled aluminium alloy are strongly dependent on the homogenization and the forming conditions [1,2], because the microstructure of the specimen will be changed by the various processing steps [3]. A powerful tool to investigate the obtained grain – subgrain structure is the backscatter electron diffraction (EBSD) technique [4]. The evolution of the microstructure of the aluminium alloy AA6082 during hot forming is presented in the following.

Specimens of the alloy AA6082 were first homogenized at 570°C for 10h to solute the Mg_2Si-phase and to induce a coagulation of the plate like β-AlFeSi-phase. Rastegaev-cylindrical compression tests of these specimens were performed at the institute for metal forming on a SERVOTEST transform simulator. A deformation degree of $\varphi = 0.8$ was chosen, because at this deformation a fully stationary subgrain structure was to be expected. The temperature (500°C and 550°C) and the deformation rate ($1s^{-1}$ and $10s^{-1}$) were selected to have similarly conditions as during hot-rolling with a DUO machine.

Cross sections of the deformed specimens were analysed in a scanning electron microscope (Zeiss DSM 982 Gemini) equipped with an EBSD system from EDAX-TSL (SIT-camera, OIM-software 4.5). An acceleration voltage of 20 kV and a probe current of 3.4 nA were used for all specimens analysing an area of 320 µm x 350 µm with a step size of 1 µm. In Figure 1 the obtained results are displayed as inverse pole figure (IPF) maps, where the white lines represent the high angle boundaries (>15°) and the black lines the subgrain boundaries (between 2° and 15°). It is clearly to see that the size of the subgrains (determined with an imaging analysis program) strongly depends on the temperature and the deformation rate.
Introducing the Zener-Hollomon-parameter Z, which is defined as $Z = \varphi \bullet (Q/(R \bullet T))$ (φ deformation rate, Q activation energy, R universal gas constant, T temperature), a decrease of the subgrains within the elongated high grain boundaries by increasing Z can be observed (see Figure 2). From this figure the line of best fit was determined and as a result the size of subgrains in the stationary range δ_{ss} can be described as a function of Z: $\delta_{ss}^{-1} = -0.8274 + 0.0318 \ln(Z)$.

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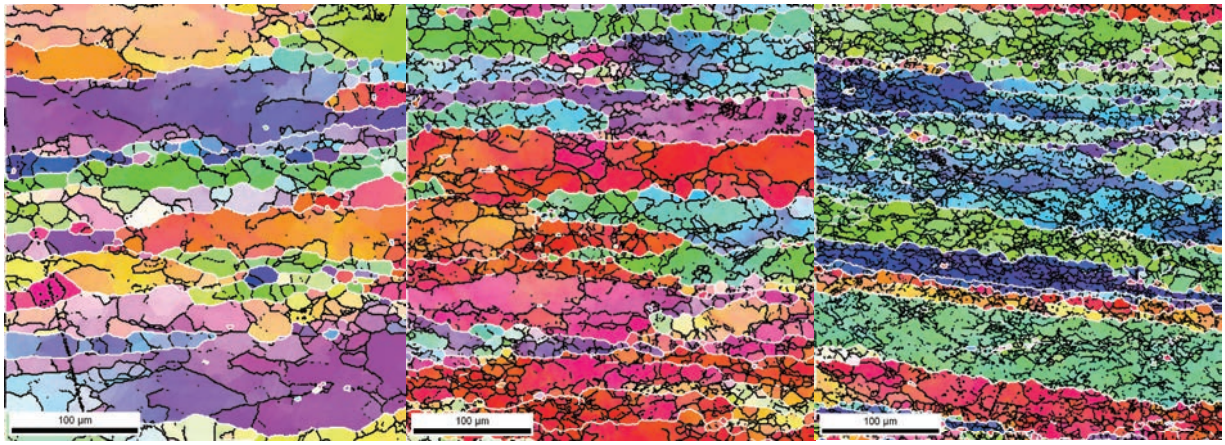

Fig. 1 Inverse pole figure maps (IPF) of the compressed specimens at T = 550°C and φ = $1s^{-1}$ (left) respectively φ = $10s^{-1}$ (centre), as well as T = 500°C and φ = $10s^{-1}$ (right); white lines mark the high angle boundaries (>15°) and black lines the subgrain boundaries (between 2° and 15°).

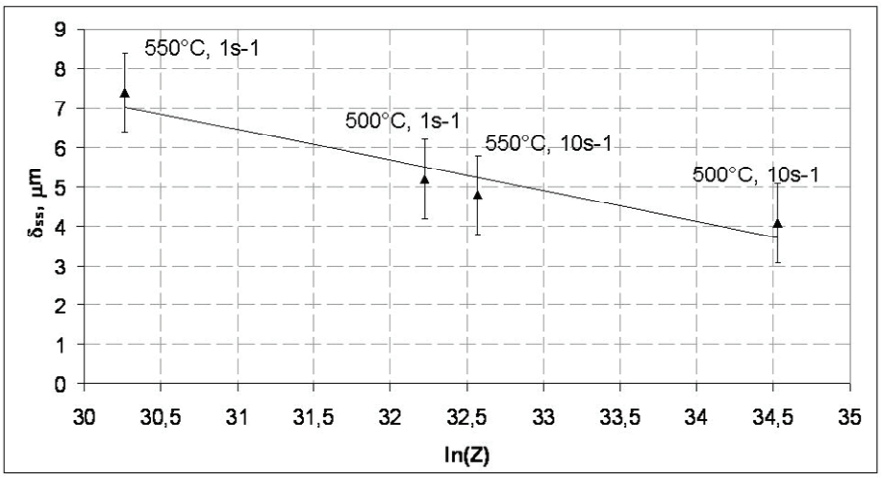

Fig. 2 Size of the subgrains (experimentally determined) in dependence on the Zener-Hollomon-parameter.

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The Interior Interfaces of a Semiconductor/metal Nanocomposite and their Influence on its Physical Properties

P. Granitzer[1], K. Rumpf[1], P. Pölt[2], M. Albu[2], B. Chernev[2]

[1] Institute of Physics, Karl Franzens University Graz, Universtätsplatz 5, 8010 Graz, Austria
[2] Institute for Electron Microscopy, University of Technology Graz, Steyrergasse 17, 8010 Graz, Austria

A semiconductor/metal hybrid system with silicon as base material and embedded metal nanostructures is fabricated in two steps by anodizing the silicon substrate in an aqueous hydrofluoric acid solution and a subsequent electrodeposition process. The achieved self-assembled nanoscopic composite system consists of a quasi-regular 3-dimensional arrangement of metal nanostructures embedded within the pores of the silicon matrix. The used metals are generally Ni, Co and their alloys. In such a hybrid system the interface between metal and silicon nanostructures is of interest in correlation with the properties of the material and can be influenced by different fabrication conditions.
The formation of PS is performed by anodizing a highly n-doped silicon wafer in an aqueous HF-solution resulting in a matrix offering a meso-/macroporous morphology with oriented pores, grown perpendicular to the sample surface which are not connected among each other [1]. Due to the employed electrochemical process parameters as current density and electrolyte concentration the pore-diameter can be varied between 30 nm and 100 nm, whereas the concomitant pore-distances diversify in a smaller range between 40 nm and 60 nm. Typical investigated specimens exhibit an average pore-diameter of 60 nm and a mean pore-distances of 40 nm. Depending on the anodization time the thickness of the porous layer is typically 30 µm - 50 µm whereas the growth rate is about 3 µm/min, resulting in an considerable aspect ratio of the pores of about 1000 (Fig. 1). Before these PS-matrices are filled with a ferromagnetic metal by electrodeposition some of the specimens are aged in air to obtain a native oxide layer (about 5 nm), others are filled with a metal immediately after the etching procedure.
The metal-precipitation within the pores is carried out in using a pulsed deposition current under galvanic conditions. As electrolyte an aqueous metal-salt solution is used whereas in case of Ni-deposition a $NiCl_2$-electrolyte or the Watts electrolyte ($NiCl_2$, $NiSO_4$) is used. The structure and physical properties of the nanocomposite system were investigated with scanning and transmission electron microscopy (SEM, TEM), optical methods (FTIR- and Raman spectroscopy) and SQUID-magnetometry.
The interior surface of the PS-matrix as well as the surface of deposited metal structures and the interface between silicon and metal are investigated with respect to oxidation because of its influence on the properties of the material. Optical methods as FTIR- and Raman-spectroscopy are performed to gain information especially about the PS-skeleton [2]. Investigating bare silicon, PS and Ni-filled PS-samples by Raman-spectroscopy a small shift of the Raman-peak from 522 cm^{-1} (silicon) to 520 cm^{-1} (PS) and a further greater shift to lower wavenumbers of 505 cm^{-1} (PS-Ni) is observed. This shift is attributed to compressive stress caused by the anodization process and the subsequent metal-deposition within the pores leading to a greater mismatch between bulk-silicon and PS.
PS samples with precipitated Ni-nanostructures have been prepared for TEM-investigations by FIB. The shown sample has been cut in order to gain a plan-view membrane of the PS-matrix. The TEM-images (Fig. 2 left) show the Ni-structures within the pores of the silicon template. In this image only a few pores filled with Ni-structures can be seen. This is caused on the one hand by the preparation of the membranes by FIB and on the other hand that this image shows only one level of the PS layer. Due to the fact that the PS membrane for TEM-investigations is very fragile some of the metal-structures which are not strongly linked to the pore-walls can loosen. Considering a SEM-picture of a cross-section of the PS-template (fig. 2 right) one sees that the porous layer is Ni-loaded over the entire thickness (pore-length) but the pores are not completely filled. The samples exhibit a statistically distributed 3-D array of

precipitated metal-nanostructures. Individual Ni-particles are also examined by HRTEM and additionally by EELS-spectroscopy to gain information about the interior surface of the PS-matrix as well as of the embedded Ni-particles. High resolution TEM-images show the Ni-particle surrounded by an oxide layer of 3 nm [2].
Magnetization measurements performed by SQUID-magnetometry have also been carried out to gain information about the interface of the precipitated Ni-particles. Field cooled measurements are used to figure out if the particles show an exchange bias effect caused by an antiferromagnetic nickel-oxide layer. Before carrying out the magnetization measurements the samples have been magnetized in an applied magnetic field of 7 T and then cooled down from 300 K to 4.2 K. Subsequently the magnetization has been detected in dependence on the applied field. The obtained hystereses loops do not show an asymmetry (not shown here) which means no exchange bias effect [4] is observed. So it can be said that the oxide shell of the Ni-structures is not antiferromagnetic or the samples are oxidized after the FIB-cut procedure by storing in air.

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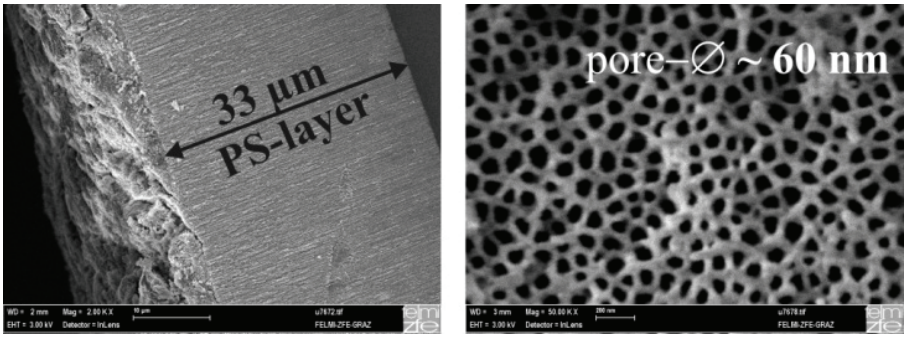
Fig. 1
Left: cross sectional SEM-image of a PS-layer of 33 µm in thickness. Right: top view of the same specimen showing an average pore-diameter of 60 nm and a mean pore-distance of 40 nm.



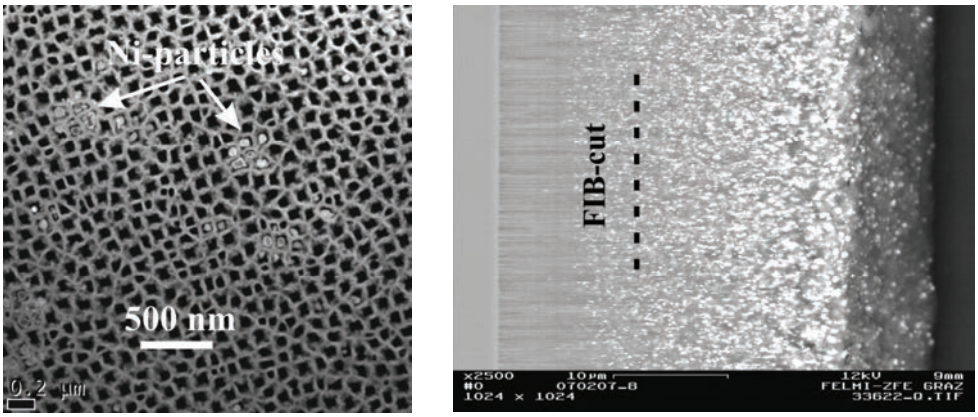


Fig. 2 Left: TEM-image of a FIB cut Ni-filled PS-sample showing precipitated Ni-particles within the pores of the PS-matrix. Right: Cross-sectional SEM-image of the entire porous layer of same sample. The level of the FIB-cut is marked.

Nano-Imprint Lithography Technique for Etch-free Processing

T. Haber[1], C. Auner[2], U. Palfinger[2], M. Sezen[1], and W. Grogger[3]

[1] Centre for Electron Microscopy, Graz, Austria
[2] Institute of Nanostructured Materials and Photonics, Joanneum Research, Weiz, Austria
[3] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology, Austria

As the field of organic electronics moves on from purely laboratory based development to standard production procedures high throughput at low costs becomes a decisive requirement. Nano-imprint lithography (NIL) appears to be a suitable tool for mass production of according devices. The goal of the NIL process is to print the device pattern onto a substrate with a sub-micron resolution independent of the applied material.
The principle of this procedure is shown in figure 1a. A silicon stamp, containing source and drain pattern, is pressed onto a stacked system of substrate (glass or flexible substrates), gate electrode, dielectric and an imprint resist. During imprinting the resist is cured and thereby the pattern is transferred irreversibly into the resist. After the release of the stamp the resist has to be etched away so that no residual layer is left at the imprinted areas (state of the scheme). Subsequently the electrode material can be sputtered, lifted off and finally the active material is applied to complete the device. However, one drawback of NIL is the anisotropic etching-step necessary to remove residues of the NIL resist after the imprint.

Here we present an improved UV-NIL process [1]: The main idea is to get rid of the etching-step by introducing a residue-free imprint. The scheme (fig. 1a) shows a sample directly after the imprint-step where the NIL resist is removed completely at the imprinted areas making the etching-step obsolete. For this achievement a new UV-NIL-resin was developed (NImpR) that shows low viscosity, is curable by UV light within seconds and moreover, it is soluble with water. Furthermore, the stamp's surface energy was adjusted to maximize the contact angle between the resin and the stamp.

To evidence the residue-free imprint, cross section lamellae were prepared using a focused ion beam instrument (FIB) and analyzed by analytical TEM investigations. Since the specimens are multi-layered compositions of inorganic and organic layers on glass or soft matters both, the preparation as well as the TEM investigation turned out to be challenging. However, bending of the lamella was kept under control by thinning only regions of interest and thus leaving the major part of the lamella as a static support as shown in figure 1b. This way the lamellae could be prepared successfully and thinned to a suitable value for energy filtered TEM (EFTEM) as evidenced by a relative thickness map shown in figure 1c. A zero-loss filtered bright field image is shown in figure 1d where the substrate, the gate electrode, the polymers and the protective layers can clearly be distinguished. However, a distinction between the individual polymer layers could not be achieved. In the presented case three polymer layers are involved, benzocyclobutene (BCB), poly-(vinyl cinnamate) (PVCi) and NImpR. Since BCB contains silicon and NImpR contains nitrogen, elemental mapping enables the visualization of the spatial distribution of the involved polymers. Figure 1e shows a superposition of four differently colored elemental maps, where the different polymers can clearly be distinguished. NImpR is obviously completely removed in the imprinted areas, even small pumps, as indicated by the white arrow, can clearly be identified as PVCi.
First sub-micron organic thin film transistors using rigid as well as flexible substrates were already fabricated applying this technique. Their transistor properties were measured and revealed good transistor behavior. Thus the new UV-NIL process is perfectly suited for organic device fabrication.

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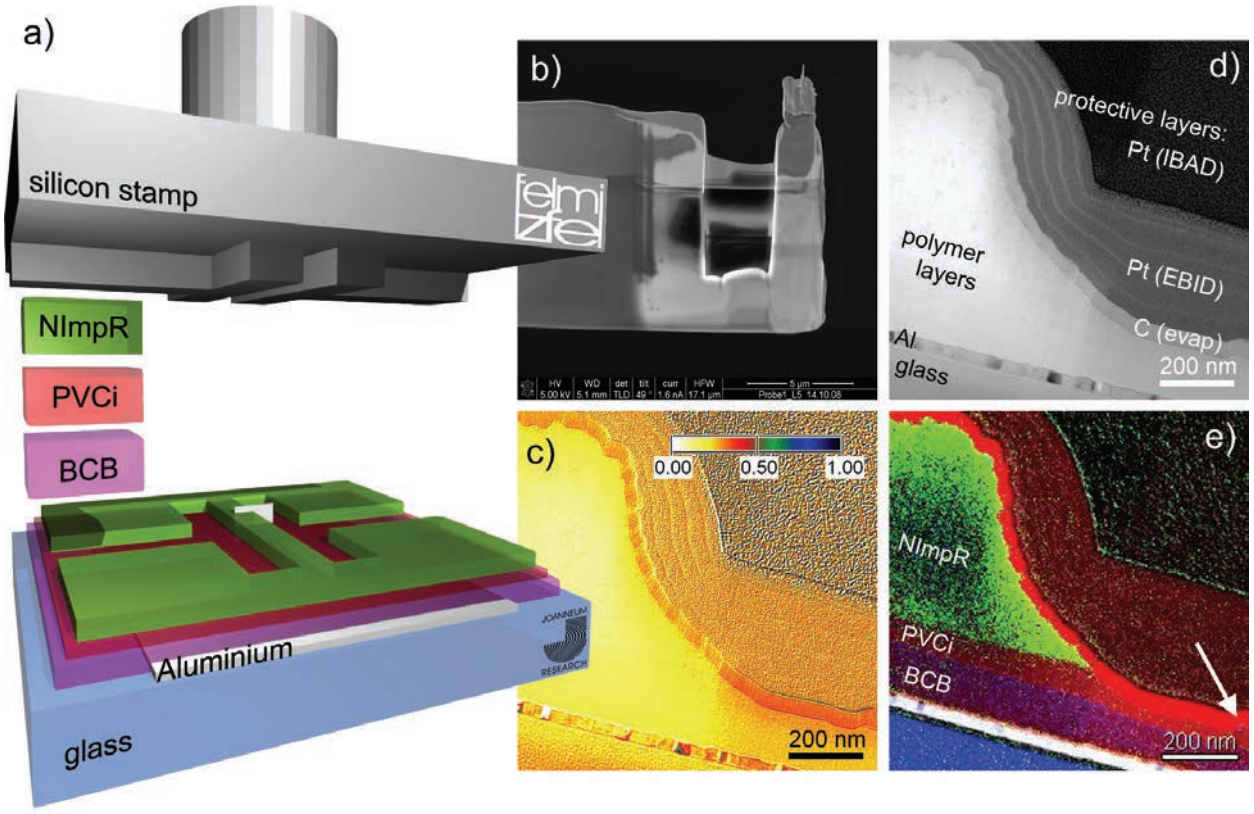

Figure 1. a) Scheme of the NIL process after the residue-free imprint step. The white plane in the center indicates the investigated area. b) FIB preparation: only the important part is thinned for further investigation to avoid bending. c) TEM relative thickness map (t/λ). d) TEM bright field image, zero-loss filtered. e) superposition of elemental maps: C…red; N…green; Si…blue; Al…white. The white arrow indicates a bump in the imprinted area, residues can be excluded.

Acknowledgements:
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Characterization, Nanostructuring and Modification of Conjugated Polymer Based Optoelectronic Devices

M. Sezen[1], H. Plank[1], B. Chernev[1], E. Fisslthaler[2], E. Tchernychova[1], W. Grogger[1], P.M. Nellen[3], S. Meier[3], E.J.W. List[2] and P. Pölt[1]

[1] Institute for Electron Microscopy, Graz University of Technology, Steyrergasse 17, A-8010 Graz, Austria
[2] Institute of Solid State Physics, Graz University of Technology, Petersgasse 16, A-8010, Graz, Austria
[3] EMPA, Swiss Federal Laboratories for Materials Testing and Research, CH-8600, Dübendorf,

The use of conjugated polymers in optoelectronic device technology is becoming increasingly attractive for industrial applications. Due to solution processability, such materials can be fabricated on flexible substrates by spin-/dip-cast processing or inkjet printing, providing a path to low-cost production [1]. Therefore they can be utilized as materials for the active medium particularly in organic light emitting devices (OLEDs) and organic field effect transistors (OFETs). Of the various soluble organic semiconductors currently in use, poly(3-hexylthiophene) (P3HT) has been studied extensively, due to its easy synthesis and relatively high charge carrier mobility. Also, highly blue emissive polyfluorenes (PF) have received remarkable attention in the recent decade as a promising class of conjugated polymers which can be used in polymer light emitting devices (PLEDs) [2].
On the other hand, electron microscopy can be considered as a comprehensive and feasible technique concerning the microstructural and chemical analysis, as well as the modification of such multilayer thin film devices. Transmission electron microscopy (TEM) enables a detailed investigation of the structures and the electronic properties of these materials, especially at the respective interfaces on a nanometer scale. Dual beam instruments, consisting of a scanning electron microscope (SEM) and a focused ion beam (FIB) column, additionally equipped with gas injection systems and micromanipulators serve as multi-functional tools both for device modification and specimen preparation for TEM. Recently, efforts have been made to use electron and ion beams for both the preparation of new and the modification of prefabricated devices based on organic semiconductors. In the frame of device modification, electron and ion beams can be used for nano-scale structuring applications. However, the irradiation of the conjugated polymers with electrons and/or ions can lead to temporary or permanent changes of their structure as the optoelectronic properties of conjugated polymers are in particular very prone to be altered by beam damage [3]. On the contrary, despite its destructive aftereffects, beam degradation can also be constructive when it is intentionally used for polymer modification, in terms of tuning the optoelectronic properties in a controlled way.
In order to understand the degradation behavior of conjugated polymers towards electron and ion beams, several experiments for different conditions of irradiation (e.g. Fig. 1a) were performed on PF and P3HT thin film samples using different microscopes such as FIB, SEM, ESEM and TEM. The evaluation of the results was based on the data acquired by Raman Spectroscopy, Electron Energy Loss Spectroscopy (EELS), Atomic Force Microscopy (AFM), Kelvin Probe Force Microscopy (KPFM), and Fluorescence Microscopy (FM). Besides, it should be remarked that the ion-beam damage formed during TEM specimen preparation by FIB, especially in the case of devices consisting of thin organic layers, should not be underestimated. The FIB cross-sections are mainly intended for imaging and microstructural analysis, as well as for atomic structure and interface analysis of the thin layers and interfaces by TEM, EELS and EFTEM (Fig. 1b). Thus, for the assessment of the damage caused by FIB, pristine and FIB processed P3HT samples were investigated by EELS. The changes in the chemical structure of the polymers were monitored at the carbon-K edge according to the dose dependent alterations and losses at the π*(C-C), σ*(C–H) and σ*(C–C) peaks.

Another emphasis in this study was put on the use of electron and ion beam degradation for the modification of the optoelectronic properties in light-emitting conjugated polymers, which allowed fine-tuning the emission color of such polymers via altering the dose of irradiation. The thin films of PF, Me-LPPP, MEH-PPV on silicon and transparent substrates were selectively irradiated using varying electron and ion doses, while photoluminescence (PL) and electroluminescence (EL) microscopy, as well as AFM and KPFM (Fig. 2a) provided supportive data for tracking the changes in the emission properties. Moreover, in terms of device modification, electron and ion beams have been used for nano-scale structuring applications, including platinum deposition to fabricate contacts and conducting paths and forming specially shaped FIB-cuts. Finally, brining many capabilities of the dual beam instrument together, a novel nano-optoelectronic device prototype was developed (Fig. 2b).

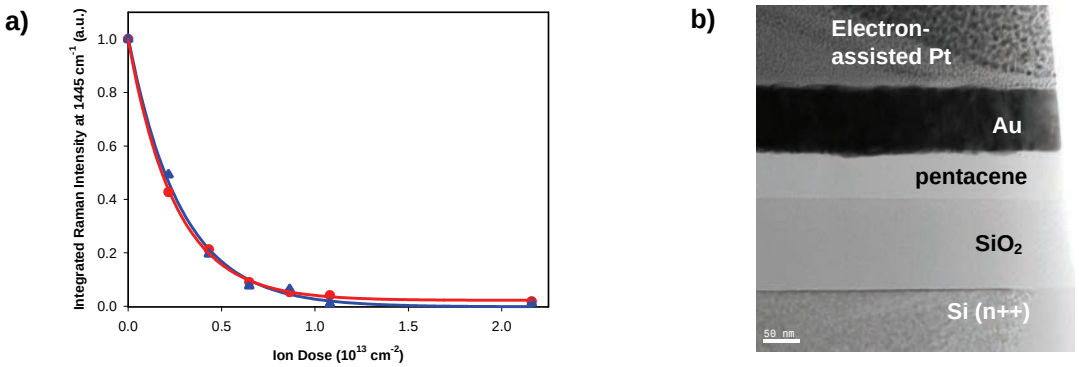

Fig. 1 a) Raman curves showing a similar irradiation behavior at room and cryo temperature of P3HT thin films towards increasing ion doses, b) TEM cross-section image of an organic thin film transistor.

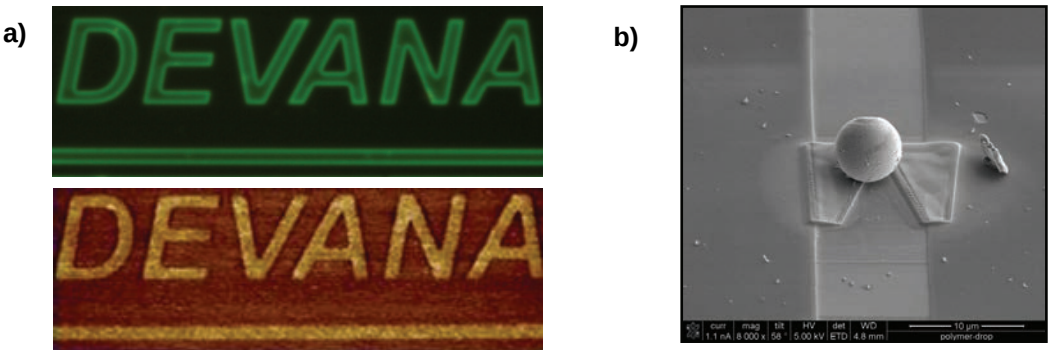

Fig. 2 a) The PL and KPFM image of an electron-beam-patterned logo on a PF thin film; the emission color was tuned from blue to green using EB irradiation, b) nano-PLED structure, fabricated by in-situ FIB prototyping.

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The authors would like to acknowledge Ullrich Scherf at University of Wuppertal, Katharina Landfester at University of Ulm and Barbara Stadlober at Johanneum Research, Weiz for the organic materials and device structures.
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A novel method for precipitates preparation using replica extraction combined with focused ion beam techniques

M. Dienstleder, H. Plank, Ch. Gspan, G. Kothleitner, F. Hofer

Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology and Graz Centre for Electron Microscopy, Steyrergasse 17, 8020 Graz, Austria

The nanoscale analysis of small precipitates via electron energy loss spectroscopy (EELS) in transmission electron microscopes (TEM) can be very complicated due to the surrounding matrix which contributes as well to the acquired data. Conventionally preparation techniques such as Ar^+ ion milling or focused ion beam (FIB) preparation are not able to remove the matrix which requires different ways for sample preparation.
An alternative technique is extraction replicas where pre-etched sample surfaces are covered with a conducting film (replica) like carbon, silicon, alumina oxide, polymer films or others [1-4]. These samples are then etched from the backside which extracts the precipitates from the matrix, while they are still fixed on the replica. However, such samples can show some drawbacks: i) replica related problems during preparation (e.g. mechanical stress) can prevent a successful extraction; ii) the chemical nature of the replica is often overlapping with the precipitates and complicates reliable EELS investigations; iii) the precipitates are often too thick and not electron transparent for TEM / EELS investigations which requires further thinning steps (e.g. FIB).

In this work we demonstrate the use of two different replica material concepts which are cast from solution and cured via UV and / or temperature. The first approach is a temporary transfer-substrate with a cross-linking polymer which withstands the following etch-step. After precipitant extraction a chemically proper metal layer is prepared and the polymer can then be removed completely which leads to a carbon free replica. The second concept is the introduction of a permanent substrate with excellent mechanical properties and a defined chemical nature with the advantage of a thick replica for an easy sample handling for post-processing. FIB technology is then used for the preparation of a plane-view lamella followed by a final thinning step to meet the requirements for EELS investigations. Both replica materials are characterized in dependence on preparation and post-treatment to find optimum process parameters. The techniques are demonstrated with chromium-carbide precipitants in steels and tested via TEM and EELS.

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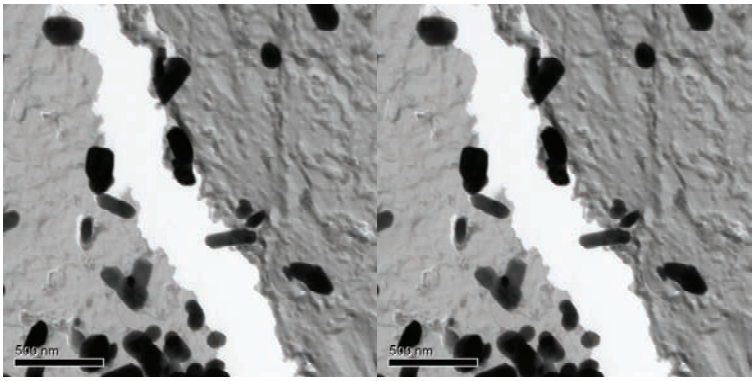

Fig. 1 TEM BF image of pre-etched precipitates covered with a carbon film.

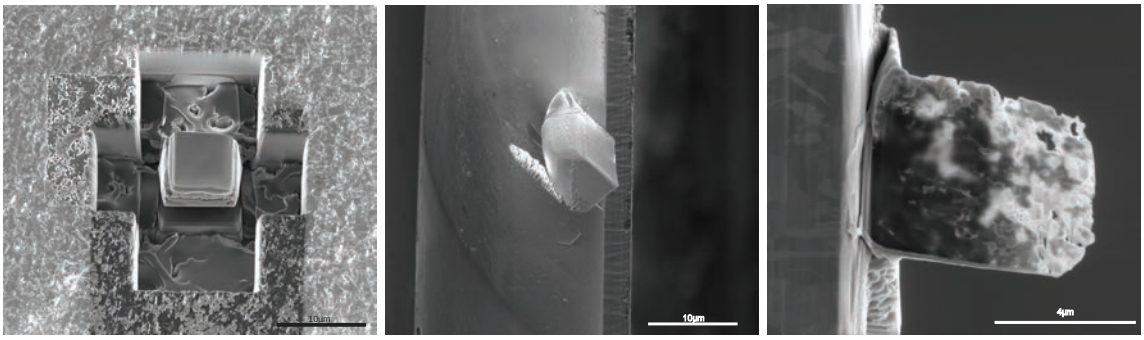

Fig. 2 a-c. SEM images of the plane view preparation in the focused ion beam instrument (FIB).

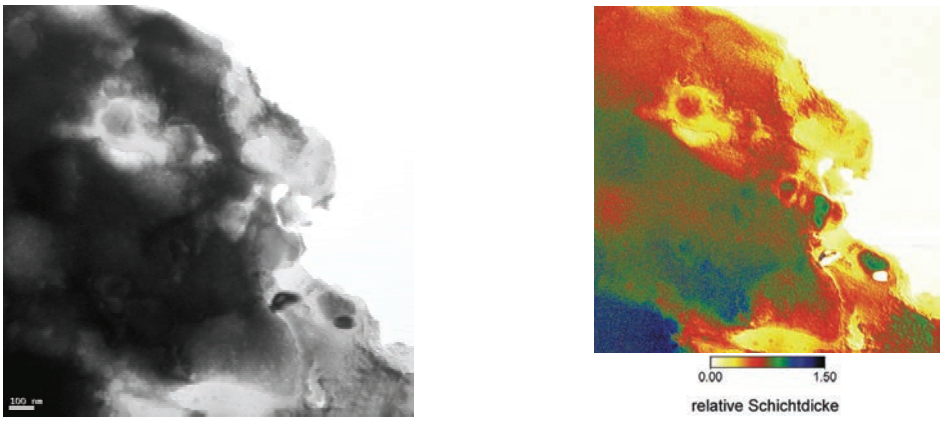

Fig. 3. TEM BF image of extraction replica with permanent polymer and EFTEM thickness map.

Correlative AFM and TEM of Soft Materials

N. Matsko[1], W. Grogger[1,2]

[1] Graz Centre for Electron Microscopy &
[2] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology, Steyrergasse 17, A-8010 Graz, Austria

The contrast in TEM-images is formed by sufficiently scattering structures that are present in the 20 to 90 nm thick volume of the section. Most of biological macromolecules (proteins, polysaccharides, nucleic acids) as well as polymer/copolymer chains mainly consist of light elements (C, H, O, N), which scatter the incident electrons rather weakly and consequently can be detected on TEM micrographs only as a grayish background. Staining of the sample can considerably improve the image contrast, but the heavy metal salts, which are used for such purpose, have a certain size and can penetrate deep into sample only when macromolecule/chain matrix is relatively disengaged. Therefore, even in stained TEM images one recognises not the whole ultrastructure, but only those cellular/polymer components which can be reached by the staining agents and which can react with the latter [1].
On the surface of the block phase investigated by AFM [2], macromolecules/polymer chains create a pronounced topographical contrast due to the relaxation of the tension inside the polymer block during or immediately after the sectioning process. AFM also detects phase shifts, which in case of biological samples can be attributed to the different density of the pure embedding resin and the copolymerised cell components surrounded by the resin [3]. In case of polymers, phase contrast usually shows chains and crystalline order, structure and distribution of the highly oriented molecules in fibers and films [4]. Therefore, the usage of AFM as a complementary microscopic technique to TEM allows the determination of the macromolecule content of biological samples embedded in epoxy resin as well as the morphology, distribution and the mechanical properties of polymer chains.
Only the investigation of exactly the same position of the specimen provides the most adequate correlative AFM/TEM analysis. Technically such data can be obtained when the block-face of the embedded sample is used for AFM imaging, while the last ultrathin section is collected, heavy metal stained (if necessary) and then used for TEM (fig. 1). The important point is that a particular organelle or macromolecule cluster is cut into two parts: one part for AFM and the other for TEM. Only such complementary pairs of images are indispensable for a serious interpretation of AFM and TEM data as well as for the discovery of new ultrastructural aspects of macromolecular/polymer chain morphology and their mechanical properties.
The complementary pair of AFM and TEM images can be used for the investigation of dynamical processes within the cell or during polymer formation/modification. At present, the description of biological ultrastructure more closely related to the living state is important as a complementary study of dynamic events in living cells by fluorescent light microscopy. In polymer science as well, many important questions concerning the structural changes accompanying melting, crystallization or glass transition are very relevant [5]. The nondestructive character of AFM and the possibility to obtain information about location, architecture and mechanical properties of macromolecules/polymer chains in a nanometer range make this technique extraordinary useful for the investigation of local changes within the sample that take place during dynamic processes. However, the identification of the area where local changes occur still requires the complementary TEM image obtained from the same area of the specimen.
In summary, the full potential of each of high resolution microscopic technique can be expressed only through the combination with other techniques, since very often the main limitation is not the technique, but the proper interpretation of the obtained results.

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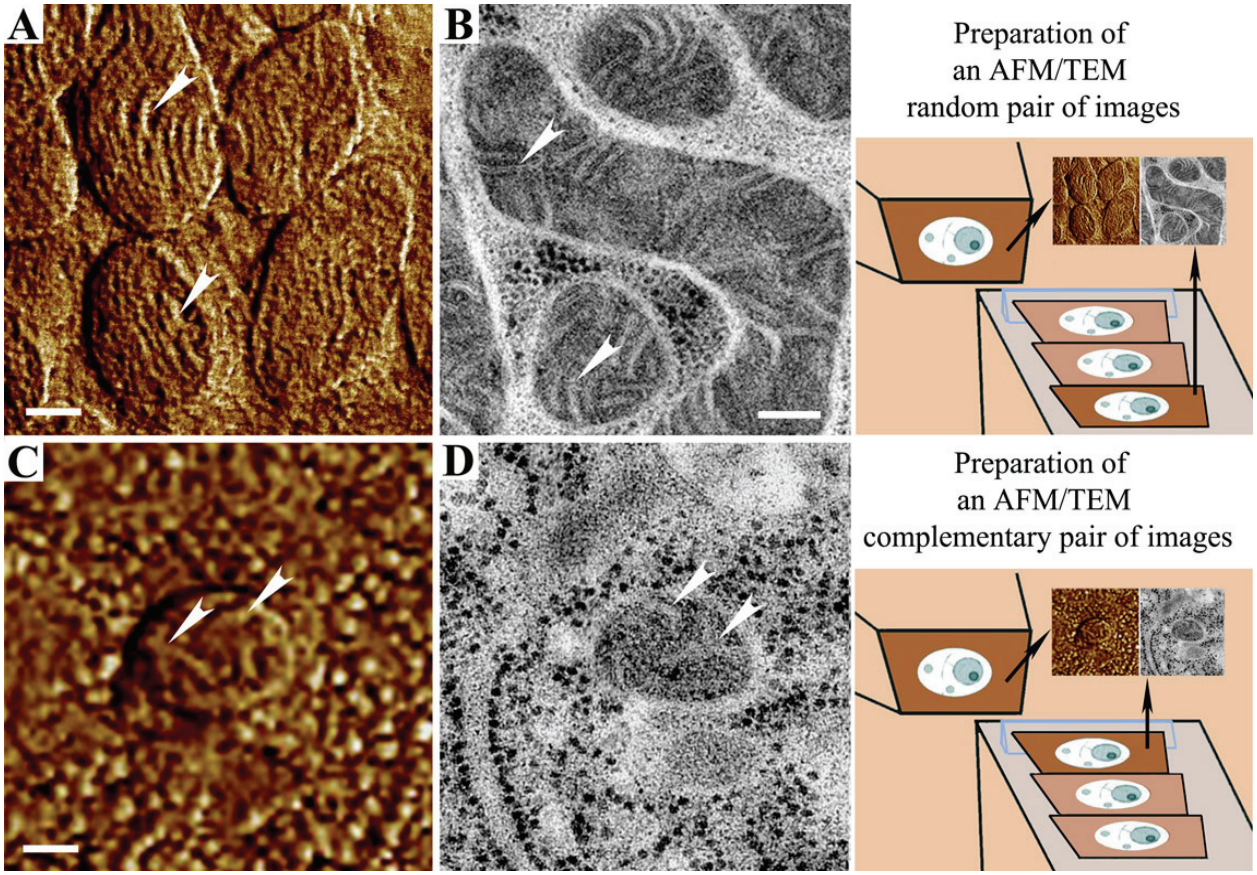

Fig.1 AFM phase (A, C) and TEM (B, D) pairs of images of HPF and epoxy FS C. elegans. (A, B) pair of images was obtained from different places of the sample; (C, D) pair of images was obtained from the same place of the sample. Scale bars: 200 nm in (A,B), and 100 nm in (C,D). Phase variation: 0 - 2 °C in (A) and 0 - 3 °C in (C).

Characterisation of Spherites in the Digestive Gland of the Snail Chilostoma (Josephinella) Lefeburianum: An Analytical Electron Microscopy Study

S. Lipovšek Delakorda[1], I. Letofsky-Papst[2], T. Novak[1], F. Hofer[2], M. Giovannelli[1], M. Šimunović[1] and M.A. Pabst[3]

[1] Department of Biology, University of Maribor, Koroška 160, Slovenia
[2] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology & Graz Centre for Electron Microscopy Graz, 8010 Graz, Austria
[3] Department for Cell Biology, Histology and Embryology, Medical University of Graz, 8010 Graz, Austria

Spherites, concentrically layered cytoplasmic inclusions, seem to be involved in detoxification processes [1] and contribute to the hardening of the exoskeleton [2, 3] in different animals.
The purpose of this study was to find out if there are seasonal-dependent changes in the structure and chemical composition of spherites in Chilostoma (Josephinella) lefeburiana (Helicidae). This middle-sized pulmonate snail, living in limestone caves, is endemic of the lowland of the Isonzo/Soča river and the adjacent Trieste karst region in Italy [4] and in the Slovene Istria [5].

The spherites were studied by a combination of TEM, EDX-spectroscopy and EFTEM before overwintering in November (a, b), in the middle of overwintering in February (c, d) and at the end of overwintering in March (e). In November, all spherites are laminated structures with electron dense and electron lucent material, containing C, O, Na, Si, P, Cl, K, Ca, Fe (a, b). In February (c) and March (e), the spherites show different morphology than in November. Their material seems to be lost during overwintering as more electron lucent “empty” layers appear and only C, O and Cl could be detected. The material was probably used in physiological processes during the non-nourishing overwintering period. In its exoskeleton (f), almost the same elements as in November spherites were found. In this study, the combined application of TEM, EFTEM and EDX-analyses provides a detailed characterization of changes in the elemental composition of spherites during ocerwintering of Ch. Lefeburiana..

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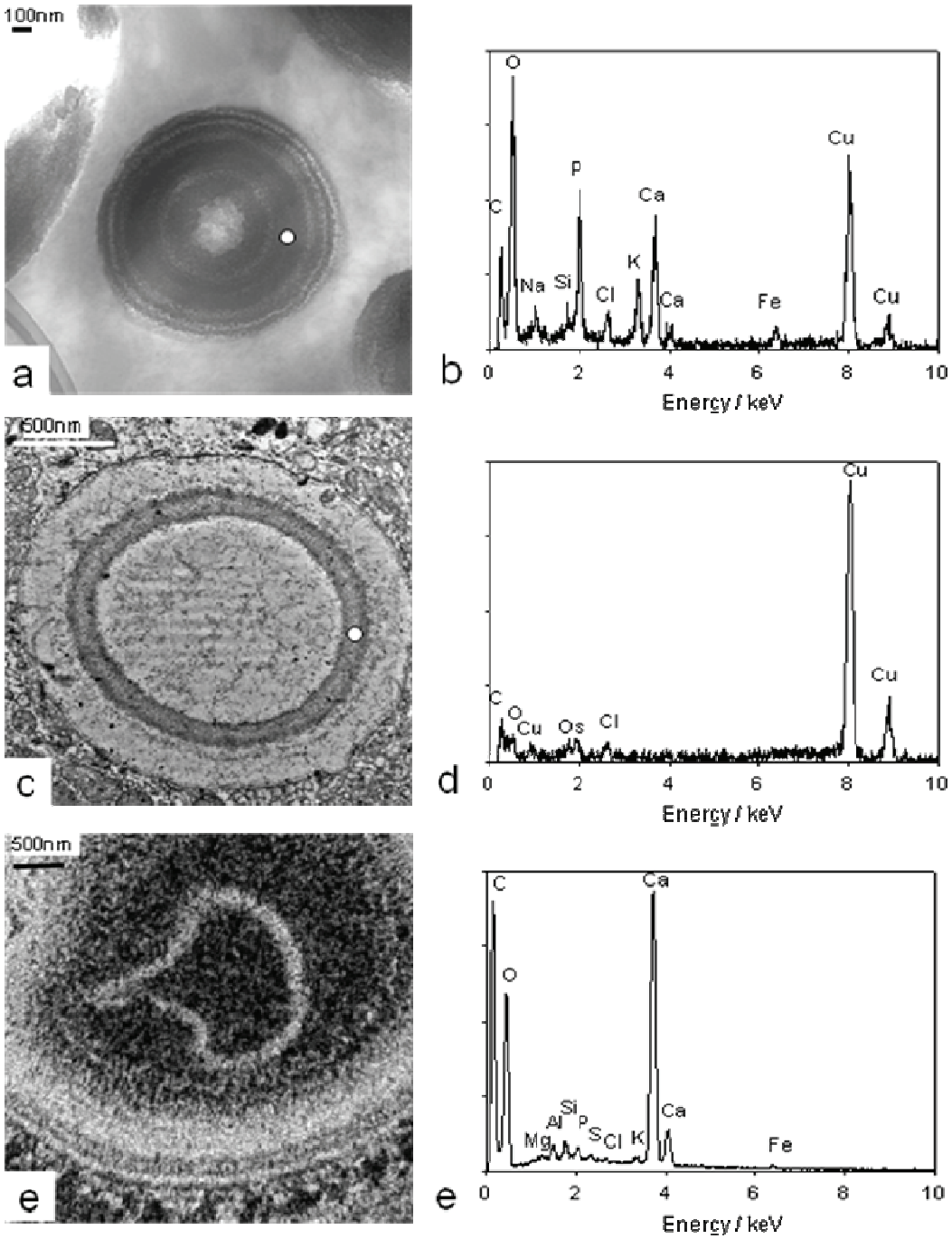

Fig.1. a) TEM bright field image of November spherite; b) EDX spectrum of November spherite (marked region in a); c) TEM bright field image of February spherite; d) EDX spectrum of February spherite (marked region in c); e) Cl-$L_{2,3}$ elemental map of March spherite; f) EDX spectrum of the exoskeleton.

Cryoelectron Microscopy of Frozen-hydrated Specimens

N. Matsko[1], W. Grogger[1], F. Hofer[1], O. Glatter[2]

[1] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology & Centre for Electron Microscopy Graz, Steyrergasse 17, A-8010 Graz, Austria
2 Institute of Chemistry, Karl-Franzens-University of Graz, Schubertstrasse 1, A-8010 Graz, Austria

Water is the most abundant component of biological material, but it is systematically excluded from conventional electron microscopy. This is because water evaporates rapidly under the vacuum conditions of an electron microscope. The development of cryoelectron microscopy of vitrified specimens has radically changed the situation during last decade [1].

The discovery of vitrification opened the door to a number of further developments which greatly facilitated cryoelectron microscopy. Simple methods were found for preparing a thin vitrified layer of aqueous specimens [2, 3]. The cutting of vitrified sections, thin enough for high resolution observation was also found possible [4, 5]. Among the various kinds of cryo specimen holders and plunge freezing machines, those which are the simplest and the easiest to manipulate, turned out to give the best results [6]. It was also observed that an optimal use of phase contrast could compensate to a large extent for the inherent low contrast of unstained vitrified specimens [7]. Furthermore, low temperature provides the possibility of rapid stopping movement in hydrated specimens without changing the structure and helps to reduce the effect of electron beam damage.

One of the basic requirements for TEM is that the specimen be very thin. This can be achieved by the technique of cryosectioning after the bulk sample has been vitrified using high pressure freezing devise. Alternatively, liquid biological suspensions can first be prepared in the form of a thin film before they are vitrified. The first method is of general use, but is very demanding. The second is simple and rapid, but limited to small (less than 200 nm) particles in liquid suspensions of not too high viscosity [1].

There are three main steps for preparing of liquid samples for cryoEM: (i) formation of the thin film of sample suspension; (ii) blotting the fluid aliquot of the specimen to a thin fluid layer prior to freezing, and (iii) rapid plunging of thin fluid layer into a suitable cryogen (see Fig. 1). An example of a cryogen that is commonly used for freezing the specimen is liquid ethane; liquid nitrogen is used to maintain the temperature of the ethane near its melting point of -183 °C [1]. With a freezing rate on the order of 1,000,000 K/sec, the fluid surrounding the specimen does not have time to form crystalline ice, which would damage the fragile sample; instead it is vitrified. Embedded within this layer of vitreous ice, the specimen is preserved in essentially its native state to near atomic resolution. Vitrification followed by cryo TEM observation is, in principle, an almost ideal preparation and observation methods. All the usual artifacts due to dehydration, staining and changing of the sample structure due to slow fixation are ruled out.

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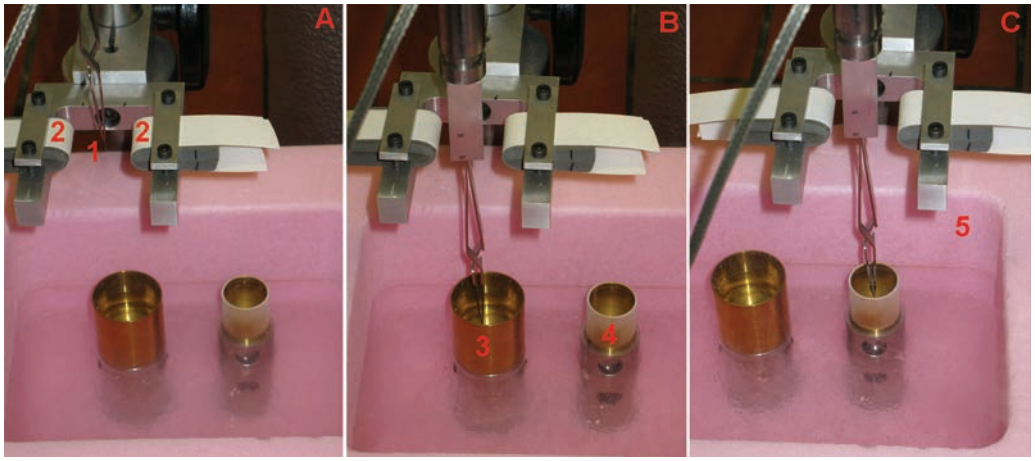

Fig. 1 Vitrification of the sample. A small aliquot of a fluid suspension is applied to the surface of a holey carbon film that is attached to the surface of a standard TEM specimen grid (A (1)). The droplet is blotted with filter paper (A (2)). After the grid is plunged into the ethane pot (B (3)), and than is transferred into liquid nitrogen pot (B (4)), the workstation (C (5)) is removed and the grid transferred to a pre-cooled cryo transfer holder for immediate viewing on the TEM.

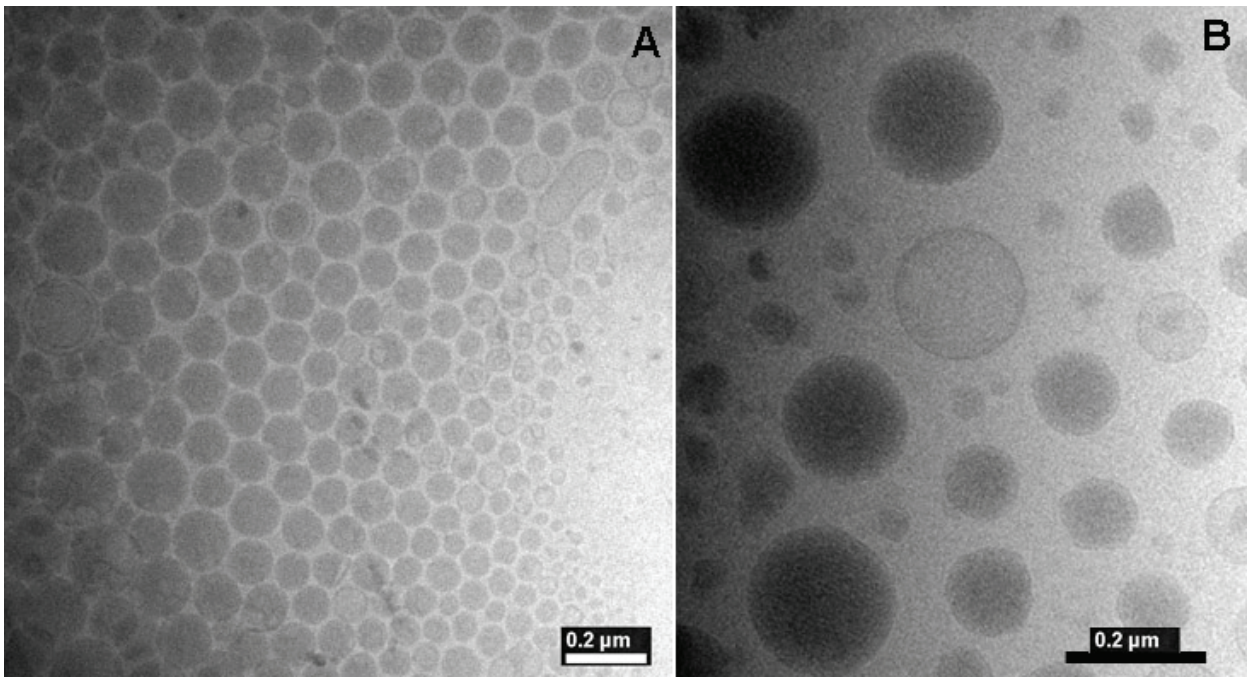

Fig. 2 (A) Cryo TEM of oil/water nanoemulsions (sample from Viktoria Klang, TU Wien) and (B) monolinolein (MLO) / water nanostructured dispersions.

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14. Author Index