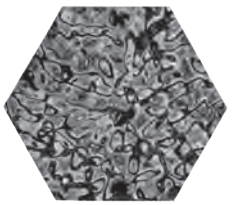
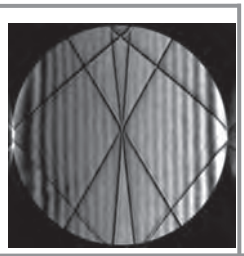
Performance Report 2005/06

Performance Report 2005/06

TU Graz
Graz University of Technology





Institute for Electron Microscopy and Fine Structure Research
Centre for Electron Microscopy Graz

AUSTRIAN COOPERATIVE RESEARCH
KOOPERATION MIT KOMPETENZ

Performance Report 2005/2006

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

Centre for Electron Microscopy Graz

www.felmi-zfe.at

Imprint

Published by

Verlag der Technischen Universität Graz
www.ub.tugraz.at/Verlag
ISBN-10: 3-902465-85-9
ISBN-13: 978-3-902465-85-9

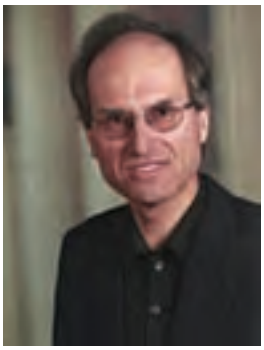
Institut für Elektronenmikroskopie und Feinstrukturforschung, Technische Universität Graz
Zentrum für Elektronenmikroskopie Graz
Steyrergasse 17,
A-8010 Graz, Austria

phone++43 316 873 8320
fax ++43 316 811 596
email sekretariat@felmi-zfe.at
web http://www.felmi-zfe.at

Printed in
A-8010 Graz, Austria

Preface

The Institute for Electron Microscopy (FELMI) is an essential part of the Faculty of Mathematical and Physical Sciences at Graz University of Technology. FELMI is an internationally visible research institute for electron microscopy and the analysis of advanced materials. The institute is very active in basic science as well as in cooperation with the local industry. Several members of FELMI are also involved in teaching for our students in physics and chemistry. The publication record of the members of FELMI is excellent and the faculty is very proud of this institute.
As the dean of the Faculty of Mathematical and Physical Sciences I wish the Institute of Electron Microscopy further successful years and I hope that the reader will enjoy this report.



O.Univ.-Prof. Dr.phil. Robert Tichy
Dean of the Faculty of Mathematical and Physical Sciences

Introduction

This biannual report contains an overview of the latest accomplishments of the Institute for Electron Microscopy (FELMI) of Graz University of Technology (TU Graz). It further includes the activities of the Centre for Electron Microscopy Graz (ZFE Graz), which is operated by the "Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung". Both institutions work in close liaison.

Over the last few years we have introduced cutting-edge instrumentation which has further increased the Centre's attractiveness and secured its international competitiveness in the field of microscopy research. However, these developments come at a price – literally: today's sophisticated tools for materials science and nanotechnology have become so expensive and complex that individual investigators are not normally able to own or adequately operate or maintain them.

This is one important reason for locating advanced microscopes in one centre which also serves other university institutes and local industry, as was recently suggested in the report "Midsize Facilities" issued by the US National Academy of Sciences. The centre has followed these principles since the early 1960s and will continue to act as a "Midsize Facility" balancing the competing purposes of basic and applied research.

The institute is now an essential player on the Austrian nanotechnology scene and it is worthwhile dedicating a section of this report to the current status and some future prospects. The institute is also increasingly engaged in teaching, e.g. in new masters and PhD courses at TU Graz, but is also developing new "life long learning" courses.

We are very grateful to all our funding agencies for their continuous support and encouragement. The scientific part of the report will certainly show evidence that all the funding provided was put to good use.

Finally, we would like to praise the enthusiasm and the dedication of our collaborators at the institute since this is indeed fundamental to our success. The key element for future progress continues to be their motivation to generate new ideas and to remain committed.

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

Contents

1. The Institute

1.1. The Institute at a Glance

The centre carries out interdisciplinary research and innovative 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 these advanced tools into practical collaboratorions 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 existing microscopy preparation and characterisation techniques and/or to develop new techniques, especially in the field of materials science. It also applies these techniques to the characterisation of all kinds of materials, providing efficient answers and solutions to materials science problems.

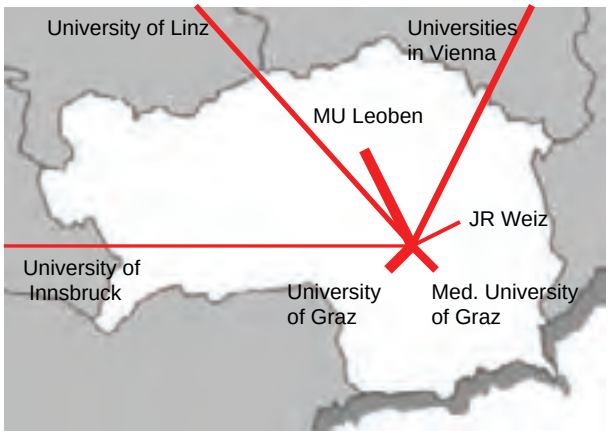
- The institute is organised in research groups focussed on a specific aspect of microscopy or materials:

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

- During 2005 and 2006 the institute cooperated with around 50 university institutes and 100

companies (mainly in Austria but also in other European countries and overseas). This technology transfer results in more than 350 visitors from other research groups and companies per year. During this period 55 graduate and PhD students (mainly from TU Graz) benefited from the support of the institute.

Active cooperations of the FELMI-ZFE with Universities and companies.



Collaborations of the institute with institutes of Austrian universities.

1.4. 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 educating our scientists and microscope operators better in order to provide state-of-the-art investigations both for university institutes and companies.

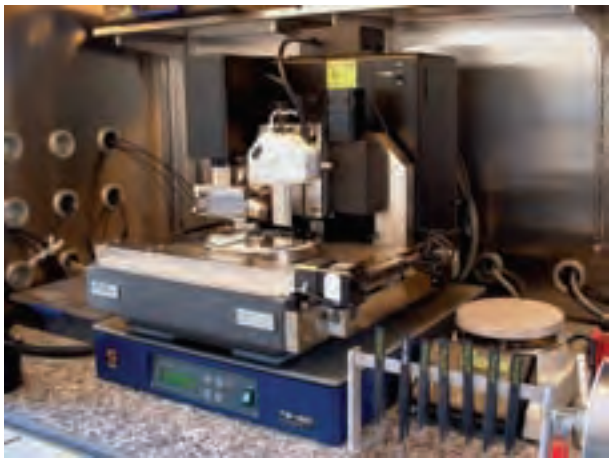
Our laboratory now includes one analytical high-resolution electron microscope, two analytical transmission electron microscopes, two environmental scanning electron microscopes, two high resolution scanning electron microscopes, one atomic force microscope as well as FTIR, Raman and advanced light microscopes. 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).

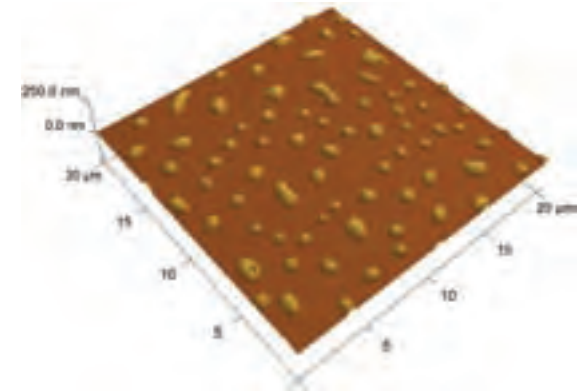
The following section provides a brief description of the main instruments introduced over the last two years:

- **Atomic force microscope (AFM)**

In 2005 we put great effort into securing the purchase of an urgently needed advanced atomic force microscope. We joined forces with the group of Prof. Dr. Emil List from the Institute for Solid State Physics (TU Graz) and finally succeeded because the Austrian Council for Research and Technology Development (RFTE) in Vienna recommended the project. After an intensive test period we decided to order a VEECO Dimension 3100 equipped with advanced scan heads. It can be used for Electrostatic Force Microscopy (EFM) and Kelvin Probe Force Microscopy (KPFM), which are comparatively new techniques for the electrical characterisation of organic semiconductor materials, blends and devices such as OLEDs, LECs, solar cells or OFETs. Both techniques are based on the implementation of the Kelvin probe principle in the Atomic Force Microscope (AFM),

which is well established in the field of modern science. The advantage of work-function determination (Kelvin probe) is thereby linked with the possibility of laterally resolved mapping (AFM), which enables a unique insight into the electronic structure of materials and electronic devices on this scale. The instrument is specifically tailored to the needs of organic semiconductor research and polymer science since it is possible to perform investigations on operating devices.





Atomic Force Microscope VEECO Dimension 3100 AFM image of polymer droplets (mLPPP/PEO) on glass substrate, spin cast preparation from THF on precleaned glass.

This option represents a crucial advantage over other *ex-situ* techniques. The EFM / KPFM *in-situ* tools thus fill a characterisation gap due to the possibility of the simultaneous observation of topography, material composition (via phase mode), and electrical behaviour.
The AFM was supplied by VEECO (USA) in April 2006 and later upgraded with a glove box for in-situ studies (Braun).

- **Low-vacuum scanning electron microscope (Environmental SEM = ESEM)**

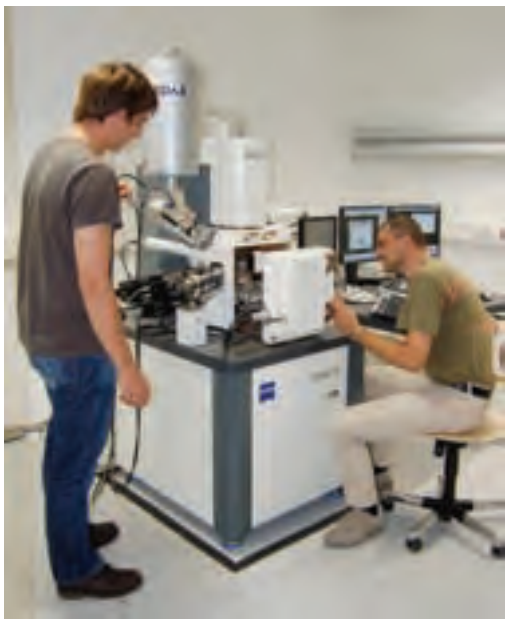
Due to the success of the FEI Quanta 600 low-vacuum scanning electron microscope, it became necessary to provide increased capacity by introducing a second ESEM.
This microscope, FEI Quanta 200, is equipped with a W cathode and enables a broad range of applications from physics and materials science to biotechnology. It can be used for the study of "wet" specimens at fairly high spatial resolution. It can also be equipped with cooling and heating stages and is therefore particularly suited for in-situ studies such as phase transitions, corrosion properties, high temperature behaviour, or drying processes. The microscope is mainly used for problem solving in the fields of physical sciences, technical chemistry, biochemistry, biology and biotechnology.
The microscope was installed in spring 2006 and is now also used for training TU Graz students. The project was launched by the "Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung".

FEI ESEM Quanta 200 with EDX-spectrometer

- **High resolution scanning electron microscope (HR-SEM)**

Recently the institute introduced a new high resolution scanning electron microscope (HR-SEM), which was installed in October 2006. This ZEISS Ultra55 is equipped with a field emission source (Schottky emitter) and a new detection system which enables simultaneous surface, compositional and crystallographic imaging.
Key advantages are the new detectors:

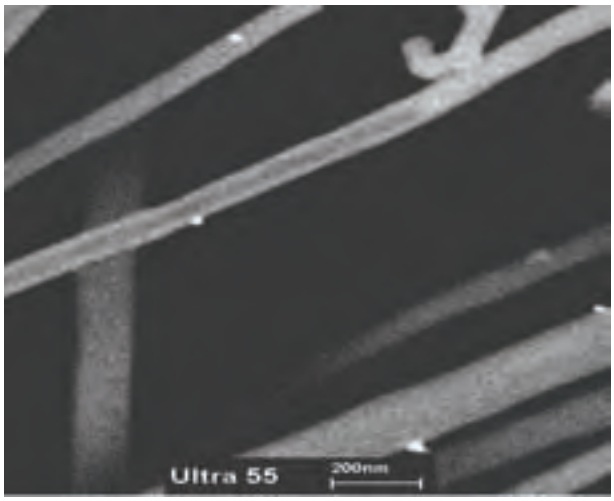
- in-lens SE (secondary electron) detector for high contrast topography imaging
- in-column ESB (energy selective backscattered electron) detector for low kV high resolution imaging with improved materials contrast (spatial resolution some nanometer with a sensitivity of 1/10 atomic number)
- integrated annular AsB angle selective backscattered) detector for compositional and crystal orientation imaging
- STEM detector for low energy transmission electron microscopy of thin films.



The new HR scanning electron microscope Zeiss Ultra 55 in the basement of Steyrergasse 17 (operated by J. Rattenberger and H. Schröttner).

The microscope is equipped with an energy-dispersive x-ray spectrometer (EDAX Genesis Si(Li) detector) for elemental microanalysis and imaging. Additionally, an electron backscattered diffraction detector (EBSD) for surface crystallography studies is attached to the microscope column.
Consequently, the microscope will be used for characterising materials at a sub-micrometer scale resolution, and will help to meet the challenges of new classes of materials and devices such as nanostructured materials, nanoparticles, semi-conductor devices and functional materials.
The project was launched by the “Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung” with financial support from the Styrian Business Promotion Agency (SFG) in Graz and the Styrian Economic Chamber (WKO) in Graz.

In a second step the microscope will be equipped with a wave-length dispersive spectrometer (WDX), which will be mainly employed for quantitative microanalysis and trace element detection in materials. The WDX system will be introduced in autumn 2007 provided that the necessary funding can be secured.



Metallic nanoparticles on carbon nanotubes recorded with backscattered electrons (EsB detector) on the Zeiss Ultra 55.

1.5. Quality Management

Development of a quality management system according to the rules of ISO 9001:2000 was started during 2004. The aim was 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.
The necessary processes were assessed and extended by the institute's first quality manager (Dr. Mario Schmied) with the help of Dr. Oliver Jöbstl (SUCCESSFACTORY, Leoben).

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

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 audit was guided by Mag. Ulrike Stürzenbecher, who has been quality manager at the institute since 2005.

The certificate confirms the high quality of our work and will help to further establish a good reputation among our cooperation partners. The institute expects to improve its standing and is now able to enter cooperations where the partners require a certified quality management system.

The institute in the building Steyrergasse 17, 2nd and 3rd floor.

1.6. The People in the Laboratory

Staff

The broad diversity of contracts and partnerships has enabled an increase in the number of employees and PhD students.

- Dipl.-Ing. Dr.techn. Christoph MITTERBAUER left the institute for a postdoctoral scholarship position at the University of California in Davis (USA). He joined FEI Company in Eindhoven (The Netherlands) in 2007.
- Mag. Dr.techn. Werner ROM was employed at the institute from 1 January 2005 to 31 July 2005. He developed and coordinated the "Styrian Nanoanalysis Handbook 2005".
- Sabrina MERTSCHNIGG joined the institute as a chemical laboratory apprentice on 1 January 2005 and now works in Dr.Kothleitner's group.
- Ferdinand HOFER was offered a C4 professorship at the Technical University Berlin (TU Berlin) in the field of nanooptics and nanoanalysis (January 2005).
- Anita ROSSMANN successfully completed her apprenticeship (chemical laboratory technician) on 22 February 2005. She has since been employed in Dr. Kothleitner's group.
- Emanuel SEIDL completed his mechanical engineering apprenticeship on 17 March 2005 and left the institute on 1 July 2005.
- Mag. Meltem SEZEN joined the institute on 1 April 2005 and works in Dr. Pölt's group.
- Mag. Ulrike STÜRZENBECHER joined the institute on 4 April 2005 and works in the administration group. She is responsible for business administration and quality management.
- Mag. Dr. Mihaela ALBU joined the institute on 18 April 2005 and works in Dr. Kothleitner's group.
- Dipl.-Ing.Dr. Mario SCHMIED left the institute on 30 April 2005 to take up a position with Treibacher Auermet GmbH, in Treibach-Althofen, Austria. Dr. Schmied was head of the scanning electron microscopy and microanalysis group and had been a member of the institute since 1999.
- Dr. Elena TCHERNYCHOVA joined the institute on 10 May 2005 and works in Dr. Grogger's group.

First barbecue party of the institute (July 2006)

- Dipl.-Ing. Katharina RIEGLER joined the institute as a doctoral student on 1 May 2005 and works in Dr. Kothleitner's group.
- Mag. Miroslava Hunkova (SCHAFFER) joined the institute on 17 May 2005. She works as a doctoral student in Ing. Schröttner's group.
- Dipl.-Ing. Siegfried KALTMANN left the institute on 31 July 2005.
- Markus SITTSAMM joined the institute on 1 September 2005 as a mechanical engineering apprentice.
- Denise ARNUSCH successfully completed her apprenticeship as a media technician and left the institute on 1 June 2006.
- Dipl.-Ing. Johannes RATTENBERGER joined the institute on 1 July 2006. He works as a doctoral student in Ing. Schröttner's group.
- Dipl.-Ing. Dr.mont. Ilse LETOFSKY-PAPST returned from maternity leave in September 2006.
- Gernot STOISER joined the institute as a media technician apprentice on 1 September 2006.
- Martin LUNELLI joined the institute as a chemical laboratory technician apprentice on 1 September 2006.
- Sanja SIMIC returned from maternity leave on 1 November 2006 and works in Dr. Pölt's group.
- Dipl.-Ing. Dr. Michael ROGERS left the institute on 31 December 2006 to take up a position at austriamicrosystems in Unterpremstätten, near Graz, Austria.
- Dipl.-Ing. Dr.techn. Karin WEWERKA has been on maternity leave since July 2004.

In memorian of members of the Advisory Board

In the last two years the following members of the Advisory Board sadly passed away. We are very grateful for the valuable support they gave us.

- Univ.-Prof. Dipl.-Ing. Dr. Harald P. FRITZER, TU Graz and
- Univ.-Prof. Dipl.-Chem. Dr. Jürgen BESENHARD, TU Graz.

Awards:

- Poster award for Dipl.-Ing. Katharina RIEGLER for her poster presentation at the "Österreichische Chemietage" conference in Leoben, September 2005.
- ACR "Cooperation Award" for ALICONA Imaging GmbH (Grambach) and ZFE Graz in the "House of Research", Vienna, 6 October 2006.
- Dipl.-Ing. Bernhard SCHAFFER completed his PhD and was awarded the title "Doctor of Technical Sciences" *sub auspiciis praesidentis*, 18 October 2006, TU Graz.
- Dr. Bernhard SCHAFFER received a special award from the Austrian Federal Ministry of Education, Science and Culture in recognition of his excellent academic achievements, Vienna, 1 December 2006.

Promotion „sub-auspiciis präsidentis": Dr. Bernhard Schaffer, Rektor Univ.-Prof. Dr. Hans Sünkel, Dr. Helmut Fischer (President of the Republic of Austria).

Organisation of scientific conferences

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

Enhanced Data Generation by Electrons
International EELS Workshop, May, 1st -5th, 2005, in Grundlsee, Austria
www.energy-loss.com

Organizing Committee:
David Muller, Ithaka, USA (program chair)
Virginie Serin, Toulouse France (program chair)
Andrew Bleloch (Cambridge, U.K.)
Nigel Browning (Berkeley, USA)
Ondrej Krivanek (Seattle, USA)
Hiroki Kurata (Kyoto, Japan)
Joachim Mayer (Aachen, Germany)
Paul Midgley (Cambridge, U.K.)
Stephen Pennycook (Oak Ridge, USA)
Ferdinand Hofer, Werner Grogger, Gerald Kothleitner (local organizer)

148 scientists from 15 nations (from USA to Japan and Australia) participated at EDGE 2005 in Grundlsee, Austria

Nanoanalytik Steiermark

- Fast Forward Workshop (SFG)
"Nanoanalytik - ein Stärkefeld der Steiermark""
June 21st, 2005 at the TU Graz, organized by Dr. Werner Rom.

- Austrian Nanoanalysis Meeting
May 11th – 12th, 2006 in Grundlsee Austria, organized in co-operation with Prof. Dr. Otto Glatter und KR Dipl.-Ing. Ulrich Santner.
www.nanoanalysis.at

The institute team at the "Kleeblatt Lauf" 2006:
Front row: Elena Tchernychova, Mrs. Schröttner, Katharina Riegler, Stefan Mitsche. Back row: Julian Wagner, Hartmuth Schröttner, Christian Gspan, Michael Rogers (from left to right).

- The "Verein zur Förderung der Elektronenmikroskopie" started to renovate the infrastructure of the institute. These activities will continue until 2009 and are strongly supported by the TU Graz. In 2006 emphasis was put on the planning and construction of the second stage of the new microscopy centre in the basement of Steyrergasse 17.
- Renovation and energy optimisation of the building facilities at Steyrergasse 17-19 based contracting agreement with Joanneum Research GmbH Graz will continue.
- The institute successfully participated in the AUSTROTEC fair in Stadthalle Graz in June 2005 (Werner Rom and Armin Zankel).
- The number of invited talks given by members of the institute increased and included not only Europe, but also America, Australia and Japan.

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 the 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 is member of the international advisory board of the Autumn School for "Electron Microscopy and Advanced Materials" at Humboldt University, Berlin, Germany.
- M. Rogers was elected as speaker of the working group on "Focused ion beam technology" of the electron microscopy societies of Austria, Germany and Switzerland (on August 29th, 2005 for a two year period).
- P. Wilhelm was elected as member of the International Advisory Board of ESIS 2006 in Lyon, France.
- F. Hofer is member of the Curricular Commission for Doctoral Studies at TU Graz.
- F. Hofer was member of the International Advisory Board of the Microscopy Conference 2005, Davos Switzerland Conference.
- F. Hofer was member of the IFSM International Advisory Board of the 16th International Microscopy Conference, Sapporo, Japan (2006).
- Staff members were active as referees for scientific institutions, 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, Chemistry of Materials, Spectrochimica Acta B, Philosophical Magazine A, Vacuum, Applied Physics Letters, Physical Review Letters, Macromolecular Symposia.

- Collaboration with research groups from the Styrian Universities in the Special Research Programme "Electroactive Materials" continued until April 2006.

G. Birnstingl at the new USV system of the institute

The institute in the news

- UNIVERSUM, „Die Küche lebt" 10/2005, pp.22-25.
- *ABSOLVENT,* „Metallbearbeitung im Elektronenmikroskop", HTL Kapfenberg, p.1.
- *Steirische Berichte* „Die Steiermark gehört zur Spitze" 5/2005, p. 24.
- *Alpenpost* „Kongress der Nanophysiker am Grundlsee", 19.5.2005.
- *Forschungsjournal der TU Graz WS O3/O4:* „RFT-Projekt: Neue Untersuchungsmethoden für Mikrosystemtechnik und Nanotechnologie"
- *www.bohmann.at* „Erstmals Nano-Handbuch für die Steiermark", 25.10.2005.
- *Austria Innovativ* „Co-operation Prize ACR", 5/2006,pp.27-29.
- *Kleine Zeitung* „Grazer Forscher schärfen den Blick" 5.12.2006, p.20
- *www.analytik-news.de* „Nanoteilchen unter Beobachtung", 11.12.2006.
- *Kronenzeitung* „In Graz stehen die besten Mikroskope", 5.12.2006, p.9.
- *CHEMIE.DE* www.chemie.de/news/d/59909/ „Steirische Forscher kombinieren Analysen-methoden neu"
- *Die Presse* „Bestes Mikroskop", 15.12.2006, p.10.

The new CCD camera (2kx2k, Gatan) mounted on the Tecnai F20; financially supported by the Wirtschaftskammer Steiermark.

1.7. Plans for the Future

Looking to the future, the centre has recently issued a three-year concept to further promote the institute as a leading Austrian research institution. The plan will increase its international visibility and the reputationof 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 2007:

- Cryo-transfer specimen holder with slow-scan CCD camera.
- Replacement of out-dated equipment, e.g. WDX and EDX spectrometer
- New computing infrastructure (1GByte network and central server)

Besides cooperation with groups from other Styrian universities established in the recently accepted ISOTEC project of the Austrian NANO Initiative, we will increase scientific exchange through cooperation with well known research institutes throughout the world.
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:

- *1st FIB-Workshop "Focused Ion Beams in Research, Science and Technology*

Organised by S.Menzel (Dresden) and M.Rogers (Graz) at the IFW Dresden, Germany, 22-23 May, 2005; in cooperation with the Austrian Society for Electron Microscopy (ASEM), Deutsche Gesellschaft für Elektronenmikroskopie (DGE), Swiss Society for Optics and Microscopy (SSOM) and Deutsche Gesellschaft für Materialkunde (DGM).

- *2nd FIB-Workshop "Focused Ion Beams in Research, Science and Technology"*

Organised by M. Rogers (Graz) and S. Menzel (Dresden) at TU Graz, 2-3 July, 2007; in cooperation with the Austrian Society for Electron Microscopy (ASEM), Deutsche Gesellschaft für Elektronenmikroskopie (DGE), Swiss Society for Optics and Microscopy (SSOM) and Deutsche Gesellschaft für Materialkunde (DGM).

- *17th European Symposium on Polymer Spectroscopy - ESOPS 2007*

Organized by P. Wilhelm (Graz), 9-12 September 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. Contributions from all fields of spectroscopy will be welcome (infrared, NIR, Raman, fluorescence, NMR, mass spectroscopy, electrical and mechanical spectroscopy). The scope of the meeting ranges from theoretical and fundamental aspects to recent advances and novel developments in characterisation and analysis of polymers.

- *„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 800 scientists mostly from the nine organising countries (Austria, Germany, Switzerland, Czech Republic, Italy, Croatia, Hungary, Slovenia, Serbia).
www.microscopy09.tugraz.at

1.8. 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
Vice Rector Hofrat Dr. Johann THEURL
Dean O.Univ.-Prof. Dr. Robert TICHY

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
- Steiermärkischer Zukunftsfonds, Graz
- Land Steiermark, Graz
- Wirtschaftskammer Steiermark, Graz
- Bundesministerium für Wirtschaft und Arbeit, Wien
- Nanotechnologie Netzwerk Steiermark

Last but not least we must particularly thank

Professor Dipl.-Ing.Dr.h.c. Helmut LIST
KR Dipl.-Ing. Ulrich SANTNER
KR 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' organization 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 resources of the university. On the other hand it allowed maintaing 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 organization 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 28 members mainly from Austria. Since the general business meeting on February 28th, 2002 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

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
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. Dr. Georg HOINKES, Karl-Franzens University of Graz
Univ.-Prof. Dipl.-Ing. Dr. Helmut CLEMENS, University of Leoben
Univ.-Prof. Dr. Hartmuth KAHLERT, Graz University of Technology
Univ.-Prof. Dr. Klaus LEDERER, University of Leoben
Univ.-Prof. Dr. Maria-Anna PABST, Medical University of Graz
Univ.-Prof. Dipl.-Ing. Dr. Franz STELZER, Graz University of Technology
Komm. Rat Dr. Theo GUMPELMAYER, Austrian Co-operative Research, Vienna
Dipl.-Ing. Dr. Armin HOLZNER, Semperit Technische Produkte, Wimpassing
Dipl.-Ing. Dr. Wolfgang NEISSL, Borealis AG, Linz
Prof. Dr. Bernhard PELZL, Joanneum Research, Graz
Min. Rat Dipl.-Ing. Dr. Stefan Kolarsky, Bundesministerium für Bildung, Wissenschaft, Vienna
Dipl.-Ing. Christian RAINER, Omya GmbH., Gummern
Vizerektor Dipl.-Ing. Dr. Johann THEURL, Graz University of Technology

Members of the "Verein zur Förderung der Elektronenmikroskopie"

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

alicona Alicona Imaging GmbH, Grambach	Montanuniversität Leoben
austriamicrosystems Austria Micro Systems AG Unterpremstätten	OMYA AG Oftringen, Schweiz
AT&S Austria Technologie & Systemtechnik Leoben	OMYA GmbH Gummern
AVL AVL List GmbH Graz	PLANSEE Plansee AG Reutte
VISHAY Vishay Bccomponents Austria GmbH	CERAM Porzellanfabrik Frauental GmbH Frauental
BÖHLER EDELSTAHL Böhler Edelstahl GmbH & Co KG Kapfenberg	SEMPERIT Semperit Technische Produkte AG Holding
BOREALIS Borealis GmbH Linz	CYTEC Surface Specialties Austria GmbH
B B Brigl & Bergmeister Papierfabrik GmbH Niklasdorf	Wattens Papierfabrik Wattens GmbH, Wattens
EPCOS EPCOS OHG Deutschlandsberg	TU Graz Technische Universität Graz
Chemson Chemson Polymer Additive AG	Constantia Constantia Packaging AG/Teich
IB STEINER Ingenieurbüro Steiner Leoben	TREIBACHER INDUSTRIE AG Treibacher Auermet GmbH Treibach-Althofen
iv Industriellenvereinigung Graz	VHI Karl-Franzens-Universität Graz
Fritz-Haber-Institut der Max-Plank-Gesellschaft	VOITH Voith Paper Service GmbH Wimpassing
Joanneum Research Graz	WKO Wirtschaftskammer Steiermark, Graz

2.2. The 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 18 full members. In 2003, they had a total of 435 full time equivalent employees and produced a turnover of 39.4 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).

Cooperation Award of ACR for Alicona Imaging and the ZFE Graz, from left to right: Dr. Ferdinand Hofer, Dr. Stefan Scherer, Dr. Manfred Prantl, Prof. Dr. Theo Gumpelmayer.

4. 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 (since April 2005)
BRUNEGGER Albert, Ing.
CHERNEV Boril, Mag. Dr.rer.nat. *
GROGGER Werner, Dipl.-Ing. Dr.techn. ao.Univ.-Prof.
INGOLIC Elisabeth, Dr.phil.
KALTMANN Siegfried Dipl.-Ing. (till August 2005)
LETOFSKY-PAPST Ilse, Dipl.-Ing. Dr.mont.
MITSCHE Stefan, Dipl.-Ing. Dr.techn.
PÖLT Peter, Dipl.-Ing. Dr.techn.
REICHMANN Angelika, Dipl.-Ing. Dr.techn. *
ROM Werner, Dipl.-Ing. Dr.techn. ** (1.1.05 – 31.7.05)
SCHAFFER Bernhard, Dipl.-Ing. Dr.techn.
SCHMIED Mario Dipl.-Ing. Dr.techn. (till April 2005)
SCHRÖTTNER Hartmuth, Ing.
TCHERNYCHOVA Elena Dr. ** (since May 2005)
WAGNER Julian, Dipl.-Ing. Dr.techn.
WEWERKA Karin, Dipl.-Ing. Dr.techn. (Maternity leave)
WILHELM Peter, Dr.phil.
ZEDLACHER Harald, Dipl.-Ing. *

PhD students

GSPAN Christian, Dipl.-Ing. *
RATTENBERGER Johannes Dipl.-Ing. * (since May 2005)
RECHBERGER Werner, Mag.Mag. *
RIEGLER Katharina Dipl.-Ing. * (since May 2005)
ROGERS Michael, Dipl.-Ing.
SEZEN Meltem Dr. ** (since April 05)
SCHAFFER Miroslava Mag. ** (since May 2005)
ZANKEL Armin, Dipl.-Ing. *

Allgemeine Mitarbeiter / General Staff

ARNUSCH Denise, Image lab-apprentice (till May 2006)
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 (since Septembre 2006)

MAYRHOFER Claudia, Ing. Chem.lab assistant *
MERTSCHNIGG Sabrina, Chem.lab-apprentice (since February 2005)
PALLER Manuel, Chem.lab assistant *
ROßMANN Anita, Chem.lab assistant *
SEIDL Emanuel, Mech. apprentice (till July 2005)
SIMIC Sanja, Microscope Operator *
SITTSAM Markus, Mech. apprentice (since Septembre 2005)
STOISER Gernot, Image lab-apprentice (since Septembre 2006)
STREUßNIG Fatima, Secretariat *
STÜRZENBECHER Ulrike Mag * (since April 2005)
WALLNER Margit, Design & image lab
WINDISCH Gerhard, Design, PC- & LAN-Admin.

* ZFE staff, ** supported by projects

Guest scientists

Prima Sarah **PRAWIRADIRAJA**, Queen Mary University of London, August-September 2005
Dr. Konstantza **LAMBRINOU**, University of Leiden, Belgium, September 2005
Dr. Saso **STURM**, Department of Nanostructured Materials, Jozef Stefan, Ljubljana, October-November 2005 and April 2006
Dr.Thomasz **MOSKALEWICZ**, AGH University of Science and Technology, Polen, November-December 2005.
Prof. Dr. David **McCOMB**, Imperial College London, U.K., March 2006.
Dr. M. David **ABAD**, Materials Science Institute of Sevilla, August-October 2006.


Mihaela Albu

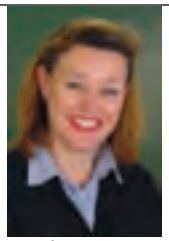

Denise Arnusch


Peter Bahr


Gerhard Birnstingl


Christian Brandl

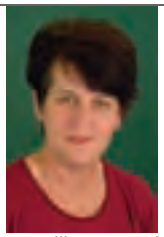
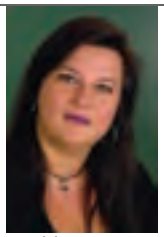
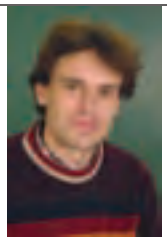
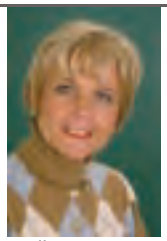
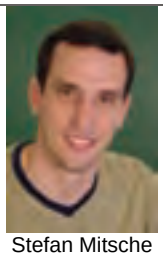
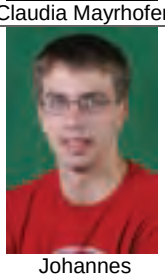
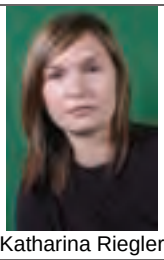
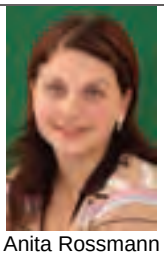
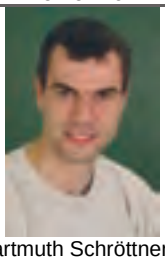

Albert Brunegger

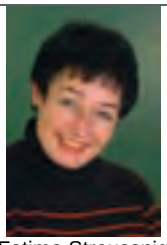

Margit Brunegger


Boril Chernev


Wolfgang Czapek


MartinaDienstleder

 Christof Elis	 Angelika Freund	 Sabine Goger	 Werner Grogger	 Christian Gspan
 Anneliese Gusmagg	 Ferdinand Hofer	 Elisabeth Ingolic	 Siegfried Kaltmann	 Gerald Kothleitner
 Elke Kranzelbinder	 Ilse Letofsky-Papst	 Martin Lunelli	 Claudia Mayrhofer	 Sabrina Mertschnigg
 Stefan Mitsche	 Manuel Paller	 Peter Pölt	 Johannes Rattenberger	 Werner Rechberger
 Angelika Reichmann	 Katharina Riegler	 Michael Rogers	 Werner Rom	 Anita Rossmann
 Bernhard Schaffer	 Miroslava Schaffer	 Mario Schmied	 Hartmuth Schröttner	 Emanuel Seidl

 Meltem Sezen	 Sanja Simic	 Markus Sittsam	 Gernot Stoiser	 Fatima Streussnig
 Ulrike Stürzenbecher	 Elena Tchernychova	 Julian Wagner	 Margit Wallner	 Karin Wewerka
 Peter Wilhelm	 Gerhard Windisch	 Armin Zankel	 Harald Zedlacher	

FELMI-ZFE booth at the NANOTEC Fair, Stadthalle, June 2005, Graz

5. Laboratory Facilities

Scanning electron microscopes (SEM)

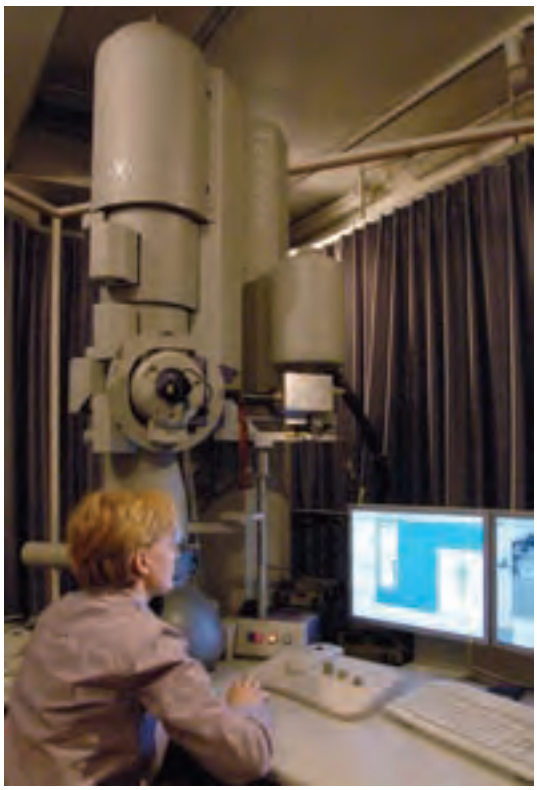
- High resolution SEM: ZEISS ULTRA 55 0,1-30kV, field emission source, with EsB detector, in-lens SE detector, STEM detector and EDAX Genisis EDX-system.
- Analytical high resolution SEM: LEO Gemini DSM986
  0.1-30 kV, field emission gun, with EDX detector Noran Voyager 3105A, with TSL EBSD detector, 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 EDX-system DX4.
- SEM-specimen stages for cryo-investigations, micro x-ray fluorescence and x-ray microscopy (developed at FELMI-ZFE).

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), EDAX Genesis EDX system, direct ion detector (CDEM).

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 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)



Transmission Electron Microscope Tecnai F20 (200 kV, FEG), FEI Company.

- Analytical TEM: FEI Tecnai 12: 120 kV, LaB_6 cathode, twin lens, and EDX Si(Li) detector with ultrathin window (EDAX), 1kX1k CCD camera
- Specimen holders: Philips double tilt holder, Philips cryo-transfer and cooling holder
  Gatan double tilt cooling holder for analytical work, low background holders, rotation and heating holders

Atomic force microscope (AFM)

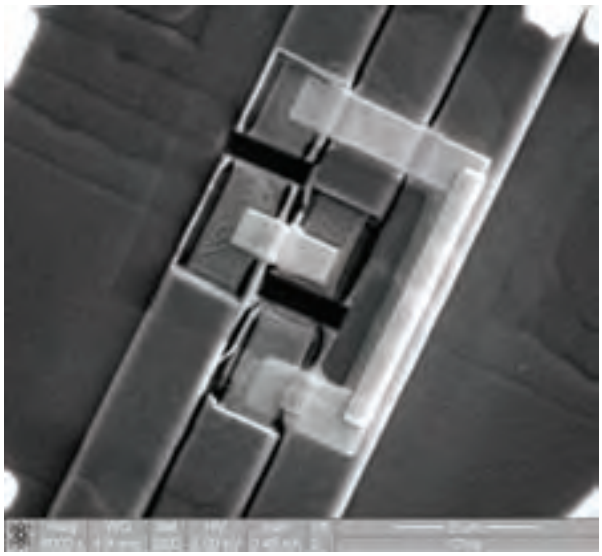
- Atomic force microscope VEECO Dimension 3100 with EFM and KPFM modes in a glove-box “Unilab MBraun 20”

Atomic force microscope VEECO Dimension 3100 in a 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)
- Stereo light microscope Leica M Z 6 for sample- preparation
- Light microscope METAVAR (Reichert)

Focused ion beam instrument FEI NANOLAB Nova 200



Nanosurgery: modification of an electronic circuit in the FIB, Milling of deep metal layers and new contacts by Pt deposition.

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-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

FELMI-ZFE calender for 2006

6. Academic Education

6.1. 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 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 GIF school at the transmission electron microscope Tecnai F20 (from left: W.Grogger, G.Kothleitner, B.Schaffer)

- 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 spectro-metry (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 condistions, 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 2006.

LIFE LONG LEARNING

6.2. Life Long Learning

FELMI staff contributes to the programme of the **University Course "Nanotechnology and Nanoanalysis"** (Degree: Master of Advanced Studies).

ULG 003: Nanoanalysis and Structural Investigations with Microscopical Methods, 2 VO (F. Hofer).

ULG 004: Micro- and Nanoanalysis in the Electron Microscope, 2 PR (W. Grogger, M. Schmied).

ULG 120: High Resolution Electron Microscopy, 2 VO (F. Hofer).

ULG 125: New Methods in Light Microscopy, 2 PR (P. Wilhelm).

ULG 126: Nanostructuring of Materials with Ion- and Electron Beams, 2 PR (P. Pölt).

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

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, semi-

conductors, 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.

M. Dienstleder demonstrates the focused ion beam instrument (FEI NANOLAB Nova 200)

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

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.

6.3. 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 250 pupils, teachers and students from other institutions visited the institute during the period 2005-06:

- 18 -19 January 2005 for physics teachers from Styria (W. Grogger).
- 16 February 2005 for pupils from the Gymnasium Ursulinen Graz (G. Kothleitner)
- 17 February 2005 for “Österreiches Gießerei-Institut”, Leoben (M. Schmied).
- 13 April 2005 for the SCA Graphic Research Board, Sweden (J. Wagner).
- 15 September 2005 for board members of the Austrian Nano Forum, (F. Hofer, in cooperation with Joanneum Research, Graz).
- 25 October 2005 for teachers from the “Pädagogische Akademie des Bundes” in Styria, (F. Hofer in cooperation with the Institute of Physics, KFU Graz).
- 9 November 2005 for new employees of the TU Graz (B. Schaffer, S. Mitsche).
- 24 November 2005 and 1 December 2005, Urania Seminar “Electron Microscopy” (W. Grogger, E. Ingolic, P. Pölt).
- 9 March 2006: for teachers from the “Pädagogische Akademie des Bundes” in Styria (W. Grogger, H. Schröttner).
- 21 March 2006: for students of the “Chemieingenieurs-Schule” in Graz (G. Kothleitner, H. Schröttner)
- 17 May 2006 for new employees of the TU Graz (B. Schaffer, S. Mitsche).
- 20 July 2006 for the Institute of Chemical Technology of the Vienna University of Technology (H. Schröttner).

- 11 September 2006 for participants of the Summer University VITA ACTIVA of the Karl Franzens University Graz (F. Hofer, S. Mitsche).
- 31 October 2006 for pupils of the BORG Deutschlandsberg (G. Kothleitner).
- 22 November 2006 for pupils of the BRG Kepler in Graz (S. Mitsche, B. Schaffer).
- 23 and 30 November 2006: Urania Seminar “Electron Microscopy” (W. Grogger, E. Ingolic, P. Pölt).

G. Kothleitner gives a demonstration for students at the transmission electron microscope CM20

Prof. Schickhofer and Prof. Steiner visit the institute

Prof. David Williams from Lehigh University (Betlehem, USA) gives a lecture in the institute.

7. Diploma and Doctoral Theses

7.1. Doctoral Theses and Diploma Theses at the FELMI

- Finished doctoral thesis:
  - Dipl.-Ing. Bernhard SCHAFFER: „Spectrum imaging with high energy resolution by energy-filtering TEM"
  - Dipl.-Ing. Stefan MITSCHE: „„Chemical and crystallographical characterization of submicron particles and thin layers in the scanning electron microscope"
- Doctoral theses in progress:
  - Dipl.-Ing. Andreas DITTMANN: "Microscopical characterization of tribological systems"
  - Dipl.-Ing. Christian GSPAN: "Electron microscopical investigation of La(Sr,Co)O_3 perowskites"
  - MMag. Werner RECHBERGER: „Characterising crystalline materials with high resolution scanning transmission electron microscopy and electron energy loss spectroscopy"
  - Dipl.-Ing. Johannes RATTENBERGER "Microscopical characterization of materials surfaces"
  - Dipl.-Ing. Katharina RIEGLER "Electron energy-loss spectrometry at high energy resolution"
  - Dipl.-Ing. Michael ROGERS: „Electron microscopical investigation of nanoparticles and functional nanostructures"
  - Mag. Miroslava SCHAFFER "3D elemental mapping with a focused ion beam instrument and x-ray spectrometry"
  - 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"
- Finished diploma thesis:
  - Katharina RIEGLER: "TEM-investigation of gold nanoparticles"
  - Johannes RATTENBERGER: "Investigation of electron-gas interaction in a low-pressure scanning electron microscope
- Master theses in progress:
  - Dipl.-Ing. Franz SCHRANK, Master Thesis
  - Dipl.-Ing. Michael ROGERS, Master Thesis

7.2. 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. Harald PLANK, Doctoral Thesis
  - Dipl.-Ing. Axel STUPNIK, Doctoral Thesis
  - Dipl.-Ing. Evelin FISSLTHALER, Doctoral Thesis
  - Dipl.-Ing. Alexander BLÜMEL, Doctoral Thesis
  - Birgit Jahn, Diploma Thesis
  - Veronika PROSCHEK, Diploma Thesis
- Institute of Experimental Physics
  - Andreas APFOLTER, Diploma Thesis
  - Dipl.-Ing. Boris WILTHAN, Doctoral Thesis
  - Julia GREINER, Diploma Thesis

- Institute for Materials Physics
  - Dipl.-Ing. Martin SAGMEISTER, Doctoral Thesis

Faculty of Chemistry, Engineering and Biotechno-logy

- Institute for Chemical Technology of Inorganic Materials
  - Dipl.-Ing. Wolfram KOHS, Doctoral Thesis
  - Dipl.-Ing. Thomas HEJZE, Doctoral Thesis
  - Dipl.-Ing. Reinhold DORNER, Doctoral Thesis
  - Dipl.-Ing. Markus THALER, Doctoral Thesis
  - Dipl.-Ing. Christiane KOREPP, Doctoral Thesis
  - Mag. Anilkumar METTU, Doctoral Thesis
  - Dipl.-Ing. Eva WALLNÖFER, Doctoral Thesis
  - Dipl.-Ing. Hosseinmardi AZARNOUSH, Doctoral Thesis
  - Dipl.-Ing. Klaus LEITNER, Doctoral Thesis
- Institute for Chemical Technology of Organic Materials
  - Dipl.-Ing. Thomas E. HAMEDINGER, Doctoral Thesis
  - Dipl.-Ing. Martin KNIENDL, Doctoral Thesis
  - Dipl.-Ing. Monika PIBER, Doctoral Thesis
  - Dipl.-Ing. Martin WEINBERGER, Doctoral Thesis
- Institute of Ressource Efficient and Sustainable Systems
  - Dipl.-Ing. Thomas BRUNNER, Doctoral Thesis
  - Dipl.-Ing. Markus JÖLLER, Doctoral Thesis
- Institute of Chemical Apparatus Design, Particle Technology and Combustion
  - Dipl.-Ing. Günter GRONALD, Doctoral Thesis
  - Dipl.-Ing. Mahmood SALEEM, Doctoral Thesis
  - Dipl.-Ing. Peter PUCHER, Doctoral Thesis
  - Dipl.-Ing. Stephan SACHER, Doctoral Thesis
- Institute for Biochemistry
  - Mag. Tibor CZABANY, Doctoral Thesis
- Institute of Biotechnology
  - M.Sc. Malene THOMSEN, Doctoral Thesis
- Institute for Paper, Pulp and Fibre Technology
  - Dipl.-Ing. Ulrich HIRN, Doctoral Thesis
- Institute of Environmental Biotechnology
  - Andrea HASEMANN, Diploma Thesis
  - Dipl.-Ing. Max SCHRÖDER, Doctoral Thesis
  - Dipl.-Ing. Anita EBERL, Doctoral Thesis

Faculty of Civil Engineering

- Institute of Applied Geosciences
  - Mag. Barbara LEGENSTEIN, Doctoral Thesis

Faculty of Mechanical Engineering and Economics

- Institute of Material Science, Welding and Forming
  - Dipl.-Ing. Bernhard SONDEREGGER, Doctoral Thesis
  - Gabriel BAMBERGER, Diploma Thesis
  - Gunter FIGNER, Diploma Thesis
  - Rene RADIS, Diploma Thesis
  - Dipl.-Ing. Peter MAYR, Doctoral Thesis

Mag. Francisca MENDEZ MARTIN, Doctoral Thesis
Dipl.-Ing. Andreas SCHWEIGER, Doctoral Thesis

- Institute of Precision Engineering
  Mag. Oskana TITARENKO; Doctoral Thesis
- Institute of Thermal Engineering
  Dipl.-Ing. Peter HASELBACHER, Doctoral Thesis

Karl-Franzens-University of Graz

- Institute of Zoology
  Bauer ANDEAS, Diploma Thesis
  Julia BAUMANN, Diploma Thesis
- Institute of Pharmaceutical Science
  Mag. Karin WERNIG, Doctoral Thesis
- Institute for Experimental Physics
  Mag. Petra GRANITZER, Doctoral Thesis
- Institute of Geosciences
  Mag. Christof BAUER, Doctoral Thesis

Medical University of Graz

- Department of Dentistry and Maxillofacial Surgery
  Irma DRAGOJVIC, Diploma Thesis
- Institute of Pathophysiology and Immunology
  Mag. Nicole SCHEER, Doctoral Thesis

University of Leoben

- Department of General, Analytical and Physical Chemistry
  Christian GROGER, Diploma Thesis
  Markus UNTERLERCHNER, Diploma Thesis
- Department of Physical Metallurgy and Materials Testing
  Dipl.-Ing. Robert FRANZ, Doctoral Thesis

Vienna University of Technology

- Institut für Straßenbau und Straßenerhaltung
  Dipl.-Ing. Klaus STANGL, Doctoral Thesis

Martin Luther University Halle, Germany

Dipl.-Ing. Michael NASE

University of Maribor, Slovenia

Iztok SVAB, Diploma Thesis
Saska LIPOVSEK, Diploma Thesis

8. Main Research Areas of the Workgroups

The institute's main research activities are devoted to developing new microscopical 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, biological tissue.

In 2005 and 2006 research funds were allocated by the FFF, the FWF within the Special Research program "Electroactive Materials", the "Steier-märkischer Zukunftsfonds" and the Austrian Nanoinitiative.

8.1. Scanning Electron Microscopy and Related Physical Methods

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 (pp. 86, 112, 115).
Its extension, the low vacuum or environmental SEM (ESEM) also allows the analysis of fluids or fluid containing specimens. Furthermore, it eliminates the need for electrically coating insulating materials with a thin conductive layer (p. 82). Yet the ESEM is not only an instrument for the analysis of materials, but also some a kind of micro-reactor. In the ESEM the chamber gas can be chosen arbitrarily and the relative humidity (if water vapour is used as chamber gas) and the specimen temperature can be varied continuously. Thus a variety of processes can be controlled, while simultaneously documenting the changes at the specimen with high magnification and great depth of focus on video (p. 80).

- Recrystallisation of Ni-base alloys by EBSD (electron backscatter diffraction) in the SEM (cooperation with the University of Leoben):
The project focuses on investigating the recrystallisation behaviour of these materials during hot forming depending on temperature and comparing the results with simulations. Another focus is on both the interaction between precipitates and the formation of recrystallisation nuclei and the impact of the precipitates on the growth of the recrystallised grains (p. 84).

- 3D-Analysis in the SEM
(cooperations: FEI Company, Eindhoven, Gatan GmbH, München and Alicona Imaging, Grambach)
The investigation and analysis of three-dimensional (3D) structures is of growing importance in a number of scientific fields. Many microscopic techniques already deliver 3D information, however, new methods are mandatory for site specific 3D analysis. Here, we follow three main directions:
1. We developed 3D-elemental mapping by combining a focused ion beam instrument with x-ray spectroscopy. This new technique is especially powerful for the 3D-characterisation of inorganic materials (p. 66).
2. Serial sectioning in an ESEM provides unique information about the phase distribution of organic matter (polymers, biomaterials, composites, p.68).
3. The 3D topography of material surfaces can be quantitativaley studied in the SEM with the MEX^{TM} software of Alicona Imaging.
These 3D 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.

- Fabrication, manipulation and characterisation of nano- and meso-sized structures (cooperation: ISOTEC project; Institute of Physics, University of Graz):
Regularly arranged magnetic nanowires in porous silicon are fabricated by etching and filled with magnetic materials like Ni (p.72). Future applications of such structures might be sensors and spintronics. In addition, optoelectronic devices on a polymeric basis will be modified and new structures created using a dual beam device (combination of a SEM and an ion gun).

- In-situ experiments in ESEM (cooperation with Borealis GmbH; Lenzing AG; Gatan):

The fracture behaviour of both polymers and textile fibres is investigated using a tensile stage mounted in the specimen chamber of the ESEM. This combination allows the fracture progress at the fracture tip to be observed at high magnification. The structures formed are strongly dependent on the microstructure of the materials (p.80).

The internal structure of the specimens can be elucidated by serial sectioning with an ultra-microtome directly installed in the ESEM and simultaneous image recording. This method permits high resolution tomography of soft materials (p.68). We were the first to apply this new method for solving materials science problems. This work is performed in cooperation with Gatan company (Pleasanton, USA).

Another research effort concentrates on the in-situ study of hot corrosion of materials. Continuous video control at high magnification facilitates observation of the onset of corrosion, the formation of structures and the changes they go through during the corrosion progress.

8.2. Analytical Transmission Electron Microscopy and High Resolution Electron Microscopy

In the past two years, FELMI-ZFE has established accessories and add-ons in the area of analytical TEM, which greatly extended the range of methods and materials for sophisticated analysis. These developments were partially driven by our industry and academic partners due to their demands for advanced investigations.

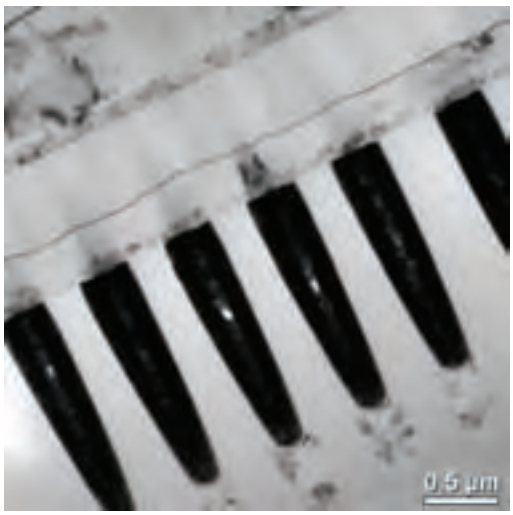
- Most importantly, the employment of a WIEN filter monochromator, incorporated in the illumination system of our field-emission TEM, improved the system energy resolution down to almost XAS level resolution of 0.2 eV. Valence electron and core-loss spectroscopy benefited directly from this improved energy resolution, opening up new possibilities for a more accurate measurement of band gaps and optical properties via the dielectric function (p.88). A more detailed analysis of the electronic structure and of bonding effects in materials has also become feasible by looking at the feature-rich near edge structures of core-loss edges. In addition, the installation of a new larger-format CCD camera in the imaging spectrometer now allows the recording of increased field-of-views at given magnifications with better digitisation depth, larger energy ranges for a specified spectrum dispersion, yet at much higher read-out speeds. The scanning performance of the (S)TEM was also augmented by attaching a second, more versatile scan unit to the microscope that now represents an integrated platform for both EFTEM, EELS and EDXS analysis. Particular progress in the area of nanocharacterisation and nanoanalysis at FELM-ZFE was made on the following types of materials and methods:

- Steels and alloys are important structural materials with a variety of industrial applications such as aircraft, chemical industry and power plants due to their mechanical properties, corrosion resistance and simple fabrication process. Although these materials have been subject to a significant amount of research to gain insight into the phase chemistry and microstructure, many fundamental questions still remain unanswered regarding the nature of the solid state chemical reactions occurring during the heat treatment of the steels, the mechanisms by which alloy additions affect the kinetics of these processes and the effect of impurities on properties. For example the ability to view precipitation or segregation at grain boundaries while simultaneously deriving chemical information make EELS and EFTEM particularly useful tools for this class of materials (p.98).

One of the problems in precipitation analysis is the influence of the matrix. Recently, the extraction replica technique has been successfully re-introduced into our specimen preparation portfolio, allowing a full chemical classification of nanometer-sized secondary carbides and nitrides by means of EELS. This analysis was made possible with the introduction of sophisticated signal deconvolution methods (multiple linear least squares fitting) assisted by advanced spectrum simulations for unambiguous phase determination.

- EFTEM studies of semiconductor device materials, interconnects, insulators and device packaging materials are gaining increasing

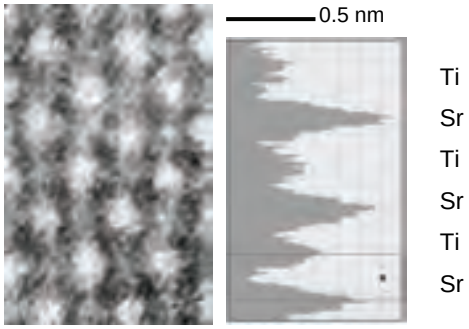
importance due to the ever shrinking size of today's devices. The process of manufacturing semiconductors typically consists of more than a hundred steps (including thermal oxidation, masking, etching, doping, metallisation, passivation etc.), during which hundreds of copies of an integrated circuit are formed on a single silicon wafer. Generally, the process involves the creation of several patterned layers on and into the substrate, ultimately forming electrically active regions in and on the semiconductor wafer surface.



TEM image of a thin cross-section of a semiconductor circuit, FIB preparation.

Due to its superior spatial and elemental resolution, EFTEM imaging can provide elemental maps of these nanometer thick layers of different elements or determine interface contamination and semiconductor defects. However, the presence of light elements in such devices and their spectral proximity create problems for the separation of their elemental signals. The acquisition of a comprehensive image data set, covering not only three but many filtered images, and the processing of multi-channel information provided a pathway for us to extract elemental and electronic information more reliably. In addition, special steps in dual-beam FIB preparation enabled us to access site-specific plan-view TEM lamellae at a thickness of less than 50 nm.

• Multilayer systems, consisting of many alternating metal (-oxide,-nitride…) layers with a thickness of only a few nanometers, frequently show unusual physical phenomena and display interesting novel properties. The distances in the multilayers and thicknesses of the adjacent layers can be selected to be comparable with the lengths of physical interactions such as the magnetic interaction length, superconducting coherence length or the electron mean free path. In magnetic multilayers for example, novel properties such as magnetic anisotropy and giant magneto-resistance can be obtained, which make these layers serious candidates for magnetic recording media and also for reading devices for magnetic tapes or disks. The beneficial properties are critically dependent on the layer chemistry, its smoothness, sharpness and structure, something that was efficiently investigated by high-annular angular dark-field scanning TEM in combination with EELS. HAADF imaging offers atomic-level resolution as well as being sensitive to chemical composition variations and compatible with different spectroscopic techniques such as EELS and energy-dispersive x-ray spectroscopy (EDXS). Spectrum imaging and line scans yielded quantitative information on diffusion profiles and gradient maps (p.94).



HR-STEM image of $SrTiO_3$ crystal in [001] direction recorded with the HAADF detector in the Tecnai F20.

Our unique equipment was essential for the investigation of this class of specimens: The Wien filter monochromator in combination with the upgraded energy filter enabled us to acquire energy loss data with superior energy resolution. Using this high quality EELS data we were able to

detect very small spectral changes such as shifts in the plasmon energies of just a fraction of an eV (p.96).

• Ceramic materials offer the potential for major improvements in component design for a wide range of applications. No other class of materials has such a wide range of electrical properties as ceramics. They can be excellent insulators or display superconducting behavior; some of them have "tunable" properties that vary predictably with composition or grain boundary chemistry. The combination of high strength-to-weight ratio, relatively inert behavior in aggressive environments, high hardness and wear resistance, as well as their ability to withstand high temperatures, also offer unique mechanical characteristics. Most of these properties, however, are determined or can be greatly influenced by the materials structure and chemical composition so that structural or chemical changes at the nanometer level can have a strong effect on their behavior.
Analytical techniques in combination with high resolution TEM were used to detect and quantify such changes and thus helped us to identify critical flaws and facilitate more accurate predictions of the failure behaviour of ceramics. In particular we used the interplay between atomically resolved TEM images and electron diffraction patterns, which resulted in valuable crystallographic information and in some cases allowed a precise structure determination of the specimen (p.100).

8.3. Polymer Microscopy and FTIR and Raman Microscopy

• The coupling of optical microscopy with vibrational spectroscopy (Infrared and Raman) enables the chemical characterisation of samples or domains as small as 10 µm (IR) or 1 µm (Raman). These techniques are applied 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).

The introduction of the focal plane array detector (FPA), which is mounted on the Bruker Hyperion 3000 IR microscope, required an intensive training and development effort. It is possible to record spectral images with lateral and infrared information in three dimensions. The FPA software enables the automatic acquisition of the spectral images within 5 to 20 minutes, but the detailed analysis of these images can be difficult and time consuming. The quality of this kind of data reduction can be considerably improved by means of chemometric methods (e.g. principle component analysis) thus enabling even quantitative analysis of the chemical constituents of a material. Since the FPA detector only works at low temperatures (liquid nitrogen) and could be harmed by frequent cooling and warming cycles, it was necessary to develop an automatic procedure which allows continuous refilling with liquid nitrogen and control via internet access.
Infrared chemical imaging was successfully introduced in order to solve advanced materials science problems, e.g. to study the composition of polymer blends and their internal interfaces, the distribution of fillers in paper (carbonates and silicates) and the distribution of active ingredients in pharmaceutical tablets.
Furthermore, the IR-polarisators in the FT-IR microscope allow the study of local orientation and crystallinity of polymers and polymer composites. Raman and IR microspectrometry are also used within the ISOTEC project of the Austrian Nano-initiative to investigate organic thin films and organic optoelectronic devices (p.70).

FT-IR microscope with FPA detector

9. Projects at the FELMI-ZFE

- "Electron microscopical characterization of electroactive, nanostructured materials", Project F00923 within the Special Research Program "Electroactive Materials" supported by FWF, Vienna, 1.5.1999–30.4. 2006.
- PROKIS-Project "Kompetenzerweiterung ZFE Graz" arranged by ACR and supported by Federal Ministry for Economic Affairs and Labour (BMWA), Vienna, 1.1.2003 – 31.12. 2006.
- „Nanoanalytical characterization of multilayered biomaterials", Wissenschaftlich-Technische Zusammenarbeit Austria-Poland, ÖAD Project 9/03, 1.1.2004 – 31.12.2005.
- „Energy-filtered TEM and high resolution STEM of nanoparticles and interfaces in materials", Wissenschaftlich-Technisches Ab-kommen Austria-Slovenia, ÖAD Project SI-A19/0405, 1.1.2004-31.12.2005.
- Multimethodenanalytik von Nanoteilchen und Nanoteilchenverbunden", supported by "Steiermärkischer Zukunftsfonds", in co-operation with University of Leoben and Karl-Franzens University Graz, 1.7.2003 – 30.10. 2006.
- "Das Bruchverhalten von Polymeren – In situ Untersuchungen im ESEM", supported by Federal Ministry for Education, Science and Culture (BMBWK), Vienna, 1.3.2005 – 30.6. 2006.
- "Nanoanalysis and Nanostructuring for Organic Optoelectronic Devices" within the ISOTEC-Project (FWF and Austrian Nanotechnology Initiative, Vienna) 1.3.2005 – 28.2.2009.
- Installation of the Network "Nanoanalysis" in Styria, supported by Joanneum Research, Graz, the Government of Styria and the Federal Ministry for Economic Affairs and Labour (BMWA), 1.10.2004 – 31.12.2006.
- „Korrosionsschutz von Magnesium-Gussbau-teilen für die Automobilaußenanwendung" COATING in cooperation with OFI Wien and ÖGI Leoben), supported by Federal Ministry for Education, Science and Culture (BMBWK), Vienna, 1.1.2006 – 31.12.2006.
- „Hochauflösungs-Rasterelektronenmikroskop" supported by Steirische Wirtschaftsförderungs GmbH (SFG) im Auftrag des Landes Steiermark, 1.9.2005-31.3.2007.
- „Mikroskopische Modifizierung von Halbleiter-bauelementen", supported by Federal Ministry for Education, Science and Culture (BMBWK), Vienna, 1.7.2006 – 30.6.2007
- "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.

10. Presentations at the Institute

22.3.2005: **Dr. Crispin HETHERINGTON** (University of Oxford, U.K.): „New Developments in Ultra-High Resolution Electron Microscopy, Including Aberration Corrected HREM“

21.6.2005: **Prof.Dr. Gerhard DEHM** (Erich Schmid Institut f. Materialwissenschaften und MU Leoben, Austria): “Mikro- und Nanodimensionen: neue Einblicke in die Werkstoffmechanik“

15.7.2005: **Dr. Erkan DEMIRCI** (Gebeze Institute of Technology, Turkey): „Coating ITO Thin Films on Glass Substrates by Sol-gel Technique“

9.8.2005: **Dr. Dieter FISCHER** (Institut für Polymerforschung Dresden, Germany): „Investigation of the orientation in composite fibres of polycarbonate with multiwalled carbon nanotubes by RAMAN microscopy“

12.10.2005: **Dr. Ludek FRANK** (Academy of Sciences, Brno, Czech Republic): “Very low energy SEM in materials science“

14.10.2005: **Dr. Saso STURM** (Department of Nanostructured Materials, Institute Jozef Stefan, Ljubljana): „HRTEM and HAADF-STEM analysis of planar faults in $SrTi0_3$“

25.11.2005: **Prof. Dr. Gerhard DEHM** (Erich-Schmid-Institut der Akademie der Wissenschaften, Leoben, Austria): “Hügel, Risse und Versetzungen: Mikroskopische Einblicke in Defektmechanismen“

31.1.2006: **Prof. Dr. LICHTE** (Institut of Structure Physics Triebenberg Laboratory, Technical University Dresden, Germany) „Electron holography: basics of electron waves and application to materials science“

3.3.2006: **Dr. Tung PHAM** (Borealis GmbH, Linz, Austria): “Interfacial strengthening of high-impact polypropylene-compounds by reactive modification“

7.4.2006: **Prof .Dr. Wolfgang JÄGER** (Christian-Albrechts-Universität Kiel, Germany): „Hochauflösende Elektronenmikroskopie von Nanomaterialien und Grenzflächen“

2.6.2006: **Ph.D. Bodil HOLST** (Institut für Experimentalphysik, TU Graz, Austria): „Helium-Mikroskopie“

20.6.2006: **Dr. Masashi WATANABE** (Department of Materials Science & Engineering, Lehigh University, USA): “Atomic-Level Characterization of Materials by Spherical Aberration Corrected Analytical Electron Microscopy“

23.6.2006: **Dr. Klaus-Jochen EICHHORN** (Institut für Polymerforschung, Dresden, Germany): „Schalten“ von thermoreversiblen Hydrogelschichten: In situ-Untersuchungen mittels ellipsometrischer Techniken“

24.10.2006: **Prof. Dr. Ute KAISER** (Central Facility of Electron Microscopy, University of Ulm, Germany): „Prospects of Transmission Electron Microscopy in Nano Science“

3.11.2006: **Prof.Dr. David B. WILLIAMS** (Department of Materials Science & Engineering, Lehigh University, Betlehem, USA): “Grain Boundary Brittle Failure; New Electron Microscopy Observations of an Old Problem”

15.11.2006: **Dr. Nadezda B. MATSKO** (Elektronenmikroskopie-Zentrum der ETH Zürich, Schweiz): “Combination of Atomic Force Microscope with the Ultramicrotome”

24.11.2006: **Prof. Dr. Horst BISCHOF** (Institut für Maschinelles Sehen und Darstellen, TU Graz, Austria): “Neue Entwicklungen in der Bildverarbeitung“

Installation of the high resolution SEM Zeis-Ultra 55 in the basement of the building Steyrergasse 17. The 2nd construction phase of the new microscopy centre (2006-07) was planned by Gerhard Birnstingl.

11. Publications of Institute Staff

11.1. Publications 2005

J. Loos, X. Yang, M.M. Koetse, J. Sweelssen, H.F.M. Schoo, S.C. Veenstra, W. Grogger, G. Kothleitner, F. Hofer
„Morphology determination of functional poly[2-methoxy-5-(3,7-dimethyloctyloxy)-1,4-phenylenevinylene]/poly[oxa-1,4-phenylene-1,2-(1-cyanovinylene)-2-methoxy,5-(3,7 dimethylocty-loxy)-1,4-phenylene-1,2-(2-cyanovinylene)-1,4-phenylene] blends“, Journal of Applied Polymer Science 97/3, (2005) 1001-1007.

T. Hejze, B.R. Gollas, R.K. Sauerbrey, M. Schmied, F. Hofer, J. O. Besenhard
“Preparation of Pd-coated polymer electrolyte membranes and their application in direct methanol fuel cells”, Journal of Power Sources 140/1 (2005) 21-27.

J. Cutrona, N. Bonnet, M. Herbin, F. Hofer
„Advances in the segmentation of multi-component microanalytical images”, Ultramicroscopy 103/2 (2005) 141-152.

K. Kimoto, G. Kothleitner, W. Grogger, Y. Matsui, F. Hofer
“Advantages of a monochromator for bandgap measurements using electron-loss spectroscopy”, Micron 36/2 (2005) 185-189.

G. Kothleitner, M. Rogers, A. Berendes, W. Bock, B. O. Kolbesen
“A combined SNMS and EFTEM/EELS study on focused ion beam prepared vanadium nitride thin films”, Applied Surface Science 252 (2005) 66-76.

M. Rogers, G. Kothleitner, A. Berendes, W. Bock, B.O.Kolbesen
„Focused ion beam preparation and EFTEM/EELS studies on vanadium nitride thin films“, Praktische Metallographie 42/4 (2005) 172-187.

S. Mitsche, P. Poelt, M. Schmied, T. Brunner, M. Jöller, I. Obernberger
“Automated particle analysis of aerosols fomed during biomass combustion by SEM/EDXS”, In: I. Obernberger, T. Brunner (eds.), Aerosols in Biomass Combustion; Series "Thermal Biomass Utilization" 6 (2005) 133-144.

W. Grogger, M. Varela, R. Ristau, B. Schaffer, F. Hofer, K. M. Krishnan
„Energy-filtering transmission electron microscopy on the nanometer length scale”, Journal of Electron Spectroscopy and Related Phenomena 143 (2005) 139-147.

V. Hacker, E. Wallnöfer, W. Baumgartner, T. Schaffer, J. O. Besenhard, H. Schröttner, M. Schmied
“Carbon nanofiber-based active layers for fuel cell cathodes - preparation and characterization”, Electrochemistry Communications 7/4 (2005) 377-382.

E. Ernst, J. Reußner, P. Poelt, E. Ingolic
“Surface stability of polypropylene compounds and paint adhesion”, Journal of Applied Polymer Science 97 (2005) 797-805.

H. Leitner, P. Staron, H. Clemens, S. Marsoner, P. Warbichler
“Analysis of the precipitation behaviour in a high-speed steel by means of small-angle neutron scattering”, Materials Science and Engineering A 398 (2005) 323-331.

D.R.G. Mitchell, B. Schaffer
“Scripting-customised microscopy tools for Digital Micrograph™”, Ultramicroscopy, 103 (2005) 319-332.

F. Hofer, B. Schaffer, W. Grogger, G. Kothleitner
„New developments in energy-filtering transmission electron microscopy”, Microscopy & Microanalysis 2005, Honolulu (Hawaii, USA), Microscopy Society of America 11(SO2) (2005) 48-49.

H. Müllner, G. Deutsch, E. Leitner, E. Ingolic, G. Daum
"YEH2/YLR020c encodes a novel steryl ester hydrolase of the yeast Saccharomyces cerevisiae", The Journal of Biological Chemistry 280/14 (2005) 13321-13328.

W.H. Binder, M.J. Kunz, E. Ingolic
"Supramolecular poly(ether ketone)-polyisobutylene pseudo-block copolymers", Journal of Polymer Science A 42 (2005) 162-172.

M. Abu Rous, E. Ingolic, K. C. Schuster
"Revelation of the pore structure of Lyocell and other cellulosics applying fluorescence and electron microscopy", Proceed. 5th World Textile Conference AUTEX 2005, Portoroz, Slovenia, pp. 64--67.

A. Zankel, P. Pölt, E. Ingolic, M. Gahleitner, C. Grein
"The fracture behaviour of polymers - in situ investigations in the ESEM", G.I.T. Imaging & Microscopy 1 (2005) 16-18.

W. Rom (editor)
"Handbuch der Nanoanalytik Steiermark 2005", Graz University of Technology, Graz (2005).

P. Wilhelm, B. Chernev, P. Pölt
"Experimental testing of lateral and depth resolution in imaging methods", Proceedings 16th European Symposium on Polymer Spectroscopy (ESOPS-16), Kerkrade, The Netherlands (2005), p. 45.

B. Chernev, P. Wilhelm
"Vibrational spectroscopy and spectral imaging techniques - special applications in the polymer sciences", Program 7th Austrian Polymer Meeting, Graz, Austria, (2005) p. 100.

P. Wilhelm, B. Chernev, P. Pölt
"A picture tells more than 4096 spectra about your polymer (spectral imaging pushing the limits)", Program 7th Austrian Polymer Meeting, Graz, Austria, (2005) p. 45.

P. Wilhelm, B. Chernev, P. Pölt
„Polymer spectral imaging: basics and applications: in: in: Wissenschaftliche Beiträge „11. Österreichische Chemietage", University of Leoben (2005) p. VO53.

B. Chernev, P. Wilhelm
„Spectral imaging techniques – a mighty tool for materials science", in: Wissenschaftliche Beiträge „11. Österreichische Chemietage", University of Leoben (2005) p. PO28.

K. Riegler, M. Rogers, B. Schaffer
„Gold nanoparticles-sysnthesis, characterization and influence of various additives on their shappe and size", in: Wissenschaftliche Beiträge „11. Österreichische Chemietage", University of Leoben (2005) p. PO37.

H. Ditlbacher, A. Hohenau, D. Wagner, U. Kreibig, M. Rogers, F. Hofer, F. R. Aussenegg, J. R. Krenn
„Silver nanowires as surface plasmon resonators", Physical Review Letters, 95 (2005) 257-403.

C. Sommitsch, M. Walter, S. Kleber, P. Pölt, S. Mitsche
"On the determination of the recrystallised fraction during hot forming", Steel Grips 3 (2005) 287-293.

M. Balog, P. Šajgalík, F. Hofer, P. Warbichler, K. Fröhlich, O. Vávra, J. Janega and J.-L. Huang
"Electrically conductive SiC–$(Nb,Ti)_{ss}$–$(Nb,Ti)C_{ss}$ cermet", Journal of the European Ceramic Society, Band 26 1259-1266.

F. Hofer, E. Ingolic, P. Pölt
"Advances in electron microscopy of polymers and organic functional materials", Program 7th, Austrian Polymer Meeting, Graz, Austria, (2005) p. 44.

J. Rez, R. Melichar, P. Pölt, S. Mitsche
"Calcite twinning lamellae stress analysis using OIM (EBSD) data", Geological Research in Moravia and Silesia in the year 2004, Brno (2005) pp. 111-114.

M. Gahleitner, B. Kretzschmar, G. Van Vliet, J. Devaux, D. Pospiech, K. Bernreitner, E. Ingolic
"Rheology / morphology interactions in polypropylene/polyamide-6 nanocomposites", Rheologica Acta, 45 (2005) 322-330.

M. Gahleitner, B. Kretzschmar, D. Pospiech, E. Ingolic, N. Reichelt, K. Bernreitner
"Morphology and mechanical properties of polypropylene-polyamide-6 nanocomposites from a two-step melt compounding process", Journal of Applied Polymer Science 100 (2005) 283-291.

G. Gronald, G. Staudinger, H. Schroettner
"Experimental investigation on agglomeration in a gas cyclone", Proceedings of the "11th Workshop on Two-Phase Flow Predictions", Merseburg, April 5-8, 2005.

V. Šrot, M. Rogers, M. Gec
"TEM sample preparation of sphalerite crystals", Proceedings 7th Multinational Congress on Microscopy, 26-30 June 2005, Portorož, Slovenia, pp. 381-382.

A. Zankel, P. Pölt, E. Ingolic, M. Gahleitner, C. Grein
„The fracture behaviour of polymers - in situ investigations in the ESEM", G.I.T. Laboratory Journal Europe 3 (2005) 30-31.

C. Sommitsch, M. Walter, S. Kleber, P. Pölt, S. Mitsche
„Über die Bestimmung des rekristallisierten Anteils bei der Warmumformung", Tagungsband Verformungskundliches Kolloquium, Planneralm, Austria (Ed. B. Buchmayr) (2005) pp.113-126.

M. Schmied, H. Schröttner, J. Wagner
„ESEM in-situ heating stage experiments on the sintering behaviour of green-body ceramics", in: Book of Abstracts, 9th Europ. Workshop on Modern Developments and Applications in Microbeam Analysis (2005), Florence, Italy, p.341.

P. Parameswaran, I. Papst, F. Hofer, W. Grogger, V.S. Raghunathan
"Application of electron energy-loss spectroscopy and energy-filtering transmission electron microscopy for microchemical studies in 2.25Cr-1Mo steel", Journal of Materials Science & Technology, 21 (2005) 361-366.

F.P. Wenzl, P. Pölt, A. Haase, S. Patil, U. Scherf, G. Leising
"Microstructure tailoring of conjugated polymer-electrolyte blends for light-emitting electrochemical cells", Solid State Ionics 176 (2005) 1747-1751.

S. Müllegger, S. Mitsche, P. Pölt, K. Hänel, A. Birkner, C. Wöll, A. Winkler
"Substrate structure dependence of the growth modes of *p*-quaterphenly thin films on gold", Thin Solid Films 484 (2005) 408-414.

W. Grogger, B. Schaffer, M. Rogers, G. Kothleitner, F. Hofer
"STEM- and EFTEM-Analysis of Nanomaterials at High Spatial and High Energy Resolution", Microscopy & Microanalysis 2005, Honolulu (Hawaii, USA), Microscopy Society of America 11(SO2) (2005) 1436-1437.

T. Bončina. A. Križman, S. Spaić, B. Markoli, I. Anžel, W. Grogger, F. Zupanič
"TEM preparation of quasicrystal-containing alloys using FIB", Proceedings 7th Multinational Congress on Microscopy, June 2005, Portorož, Slovenia, pp.379-380.

B. Schaffer, W. Grogger, F. Hofer, G. Kothleitner
"New developments in energy-filtering transmission electron microscopy", Proceedings 7th Multinational Congress on Microscopy, June 2005, Portorož, Slovenia, pp. 51-54.

M. Schmied, H. Schroettner, J. Wagner
"ESEM - the ultimate in-situ observation tool at high and low temperatures", Microscopy &.Microanalysis 2005, Honolulu (Hawaii, USA), Microscopy Society of America 11(SO2) (2005) 412-413.

C. Mitterbauer, W. Grogger, G. Kothleitner, R. Spehr, P. Tiemeijer, F. Hofer
"0.60 eV energy resolution in EELS using a thermionic LaB_6-cathode", Microscopy &.Microanalysis 2005, Honolulu (Hawaii, USA), Microscopy Society of America 11(SO2) (2005) 712-713.

G. Kothleitner, B. Schaffer, W. Grogger, F. Hofer
„EELS spectrum imaging: the next steps", Microscopy &.Microanalysis 11(SO2) (2005) 34-35.

P. Wilhelm, B. Chernev, P. Pölt
"Experimental testing of lateral and depth resolution in imaging methods", Macromolecular Symposia 230 (2005) 105-109

T. Pabel, G. Geier, H. Rockenschaub, M. Hopfinger, G. Kothleitner
„Beschleunigung der Auslagerungsvorgänge der Druckgusslegierung", Druckgusspraxis 4 (2005) 137-146.

M. Rogers, G. Kothleitner, B. Schaffer, W. Waldhauser
„A comprehensive EELS-EFTEM study of FIB-prepared samples of nitride and oxide thin films", in: Book of Abstracts "Enhanced Data Generated by Electrons – EDGE 2005", Grundlsee, Austria, May 1^{st} – 5^{th}, 2005, p. 48.

M. Pabst, I. Letofsky-Papst, E. Bock, K. Spindler, S. Guillen, F. Hofer
„Bacterial spores in the skin of a 1000 year old Peruvian mummy", in: Proceedings Microscopy Conference 2005 "Dreiländertagung für Elektronenmikroskopie" Davos, Switzerland, p.145.

M. Pabst, I. Letofsky-Papst, P. Wilhelm, E. Bock, K. Spindler, S. Guillen, F. Hofer
„Preservation of the dermis of a 1000-year old mummy from Chiribaya Alta, Peru", in: Prceddings Microscopy Conference 2005 "Dreiländertagung für Elektronenmikroskopie" Davos, Switzerland, p.147.

C. Gspan, W. Grogger, I. Letofsky-Papst, F. Hofer, E. Bucher, W. Sitte
„Superstructure and Domains in $La_{0.4}Sr_{0.6}CoO_{2.71}$", in: Proceedings Microscopy Conference 2005 "Dreiländertagung für Elektronenmikroskopie" Davos, Switzerland, p. 343.

M. Rogers, B. Schaffer, G. Kothleitner
"Behaviour of EBID Pt/C nanowires under mechanical stress", in: Proceedings Microscopy Conference 2005 "Dreiländertagung für Elektronenmikroskopie" Davos, Switzerland, p. 281.

C. Schuster, M. Abu-Rous, U. Schädel, P. Hosemann, C.Teichert, E. Ingolic, T. Bechtold
"Characterisation of cellulose fibres by modern imaging methods: Fibrillar and pore structure in the dry and wet state", in: Japanese-European Workshop on Cellulose a. Functional Polysaccharides Manchester (2005) U.K. , p.240.

H. Schröttner, M. Schmied, S. Scherer
„Comparison of 3D surface reconstruction data obtained by conventional SE; ESEM and an infinite focus microscope", Book of Abstracts, 9^{th} Europ. Workshop on Modern Developments and Applications in Microbeam Analysis (2005), Florence, Italy, p.339.

M. Stöger-Pollach, S. Lazar, B. Schaffer, P. Schattschneider, H. Zandbergen
„Cerenkov-losses in valence EELS", Book of Abstracts "Enhanced Data Generated by Electrons – EDGE 2005", Grundlsee, Austria, May 1^{st} – 5^{th}, 2005, p. 74.

G. Völksch, R. Harizanova, S. Mitsche, P. Pölt, C. Rüssel
„EBSD investigations of high iron oxide containing glass ceramics", Proc. Europ. Workshop on Modern Developments and Applications in Microbeam Analysis (2005), Florence, Italy, p.282.

B. Schaffer, W. Grogger, G. Kothleitner, F. Hofer
„Development of advanced EFTEM-SI methods and first applications",Book of Abstracts "Enhanced Data Generated by Electrons – EDGE 2005", Grundlsee, Austria, May 1^{st} – 5^{th}, 2005, p. 11.

F. Hofer
"Elektronenmikroskopie an den Grenzen örtlicher und energetischer Auflösung" in: Wissenschaftliche Beiträge „11. Österreichische Chemietage", Montanuniversität Leoben (2005) p. PV3.

W. Rechberger, G. Kothleitner, F. Hofer
„Energy resolution and spatial resolution on a monochromated (S)TEM", in: Proceedings Microscopy Conference 2005 "Dreiländertagung für Elektronenmikroskopie" Davos, Switzerland, p. 82.

F. Hofer, W. Grogger, B. Schaffer
„Energy-filtering TEM in semiconductor research“, in: Proceedings Microscopy Conference 2005 “Dreiländertagung für Elektronenmikroskopie” Davos, Switzerland, p.323.

J. Wagner, H. Schröttner, A. Reichmann, S. Mitsche, M. Schmied
„ESEM in-situ heating stage experiments on the sintering behaviour of green-body ceramics“, in: Proceedings Microscopy Conference 2005 “Dreiländertagung für Elektronenmikroskopie” Davos, Switzerland, p.356.

W. Grogger, G. Kothleitner, B. Schaffer, F. Hofer
„Exploring the resolution limits with a monochromated TEM“, Proceed. Xth Int.Conf. on Frontiers of Electron Microscopy in Materials Science, Kasteel Vaals Broek, The Netherlands (2005) p. F2.

P. Granitzer, K. Rumpf, P. Pölt, A. Reichmann, S. Surnev, H. Krenn
„Fabrication of bimodal ferromagnetic nanosystem in an etched silicon structure and ist magnetic and magneto-optic characterisation”, in: Mat. Res. Soc. Symp. Proceedings, vol.872 (2005) J13.13.

K. Riegler, M. Rogers, B. Schaffer, G. Kothleitner, F. Hofer
„Gold nanopartciles – synthesis, characterisation and influence of various additives on their shape and size“, in: Proceedings Microscopy Conference 2005 “Dreiländertagung für Elektronen-mikroskopie” Davos, Switzerland, p.259.

B. Schaffer, W. Grogger, G. Kothleitner
„“High energy resolution spectrum imaging by energy-filtering TEM“, in: Proceedings Microscopy Conference 2005 “Dreiländertagung für Elektronenmikroskopie” Davos, Switzerland, p. 84.

A. Zankel, E. Stabentheiner, M. Schmied, P. Pölt
„in-situ tensile testing in the ESEM – a versatile method for polymer and life sciences“, in: “, Book of Abstracts, Europ. Workshop on Modern Developments and Applications in Microbeam Analysis (2005), Florence, Italy, p. 368.

A. Reichmann, S. Mitsche, J. Obenholzner, P. Pölt
„Investigation of volcanic particles by SEM-EDS“, in: Book of Abstracts, 9th Europ. Workshop on Modern Developments and Applications in Microbeam Analysis (2005), Florence, Italy, p. 330.

P. Granitzer, K. Rumpf, P. Pölt, A. Reichmann, H. Krenn
„Micromagnetic and structural investigations on a ferromagnetic nanocomposite sytem in porous silicon“, in: Tagungsprogramm 55th Jahrestagung der Österr. Physikalischen Gesellschaft, Wien (2005), p. 50.

F. Hofer, W. Grogger, G. Kothleitner
„New developments in energy-filtering transmission electron microscopy”, in: Abstracts XIIth Int. Conf. Electron Microscopy of Solids, (2005), p. 33.

T. Moskalewicz, W. Grogger, A. Czryska-Filemonowicz
“Microstructural Characterisation of nitrided Timetal 834”, in: Abstracts XIIth Int. Conf. Electron Microscopy of Solids, (2005), p.111.

M. Dienstleder, M. Rogers, G. Kothleitner, F. Hofer, B. Kolbesen
„Optimisation of the FIB milling conditions for RTP-processed niobium and tantalum thin films on silicon substrates“, in: Abstracts Microscopy Conference 2005 “Dreiländertagung für Elektronenmikroskopie” Davos, Switzerland, p. 90.

P. Pölt
“Poymers and SEM-EDX –results or damage”, in: Book of Abstracts, 9th Europ. Workshop on Modern Developments and Applications in Microbeam Analysis (2005), Florence, Italy, p. 325.

C.Mitterbauer, W. Grogger, P. Wilhartitz, F. Hofer
„The formation of molecular nitrogen in chromium nitrides monitored by EELS“, Proceedings 16th International Plansee Seminar 2005, (eds. G. Kneringer, P. Rödhammer, H. Wildner) Plansee, Reutte, Austria, vol.1 (2005), pp.951-957.

11.2. Publications 2006

C. Mitterbauer, W. Grogger, P. Wilhartitz, F. Hofer
"Electron-irradiation damage in chromium nitrides and chromium oxynitride thin films", Micron 37 (2006) 385-388.

M. Balog, P. Sajgalik, F. Hofer, P. Warbichler, K. Fröhlich, O. Vavra, J. Janega, J.-L. Huang
"Electrically conductive SiC–(Nb,Ti)ss–(Nb,Ti)Css cermet", Journal of the European Ceramic Society, 26 (2006) 1259-1266.

B. Schaffer, G. Kothleitner, W. Grogger
„EFTEM spectrum imaging at high-energy resolution", Ultramicroscopy 106 (2006) 1129-1138.

B. Chernev, P. Wilhelm
"Vibrational spectroscopy and spectral imaging techniques - special applications in the polymer science", Chemical Monthly 137 (2006) 963-967.

M. Gahleitner, B. Kretzschmar, D. Pospiech, E. Ingolic, N. Reichelt, K. Bernreitner
"Morphology and mechanical properties of polypropylene/polyamide 6 nanocomposites prepared by a two-step melt-compounding process", Journal of Applied Polymer Science 100 (2006) 283-291.

S. Mitsche, C. Sommitsch, P. Pölt
"Quantitative metallography: electron backscatter diffraction in a FEGSEM", G.I.T. Imaging & Microscopy 1 (2006) 38-39.

T. Haber, M. Oehzelt, A. Andreev, A. Thierry, H. Sitter, D.-M. Smilgies, B. Schaffer, W. Grogger, R. Resel
"Single crystalline nature of para-sexiphenyl crystallites grown on KCl(100)", Journal of Nanoscience and Nanotechnology 6 (2006) 698-703.

P. Poelt, C. Sommitsch, S. Mitsche, M. Walter
"Dynamic recrystallization of Ni-base alloys - experimental results and comparisons with simulations", Materials Science & Engineering, A420 (2006) 306-314.

P. Pölt, A. Zankel, M. Gahleitner, C. Grein, E. Ingolic
"The fracture of polymers - tensile tests in the environmental scanning electron microscope", DYFP 2006 (13th Int. Conference on Deformation, Yield and Fracture of Polymers), Book of Abstracts (2006) pp. 331-334.

T. Moskalewicz, W. Grogger, A. Czyrska-Filemonowicz
"Microstructural characterization of nitrided Timetal 834", Journal of Microscopy 223 (2006) 195-199.

K. Rumpf, P. Granitzer, P. Pölt, A. Reichmann, H. Krenn
"Structural and magnetic characterization of Ni-filled porous silicon", Thin Solid Films 515 (2006) 716-720.

S. Mitsche, C. Sommitsch, P. Pölt, S. Kleber
"EBSD analysis of the recrystallization behaviour of the nickel based alloy 80 A during hot forming", Prakt. Metallographie - special issue 38 (2006) 43-50.

M. Abu-Rous, E. Ingolic, K.C. Schuster
"Visualisation of the fibrillar and pore morphology of cellulosic fibres applying transmission electron microscopy", Cellulose 13 (2006) 411-419.

H. Cerjak, B. Sonderegger, I. Holzer, E. Kozeschnik, C. Pein, G. Dimmler, G. Kothleitner, S. Mitsche, „Einfluss langer Betriebszeiten uaf die Mikrostruktur und das zeitstandsverhalten:", in 32. MPA Seminar (2006), pp. 19.1-19.23.

J. Wagner
„3D-Elementverteilungsbilder von Bauelementen und Biomaterialien mittels Focused Ion Beam Anlage und Röntgenspektrometrie", TUG Forschungsjournal, WS 05/06, pp.14-15.

M. Stöger-Pollach, H. Franco, P. Schattschneider, S. Lazar, B. Schaffer, W. Grogger and H.W. Zandbergen
"Čerenkov losses: a limit for bandgap determination and Kramers-Kronig Analysis", Micron 37 (2006) 396-492.

M. Thaler, V. Hacker, M. Anilkumar, J. Albering, J.O. Besenhard, H. Schröttner, M. Schmied
"Investigations of cycle behaviour of the contact mass in the RESC process for hydrogen production", International Journal of Hydrogen Energy 31/14 (2006) 1-7.

E. Richtig; B. Leinweber; Y. Ghanimi; P. Pölt; A. Di Stefani; R. Hofmann-Wellenhof
"Speckled posttraumatic hyperpigmentation by foreign bodies on the lower legs after welding", Archives of Dermatology 142 (2006) 790-792.

M. Gahleitner, B. Kretzschmar, G. Van Vliet, J. Devaux, D. Pospiech, K. Bernreitner, E. Ingolic
"Rheology / morphology interactions in polypropylene/polyamide-6 nanocomposites", Rheologica Acta, 45 (2006) 322-330.

H. Schroettner, M. Schmied, S. Scherer
"Comparison of 3D surface reconstruction data from certified depth standards obtained by SEM and an Infinite Focus measurement instrument (IFM)", Microchimica Acta 155 (2006) 279-284.

J. Wagner, M. Hunkova, M. Schmied, H. Mulders, M. Novak
"3D Elemental mapping using X-ray spectrometry in a Dual Beam-Focused Ion Beam", Microscopy & Microanalysis 12, Chicago, USA, Microscopy Society of America (2006) 1242-1243.

P. Poelt, A. Reichmann, G. Marinitsch
"In situ investigations of hot corrosion in ESEM", Microscience 2006, London, Royal Microscopical Society, Conference Programme (2006) p.47.

C. Sommitsch, P. Pölt, S. Mitsche, G. Rüf
"On the modelling of the interaction of materials softening and ductile damage during hot working of Alloy 80A", Journal of Materials Processing Technology 177 (2006) 282-286.

S. Mitsche, P. Poelt, J. Wagner
"Analysis of submicron particles by scanning electron microscopy and energy-dispersive X-ray spectrometry - accuracy of size measurement", Scanning 28 (2006) 282-288.

P. Poelt, A. Zankel, M. Gahleitner, E. Ingolic, C. Grein
"ESEM and tensile stage - an Ideal couple for the investigation of polymer fracture", 16th International Microscopy Conference IMC16, Sapporo, Japan, (2006), p.1802.

D.M. Többens, V. Kahlenberg, C. Gspan, G. Kothleitner
"Atomic and domain structure of the low temperature phase of barium metagermanate", Acta Crystallographica B 62 (2006) 1002-1009.

J. Neidhardt, M. O'Sullivan, A.E. Reiter, W. Rechberger, W. Grogger, C. Mitterer
"Structure–property–performance relations of high-rate reactive arc-evaporated Ti–B–N nanocomposite coatings Structure", Surface & Coatings Technology 201 (2006) 2553-2559.

M. Karlík, P. Haušild, C. Prioul, M. Stöger-Pollach, M. Rogers, W. Rechberger
"TEM observation of the microstructure of reactor pressure vessel steel close to the ductile tearing and cleavage fracture surfaces", Proceed. Fractography 2006, Stará Lesná, Slovakia, October 15-18 (2006) pp. 40-51.

M. Sezen, E. Fissthaler, P. Poelt, W. Grogger, E. Tchernychova, E.J.W. List, B. Chernev, F. Hofer
"The effect of ion / electron irradiation on polymer based organic optoelectronic devices", Microscopy & Microanalysis 12, Chicago, USA, Microscopy Society of America (2006) pp. 1300-1301.

W. Grogger, G. Kothleitner, B. Schaffer, F. Hofer
"Electron energy-loss spectroscopy with a monochromated TEM", Microscopy & Microanalysis 12 Chicago, USA, Microscopy Society of America (2006) pp. 1146-1147.

C. Hébert, A. Satz, G. Kothleitner, C. Eisenmenger-Sittner, P. Schattschneider
"Lifetime of hot electrons in metals retrieved from low loss EELS", Microscopy & Microanalysis 12 Chicago, USA, Microscopy Society of America (2006) pp. 1168-1169.

B. Sonderegger, G. Kothleitner, W. Rechberger, H.-H. Cerjak
"EFTEM investigations on precipitates in a creep resistant 9-12% Cr steel", Prakt. Metallographie - special issue 38 (2006) 207-214.

G. Fruhwirt, A. Moumtzi, A. Loidl, E. Ingolic, A. Hermetter
"The oxidized photolipids POVPC and PGPC inhibit growth and induce apoptosis in vascular smooth muscle cells", Biochim. Biophys. Acta 1761 (2006) 1060-1069.

B. Sonderegger, G. Kothleitner, W. Rechberger, H.-H. Cerjak
"EFTEM investigations on precipitates in a creep resistant 9-12% Cr steel", Materials for Advanced Power Engineering, (2006), pp.1217-1226.

B. Sonderegger, S. Mitsche and H. Cerjak
"Microstructural analysis on a creep resistent martensitic 9-12% Cr steel using the EBSD method", Materials for Advanced Power Engineering (2006) pp.119-1205.

S. Mitsche, C. Sommitsch, P. Pölt, S. Kleber
"Recrystallization behaviour of the nickel based alloy 80A during hot forming", EBSD 2006, Conference Proceedings, University of Oxford, England April 3rd to April 4th (2006) p.7.

T. Moskalewicz, B. Schaffer, J. Wagner, A. Czyrska-Filemonowicz
"Microstructure characterisation of the Ti-48Al-3Ag layer on Timetal 834", Proceed. XXth Conference on Applied Crystallography, September, 11th to 14th, Wisła, Poland (2006) p.58.

D.M. Többens, V. Kahlenberg, C. Gspan, G. Kothleitner
"Atomic and domain structure of the low temperature phase of barium metagermanate", 23rd European Crystallographic Meeting, ECM 23, Leuven; Acta Cryst. A62 (2006) p.237.

N. Hochgatterer, P. Raimann, C. Korepp, K.-C.Möller, M.Winter, J.Besenhard, H. Schröttner
„Monitoring dynamics of electrode reactions in Li-ion batteries by in-situ ESEM", Ionics 12 (2006) 253-255.

K. Leitner, J. Besenhard, J.-H. Han, A. Hosseinmardi, A. Trifonova, F. Hofer, B.R. Gollas, K.-C. Möller, M. Winter, "A novel coating technique fort he preparation of core-shell materials and thin film electrodes", Proceed. Int. Workshop "Portable and Emergy Energy-Sources – From Materials to Systems (2006), pp.L9-1- L9-22.

P. Granitzer, K. Rumpf, P. Pölt, A. Reichmann, M. Hofmayer, H. Krenn
„Self organized quasi-regular mesoporous silicon templates for electrodeposition of ferromagnetic nanostructures", in: Tagungsband 56. Jahrtestagung Österr. Physikal. Gesellschaft, Graz (2006) Graz, p. 39.

A. Reichmann, P. Poelt
„In-situ investigations of hot corrosions in ESEM", in: Tagungsprogramm 56th Jahrestagung der Österr. Physikalischen Gesellschaft, Graz (2006), p. 111.

K. Rumpf, P. Granitzer, P. Poelt, A. Reichmann, H. Krenn
„Optical properties of an effective magnetic medium consitiing of ferromagnetic nanaostructures incorporated into mesoporous silicon, in: Tagungsprogramm 56th Jahrestagung der Österr. Physikalischen Gesellschaft, Graz (2005), p. 131.

W. Grogger, G. Kothleitner, F. Hofer
„Advantages of a monochromated transmission electron microscope in solid state physics", in: Tagungsprogramm 56th Jahrestagung der Österr. Physikalischen Gesellschaft , Graz (2005), p. 36.

C. Gspan, W. Grogger, I. Letofsky-Papst, F. Hofer, E. Bucher, W. Sitte
„Superstructure and Domains in $La_{0.4}Sr_{0.6}CoO_{2.71}$", in: Tagungsprogramm 56th Jahrestagung der Österr. Physikalischen Gesellschaft , Graz (2005), p. 125.

M.A. Pabst, C. Rentenberger, E. Bock, R. Schmied, M. Lackner, I. Letofsky-Papst, P. Wilhelm, K. Crailsheim, H.P. Karnthaler, "Protein crystals in the testes of the adult apis mellifera at different ages, 16th International Microscopy Conference IMC16, Sapporo, Japan (2006), p.534.

J. Besenhard, K. Leitner, A. Basch, A. Hosseinmardi, P. Raimann, C. Korepp, E. Lanzer, L.Nussbaumer, R. Saf, F. Hofer, B.R. Gollas, K.-C. Möller, M. Winter. „Thin film electrodes and improved percolation paterrn composites, prepared by atmospheric pressure ion deposition and subtrate induced coagulation", In: Proceed. 7th China International Battery Fair (CIBF 2006) pp. 197 – 209.

M. Abu-Rous, E. Ingolic, K. Schuster
"Visualisation of the nanostructure of Lyocell and other cellucosics for a basic understanding of their functional and wellness properties", in: Proceed. 5th Int. Textile Conference (2006), Instanbul, Turkey, pp. 17-21.

G. Kothleitner, F. Hofer, W. Grogger
„About the concept, the operation and uses for TEM gun monochromators", Abstracts 5th Workshop on Electron Energy Loss Spectroemtry and Energy Filtering, Technische Universität Wien, Wien (2006) p. 14.

P. Pölt
"Der Film vom Riss", in "Annual Report 2005" Austrian Cooperative Research, Vienna (2006), pp.59-60.

G. Mori, S. Sutthiruangwong, B. Schaffer, W. Grogger
„Passivation of WC-Co Based Cemented Carbides", in: Grundlagen und Anwendungen der Elektrochemischen Oberflächentechnik, eds. J.Besenhard & J.Russow:, Proceedings „Angewandte Elektrochemie" GDCh 2004, Monographie vol. 32, GDCh, Frankfurt/Main (2005) pp.197-205

The renovation of the institute continues in 2006 (H. Schröttner, J. Wagner)

Dr. Klaus Weikhard (right) congratulates the winners of the "Nano-Gewinnspiel" which was organised in cooperation between the institute and the company "Weikhard" in Graz.

12. Oral Presentations of Institute Staff

12.1. Oral Presentations 2005

S. Mitsche, H.Schröttner
"Neue Methoden der Rasterelektronenmikroskopie", Böhler Welding, Kapfenberg, January 5, 2005.

C. Gspan
„Structural investigation of $La_{0.4}Sr_{0.6}Co0_{2.71}$ by electron microscopy", SFB-Seminar, Reinischkogel, Austria, February 10-12, 2005.

B. Schaffer
„Entwicklung neuer EFTEM-Spectrum Imaging Methoden und erste Anwendungen", SFB-Seminar, Reinischkogel, Austria, February 10-12, 2005.

A. Zankel
„ESEM - application report from the hot & cold in situ lab"
Annual ESEM Userclub Meeting, Eindhoven, The Netherlands. March 16-18, 2005.

A. Zankel
„In situ investigations in the ESEM - a challenge for both the operator and the engineer"
Annual ESEM Userclub Meeting, Eindhoven, The Netherlands. March 16-18, 2005.

S. Mitsche
„Automated particle analysis of aerosols formed during biomass combustion by SEM/EDX"
International Workshop „Aerosols in Biomass Combustion",TU Graz, Austria, March 18th, 2005.

F. Hofer (invited)
„Energiefilterungs-Transmissionselektronenmikroskopie: Grundlagen und Anwendungen im Bereich der Bio- und Materialwissenschaften" Symposium, University of Innsbruck, Austria, April 11, 2005.

M. Rogers
„Nanostructuring and low voltage TEM specimen polishing on a Nova 200", European FIB/Dual Beam™ Userclub Meeting Eindhoven, The Netherlands, April 24-28, 2005.

J. Wagner
„Nanolab 200 applications on semiconductor devices", European FIB/Dual Beam™ Userclub Meeting Eindhoven, The Netherlands, April 24 to 28, 2005.

B. Schaffer
„EFTEM spectrum imaging at high energy resolution - methods and applications", International EELS-Workshop, Grundlsee, Austria, May 1 to 5, 2005.

F. Hofer
„Mikro- und Nanoanalytik von funktionellen Werkstoffen und Bauelementen"
Erich-Schmid-Institute, University of Leoben, Austria, May 18, 2005.

E. Ingolic
„Strukturanalyse von Holz-Kunststoff-Verbunden mit mikroskopischen Methoden", Veranstaltung Holz und Kunststoff im Verbund, HTBL Kapfenberg, Austria, May 19, 2005.

F. Hofer (invited)
"EFTEM and EELS investigations at high spatial and high energy resolution", Hungarian Eletron Microscopy Conference, Balatonalmádi, Hungary, May 26-28, 2005.

P. Wilhelm (invited)
„Experimental testing of lateral and depth resolution in imaging methods", ESOPS 16 (Europ.Symp.on Polymer Spectroscopy), Kerkrade, The Netherlands, May 29 - June 1, 2005.

F. Hofer (invited)
"New developments in energy-filtering transmission electron *microscopy* "
XII International Conference on Electron Microscopy of Solids, Kazimierz Dolny, Poland, June 5-9, 2005.

F. Hofer
„Ortsaufgelöste Methoden der Nanoanalytik“, Fast Forward Workshop „Nanoanalytik – ein Stärkefeld der Steiermark“, TU Graz, Austria, June 21, 2005.

W. Rom
„Im Reich der Zwerge – Eine Nano-Einführung“, Fast Forward Workshop „Nanoanalytik – ein Stärkefeld der Steiermark“, TU Graz, Austria, June 21, 2005.

W. Grogger (invited)
„EFTEM und EELS an der Auflösungsgrenze“, Leibniz-Institut für Festkörper- und Werkstoff-forschung, Dresden, Germany, June 21, 2005.

F. Hofer (invited)
„New developments in energy-filtering transmission electron microscopy“, 7th Multinational Congress on Microscopy, Portoroz, Slovenia, June 26-30, 2005.

F. Hofer (invited)
“Advances in Electron Microscopy of Polymers and Organic Functional Materials”, 7th Austrian Polymer Meeting, TU Graz, Austria, July 4-6, 2005.

P. Wilhelm
„A picture tells more than 4096 spectra about your polymer (spectral imaging pushing the limits)", 7th Austrian Polymer Meeting, TU Graz, Austria, July 4-6, 2005.

F. Hofer (invited)
„New developments in energy-filtering transmission electron microscopy“, Microscopy & Microanalysis 2005, Honolulu, Hawaii,USA, July 31 - August 4, 2005.

G. Kothleitner (invited)
„EELS spectrum imaging: the next steps“, Microscopy & Microanalysis 2005, Honolulu, Hawaii,USA, July 31 - August 4, 2005.

W. Grogger (invited)
„STEM- and EFTEM-analysis of nanomaterials at high spatial and high energy resoluton“, Microscopy & Microanalysis 2005, Honolulu, Hawaii,USA, July 31 to August 4, 2005.

M. Schmied (invited)
„ESEM – the ultimate in-situ observation tool at high and low iemperatures“, Microscopy & Microanalysis 2005, Honolulu, Hawaii,USA, July 31 - August 4, 2005.

F. Hofer (invited)
„Energy-filtering TEM in semiconductor research“, Dreiländertagung Mikroskopie, Davos, Switzerland, August 28 - September 2, 2005.

M. Rogers
“Behaviour of EBID Pt/C nanowires under mechanical stress”,
Dreiländertagung Mikroskopie, Davos, Switzerland, August 28 to Septembre 2, 2005.

B. Schaffer (invited)
“High energy resolution spectrum imaging by EFTEM: advanced methods”, Dreiländertagung Mikroskopie, Davos, Switzerland, August 28 - September 2, 2005.

G. Kothleitner (invited)
„Local structure and bonding determined by EELS“, ELCRYST 2005, Brüssel, Belgium, September 2 - 9, 2005.

W. Grogger (invited)
„TEM, EELS and EFTEM: application to semiconductor materials and device characterization“ , “Crystalline Defects and Contamination: Their Impact and Control in Decive Manufacturing IV” DECON 2005, Grenoble, France, September 15 - 16, 2005.

F. Hofer (invited)
“ Elektronenmikroskopie an den Grenzen örtlicher und energetischer Auflösung”, 11. Österreichische Chemietage, University of Leoben, Austria, September 19 - 22, 2005.

P. Wilhelm
„Polymer spectral imaging (IR and Raman): basics and applications“, 11. Österreichische Chemietage, University of Leoben, Austria, September 19 - 22, 2005.

W. Grogger (invited)
„Exploring the resolution limits with a monochromated (S)TEM“, Xth International Conference on Frontiers of Electron Microscopy in Materials Science FEMMS 2005 Kasteel Vaalsbroek, The Netherlands, September 25 - 30, 2005.

F. Hofer (invited)
„Elektronenenergieverlustspektroskopie in der Materialforschung“
IFW-Kolloquium „65. Geburtstag Prof. Klaus Wetzig“ at Leibniz-Institut für Festkörper- und Werkstoffforschung , Dresden, Germany, September 30th, 2005.

F. Hofer
„Neue Entwicklungen in der elektronenmikroskopischen Untersuchung von Polymeren und funktionellen Nanostrukturen“, Sitzung Fachausschuß „Gefüge und Eigenschaften von Polymerwerkstoffen“ der DGM, Vienna, Austria, October 13 - 14, 2005.

F. Hofer
“Elektronenmikroskopie”, Veranstaltung des Pädagogischen Institutes des Bundes in Steiermark Graz, Austria, October 25, 2005.

A. Zankel (invited)
„In situ experiments in the ESEM“ – a challenge for both the scientist and the engineer“, Cavendish Laboratory, University of Cambridge, U.K., November 10th, 2005.

F. Hofer (invited)
"Energiefilterungs-Transmissionselektronenmikroskopie: Neue methodische Ent-wicklungen und Anwendungen in der Festkörperforschung", University of Ulm, Germany, November 21st, 2005.

B. Chernev
„Chemometrische Auswertung von 3D-Daten“, 2. Bruker Anwendertreffen, Ettlingen, Germany, November 28 - December 2, 2005.

F. Hofer (invited)
„Über aktuelle Entwicklungen in der analytischen Transmissionselektronen-mikroskopie“, Physikalisches Kolloquium des Fachbereiches Physik, Westfälische Wilhelms-University Münster, Germany, December 1st, 2005.

12.2. Oral Presentations 2006

F. Hofer (invited)
“Energy filtering TEM – new methods and applications”, Annual Conference of the Czechoslovak Microscopy Society, Nove Mesto, February 16 - 17, 2006.

F. Hofer
“Neue Möglichkeiten der Elektronenmikroskopie in der Polymer- und Gummiforschung”, Semperit Technische Produkte AG, Wimpassing, Austria, February 8, 2006.

F. Hofer
„Über den Einsatz der Elektronenmikroskopie in der Bausforschung“, Bautechnische Versuchs- und Forschungsanstalt Salzburg, Austria, February 27, 2006.

W. Grogger, H. Schröttner
„Elektronenmikroskopie in der Materialforschung“, Böhlerit GmbH, Kapfenberg, Austria, March 7, 2006.

P.Pölt
„About the fracture behaviour of polymers“, Borealis GmbH, Linz, Austria, March 14, 2006.

E. Ingolic
“Kryoultramikrotomie in der Materialforschung”, Borealis GmbH, Linz, Austria, March, 14, 2006.

W. Grogger (invited)
„Nanoanalysis of organic and inorganic semiconductor devices“, Seminar NANO and PHOTONICS 2006, Mauterndorf, Austria, March 15 - 17, 2006.

C. Gspan
“Electron diffraction and HRTEM study of the perovskite $La_{0.4}Sr_{0.6}Co0_{2.71}$”, SFB-Workshop, Stainz, Austria, March 16, 2006.

K. Riegler
„Camera characteristics and detection limits on a monochromated TEM“, SFB-Workshop, Stainz, Austria, March 16, 2006.

F. Hofer (invited)
“Advanced electron microscopy for materials research“, National Institute of Chemistry, Ljubljana, Slovenia, March 16, 2006.

P. Pölt
„Das Environmental Scanning Electron Microscope – Grundlagen und Anwendung“, Institut für Experimentalphysik, TU Graz, March 30, 2006.

H. Schröttner
“Neue methodische Entwicklungen vom FELMI-ZFE in Graz”, Böhler Welding, Kapfenberg, March 30, 2006

S. Mitsche
„Recrystallization behaviour of the nickel based alloy 80 A during hot forming“, Workshop on EBSD, Oxford, U.K., April 2 - 5, 2006.

G. Kothleitner (invited)
“Introduction to energy-filtering TEM” and „EFTEM applications“, GIF-School, GATAN Inc., Pleasanton, CA, USA, April 3 - 11, 2006.

P. Wilhelm (invited)
„IR and Raman Imaging –applications comparisons, limitations“, ESIS 2006, Lyon, France, April 4 - 6, 2006.

M. Rogers
„Dual Beam and analytical electron microscopy – applications in materials science”, European FEI FIB / DualBeam ™ Users Club Meeting, Eindhoven, The Netherlands, April 24 - 26, 2006.

J. Wagner
“Difficulties and solutions of tomographic investigations“
European FEI FIB / DualBeam ™ Users Club Meeting, Eindhoven, The Netherlands, April 24 to 26, 2006.

G. Kothleitner (invited)
„Analytische Transmissions-Elektronenmikroskopie: Ein unentbehrliches Werzeug in der modernen Werkstoffforschung“, University of Innsbruck, Austria, April 27, 2006.

M. Sezen
„Nanoanalysis and nanostructuring of organic semiconductor based optoelectronic devices“. 1. Österr. Nanoanalytik-Symposium, Grundlsee, Austria, May 11 - 12, 2006.

M. Hunkova
“Röntgenspektroskopie im Focussed Ion Beam (FIB) Mikroskop – ortsaufgelöste Elementanalyse in drei Dimensionen”, 1. Österr. Nanoanalytik-Symposium, Grundlsee, Austria, May 11 - 12, 2006.

W. Rechberger

„Hochauflösungs-Rastertransmissionselektronenmikroskopie“, 1. Österr. Nanoanalytik-Symposium, Grundlsee, Austria, May 11 - 12, 2006.

F.Hofer (invited)

„Recent developments in analytical electron microscopy“, Einweihung Ernst-Ruska- Zentrum, Jülich, Germany, May 18 to 19, 2006.

M. Hunkova

“X-ray elemental analysis in three dimensions using a DualBeam system“, FIB-Workshop, Focused Ion Beams in Research, Science and Technology, Dresden, Germany, May 22 - 23, 2006.

F. Hofer (invited)

„Analytische Elektronenmikroskopie in der Materialforschung“, TF-Kolloquium, Christian-Albrechts-University, Kiel, Germany, June 12, 2006.

P. Wilhelm (invited)

„Polymeranalytik: IR-Bildgebung in der Praxis und im Methodenvergleich“, Bruker Information-Meeting, Wien, Austria, June 20 to 21, 2006.

P. Pölt

„In situ experiments in environmental scanning electron microscopy“, ESEM VII (7th Annual European ESEM Userclub Meeting), London, U.K., June 26, 2006.

S. Mitsche (invited)

„Gefügeuntersuchungen der Nickel-Basislegierung 80A nach Warmumformungs-prozessen mittels EBSD“, University of Leoben, Austria, June 27, 2006.

G. Kothleitner (invited)

„Focussed Ion Beam und Analytische Transmissions-Elektronenmikroskopie: Unentbehrliche Werkzeuge in der modernen Werkstoffforschung“, Johann-Wolfgang-Goethe University Frankfurt am Main, Germany, July 4, 2006.

W. Grogger

“Einführung in die analytische Elektronenmikroskopie”, ISOTEC-Seminar, Reinischkogel, Austria, July 6 - 8, 2006.

G. Kothleitner, H. Schröttner

„Elektronenmikroskopie in der Materialforschung“, Wolfram AG, Bergla July 14, 2006.

F. Hofer (invited)

“Electron energy-loss spectroscopy with a monochromated TEM”, Microscopy & Microanalysis Conference 2006, Chicago, USA, July 30 - August 3, 2006.

M. Sezen

“The effect of ion / electron irradiation on polymer based organic optoelectronic devices“, Microscopy & Microanalysis 2006, Chicago, USA, July 30 - August 3, 2006.

J. Wagner

„3D elemental mapping using X-Ray spectrometry in a Focused Ion Beam instrument“, Microscopy & Microanalysis Conference 2006 , Chicago, USA, July 30 - August 3, 2006.

P. Pölt

“ESEM and tensile stage – an ideal couple for the investigation of polymer fracture“, 16th Int. Microscopy Congress, Sapporo, Japan, September 3rd – 8th, 2006.

E. Tchernychova

“TEM characterization of optoelectronic devices based on conjugated polymers: can FIB specimen preparation help?“, 16th Int. Microscopy Congress, Sapporo, Japan, September 3rd – 8th, 2006.

F. Hofer

„Die wunderbare Welt des Elektronenmikroskops“, Sommeruniversität Vita Activa 2006, Graz, Austria, September 11 - 15, 2006.

W. Grogger (invited)
"Advantages of a monochromated TEM for Solid State Physics", 56th Jahrestagung der Österr. Physikalischen Gesellschaft, Graz, Austria, September 18 - 21, 2006.

F. Hofer (invited)
„Charakterisierung von Nanopulvern mittels elektronenmikroskopischer Methoden", Seminar NANOPULVER, University of Leoben, Austria, September 20, 2006.

A. Zankel
„The fracture behaviour of polymers – in situ tensile testing in the environmental scanning electron microscope", 8th Austrian Polymer Meeting, Linz, Austria, September 20 - 22, 2006.

W. Grogger (invited)
"Chemical mapping in EFTEM and spectrum imaging mode", UCA Summer Workshop on Nanoparticle Research, Cadiz, Spain, September 20 - 26, 2006.

S. Mitsche
„EBSD-Untersuchungen des Rekristallisationsverhaltens der Nickelbasis-Legierung Alloy 80A während der Warmformung" 12.Int. Metallographie-Tagung 2006, University of Leoben, Austria, September 27 to 29, 2006.

G. Kothleitner (invited)
"About the concept, the operation and uses for TEM gun monochromators", 5th Workshop on Electron Energy Loss Spectrometry and Energy Filtering, Vienna, Austria, September 27 - 29, 2006.

B. Schaffer (invited)
"Scripting in Digital Micrograph", 5th Workshop on Electron Energy Loss Spectrometry and Energy Filtering, Vienna, Austria, September 27 - 29, 2006.

A. Zankel
„In situ microtomy in the ESEM – a versatile method for materials science, with special applications in polymer science", Conference Polymerwerkstoffe 2006, Martin-Luther-University Halle-Wittenberg, Germany, September 27 - 29, 2006.

M. Rogers (invited)
„Introduction of the three-countries FIB workgroup", FIB User Group Meeting 2006, University of Wuppertal, Germany, October 1 - 3, 2006.

E. Tchernychova (invited)
„Characterization of polymer based optoelectronic devices in TEM: specimen preparation and irradiation effects", Sabanci University, Istanbul, Turkey, October 17 - 20, 2006.

P. Pölt (invited)
„Polymere – Zugversuche im ESEM", Fachausschuß der DGM, „Gefüge und Eigenschaften von Polymerwerkstoffen", University of Bayreuth, Germany, November 9 - 10, 2006.

A. Reichmann
„Charakterisierung und Schadensanalyse von Baustoffen mit Hilfe der Raster-elektronenmikroskopie", Kolloquium „Forschung und Entwicklung für Zement und Beton", Wirtschaftskammer Österreich, Wien, Austria, November 15, 2006

A. Zankel (invited)
„In situ microtomy in the ESEM, a versatile method for life sciences and materials science", 2. Wiener Biomaterialsymposium, Vienna, Austria, November 22 - 24, 2006.

M. Rogers (invited)
"The FIB – a versatile tool in materials and life science", Joint Meeting of the Belgian and Dutch Societies for Microscopy, Lunteren, The Netherlands, November 26 - 29, 2006.

E. Tchernychova
"EELS analysis of organic semiconductors in TEM", MRS Fall Meeting, Boston, USA, November 27 - December 1, 2006.

M. Sezen

"Modification of polymer based optoelectronic devices by electron and ion beams", MRS Fall Meeting, Boston, USA, November 27 - December 1, 2006.

F. Hofer

"Neue Möglichkeiten der Elektronenmikroskopie in der Materialforschung", AVL List GmbH, Graz, Austria, December 4, 2006.

H. Schröttner

"Untersuchungsmöglichkeiten am Institut für Elektronenmikroskopie", Institut für Papier-, Zellstoff- und Fasertechnologie, TU Graz, December 12, 2006.

A. Reichmann

„Charakterisierung und Schadesnanalyse von Baustoffen mit Hilfe der Rasterelektronenmikroskopie", Bautechnische Versuchs- und Forschungsanstalt, Salzburg, Austria, December 20, 2006.

Invited speakers from the FELMI-ZFE at Microscopy & Microanalysis 2005, Honolulu, Hawaii, USA.
(F. Hofer, M. Schmied, G. Kothleitner, W. Grogger and Niki Grogger)

Participants from the FELMI-ZFE at the Microscopy Conference 2005 in Davos, Switzerland.
(M. Rogers, F. Hofer, W. Rechberger, J. Rattenberger, C. Gspan, J. Wagner, K. Riegler, M. Dienstleder)

13. Poster Presentations by Institute Staff

13.1. Poster Presentations 2005

A. Zankel
"In situ tensile testing in the ESEM – a challenge for the engineer", Annual ESEM Userclub Meeting, Eindhoven, The Netherlands. March 16 - 18, 2005.

M. Rogers
„A comprehensive EELS/EFTEM study of focused ion beam prepared samples of nitride- and oxide thin films", International EELS-Workshop, Grundlsee, Austria, May 1 to 5, 2005.

H. Schröttner
„Comparison of 3 D surface reconstruction data obtained by conventional SEM, ESEM and an infinite focus microscope (IFM)", EMAS 2005 9th European Workshop on Modern Developments and Applications in Microbeam Analysis, Convitto della Calza, Italy, May 22 - 26, 2005.

A. Zankel
In situ tensile testing in the ESEM – a versatile method for polymer science and life science", EMAS 2005 9th European Workshop on Modern Developments and Applications in Microbeam Analysis, Convitto della Calza, Italy, May 22 - 26, 2005.

P. Pölt
„Polymers and SEM/EDXS – results or damage?", EMAS 2005 9th European Workshop on Modern Developments and Applications in Microbeam Analysis, Convitto della Calza, Italy, May 22 - 26, 2005.

A. Reichmann
„Investigation of volcanic particles by SEM/EDXS", EMAS 2005, 9th European Workshop on Modern Developments and Applications in Microbeam Analysis, Convitto della Calza, Italy, May 22 - 26, 2005.

B. Chernev
„Spectral imaging techniques – special applications", ESOPS 16 (Europ.Symp.on Polymer Spectroscopy), Kerkrade, The Netherlands, May 29 - June 1, 2005.

B. Schaffer
„The formation of molecular nitrogen in chromium nitrides monitored by electron energy-loss spectroscopy", 16th International Plansee Seminar 2005, Reutte, Austria, May 30 - June 3, 2005.

B. Chernev
„Vibrational spectroscopy and spectral imaging techniques – special applications in the polymer science", 7th Austrian Polymer Meeting, TU Graz, Austria, July 4 - 6, 2005.

M. Dienstleder
„Optimization of the FIB milling conditions for RTP-processed niobium and tantalum nitride thin films on silicon substrates", Dreiländertagung Elektronenmikroskopie, Davos, Switzerland, August 28 - September 2, 2005.

C. Gspan
„Superstructure and domains in $La_{0.4}Sr_{0.6}Co0_{2.71}$", Dreiländertagung Elektronenmikroskopie, Davos, Switzerland, August 28 - September 2, 2005.

W. Rechberger
„Energy resolution and spatial resolution on a monochromated (S)TEM", Dreiländertagung Elektronenmikroskopie, Davos, Switzerland, August 28 - September 2, 2005.

K. Riegler
„Gold nanoparticles – synthesis, characterization and influence of additives on their size and shape". Dreiländertagung Elektronenmikroskopie, Davos, Switzerland, August 28 - September 2, 2005.

J. Wagner
"ESEM in-situ heating stage experiments on the sintering behaviour of green body ceramics" Dreiländertagung Elektronenmikroskopie, Davos, Switzerland, August 28 - September 2, 2005.

K. Riegler
„Gold nanoparticles – synthesis, characterization and influence of varoius additives on their size and shape", 11. Österreichische Chemietage, University of Leoben, Austria, September 19 - 22, 2005.

B. Chernev
"Spectral imaging techniques – a mighty tool for the materials science", 11. Österreichische Chemietage, University of Leoben, Austria, September 19 - 22, 2005.

13.2. Poster Presentations 2006

P. Pölt
"The fracture of polymers-tensile tests in the environmental scanning electron microscope", 13th Int. Conference on Deformation, Yield and Fracture of Polymers, Kerkrade, The Netherlands, April 10 - 13, 2006.

P. Pölt
„In situ investigations of hot corrosion in ESEM", MICROSCIENCE 2006 and ESEM User Club Meeting, London, U.K., June 27 - 29, 2006.

C. Gspan
"Superstructure and domains in $La_{0.4}Sr_{0.6}Co0_{3-d}$", 56th Jahrestagung der Österr. Physikalischen Gesellschaft, Graz, Austria, September 18 to 21, 2006.

W. Rechberger
"Monochromated scanning transmission electron microscopy", 56th Jahrestagung der Österr. Physikalischen Gesellschaft, Graz, Austria, September 18 - 21, 2006.

A. Reichmann
"In situ investigations of hot corrosion in ESEM", 56th Jahrestagung der Österr. Physikalischen Gesellschaft, Graz, Austria, September 18 - 21, 2006.

B. Chernev
„Characterization of APID thin polymer films by vibrational microspectroscopic techniques", 8th Austrian Polymer Meeting, Linz, Austria, September 20 - 22, 2006.

C. Gspan at a poster presentation in Davos 2005.

14. Further Education for Staff Members

2005

February 2005
- H. Schröttner, Course „LAB-View Basic“ National Instruments, TU Graz

March 2005
- S. Kaltmann, G. Birnstingl, „Internationale Handwerksmesse“, München, Germany

May 2005
- M. Wallner, Windows XP Workshop, TU Graz
- B. Chernev, C. Brandl, BRUKER-Conference „Infrarot und Raman“, Wien, Austria
- B. Chernev, C. Brandl, Perkin-Elmer Seminar 2005 „Nachhaltige Produktoptimierungen durch moderne Analysenmethoden“ Graz
- G.Birnstingl, WIFI Course “PC-Einsteiger”,Graz

June 2005
- U. Stürzenbecher, Seminar for QM-Managers, TU Graz
- U. Stürzenbecher, Seminar „Steuerrecht“, Salzburg, Austria
- M. Paller, A. Rossmann, „ASECOS-Sicherheits-Demo“, TU Graz

July 2005
- P. Pölt, H. Schröttner „Demonstration Hochauflösungs-Rasterelektronenmikroskop“, ZEISS Oberkochen, Germany

September 2005
- U. Stürzenbecher, „Einführung in die Bilanz und G&V“, TU Graz

October 2005
- W. Rechberger, „NORAN-Anwendertreffen Mikroanalyse“, Bochum, Germany
- U. Stürzenbecher, „SAP-Einführungskurs“, TU Graz
- S. Goger, SAP „Grundkurs, Bestellwesen“, TU Graz
- A. Gusmagg, SAP „Auffrischung und Vertiefung“, TU Graz

November 2005
- P.Wilhelm, B.Chernev, WITec Workshop, “Introduction to Confocal Raman Microscopy”, Ulm, Germany

December 2005
- P. Pölt, H. Schröttner „Demonstration Hochauflösungs-Rasterelektronenmikroskop)
  FEI Eindhoven, The Netherlands

2006

January 2006
- U. Stürzenbecher, S. Goger, „Access 2003 /Einführungskurs“, TU Graz
- U. Stürzenbecher, „Einführung in EU-Projektmanagement“, TU Graz

February 2006
- C. Brandl, „ERSTE HILFE Auffrischungskurs“, TU Graz
- W. Rechberger, Department for Nanostructured Materials, Jozef Stefan Institute (Prof. Miran Ceh), Ljubljana, Slovenia
- A. Zankel, Seminar „Mechanische Kunststoffprüfung“, Martin-Luther-University, Merseburg, Germany

March 2006
- G. Birnstingl, „Brandschutzseminar“, TU Graz

April 2006
- C. Gspan, W. Grogger, „Demonstration of Cs-corrected TEM“, Forschungszentrum Jülich, Germany
- U. Stürzenbecher, Course „SAP-Vertiefung Berichtswesen“, TU Graz
- U. Stürzenbecher, ACR-Course „Haftungsfragen für Institutsleiter“, Vienna
- U. Stürzenbecher, TÜV Course „Balanced Scorecard“, Vienna

May 2007
- F. Streußnig, “MS Word 2003 Advanced (Modul AM3 ECDL)”, WIFI Graz
- A. Reichmann, Course “Rasterelektronenmikroskopie und Analyse von Mikrobereichen und Oberflächenschichten”, Pfäffikon, Switzerland

August 2006
- C.Gspan, Course „X-EL 2006“, Antwerpen, Belgium

September 2006
- H. Schröttner, H. Rattenberger, Gemini-User-Meeting, Bad Boll, Germany
- J.Rattenberger, H. Schröttner, IFM Training, ALICONA Imaging, Grambach.

October 2006
- A. Gusmagg, SAP „Einführung Rechnungswesen u. Finanzen“, TU Graz
- M. Albu, Course „Communication Skills 1 (Intermediate)“, TU Graz

A. Gusmagg, Course „SAP-Auffrischung u. Vertiefung“, TU Graz

M. Albu, Course „Einführung in die Projekt- und Businessplanung für Wissenschafter“, TU Graz

November 2006

M. Albu, Course „Scientific Proposal and Paper Writing“, TU Graz

A. Reichmann, H. Schröttner, Course „Hochtemperaturkorrosion“, Forschungszentrum Jülich; Germany

B. Chernev, „Bruker Anwenderseminar“, Ettlingen, Germany

C. Mayrhofer, Course LEICA Microsystems: „Ultramicrotomy of Industrial Materials“, Wien, Austria

December 2006

B. Chernev, Course „Schicht- und Oberflächenanalytik“, University of Kaiserslautern, Germany

H. Schröttner, J. Wagner „Demonstration of new X-ray spectrometers“, Bruker-AXS, Berlin, Germany.

Handbook of Nanoyanalysis Styria (Editor Werner Rom) 2005
The Handbook is a compilation of the available resources in Styria and introduces the methods and instrumentation, problem solving and active companies and university institutes.
www.nanoanalytik.at

15. Abstracts of Scientific Main Results 2005-2006

With the next pages we try to give an impression of the many activities that have characterized 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.

3D Elemental Mapping using X-Ray Spectrometry in a Dual Beam-Focused Ion Beam

J. Wagner,* M. Schaffer,* M. Schmied,* H. Mulders,** and M. Novak**

*Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology; Centre for Electron Microscopy Graz
** FEI Company, 5600 KA Eindhoven, The Netherlands

While micro- and nanotechnology present many exciting opportunities for materials science, they also present significant challenges for characterisation. These challenges arise because optimising the functionality of materials often depends on a precise control of the size, shape, crystal structure and composition of the material being synthesised. Therefore many analysing methods have been established in order to characterise solids in an appropriate way [1].

In this work we present the development of a new tomographic method for elemental analysis using a focused ion beam (FEI NANOLAB Nova200). We aim at collecting three dimensional data from a maximum volume of 20 µm × 20 µm × 20 µm and the method should be able to run fully automated. The FIB instrument combines scanning electron microscopy and precisely focused ion beam etch and deposition. It is a complete nanotechnology laboratory in one tool and can be used for nanoscale prototyping, nano-machining, characterisation and nanoanalysis.
3D elemental maps can be acquired by cutting the specimen using the focused ion beam slice by slice. After each slice the visible cross section is imaged and mapped via the electron beam and the energy dispersive x-ray spectrometer (EDXS - EDAX Genesis) as shown in Figure 1a. In this context a number of challenges must be met:
Specimen drift (Fig. 1b), contrast change, the logistic work cycle, shadowing and redeposition effects (Fig. 2) together with the correct milling setups, the right take-off angle, post-processing tools and correction methods must be properly adjusted and therefore tested and documented separately. Shadowing and redeposition effects for instance can be avoided by milling a “U-pattern” surrounding the area of interest (Fig. 3a) [2,3].

A great amount of work is also involved in post-processing. For example, the specimen drift and changes in contrast between two slices are corrected with a specially developed script in the program ”Digital Micrograph” (GATAN, Pleasanton, USA) [4]. First results are shown in Fig. 3b and illustrate the calcium distribution in a calcium-magnesium-titanium oxide ceramic (specimen EPCOS, Deutschlandsberg, Austria; reconstructed using AMIRA software, TGS, San Diego, USA).
In the near future this analysis method could, however, prove to be a helpful tool for investigating the functional properties hidden in new structures, composites, nanomaterials and biological tissues.

References

[1] B. J. Inkson et al., Journal of Microscopy, Vol. 201, Pt 2, February 2001, pp. 256-269
[2] L. Holzer et al., Journal of Microscopy, Vol. 216, Pt 1, October 2004, pp. 84-95
[3] M. Schaffer, J. Wagner, B. Schaffer, M. Schmied, H. Mulders, Ultramicroscopy 107 (2007) 587-597.
[4] The aid of Digital Micrograph scripts written by Bernhard Schaffer is gratefully acknowledged.

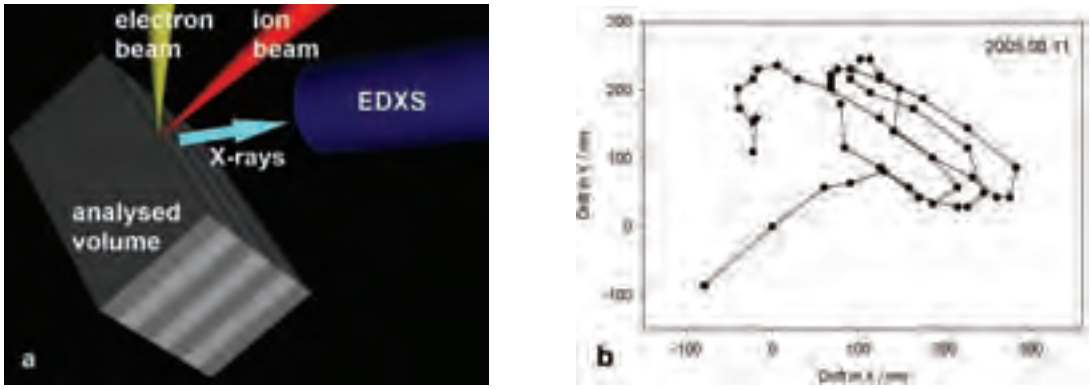


Fig.1a. Scheme of the 3D elemental map process, b. drift during “Auto slice & view “ procedure of a (Ca,Mg)TiO_x ceramic (500 minutes, 50 slices, 20x15x6 µm).

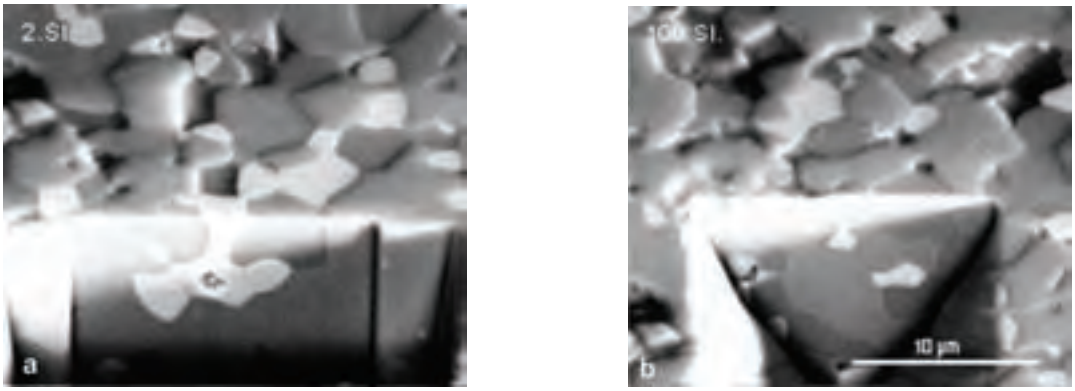


Fig.2 BSE images of the (Ca,Mg)TiO_x ceramic showing shadowing and redeposition effects after a. 2 slices and b. 100 slices (15 x 5 x 20 µm).

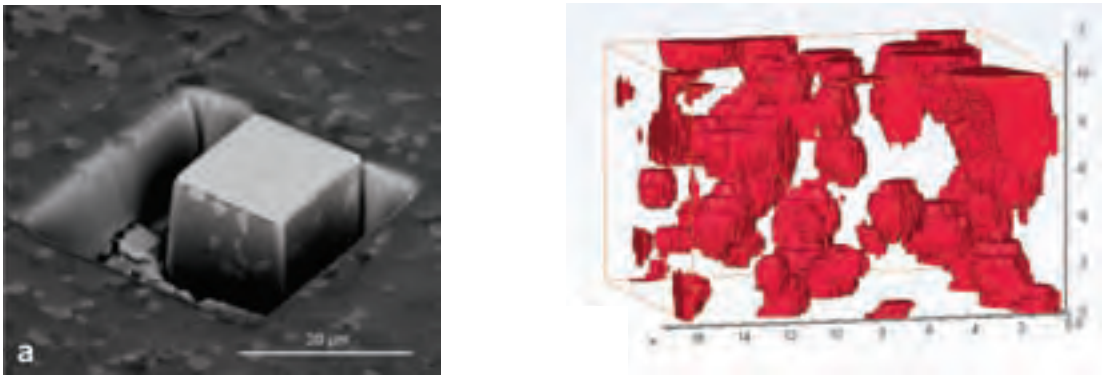


Fig.3a. “U-pattern” surrounding the interesting area to avoid shadowing and redeposition, b. 3 dimensional Ca distribution in a (Ca,Mg)TiO_x ceramic reconstructed using AMIRA software and EDXS elemental mapping (in microns).

In situ Microtomy in the ESEM – A Versatile Method for Materials Science

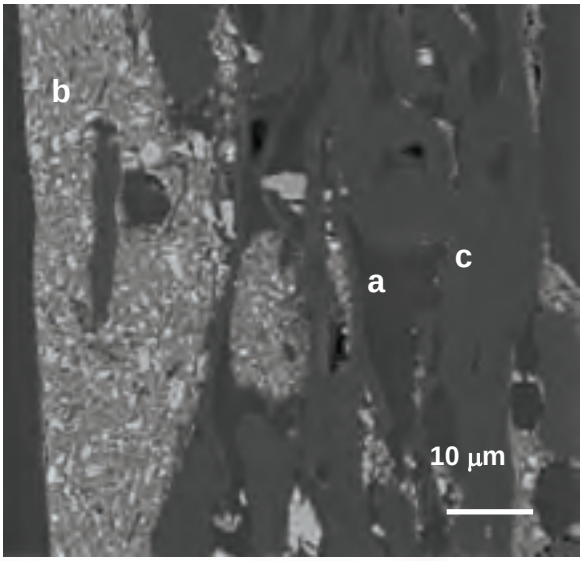
A. Zankel[1], E. Ingolic[1], B. Kraus[2], P. Poelt [1], M. Schaffer[1], G. Kothleitner[1]

[1] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology, Centre for Electron MicroscopySteyrergasse 17, A-8010 Graz, Austria.
[2] Gatan GmbH, Ingolstädter Straße 12, D-80807 München, Germany

The investigation and analysis of three-dimensional (3D) structures is an important issue in a number of scientific fields. Although many microscopic techniques already deliver 3D information, this is still a dynamic area with newly developed methods that make use of several types of signal detection (e.g. light, electrons, ions and x-rays).
A promising new method to obtain 3D information on sub-micrometer scale is serial sectioning of specimens directly in a scanning electron microscope (SEM) with automated 3D acquisition [1]. For non-conducting specimens it is necessary to perform the serial sectioning in an environmental scanning electron microscope (ESEM), which operates at higher chamber pressures (low vacuum mode, nominally from 10 to 300 Pa) and allows the specimen to be imaged without coating.
In this work we present first systematic experiments with the prototype of the 3View^{TM} microtome of Gatan, Inc. It is a specially designed ultramicrotome operating *in situ* within the ESEM FEI Quanta 600 equipped with a field emission gun. The principle of this new *in-situ* method is to cut the specimen using a small diamond knife of special geometry, to take an image of the fresh block face, move the specimen towards the column of the microscope by the chosen slice thickness, cut it again and repeat this for a chosen number of slices.
In order to demonstrate the advantages of serial sectioning in materials science we have investigated the 3D microstructure of a paper sample. It is well known that the paper quality is strongly influenced by the spatial distribution of the raw materials in the paper sheet like fibres, paper fragments, filler particles and the coating layer [2]. A series of 100 slices was cut from a paper specimen and each slice was imaged with the back-scattered electron detector (10 kV). The chosen slice thickness of 200 nm and the imaged area of 60x60 μm^2 with an image size of 1000x1000 pixels enable a resolution of around 200 nm^3 / pixel. The brightest region (b in Fig.1) is the coat ($CaCO_3$), while the darkest region is the resin (a, EPOFIX^{TM}). The medium grey regions represent the fibre material (c, cellulose). These intensity ranges were used to separate the three phases with a threshold technique using the 3D visualisation software AMIRA 3.1^{TM}. The 3D models of the phases were calculated by triangulation and the specific volume and surface of each phase can be determined with an error of around 3% (mostly due to thresholding).
The finely focused electron probe of the SEM and the small interaction volume offer a spatial resolution with an order of magnitude better than confocal microscopy, in principle even approaching the nanometer scale. As there is no depth probing, this 3D resolution is maintained throughout the entire sectioning depth.
The 3D microtome sectioning method in the ESEM has since been successfully applied to the analysis of crack propagation in polymers, wood composites and botanical samples.

References:
[1] Denk W., Horstmann H., PLoS Biol. 2 (11) e329.
http://biology.plosjournals.org/perlserv/?request=get-document&doi=10.1371/journal.pbio.002032
[2] M. Wiltsche, M. Donoser, Papier aus Österreich (2004) 6, 30-31.

Fig.1 BSE-micrograph of the surface of a paper specimen, wich was embedded in resin and sectioned with the diamond knife of the in-situ microtome.
Material contrast reveals the different phases of the paper specimen, a. resin; b. coat; c. cellulose fibers.



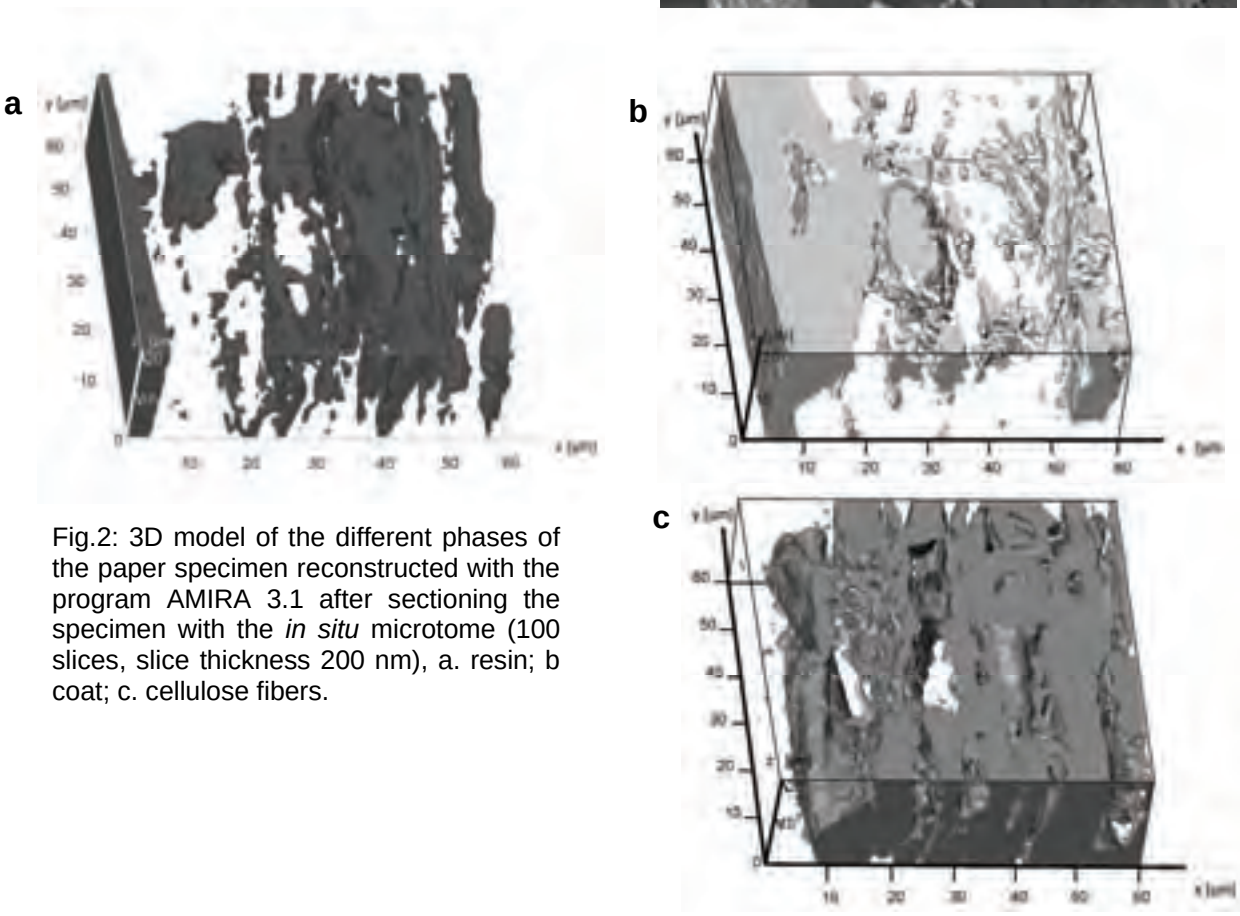


Fig.2: 3D model of the different phases of the paper specimen reconstructed with the program AMIRA 3.1 after sectioning the specimen with the *in situ* microtome (100 slices, slice thickness 200 nm), a. resin; b coat; c. cellulose fibers.

The Effect of Ion / Electron Irradiation on Polymer Based Organic Optoelectronic Devices

M. Sezen*, E. Fisslthaler**, P. Poelt*, W. Grogger*, E. Tchernychova*, E.J.W. List**, B. Chernev*

* Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology, Centre for Electron Microscopy Graz
** Institute of Solid State Physics, Graz University of Technology, Petersgasse 16, A-8010, Graz,

The use of conjugated polymers in optoelectronic devices is becoming increasingly attractive for industrial applications. They can be utilised as the active medium in organic light emitting devices (OLEDs), solar cells and organic field effect transistors (OFETs). Since most of these devices are assembled into thin-film multilayered structures, electron microscopy offers a convenient tool for probing the metal-polymer interfaces. Due to the low melting point of organic materials, however, it is commonly a difficult and challenging task to carry out electron microscopic investigations, because both electron and ion irradiation will generally result in the degradation of the structures. Especially during the sample preparation for Transmission Electron Microscopy (TEM) of these multilayered devices with Focused Ion Beam (FIB) milling, the samples are exposed to high doses of ion irradiation, which can alter the chemical structure of the conjugated polymers, leading to a change in the electronic and optical properties.

The occurrence of irradiation damage in organic materials is very common and already well known [1]. Special focus in this investigation was put on the conjugated bonds. In the study, samples of poly-(3-hexylthiophene) (P3HT) and polyfluorene (PF) based optoelectronic devices were examined. Polyfluorene is an electroluminescent polymer, with an emission maximum at about 440 nm and a glass transition temperature of 70°C, which is often used for blue OLEDs. P3HT is a common material used for OFET applications. It has a high charge carrier mobility due to its nanocrystalline structure, and it experiences a phase transition to an amorphous state at temperatures higher than 220°C. Polyfluorene and P3HT are both conjugated polymers, and the uninterrupted conjugation is crucial for the optical and electronic properties. Most conjugated polymers lose their electronic properties due to structural degradation when heated above ~220°C. Additionally, glass transitions (TG) and melting can occur during FIB treatment, hampering preparation. It has been shown that similar polymers are prone to electron irradiation damage already at very low electron energies and low electron doses [2].

A potential ion and electron irradiation damage of the polymers was investigated by Raman spectroscopy, which is a convenient method to determine the alterations in chemical structures with a lateral resolution down to 1 µm. The effects of sputtering and electron and ion assisted chemical vapour deposition (CVD) of protective metal coatings were also analysed. Alternatively, both FIB milling and ultramicrotomy were applied for the preparation of the polyfluorene sample; and irradiation artifacts of FIB based nanomachining were compared to mechanical sectioning. Ultramicrotomy is a common sample preparation technique that allows very thin sections of embedded samples to be cut using diamond knives without causing any irradiation damage to the material. However, ultramicrotomy cutting requires suitable “soft” substrates and thus cannot be applied to the organic devices fabricated on silicon substrates. In a control experiment, a corresponding bulk polyfluorene specimen was used for ultramicrotomy sectioning. Concerning the Raman spectra, the peak indicating the double bonds vanishes during both electron irradiation and FIB milling, with the amount of damage being dose- and time-dependent. This clearly demonstrates the loss of chemical stability of the investigated materials. On the contrary, sputter coating with the diode sputter coater has left the specimen undamaged.

References

[1] R.F. Egerton et al., Micron, 35 (2004) 399.
[2] H. Ahn et al., Macromolecules, 37 (2004) 3381.
[3] This work was performed within the "ISOTEC" project funded by the Austrian NANO initiative (project DevAna 0706). The support by Dr. Elisabeth Ingolić is gratefully acknowledged.

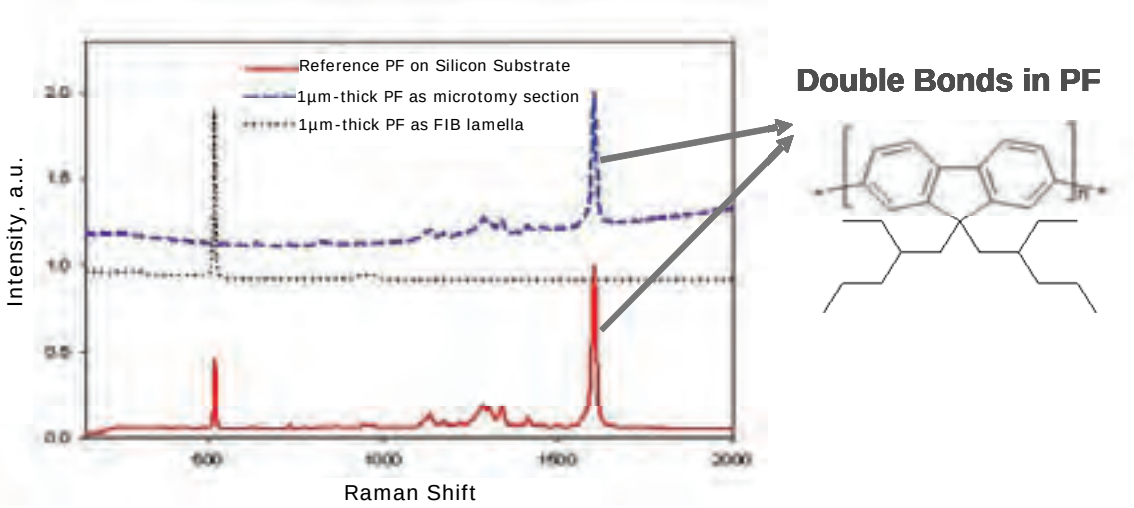


Fig.1. Comparison of Raman spectra, showing the effect of TEM sample preparation techniques on the chemical stability of polyfluorene. band at 520 cm^{-1} originates from the silicon substrate. The spectrum of the reference PF sample has a higher intensity compared to that of the microtome section because of the highly refractive silicon substrate. The signal/noise ratios are similar for both spectra; the band at 520 cm^{-1} originates from the silicon substrate.

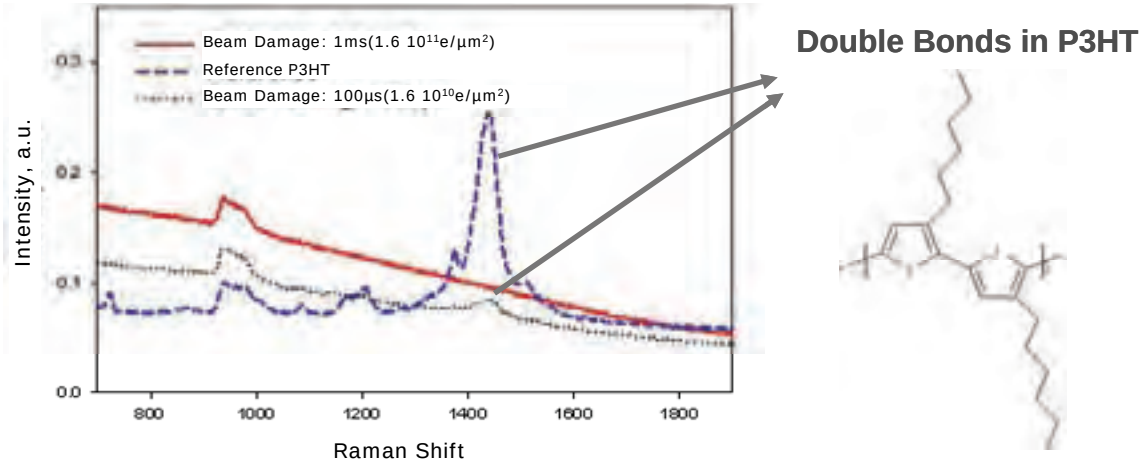


Fig.2. Raman spectra from P3HT samples irradiated with electrons at dwell times of 100 µs and 1 ms per pixel, with total irradiation times of 58 sec. and 9:42 min. respectively, (E_0=5keV; total area: 100 μm^2). The peak at 1439 cm^{-1} corresponds to the double bonds. The background differences are due to sample fluorescence, presumably generated by the products of polymer degradation.

Porous Silicon – Matrix for Ferromagnetic Nanostructures

P. Granitzer[a], K. Rumpf[a], P. Poelt[b], A. Reichmann[b], H. Krenn[a]

[a] Institute of Physics, Karl Franzens University Graz, Universitätsplatz 5, 8010 Graz, Austria
[b] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology, Centre for Electron Microscopy Graz

Porous silicon (PS) consists of parallel channels grown perpendicular to the surface and can be used as a template for magnetic nanostructures. The fabrication of porous silicon is carried out electrochemically during an anodisation process in an aqueous hydrofluoric acid solution, where the pore growth generally leads to a sponge-like structure with interconnected, branched pores in the diameter range between 2 and 4 nm. Despite the usual dendritic growth of mesopores, highly oriented and clearly separated channels with diameters around 50 nm and a length up to 50 µm have been fabricated (Fig. 1a). The direction of pore growth depends on the crystal orientation of the silicon substrate, pores oriented perpendicular to the surface are achieved by etching a (100) wafer. The quasi-regular self-assembled pore arrangement is characterised by a quite homogeneous pore size distribution (Fig. 1b).
The formation of the pores is a self-assembling process, which can be influenced by varying the electrochemical parameters like current density, HF-concentration and bath temperature as well as the doping density of the wafer. The pore diameter is mainly influenced by the applied current and increases with increasing current density. The interpore spacing of this quite regular system is usually of the same size as the pores (typical pore densities around 6.9×10^9 pores/cm^2), but nevertheless the pitch of the pore array depends slightly on the HF concentration and current density and can additionally be varied within a narrow range. To achieve oriented pore growth with rather smooth pore walls the distance between two channels has to be smaller than twice the space charge region (SCR) to suppress dendritic growth. This low-cost fabrication process is suitable for producing tailored samples with desired characteristics within a certain regime by accurate control of the electrochemical parameters of the procedure. The characterisation of this PS system is carried out by structural analysis methods (SEM, EDXS) and by FTIR spectroscopy to investigate the optical behaviour.
In a second electrochemical step this self-assembled template is filled with a ferromagnetic material to achieve an array of incorporated magnetic nanostructures. Using a Ni-salt solution as electrolyte leads to a distribution of Ni-nanostructures within the pores (Fig. 2a, b).
Metal deposition is performed by pulsed deposition to avoid the exhaustion of the electrolyte during the galvanic process. Variation of the deposition conditions strongly influences the ferromagnetic filling. In this manner the Ni-filling status can be varied between more or less elongated Ni-structures as well as different distributions among them.
Magnetic characterisation of the resulting PS/Ni-nanocomposite system is performed by SQUID magnetometry (Quantum Design MPMS XL7) exhibiting a strong magnetic anisotropy at low magnetic fields due to the shape of the deposited Ni-structures (Fig.3a). At high magnetic fields, far above the saturation magnetisation of Ni (0.62 T), the samples show a non-saturating paramagnetic-like behaviour, which seems to be due to an orbital magnetism which occurs in addition to the spin magnetism (Fig.3b). This kind of orbital magnetism has already been observed at surfaces and interfaces reported in the literature.

Reference:
P. Granitzer, K. Rumpf, P. Pölt, A. Reichmann, H. Krenn, PHYSICA E 38 (2007), 205-210
This work has been financially supported by the FWF in Vienna.

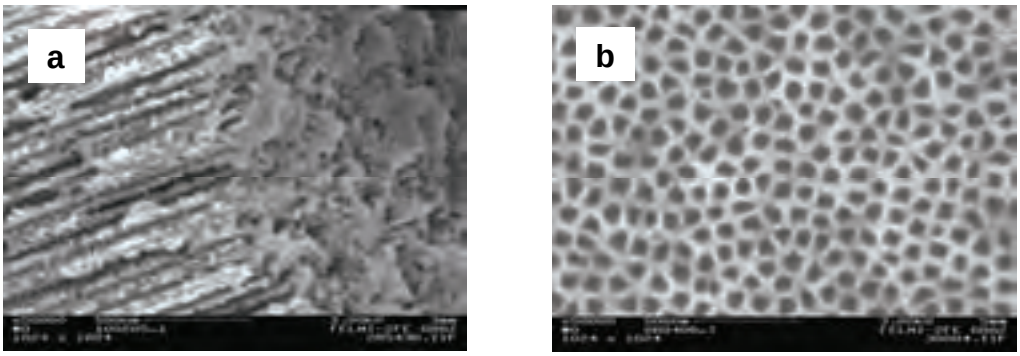


Fig.1a. SEM image of a mesoporous sample showing oriented pores (diameter 55 nm) grown perpendicular to the sample surface,b. SEM-image of the top view of the same PS-sample without top-layer.

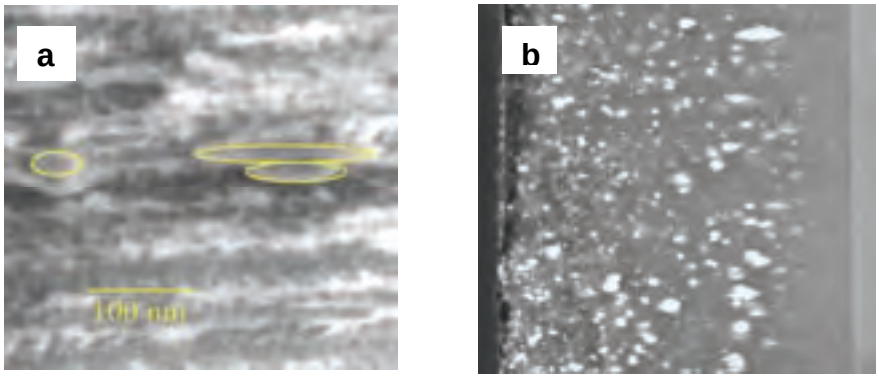


Fig.2a. BSE-image with 3 precipitated Ni-particles within the channels of the porous silicon layer, particle lengths between 50 and 150 nm, the diameter corresponds to the pore diameter. b. Ni-distribution within the entire PS-template with a concentration gradient between surface and pore tips (right side of the picture).

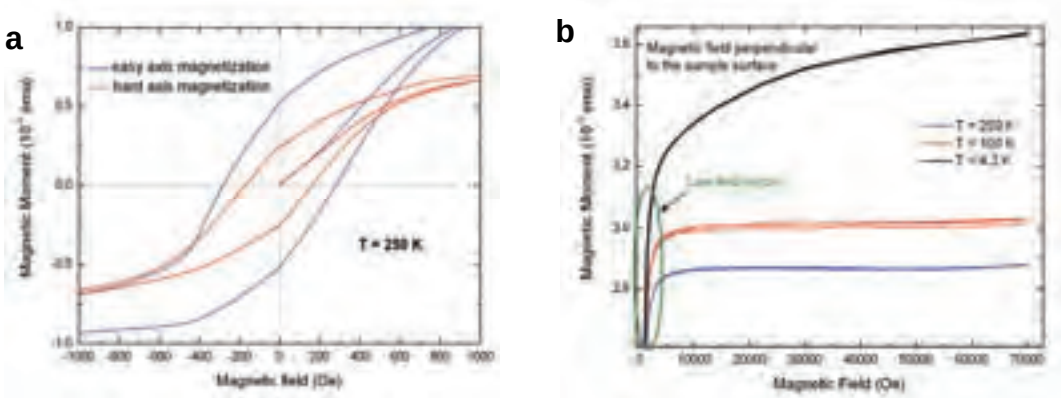


Fig.3a. Hysteresis loop between ± 0.1 T for easy axis magnetization corresponding to an external magnetic field perpendicular to the surface (blue curve) and hard axis magnetization with the magnetic field applied parallel to the surface (red curve), b. High field magnetization (0-7 T) shows a supplementary non-saturating paramagnetic term at 4.2 K.

Vibrational Spectroscopy and Spectral Imaging Techniques – Special Applications in the Polymer Science

Boril Chernev, Peter Wilhelm

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

FTIR microspectroscopy is a major technique for investigating polymer samples in terms of composition as well as constituent distribution. Generally, one has the possibility to perform either a multiple measurement (point-by-point mapping) or a single imaging measurement. The chemical images which are the visual result of the latter represent distributions of different species and thus make it possible to present the data to third parties such as non-professionals in an easy and understandable way. Apart from different chemical distributions in the sample, the spectroscopist is also able to visualise areas with different degrees of crystallinity or preferred orientation and by these means ensure reliable data about the quality of the investigated sample, manufacturing process, etc.. This makes that approach an interesting alternative for materials characterisation in terms of morphology and microstructure.
Since there are only a few studies available about the use of a polariser in combination with an imaging FPA detector [1-3], we explored this method for applications in polymers.

Semi-thin cross-sections (about 8 µm thick) were prepared from an injection moulded polypropylene PT 551 sample by means of an Ultracut E (Reichert-Jung) ultramicrotome, supplied with a 45° diamond knife. The sections were cut parallel to the flow direction from the region immediately below the moulding surface. Polarised FTIR images were recorded in transmission mode using a Bruker Hyperion 3000 infrared microscope with x15 Cassegrainian objective. The microscope was coupled to a Bruker Equinox 55 scientific FTIR spectrometer. The detector was a Bruker focal plane array (FPA) MCT detector with liquid nitrogen cooling. Spectral resolution was 4 cm^{-1}, 32 images were taken. The polarisation of the incident IR beam was achieved by using a KRS-10 polarising unit. The data for the images was calculated using the OPUS software (Bruker Optics); some data was further processed using Sigma Plot 7.0 and in-house -developed scripts for data processing. Dichroic ratios *D* were calculated as the ratio between the parallel and perpendicular polarised intensities (1):

(1) $$D = \frac{A_{\parallel}}{A_{\perp}}$$

and the orientation function f_c was calculated (2):

(2) $$f_c = \left(\frac{2}{3\cos^2\alpha - 1}\right)\left(\frac{D-1}{D+2}\right)$$

By those means we were able to visualize the crystallinity index and orientation function distributions resulting from the occurrence of flow lines in the molding over an area of 270 x 270 µm² which makes polarized FTIR imaging a comfortable tool for revealing the morphology of molded items with reasonable spatial resolution.

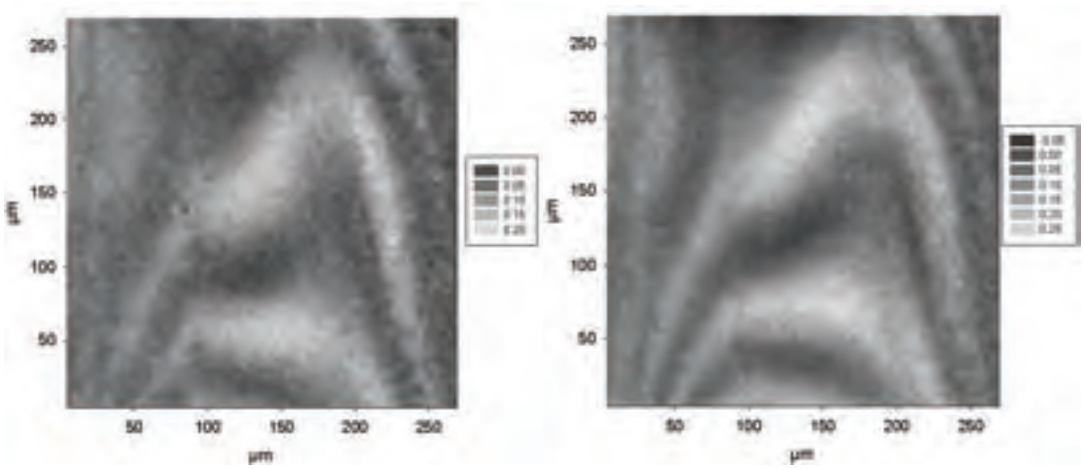


Fig.1 Distribution of the orientation function f_c, calculated for the amorphous phase (left) and for the crystalline phase (right), flow direction parallel to the y axis. As expected the obtained values for the crystalline phase are slightly higher than those for the amorphous phase (cf. both grey shade bars)

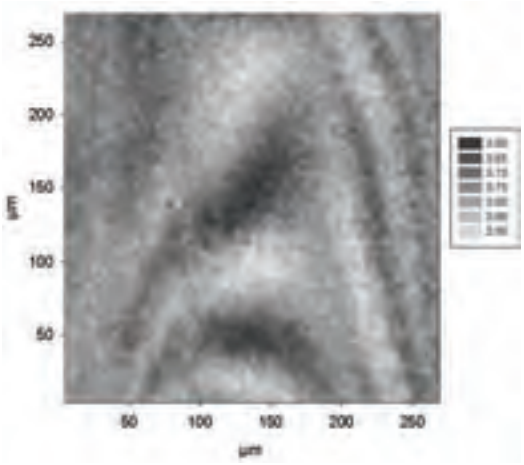


Fig.2 Distribution of the crystallinity index, flow direction parallel to the y axis.

References:

[1] Snively, C.M.; Koenig, J.L., Journal of Polymer Science, Part B: Polymer Physics 37(17) (1999) 2353-2359.
[2] Zheng, Ai-guo; Zhao, Ying; Xu, Yi-zhuang; Wang, Du-jin; Zhang, Xiu-qin; Guangpuxue Yu Guangpu Fenxi 24(7) (2004) 803-805.
[3] Camacho, N.P.; West, P.; Torzilli, P. A.; Mendelsohn, R. Biopolymers (Vol. 2001) 62 (1) (2000) 1-8.
[4] *Polypropylene*, A-Z Reference, ed. by J. Karger-Kocsis, Kluwer Academic Publisher (1999).
[5] Lamberti, G.; Brucato, V., Journal of Polymer Science B: Polymer Physics 41 (2003) 998-1008.
[6] Chernev, B., Wilhelm, P., Chemical Monthly 137 (2006), 963-967.

Vibrational Spectroscopic Investigations on Thin Polymer Films

Boril Chernev[1], Peter Wilhelm[1], Lydia Nußbaumer[2], Robert Saf[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 for Chemistry and Technology of Organic Materials, Graz University of Technology, Stremayrgasse 16, 8010 Graz

Thin polymer films have wide application in different industrial sectors, especially in optoelectronics. Since the quality of such films is often a crucial issue there is a need for exact and rapid characterisation methods either for proving the existence of the films or for proving their quality and/or imperfections such as layer inhomogeneity and other defects.

We investigated thin films of ADS 120 BE (Fig. 1), obtained by means of atmospheric-pressure ion deposition (APID) [1,2] with thicknesses ranging from 3 to 30 nm. APID is a fairly novel technique that allows highly controllable and soft processing of various organic and inorganic materials, including polymers, into thin structured films. This technique implies an electrospray process: microdroplets are initially formed and dried, generating ions that are extracted by electrostatic lenses. Thin structured films are then produced by the deposition of the resulting ion beam onto a movable target. For our experiments we used a glass slide, covered with a fairly thick gold layer.

The samples obtained were analysed using special investigation techniques, such as grazing incidence reflection-absorption FTIR spectroscopy (GIR-FTIR) and Raman microspectroscopy. GIR is a technique for investigating ultrathin layers on a reflecting surface [3]; the investigated spot is typically 800 x 400 µm² with a x15 GIR objective. Raman mapping turned out to be a convenient technique if the layer quality was of primary interest. The spot size on the sample was about 3 µm across with a x50 objective for Raman microscopy, which is a fairly good spatial resolution.
Thus, we were able to characterise the samples with respect to the layer thickness. We found a linear correlation between the integrated GIR-FTIR absorptions and the measured layer thickness values, thus making GIR also a valuable method for quick layer thickness estimation after appropriate calibration.

References:
[1] Saf R.; Goriup M.; Steindl T.; Hamedinger T. E; Sandholzer D.; Hayn G., *Thin organic films by atmospheric-pressure ion deposition*, Nature materials 3(5) 323-329 (2004)
[2] Hamedinger, T. E.; Steindl, T.; Albering, J.; Rentenberger, S.; Saf, R. *Direct patterning of functional materials via Atmospheric-Pressure Ion Deposition*, NSTI Nanotech 2005, 2 467-470
[3] http://www.brukeroptics.de/microscopy/grazing_angle.html

We gratefully acknowledge financial support by the *Fonds zur Förderung der wissenschaftlichen* Forschung, *Vienna, Austria* within the Special Research Program "*Electroactive Materials".*

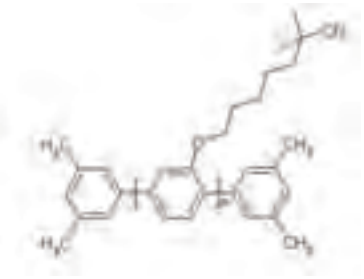


Fig.1 ADS 120 BE - poly[2-(6-cyano-6-methylheptyloxy)-1,4-phenylene]

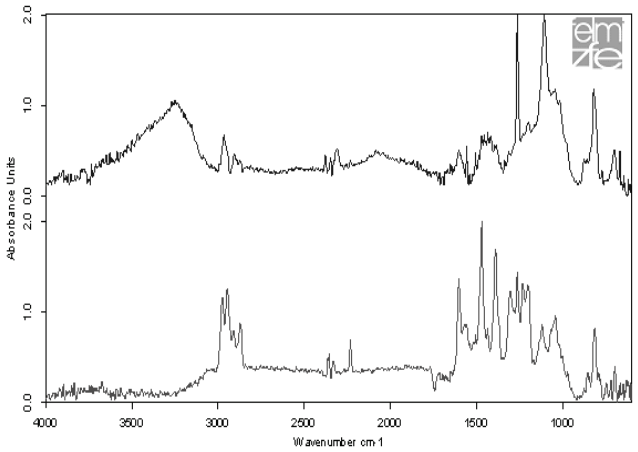


Fig.2 Normalized GIR-FTIR spectra (3 and 30 nm films); GIR allows to detect monolayers of the analyte on the reflecting sample surface.

Fig.3. Raman spectra for several APID films and Raman mapping of an artificially caused defect (scratch) on the 28 nm film.

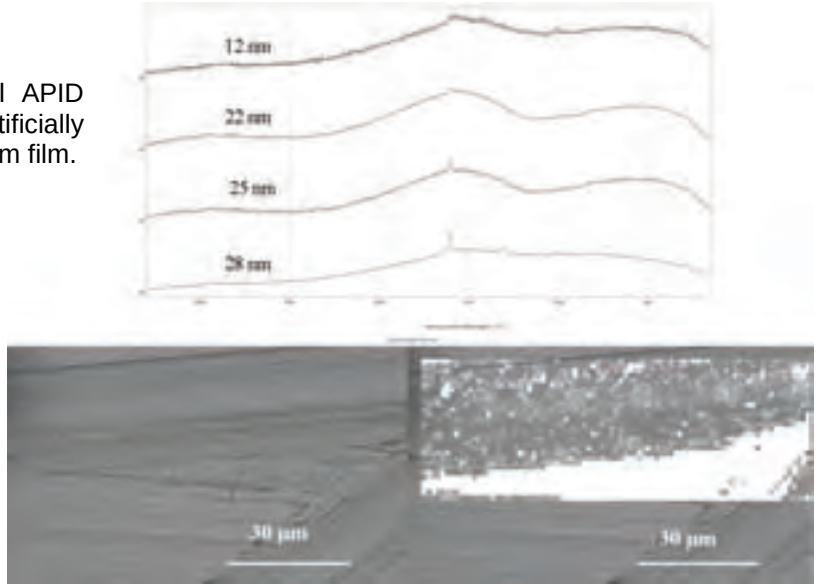
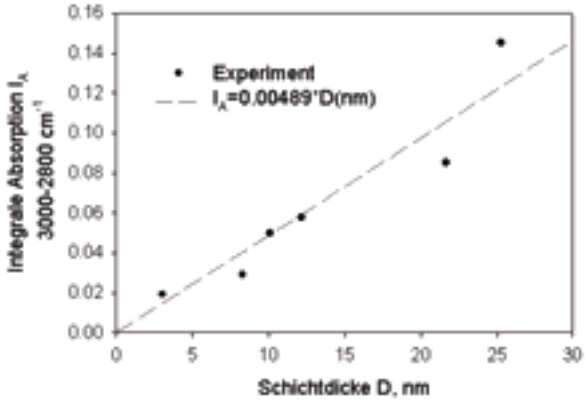


Fig.4 Correlation of integral GIR absorption vs. layer thickness in nm



Behavior of EBID Pt/C Nanowires under Mechanical and Electrical Stress

Michael Rogers, Bernhard Schaffer, Gerald Kothleitner

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

Electron beam induced deposition (EBID) is a promising method for controlled preparation and modification of functional nanostructures such as field emitter tips or scanning probe microscopy tips [1]. A focused electron beam (FIB) is scanned across the specimen surface while a precursor gas is injected into the chamber and onto the specimen. The precursor gas molecules adsorbed on the surface and near the surface are cracked by electron beam irradiation and deposit on the surface. This permits site-specific deposition of structures of very small dimensions (typically 10 - 100nm). The smallest achievable structural size is dependent on various factors like the type of precursor gas or the electron beam diameter. A very common precursor gas is cyclopentadienyldimethylplatinum, which is mostly used in focused ion beam (FIB) systems to deposit protective layers of platinum for transmission electron microscopy (TEM) specimen preparation.

While the electronic properties like the electric resistance of Pt nanostructures prepared by both FIB-induced deposition and EBID are already known [2], there is not much knowledge about the mechanical behavior of EBID Pt/C nanostructures. The main obstacle for obtaining information about the mechanical properties of nanostructures are the small dimensions of these structures, which make it virtually impossible to apply conventional methods like fracture tests or bending tests.

In this work a DualBeam™ FIB/SEM instrument (FEI Nanolab Nova 200) was used to prepare Pt/C nanotips of varying diameter via EBID, with cyclopentadienyltrimethylplatinum as the precursor. The test structures were deposited on a TEM grid in order to allow an easy subsequent study in the analytical TEM. Testing of mechanical behavior was achieved by application of a fine tungsten micromanipulator tip, and the process was simultaneously imaged using the electron beam. To obtain information about possible changes in morphology and chemistry after the bending process, the nanotips were investigated via energy filtering transmission electron microscopy (EFTEM).

The mechanical bending tests showed that the Pt/C nanowires could be bent to at least 45°, as shown in Fig.3. This elastic behavior could be attributed to the heterogeneous morphology and the high carbon content (which could be estimated to ~95 at% according to EFTEM results). The mechanical stress seemingly does not change the morphology of the nanowire as the heterogeneous nanoparticular structure is preserved (as shown in fig. 3c).

However, touching a charged Pt/C nanowire (e-beam charging) with a grounded tungsten tip results in its immediate fracture. Fig.4 shows EFTEM images of a broken nanowire, showing the structural changes. We assume that the cause is an electric discharge resulting in melting of the nanocrystallites, which is forming a core-shell like structure.

The elementary conclusions from this study are that the highly elastic behavior of Pt/C nanowires makes them suitable for applications like atomic force microscopy, and that the structure of Pt/C nanowires can be changed by electric discharge.

References:

[1] H. Koops et al., Jpn. J. Appl. Phys. 33 (1994) 7099

[2] L. Rotkina, J.-F. Lin, and J.P. Bird, Appl. Phys. Lett. 83 (2003) 4426

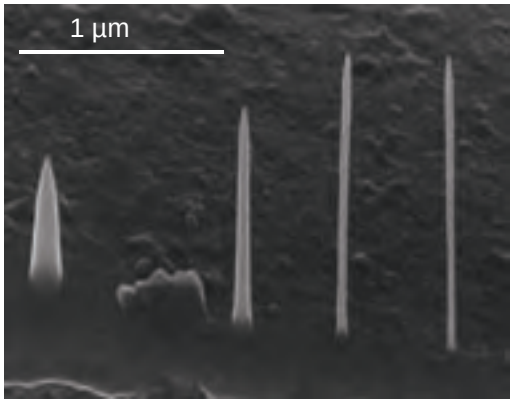


Fig. 1 SE image of Pt/C composite nanotips fabricated by electron beam induced deposition in the FIB.

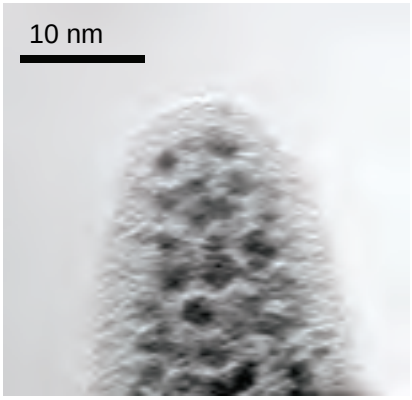


Fig. 2 TEM image of an EBID platinum nanotip showing heterogeneous morphology.

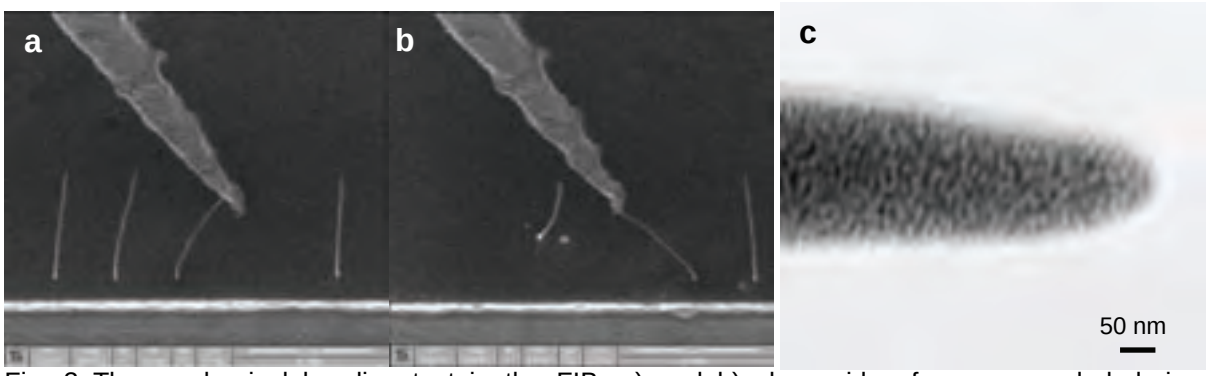


Fig. 3 The mechanical bending test in the FIB; a) and b) show video frames recorded during the mechanical test; c) TEM bright field image of the tip of Pt/C nanowire after mechanical bending.

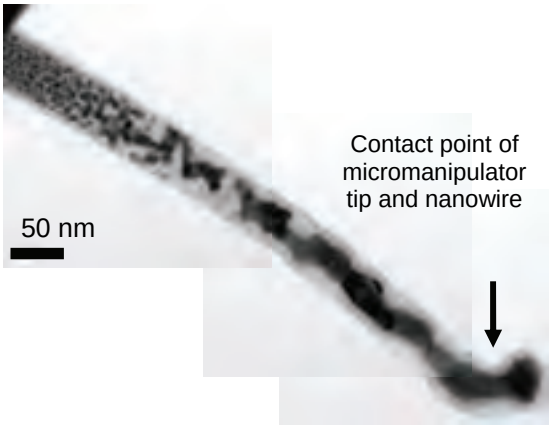


Fig.4 TEM imageof a Pt/C nanowire after electric discharge induced by electron beam irradiation; and subsequent contact with the grounded W micromanipulator tip.

Fig.5 EFTEM images of the Pt/C nanowire after electric discharge showing the Pt nanocrystals which are covered by an amorphous carbon.

ESEM and Tensile Stage – An Ideal Couple for the Investigation of Polymer Fracture

Peter Poelt[1], Armin Zankel[1], Markus Gahleitner[2], Elisabeth Ingolic[1], Christelle Grein[2]

[1] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology & Centre for Electron Microscopy Graz
[2]Borealis GmbH, St.-Peter Straße 25, A-4021 Linz, Austria

The elastic and plastic properties of polymers are usually determined by tensile and bending tests. The elastic modulus can be determined from the stress-strain diagrams. Yet both the diagrams and the parameters deduced from them are global macroscopic values, averaged over the whole material, providing scarce information about the microstructure of the material. But if the tensile stage is operated in an ESEM (Environmental Scanning Electron Microscope), video recordings of the crack propagation at the tip are possible simultaneously with the measurement of the stress-strain diagrams with high magnification and high depth of focus. However, the processes taking place at the tip of the crack and the structures formed there are strongly dependent on the microstructure of the respective material, thus enabling a correlation between the microstructure of the specimen and its macroscopic data.
V-notched specimens of iPP (isotactic polypropylene) and PE/EPR (polyethylene / ethylene propylene rubber) modified iPP with EPR particles of different sizes were investigated. The specimens were characterised both before and after the tensile tests by light and electron microscopy. All specimens were also precracked immediately before the tensile tests, which were performed by use of a Deben M5000 tensile stage mounted in an ESEM Quanta 600 FEG from FEI. In the case of polymer blends a modified heating/cooling platform allowed the choice of a temperature between the glass points of the individual components, thus making it possible to deactivate one component of the blend and to track the behaviour of the ductile remainder of the polymer.
The evolution of the structures at the tip of the crack is dependent both on the test parameters (e.g. jaw speed) and the properties and microstructure of the material, like viscosity [1], type and distribution of the spherulites [2] and the size and volume share of the EPR particles. At temperatures above the glass point, crack formation always started with strong tip blunting accompanied by the formation of fibrils at the tip. It was also observed that a kink in the stress-strain diagram followed by a shift in direction of the characteristic is caused by the breaking of a bundle of fibrils (Fig. 1). This is an indication that the processes taking place directly at the tip have a significant influence on the shape of the stress-strain diagram.
The formation of the fibrils starts from around 25% yield. The exact onset of fibril formation could not be determined because of the geometry of the specimens used, as the width of the crack for low strains was too small to observe the processes at the tip. Fig. 3 shows the evolution of tip blunting and fibril formation for two EPR-modified iPP specimens, with the size and distribution of the corresponding EPR particles given in Fig. 2. Significant differences can be observed in the fracture behaviour, both in the amount of tip blunting and the arrangement of the fibrils. Additionally, the fibrils seem more elastic and extendable in the case of small particles. Although the specimens differed in their overall viscosity, with the viscosity of the matrix being the same (MFR=10) in both cases, additional tests with other specimens demonstrated that the differences found in the crack behaviour can be mainly attributed to the differences in the size of the EPR particles.

[1] Zankel A., Poelt P., Gahleitner M., Grein C., Ingolic E. Proc. EMC 2004, Vol. 2, G. Van Tendeloo, ed., (2004) 361-362.
[2] A. Zankel, P. Poelt, E. Ingolic, M. Gahleitner, C. Grein. GIT Imaging & Microscopy 1 (2005) 16-18.
This work was financially supported by the Austrian Research Promotion Agency (FFG, Project 809817)

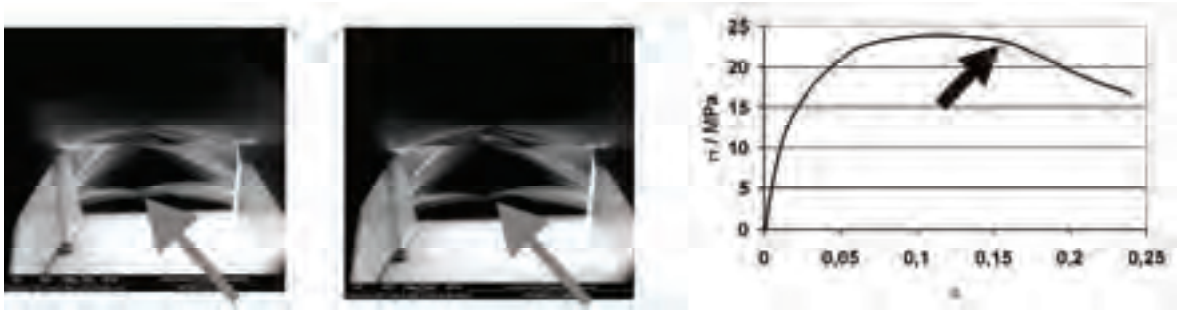
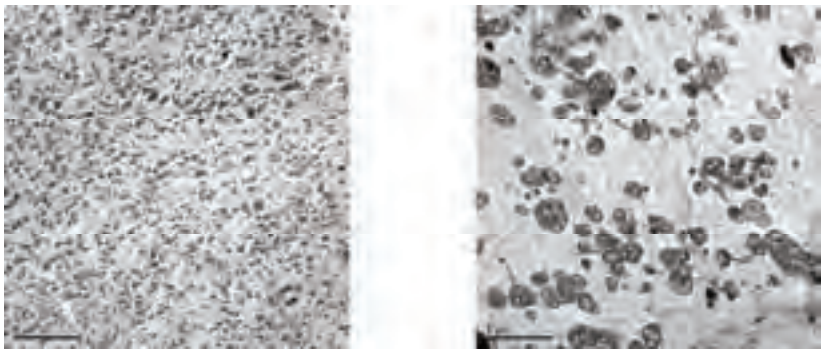


Fig.1 Images cut out from a video to the left (left) and the right (center) of the kink marked by an arrow in the corresponding stress-strain diagram (right); image width: 6.2 mm.

Fig.2. TEM images of cross – sections of 2 EPR-modified iPP specimens with MFR=6.9 (left) and MFR=2.3 (right); image width: 26 µm.



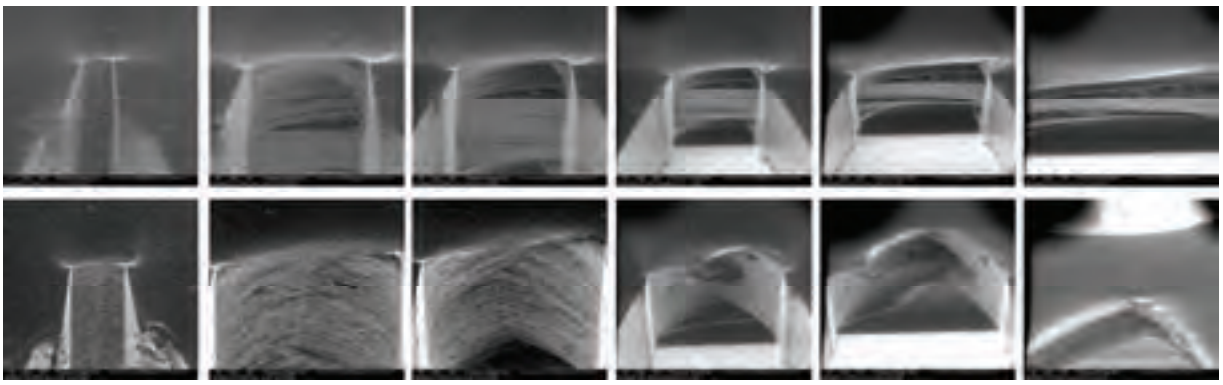
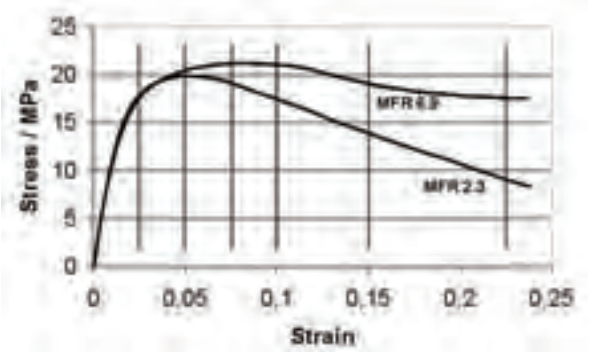


Fig.3 Images cut out from videos of tensile tests (top: MFR=6.9, bottom: MFR=2.3) at the strains marked in the stress-strain diagram at the right. The size and distributions of the EPR-particles are shown in Fig.2.
The magnification varies along the rows, but is always the same in a column.



Advanced Investigations of Electron-Gas Interaction in an ESEM used for Contrast Formation

J. Rattenberger, J. Wagner

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

In an environmental scanning electron microscope (ESEM) the specimen chamber is at a defined vacuum (10 Pa to 10 kPa) which permits the imaging of non-conductive, organic, biological and even wet samples without sophisticated preparation.
In the ESEM the secondary electrons are accelerated by the electric field of the large field detector (LFD) or the gaseous secondary electron detector (GSED) through the gas towards the detector. In this way a cascade effect takes place and the original signal can be amplified over 1000 times [1].
The positive ions generated are swept towards the detector and remove excess electronic charge. But the gaseous environment inside the specimen chamber limits the capability of the microscope concerning scattering of the primary beam electrons and the excess quantity of ions complicates specimen imaging and microcharacterisation [2,3,4].
Models of the gas amplification avalanche which occurs in ESEM usually assume that the cascade behaves like a linear amplifier, although the positive ions inside the specimen chamber damp the amplification [4,5]. Especially the recombination of secondary electrons, and ions and the buildup of positive space charge affect image formation and lead to contrast mechanisms that are not yet fully understood [5].
The experiments were performed on an FEI Quanta 600 and the influence of various parameters such as working distance (2-32 mm), detector bias (150-520 V), chamber pressure (70-665 Pa) and detector type (LFD, GSED) on the equilibrium state of the gas amplification as well as the time dependent influence on the positive ion current was investigated. The primary beam electrons inside the specimen chamber are fractionally scattered by the imaging gas molecules depending on the gas type, the electron energy, the beam gas path length and the chamber pressure. These electrons increase the noise signal and complicate image formation and microcharacterisation of wet samples in particular. Therefore, a new Faraday cup was developed for determining the total scattering cross-section of the imaging gas (water vapour) for different electron energies (see Fig.1).
Time resolved automated measurements (scripting) and a quantitative analysis of the buildup and breakdown of this space charge are presented. These investigations are the basis for a better understanding of the complex ion concentration distribution inside the specimen chamber and the influence of the ion drift velocity on contrast mechanisms. The results will help to improve the quantitative image interpretation in an ESEM. In addition a procedure is outlined for investigating the secondary electron and ion recombination which is primarily responsible for the self-damping of the gas amplification (see Fig.2) [6].

[1] A.L. Fletcher, B:L: Thiel and A:M: Donald, J.Phys. D: Appl. Phys. 30 (1997) 2249-2257.
[2] G.D. Danilatos, Mikrochim. Acta 114/115 (1994) 143-155.
[3] G.D. Danilatos, Scanning Microscopy 4 (1990) 799-823.
[4] E. Nasser, Fundamentals of Gaseous Ionization and Plasma Electronics, Wiley, New York, 1971.
[5] S.W. Morgan and M.R. Phillips, Microsc. Microanal. 7 (Suppl 2) (2002) 788-789.
[6] J.P. Craven; F.S. Baker, B.L. Thiel and A.M. Donald, J. Microsc. 205 (2002) 96-105.
We gratefully acknowledge financial support by the *Fonds zur Förderung der wissenschaftlichen* Forschung, *Vienna, Austria* within the Special Research Program *Electroactive Materials.*

$$m = \frac{\sigma \cdot p \cdot BGPL}{k \cdot T} \quad [1,2]$$

m average number of collisions per electron
p pressure [Pa]
$BGPL$ beam gas path length travelled by each electron in the imaging gas [m]
k Boltzmann constant
T gas temperature [K]
σ total scattering cross section [m^2]

$$I_{ion} \approx \Gamma \cdot I_{PE} \cdot e^{-\psi \cdot I_{PE}} \quad [5]$$

I_{ion} ion current [A]
I_{PE} primary beam current [A]
Γ linear amplification factor
ψ coefficient of self-damping [1/A]

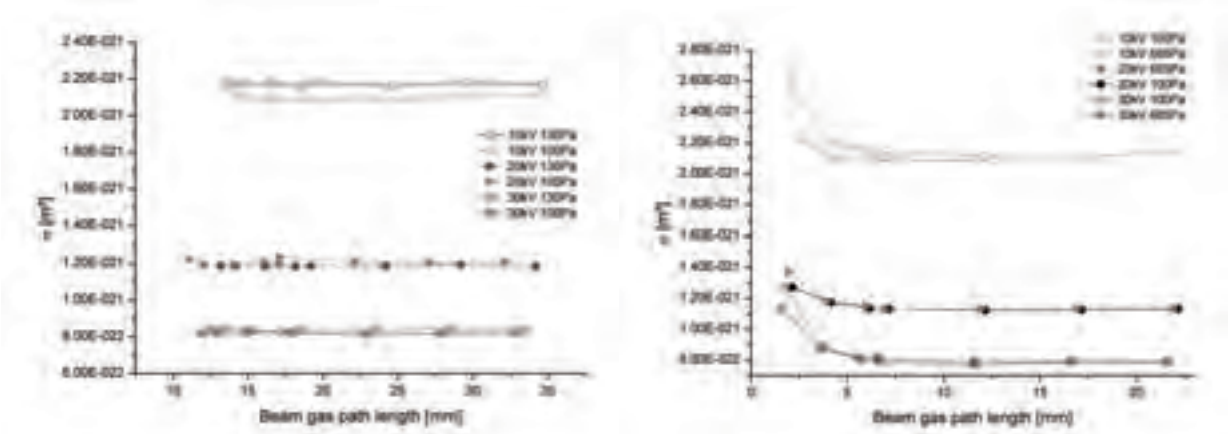


Fig.1 a. Total scattering cross section with large field detector (LFD); b. total scattering cross section with gaseous SE detector (GSED).

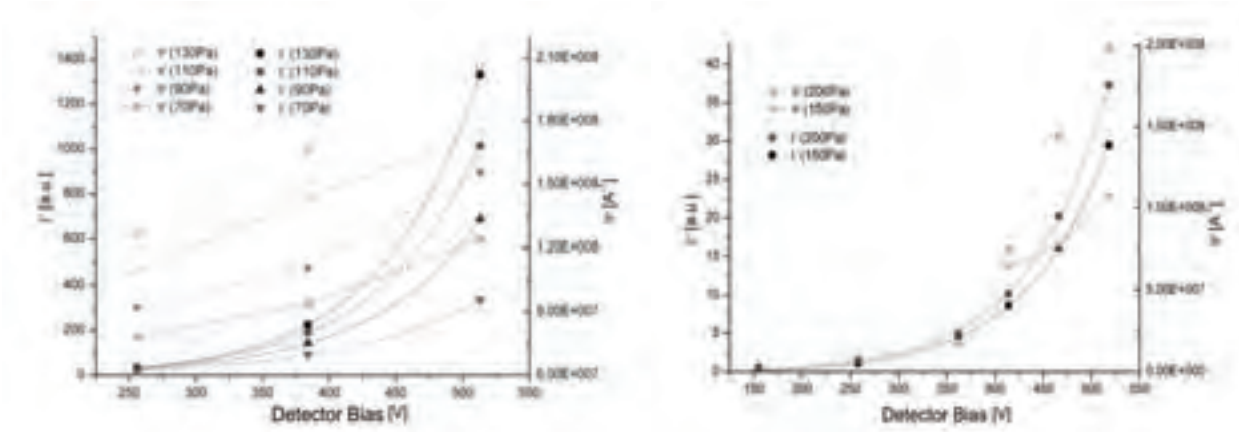


Fig.2 Ψ and Γ as a function of detector voltage for the LFD (a) and the GSED (b) detectors.

EBSD Analysis of the Recrystallization Behaviour of the Nickel based Alloy 80A during Hot Forming

S. Mitsche*, C. Sommitsch**, P. Poelt*

* Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology, A-8010 Graz, Austria
** Christian Doppler Laboratory for Materials Modelling and Simulation / Chair of Metal Forming, University of Leoben, A-8300 Leoben, Austria

Recrystallisation of metals and alloys can take place due to hot deformation or due to cold working followed by annealing processes and is often used to tune the properties of the respective material. The recrystallisation process predominantly changes the microstructure and therefore the mechanical properties and formability of the materials, whereas the physical properties are changed only slightly. The underlying mechanisms are dynamic (DRX), meta-dynamic (MDRX) as well as static recrystallisation (SRX) [1].
In order to distinguish between these three recrystallisation mechanisms for the nickel based alloy 80A, specimens were cut from hot rolled pieces, thus ensuring a completely recrystallised, fine-grained and homogeneous microstructure. This was followed by a solution heat treatment at 1220°C for 60 seconds. This short annealing time was chosen to avoid excessive grain growth and led to an initial grain size of about 120 µm (see Fig. 1a). Hot compression tests were carried out on a Gleeble-3800 Hydro Wedge testing system. The specimens were cooled down to the test temperature of 1120°C and were compressed at a constant strain rate of 0.1 s^{-1} to different strains (up to 0.92). Fig. 1b shows the relevant stress-strain curve. After a soak time of between 0 and 100s the specimen was cooled down to room temperature. An example of the microstructure obtained for a specimen with a pre-strain of 0.3 and without subsequent soaking is displayed in Fig. 1c. The cylindrical compression samples (h = 12 mm, d = 10 mm) were cut into both longitudinal (specimen centre) and transversal cross-sections. The latter sections were chosen at one fourth of the specimen height, since finite element calculations of the compression tests had shown that the local and the global strain rate correspond in this section from the centre to half the radius [2]. The resultant microstructures were investigated by a Zeiss DSM 982 Gemini equipped with an EBSD system from EDAX-TSL.
The recrystallised and the non-recrystallised grains were differentiated based on the grain orientation spread [3]. A nearly linear increase of the recrystallised fraction was obtained for the dynamic recrystallisation process as a function of strain (Details see Poelt et al [4]). The results of the specimens with different pre-strains and soak times are given in Fig. 2. The left diagram clearly indicates that the major part of the recrystallised grains in the pre-strain range investigated is caused by SRX. A noticeable contribution of DRX and MDRX (results obtained at 1 second soak time) can only be observed for pre-strains higher than 0.17. At very low strain (0.07), where no DRX and MDRX can take place, a strong dependence of the recrystallised fraction on the soak time was found with a value of more than 80% at a soak time of 100 sec. Additionally, the grain size of the recrystallised grains strongly depends on the soak time (see Fig. 2 right). In contrast, the pre-strain only slightly influences the size of the recrystallised grains. Fig. 3 demonstrates the dependence of the microstructure with a similar fraction of recrystallised grains on the recrystallisation mechanism.

[1] N. Srinivasan and Y.V.R.K. Prasad, Mater. Sci. Technol. 8 (1992) 206-212.
[2] C. Sommitsch, M. Walter, S. Kleber, P. Pölt, S. Mitsche, STEEL GRIPS 3 (2005) 287-293.
[3] S. Mitsche, P. Poelt, C. Sommitsch, M. Walter, Microsc.. Microanal. 9 (2003) 344-345.
[4] P. Pölt, C. Sommitsch, S. Mitsche, M. Walter, Mater. Sci. Eng. 420A (2006) 306-314.

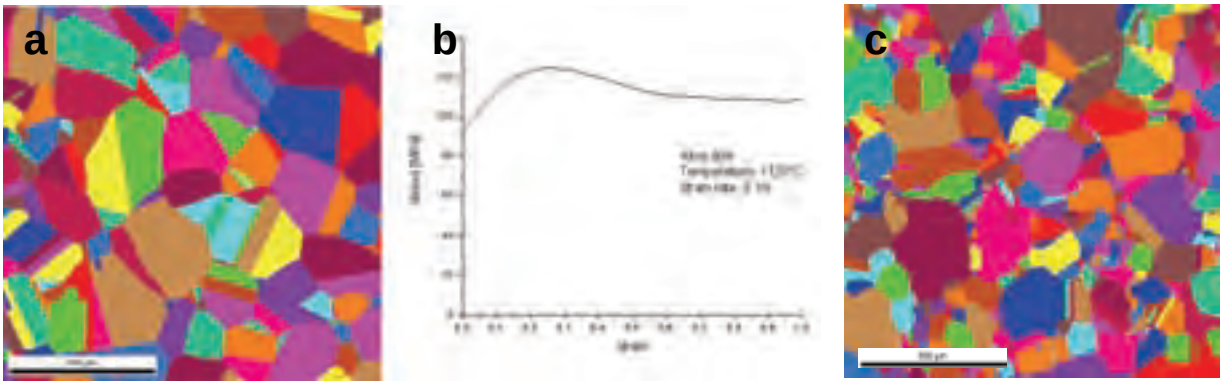


Fig.1 a) Grain map of a hot rolled nickel based alloy 80A specimen after 60 sec solution heat treatment at 1220°C, b) Stress-strain curve for T = 1120°C and $\dot{\varepsilon}$ = 0.1 s^{-1}, c) Grain map of a specimen with a pre-strain of 0.17 and a soak time of 10 sec.

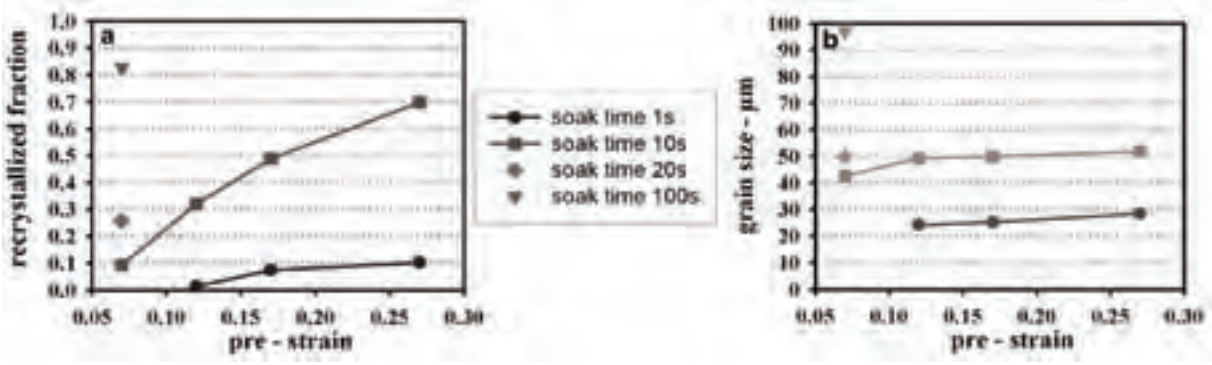


Fig.2 left: Recrystallized fraction as a function of pre-strain right: Average diameter of the recrystallized grains as a function of pre-strain (all twins removed)

predominant DRX | combined DRX-MDRX-SRX | predominant SRX

Fig.3 Inverse pole figure map showing the dependence of the microstructures with similar recrystallized fractions on the recrystallisation mechanism (image width 700 µm, black deformed grains, coloured recrystallized grains).

Cross Sectional Pigment Distribution of Calcium Carbonate and Kaolin in Paper

U. Hirn[1], M. Schmied[2]

[1] Insititute for Paper, Pulp and Fibre Technology, Graz University of Technology, Kopernikusgasse 24, 8010 Graz, Austria
[2] Institute for Electron Microscopy, Graz University of Technology & Centre for Electron Microscopy Graz, Steyrergasse 17, 8010 Graz, Austria

Printing paper is composed of fibres and, up to 30% of total mass, filler pigments like calcium carbonate or kaolin. These pigments improve surface smoothness, brightness and printing properties. The distribution of the filler pigments over the paper cross-section is determined by the dewatering characteristics during the production process as well as the size and shape of the filler particles. An automated microscopy procedure as well as digital image analysis was developed in order to investigate the differences in calcium carbonate and kaolin distribution.

The paper samples were embedded in epoxy resin and cross-sections were made. Automated elemental maps were acquired at different positions on the cross-section using a specially designed script on the Noran Voyager EDX system attached to a Zeiss Gemini DSM 982. Elemental maps of Ca, C, Al, Si, S and O were recorded with an acceleration voltage of 10 keV and a resolution of 512 x 512 pixels. Once the starting and end-point had been defined the specially designed script enabled the acquisition of a series of elemental maps. Six different regions were selected from each sheet of paper and a minimum of three elemental maps per section were recorded.

Fig.1a shows the distribution of calcium and aluminium in a sample, indicating the presence of the filler pigments calcium carbonate and kaolin. Digital image analysis of the C, Ca and Al pictures from the EDXS delivers the pigment profile across the paper sample: The carbon picture is used to identify the paper region within the embedding resin. A grid with ten partitions across the paper cross-section is positioned over the paper region, see Fig.1b, the first partition being closest to the bottom edge of the paper and the last, tenth partition closest to the top edge. The intensity of Ca and Al is determined in each segment of the grid, yielding the filler distribution over the cross-section of the paper. Fig. 2 and Fig. 3 show such distributions computed from 100 pictures, each picture representing a length of 150µm. The intensity is scaled to the average intensity, so no information about absolute concentration of fillers can be extracted. However the relative distribution can be determined.

The symmetric filler distribution in Fig.2 shows exactly the same concentration profile for calcium carbonate and for kaolin. In the asymmetric profile (Fig.3) the profile of calcium carbonate (red) is more distorted. That could be explained by the smaller particle size of calcium carbonate filler pigments compared to kaolin filler pigments.

Acknowledgements: This work was supported by the Austrian Industrial Research Promotion Fund (FFG), Project 806417.

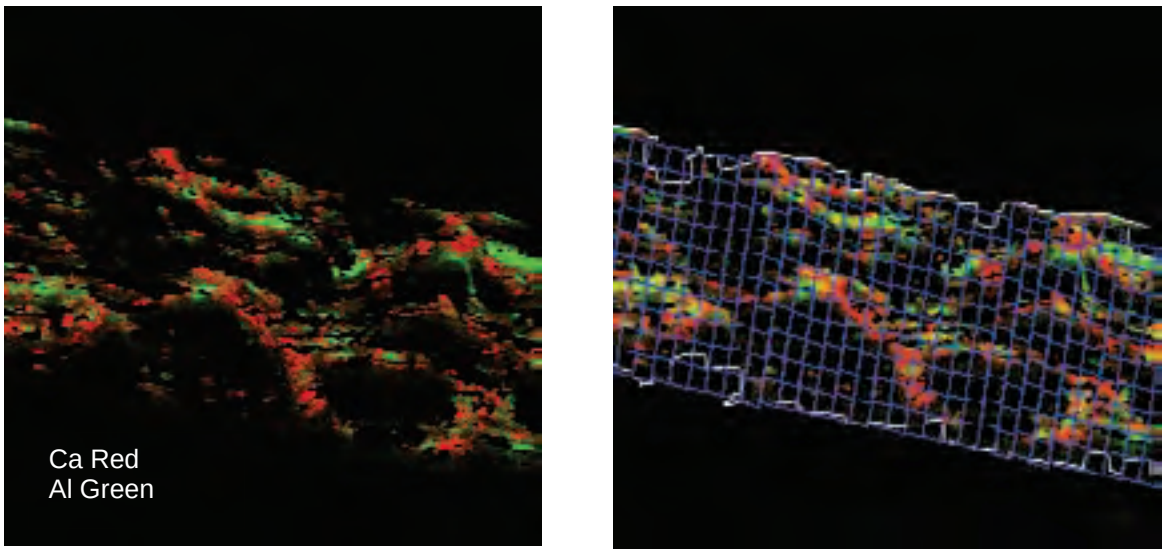
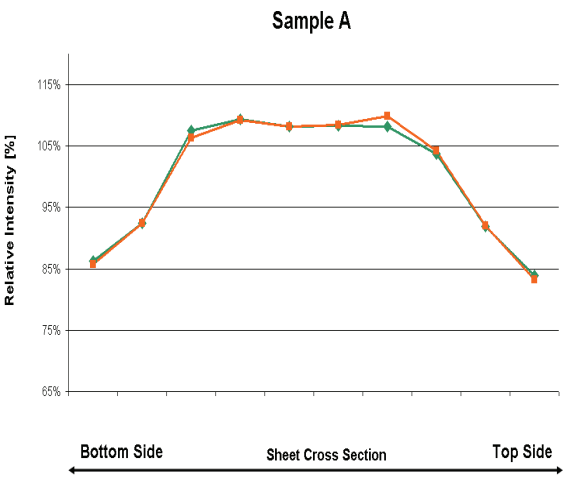


Fig.1a. Paper cross section distribution of Ca and Al, combination of two EDXS maps; b. image analysis of intesity distribution by superimposing a grid (blue) and analyzing the intensity in ten partitions over the paper sheet cross section.



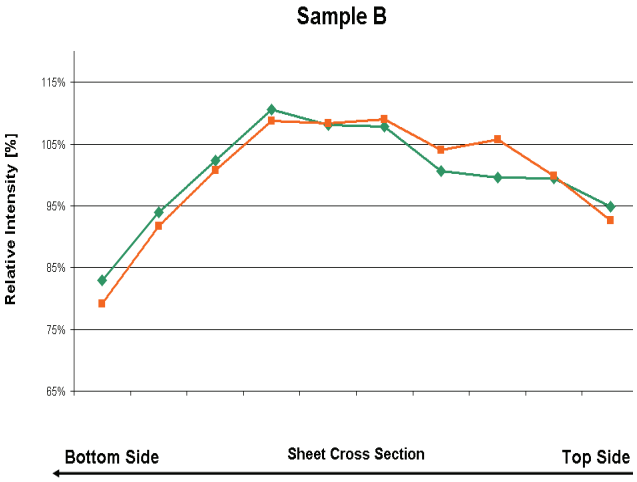


Fig.2 Symmetric filler pigment distribution, presumably due to more intense initial dewatering to the bottom side of the sheet.

Fig.3 Asymmetric filler pigment distribution,

Electron Energy-Loss Spectroscopy with a Monochromated TEM

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

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

The characterisation of nanostructured devices and functional materials on a nanometer scale is paramount to understanding their physical or chemical properties. The introduction of the monochromated transmission electron microscope equipped with a high resolution energy filter has made it possible to solve advanced materials science problems which could not be addressed before. In particular, one can obtain improved information not only about the chemical composition of a sample, but also about chemical bonding and optical properties.
The experiments presented here were carried out using an FEI Tecnai F20 microscope (200 kV, Schottky emitter). The system is equipped with a pre-specimen monochromator (Wien filter) [1], a high resolution imaging filter (Gatan) [2] and a stabilised high voltage supply.
This setup allows EELS spectra to be recorded with an energy resolution in the 100-200 meV range (Fig.1) [3, 4]. The improved energy resolution opens new possibilities for studying detailed electronic structure and bonding effects evaluated from near edge fine structures [5, 6], band gap [7] and dielectric function measurements via the low-loss part of the spectrum. A typical example of the potential of high resolution EELS in materials science is shown in Fig.2. The first peak of the Al $L_{2,3}$ edge of α-Al_2O_3 shows a separation of 0.49 eV which can be attributed to spin-orbit splitting (L_3 and L_2) [8]. These results agree with x-ray absorption spectroscopy measurements. We used STEM imaging to acquire EELS spectra with an energy resolution of less than 0.25 eV, but the spatial resolution was limited to about 2 nm by the properties of the monochromated illumination system of the TEM.
The monochromated TEM also offers advantages for EFTEM spectrum imaging [9], because the microscope is equipped with a high resolution energy filter which is corrected for higher order spectral aberrations. In this work we show that EFTEM spectrum images can be recorded with an energy resolution of less than 1 eV for suitably small slit widths. However, at increased energy resolution aberrations, energy and spatial drift play a more pronounced role. Therefore we have developed 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 [10], simultaneous energy-drift, non-isochromaticity and spatial drift correction [11]). The spectral information now available with high energy resolution allows methods previously developed for EELS analysis to be applied to map spectral features over a large field of view. Fig. 3 shows an EELS spectrum of GaN which was recorded with an EFTEM spectrum image in comparison with a spectrum acquired in spectroscopy mode. We will demonstrate the use of the methods with practical application examples from solid state technology.

References

[1] P.C. Tiemeijer, *Inst. Phys .Conf. Ser.* 161 (1999) 191.
[2] H.A. Brink et al., *Microsc. Microanal*. Vol. 7, Suppl. 2 (2001) 908.
[3] W. Grogger et al., *Proc. 13th Eur. Microsc. Conf.* (2003) Antwerp Vol.1, 271.
[4] K. Kimoto et al., *Micron* 36 (2005) 185.
[5] C. Mitterbauer et al., *Ultramicroscopy* 96 (2003) 469.
[6] G.A. Botton et al., *Ultramicroscopy*, in press.
[7] S. Lazar et al. *Ultramicroscopy* 96 (2003) 535.
[8] F. Hofer et al., *Proc. 13th Eur. Microsc. Conf.* (2003) Antwerp Vol.2, 725.

[9] G. Kothleitner, F. Hofer, *Micron* 34 (2003) 211.
[10] B. Schaffer et al., *Ultramicroscopy* 102 (2004) 27.
[11] B. Schaffer et al., *Ultramicroscopy 106 (2006)* 1129.
This work was financially supported by the special research program ”Electroactive Materials” of the *Austrian Science Fund* (FWF, Vienna, Austria).

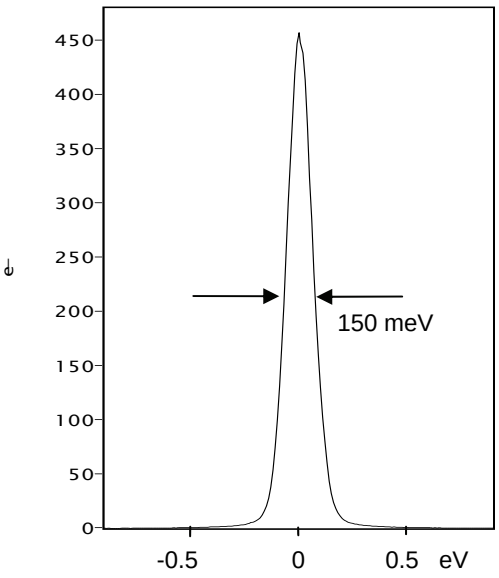


Fig.1 Zero-loss peak measured with the monochromated Tecnai F20 as installed in Graz, acquisition time 0.5 sec.

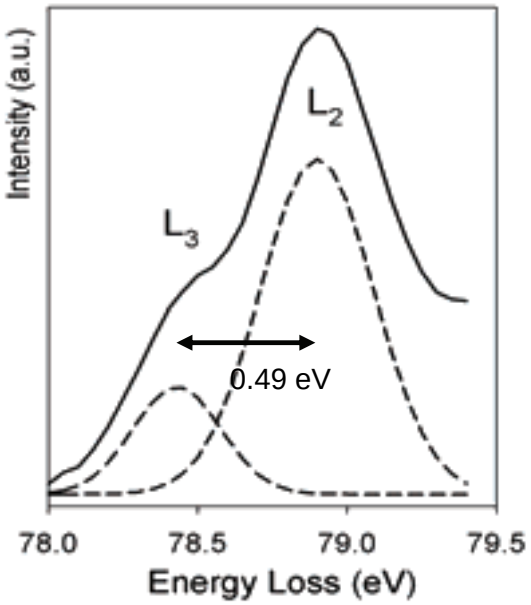
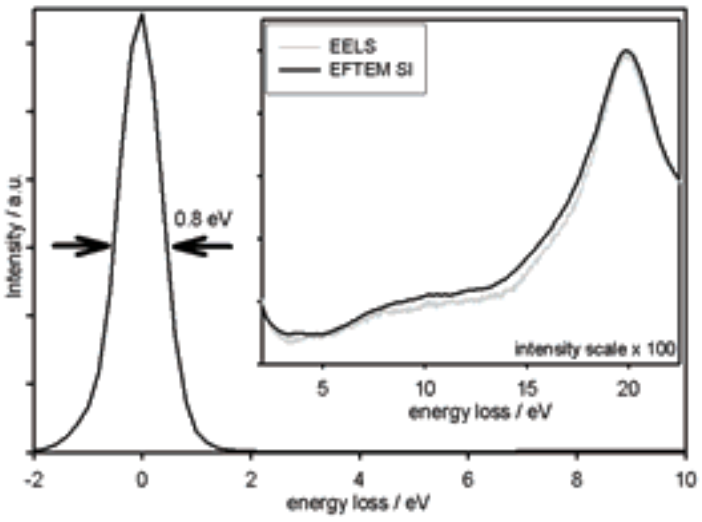


Fig.2 Al-$L_{2,3}$ ELNES of α-Al_2O_3 fitted with Gaussians revealing spin-orbit splitting (separation of L_3 and L_2).

Fig.3 Comparison of EELS spectra of GaN acquired in spectroscopy mode with a spectrum extracted from an EFTEM spectrum image of the same sample region.



Energy Resolution and Spatial Resolution on a monochromated (S)TEM

W. Rechberger, G. Kothleitner and F. Hofer

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

The energy resolution in electron energy-loss spectroscopy (EELS) is largely limited by the stability of the high voltage supply, by the resolution of the spectrometer and by the energy spread of the source. To overcome this limitation a Wien filter monochromator was recently introduced with commercially available transmission electron microscopes (TEM) [1], providing increased resolution of EELS fine structures, which contain valuable bonding information. The method of Z-contrast imaging within an (S)TEM, utilising a high-angle annular dark-field (HAADF) detector perfectly complements the better energy resolution, since both can be collected simultaneously. In combination with a monochromator microscope this allows not only high spatial resolution images but also high energy resolution EELS spectra to be recorded.
In this work we investigated the STEM performance of a 200 kV monochromated Tecnai F20 with a high resolution Gatan Imaging Filter (HR-GIF). The test sample was an ODS-niobium alloy [2] with embedded TiO_X precipitates. In conventional STEM (monochromator off, no dispersion of the beam, 0.55 eV energy resolution) the microscope is in nanoprobe-mode and the probe diameter can be as small as 0.2 nm which allows atomic resolution imaging. In filtered STEM mode (monochromator on, dispersion of the beam, 0.24 eV energy resolution), however, the probe size is enlarged to approximately 4 nm due to electron optical limitations of the condenser system of the Tecnai F20.
Titanium oxides are known to display a variety of different EELS fine structures, which can only be adequately resolved in monochromated EEL spectra as shown in [3] and therefore are suitable candidates for challenging the monochromator also in terms of energy resolution.
Fig. 1a shows a zero-loss filtered TEM bright field image of an ODS-niobium alloy with embedded titanium oxide particles, in which the particles are hardly visible due to diffraction effects and bend contours. Only in the EFTEM jump ratio images recorded at the Nb-M_{45} and Ti-L_{23} edge can the precipitates be detected. For comparison, a conventional bright field STEM image was recorded of the same area of view (Fig. 2a). Similar to the bright field TEM image, it does not allow differentiation between matrix and precipitates. In the HAADF STEM images (Fig.2b and 2c) the precipitates show up dark and the Nb matrix bright. The conventional HAADF STEM image offers better resolution (0.4 nm) than the monochromated HAADF STEM image (4 nm). EELS spectra of TiO_X precipitates of the size of 5-10 nm were recorded by positioning the monochromated STEM probe on the single precipitates, offering an energy resolution of 0.24 eV. Fig. 3 presents the comparison of the EELS spectra of a TiO_X precipitate with monochromated reference spectra of different titanium oxides at the Ti-L_{23} edge. Because the EELS spectra of the particle correspond with the reference spectrum of Ti_2O_3, it was possible for the first time to prove that the small TiO_X precipitates consist of Ti_2O_3.

References:
[1] P.C. Tiemeijer, Ultramicroscopy 78 (1999), p. 53.
[2] F. Hofer, P. Warbichler, W. Grogger, Ultramicroscopy 59 (1995), p. 15.
[3] C. Mitterbauer, PhD Thesis (2003), TU-Graz, Austria
The authors would like to thank the “Steiermärkischer Zukunftsfonds” for financial support (PN119)

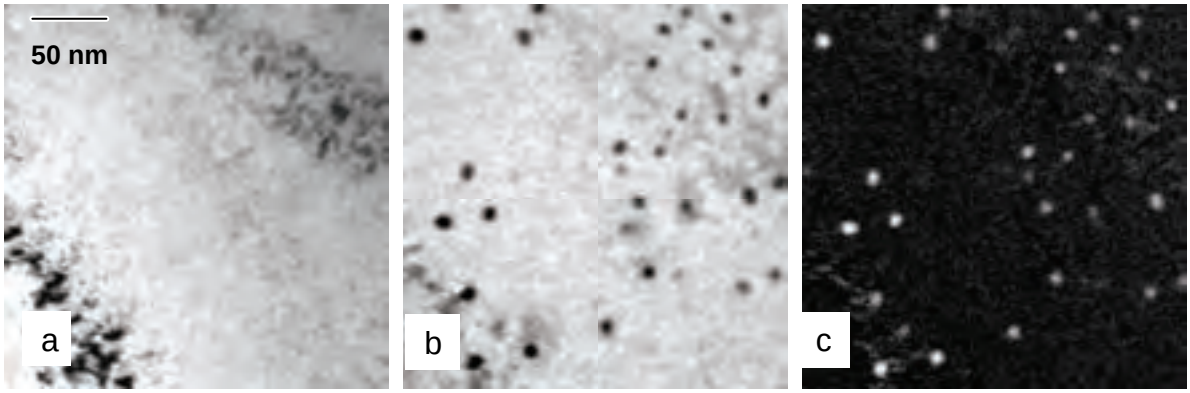


Fig.1 EFTEM images of titanium-oxide particles in Nb, a. zero-loss filtered TEM bright field image, b. Nb-M_{45} jump ratio image and c. Ti-L_{23} jump ratio image.

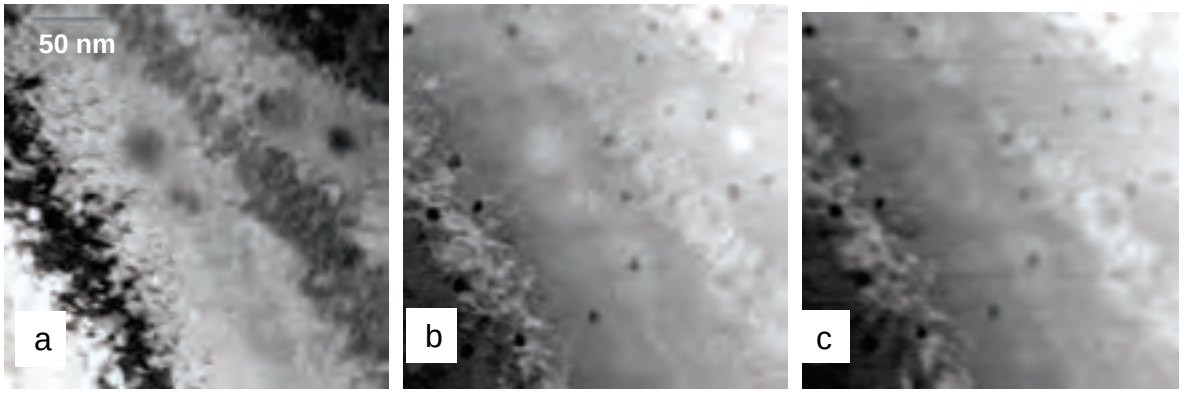


Fig.2 STEM images of titanium-oxide particles in Nb, a. bright field STEM image, b. HAADF STEM image, c. monochromated HAADF STEM image.

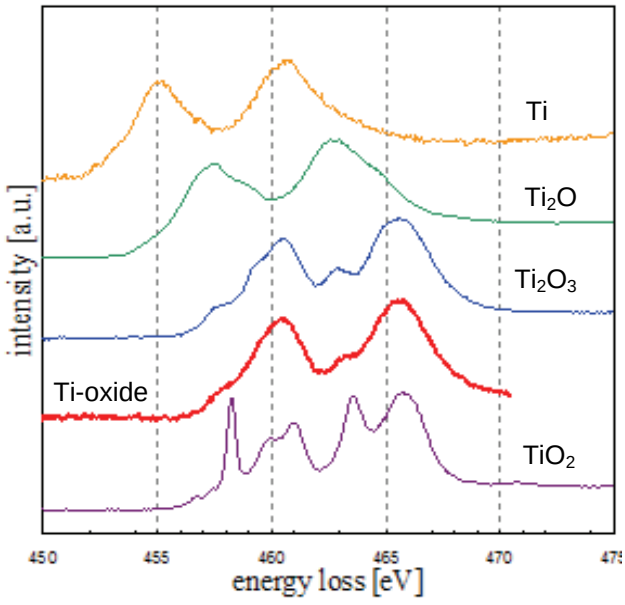


Fig.3 Comparison of monochromated EELS reference spectra of different titanium-oxides with a monochromated spectrum of a titanium-oxide particle in an Nb alloy.

STEM- and EFTEM-Analysis of Nanomaterials at High Spatial and High Energy Resolution

W. Grogger[1], B. Schaffer[1], M. Rogers[1], H. Kirmse[2], W. Neumann[2], G. Kothleitner[1], and F. Hofer[1]

[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 Physics, Humboldt University Berlin, Germany

The full characterisation of nanostructured devices and functional nanomaterials on a nanometer scale is paramount to understanding their electrical, optical, or mechanical properties. Transmission electron microscopy (TEM) offers an excellent tool for this task, in terms of structural and chemical analysis on a nearly atomic scale. In particular, electron energy loss spectrometry (EELS) can obtain information not only about the chemical composition of a sample, but also about chemical bonding (via energy loss fine structure, ELNES) and optical properties (through valence loss EELS, VEELS) [1-4]. Recent instrumental improvements like TEM monochromators have made it possible to considerably improve energy resolution for EELS, which makes the spectral quality comparable to that of x-ray absorption spectrometry and allows for the detection of subtle ELNES changes [5]. Another strong point of the method lies in the combination with a fine electron probe (STEM), where high spatial resolution can be combined with high energy resolution EELS. A special advantage of this combination is the simultaneous acquisition of spectral and spatial information, which can be done spectrum by spectrum (STEM-EELS spectrum imaging) or image by image (EFTEM spectrum imaging).
All results presented in this paper were obtained on a monochromated 200 kV FEG TEM/STEM (FEI) with a high resolution imaging filter attached to it (HR-GIF, Gatan) [6, 7]. The obtainable energy resolution of this instrument is 0.2 eV. We have done experiments using both approaches in order to fill the three-dimensional data cube. For EFTEM spectrum imaging we have developed acquisition and correction algorithms that are necessary for measuring a reliable data set as well as for interpreting the data correctly and accurately. In this mode, with the monochromator switched off, we were able to obtain spatial resolution values below 1 nm and an energy resolution of 0.8 eV due to the precise slit assembly of the HR-GIF. In addition we also used STEM spectrum imaging in combination with the monochromator. In this mode spatial resolution was limited to about 2 nm by the properties of the monochromated illumination system of the TEM. At the same time EEL spectra with less than 0.25 eV were acquired.
As a real world application example we investigated a VN_x coating on a Si substrate in STEM mode (Fig. 1a) [8, 9]. The energy resolution in this particular case was 0.27 eV measured as the FWHM of the zero loss peak. By looking at the ELNES of the N-K ionisation edge we were able to clearly differentiate between VN and V_2N (fig. 1b) and compare these results with theoretical simulations. Fig. 2a shows a bright field image of an InAs/In(P,As) multilayer structure. The nominal thicknesses are 4 ML for the InAs and 20 nm for the In(P,As). There is a slight plasmon shift of about 0.2 eV between the two compounds. Using an EFTEM series with an extremely small slit width of 0.4 eV in the energy range up to 25 eV, this region was evaluated in terms of the position of the plasmon with superb energy precision (Fig. 2b).
The presentation of the paper will focus on the aspects important for spatial and energy resolution. We will point out the limitations of current commercially available TEM instruments and demonstrate the use of the methods with some practical application examples [10].

[1] F. Hofer, P. Warbichler, in *Transmission Electron Energy-Loss Spectroscopy in Materials Science*, Plenum Press, New York, 2004.
[2] W. Grogger et al., Ultramicroscopy 96 (2003) 469.
[3] R. Brydson, Mat. Sci Technol. 16 (2000) 1187

[4] R.H. French, J. Am. Ceram. Soc. 73 (1990) 477.
[5] C. Mitterbauer et al., Ultramicroscopy 96 (2003) 469.
[6] P. Tiemeijer et al., Microsc.Microanal. 7 (Suppl. 2: Proc.) (2001) 1130.
[7] H.A. Brink et al., Ultramicroscopy 96 (2003) 367.
[8] G. Kothleitner, M. Rogers, B.O.Kolbesen, Appl. Surf. Sci. 252 (2005) 66-76.
[9] M. Rogers, G. Kothleitner, A. Berendes, B.O.Kolbesen, Prakt. Metallogr. 42/4 (2005) 172-187.
This work was financially supported by the SFB “Electroactive Materials”, *Austrian Science Fund,* Vienna.

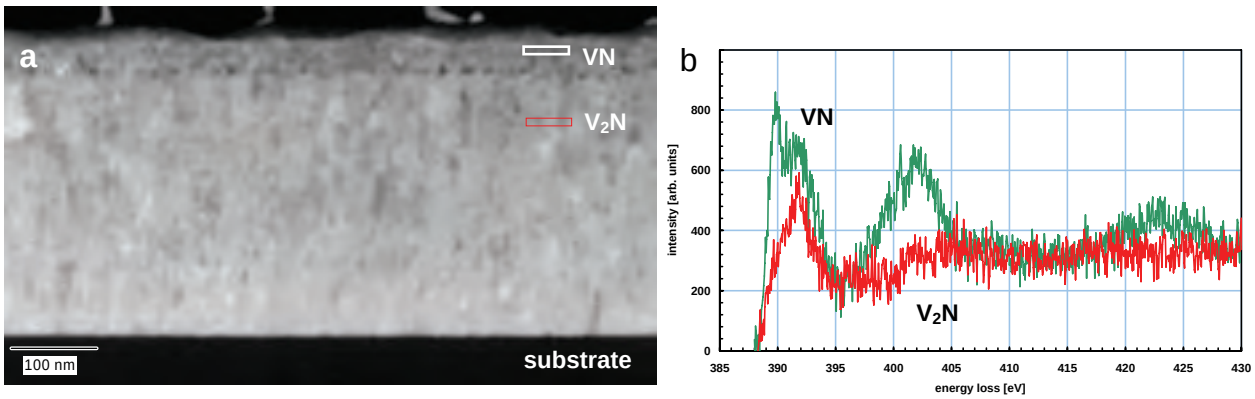


Fig.1 a. HAADF STEM image of VN_x coatings on silicon; b. ELNES of the N K ionization edge for VN and V_2N acquired with 0.27 eV energy resolution.

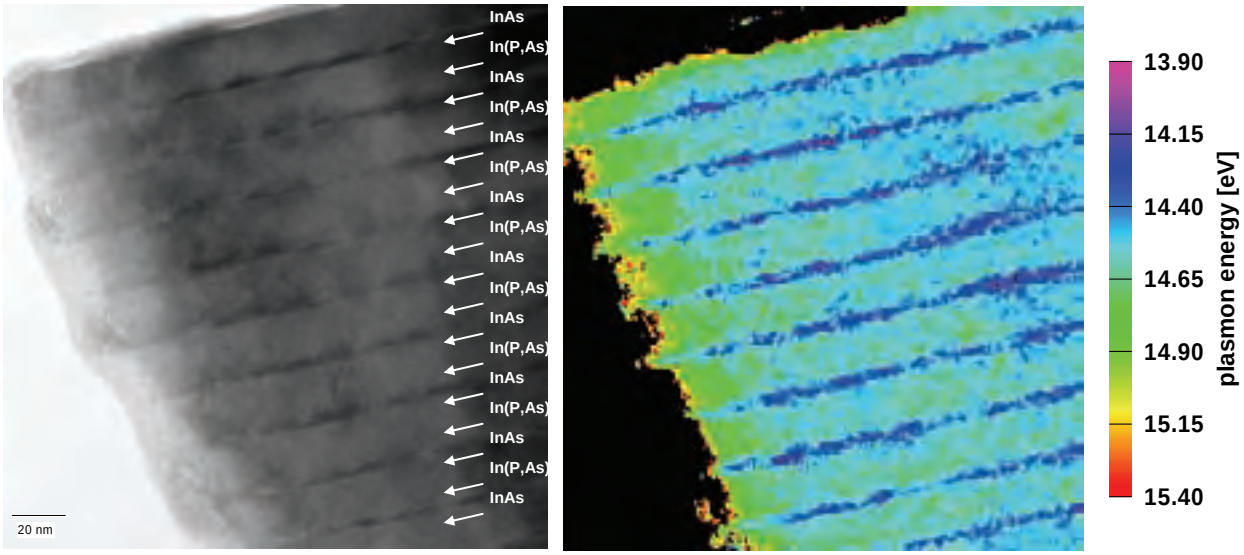


Fig.2 a. TEM bright field image of a InAs/In(P,As) multilayer; b. plasmon energy map calculated from a high energy resolution EFTEM series. The energy difference of the plasmons of InAs and In(P, As) is only 0.2 eV.

EELS Spectrum Imaging: The Next Steps

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

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

With the advent of energy-filtering transmission electron microscopes, in combination with fast two-dimensional EELS detection systems, more powerful acquisition and processing software and the enormous increase in computing power, there has been growing interest in exploiting the potential imaging capabilities of electron energy-loss spectroscopy for the comprehensive compositional and electronic characterisation of modern materials. EELS spectrum imaging has gained especially widespread acceptance as a tool that not only provides better mechanisms to cross-check the data quality but also allows for increased sophistication in processing the data compared to conventional 3-window elemental mapping or EELS point analysis.
The idea of EELS spectrum imaging is to collect an almost complete data set in the spatial and energy-loss dimensions. This can be achieved by either recording an entire energy-loss spectrum from each pixel in an image (STEM SI) [1] or by recording the spatial distribution of the intensity at each energy loss (EFTEM SI) [2]. Various forms of spectrum image data processing like thickness deconvolution [3], Kramers-Kronig analysis, MLS fitting for quantitative compositional image analysis [4,5] or automated elemental occurrence mapping [5] have already been successfully demonstrated. However, depending on the acquisition mode, most spectrum imaging applications so far were limited in energy resolution and/or field of view. With the latest generation of monochromated TEMs, providing energy resolutions of as low as 0.1-0.3eV [6,7], spectrum imaging has reached the next challenge level.
The EFTEM spectrum imaging approach has several advantages for applications in the low-loss region. Benefits include the fact that the imaged areas can be larger and the exposure times can still be kept relatively low even when the slit widths used are very small (~0.2eV) or the monochromator is switched on. Although current imaging filters are corrected up to 3rd and partly 4th order [8], higher-order spectral aberrations (non-isochromaticity) can become visible under these conditions, preventing narrow low-loss features to be mapped uniformly. Provided that there was no sample drift between the image slices, the contribution of energy warping to the individual image plane intensities can be determined and corrected. In the case of drift each image plane has its individual displacement vector, which shifts the respective non-isochromatic surface correspondingly. Therefore, a combined spectral aberration and spatial drift correction is not straightforward but always has to be carried out simultaneously, in order to avoid the introduction of additional artifacts. In this paper we will give an overview of recent EELS spectrum imaging applications, and present a new artifact correction scheme to comply with the higher resolution requirements of present-day applications.

References

[1] C. Jeanguillaume and C. Colliex *Ultramicroscopy* 28 (1989) 252.
[2] J.L. Lavergne et al *Microsc. Microanal. Microstruct.* 3 (1992) 517.
[3] P.J. Thomas and P.A. Midgley *Ultramicroscopy* 88 (2001) 187.
[4] M. Tencé et al *Ultramicroscopy* 58 (1995) 42.
[5] G. Kothleitner and F. Hofer *Ultramicroscopy* 96 (2003) 491.
[6] G. Kothleitner and F. Hofer *Micron* 34 (2003) 211.
[7] S. Lazar et al *Ultramicroscopy* 96 (3-4) (2003) 535.
[8] H. A. Brink et al *Ultramicroscopy* 96 (3-4) (2003) 367.

Special thanks to Dr. A. Trampert (P. Drude Institute Berlin, Germany) for providing the AlN/GaN sample and to the SFB “Electroactive Materials” for financial support.

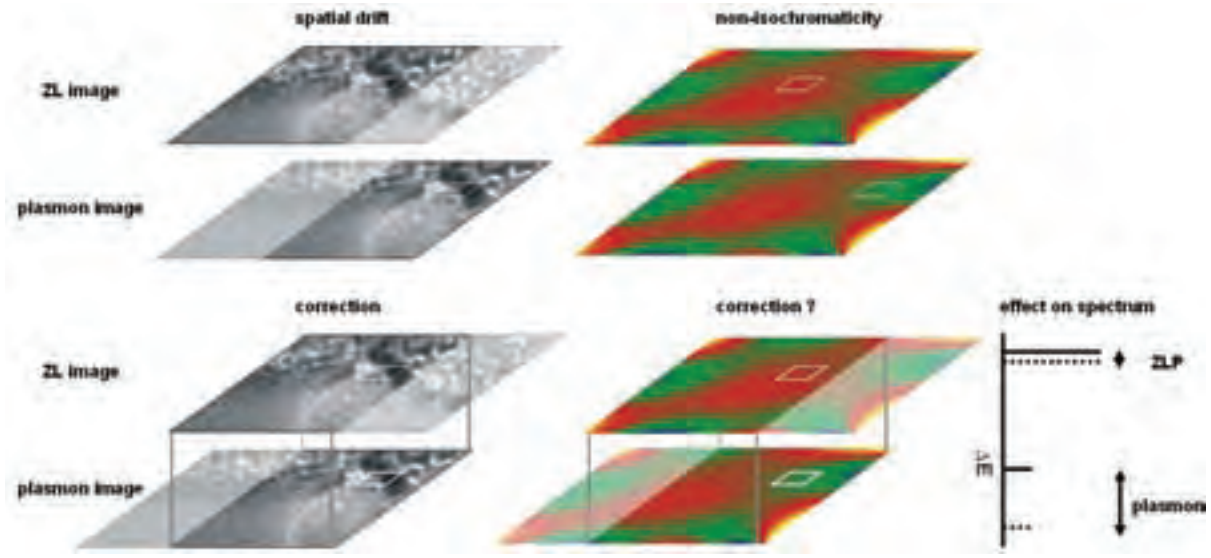


Fig.1. Graphics, showing the implications of simultaneous spatial drift and spectral aberration correction.

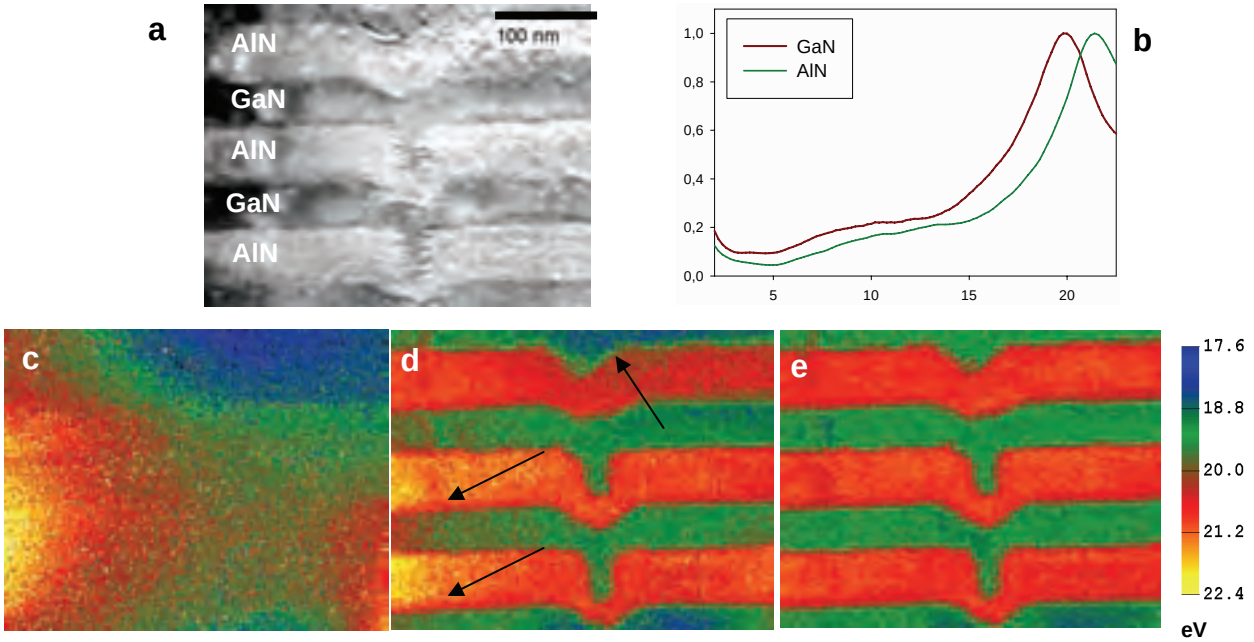


Fig.2a. ZL-filtered image of a layered AlN/GaN stack, b. corresponding plasmon spectra, c. spectral aberrations together with d. incorrectly and e. correctly calculated plasmon shift map.

EFTEM Spectrum Imaging – At the Limits of Energy Resolution

B. Schaffer, W. Grogger, G. Kothleitner

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

In general, spectrum imaging is a method of collecting an almost complete data set in both spatial and spectral dimension. Thus, an electron energy-loss spectrum image (EELS SI) is a three-dimensional data set containing EELS spectra for each point on the sample and energy-filtered images for each energy-loss value [1]. Accordingly, such a spectrum image can be acquired either by recording a series of spectra for each point of the sample in scanning mode (STEM-SI), or by combining a series of EFTEM images closely spaced on the energy-loss axis (EFTEM-SI). While STEM-SI usually yields data with excellent energy resolution but coarse spatial sampling or limited field of view, EFTEM-SI creates high quality image information at a limited energy resolution as shown schematically in Figure 1.
In our work we have shown that modern imaging filters with higher order aberration correction also allow energy resolutions less than 0.8 eV to be achieved in EFTEM-SI, if ultra-small energy selecting slit widths down to 0.1 eV are used. Figure 2 shows the theoretically obtainable resolution as a function of the selected slit width and the energy resolution performance of the system in spectroscopy mode. On the right side, an extracted spectrum from EFTEM-SI acquired with a 0.4 eV slit is directly compared to a spectrum of the same specimen acquired in spectroscopy mode. As can be seen, the detail of spectral information is similar.
Contrast in ratio images of energetically closely spaced spectral features depends strongly on the energy resolution as shown in Figure 3, which compares data acquired with three different slit widths. The two maps on the right show material contrast of GaN and AlN in such ratio images as a function of the energy loss of the two images concerned. It can be seen that highest contrast can be achieved with the smaller slit width of 0.4 eV and the two images extracted at the energy position of the GaN and AlN plasmon energy. For the larger slit width, contrast is lower and the two optimum energy positions have also shifted to higher values.
More importantly, the full spectral information at high energy resolution allows methods previously developed for EELS analysis to be applied also to map spectral features over a larger field of view at good spatial resolution.

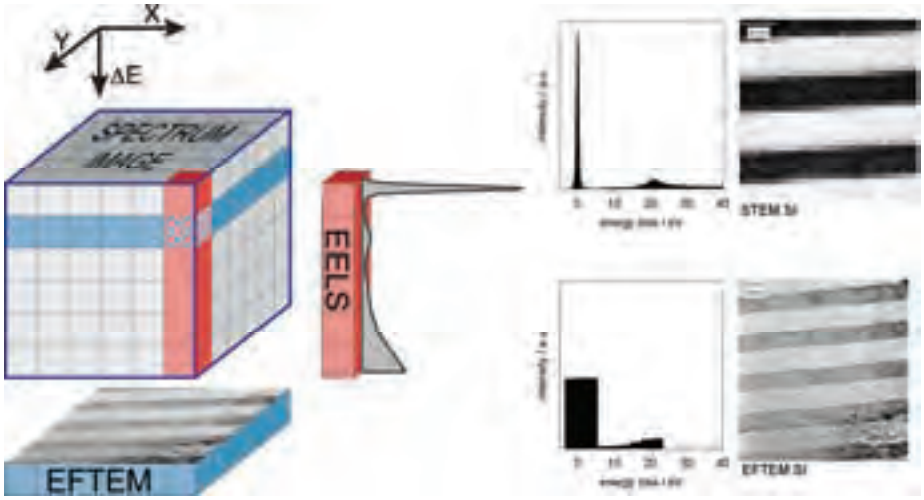


Fig.1 Schematics of EELS spectrum imaging, showing the 3 D data cube which can be acquired either column-by-column in scanning mode (STEM-SI) or slice-by-slice in energy-filtered mode (EF-TEM-SI).

For example, it becomes possible to map the energy position of volume and surface plasmon peaks and relate this with other physical properties of the sample [2,3]. The low-loss information can also be used to calculate the energy dependent dielectric constant of the sample via Kramers-Kronig transformation, and maps of corresponding properties can be generated with high spatial resolution.

References:
[1] J.L. Lavergne et al., Microsc. Microanal. Microstruct. 3 (1992) 517.
[2] W. Sigle et al., Ultramicroscopy 96 (2003) 565.
[3] H.R. Daniels et al., Ultramicroscopy 96 (2003) 547.
We gratefully acknowledge financial support by the *Fonds zur Förderung der wissenschaftlichen* Forschung, *Vienna, Austria* within the Special Research Program *Electroactive Materials.*

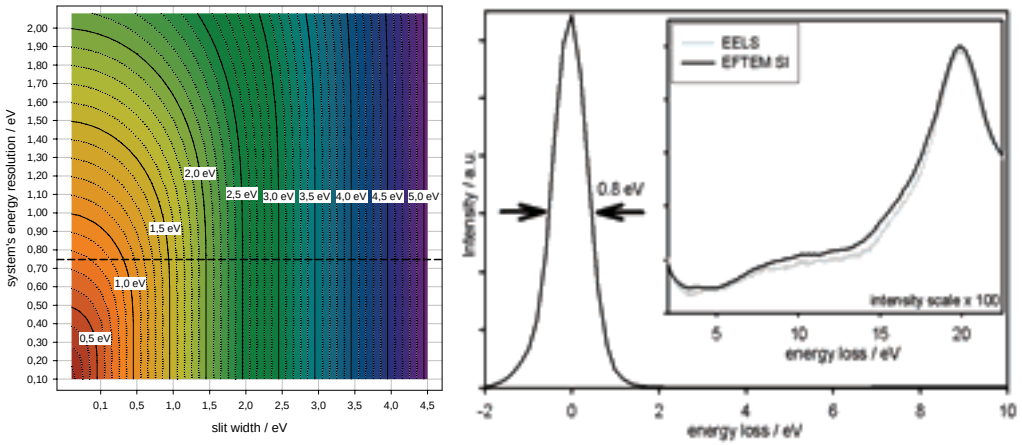


Fig.2 Achievable energy resolution in an EFTEM SI as a function of the energy-filtering slit width and the system energy resolution; comparison of a GaN EEL spectrum acquired in spectroscopy mode with a spectrum extracted from EFTEM-SI.

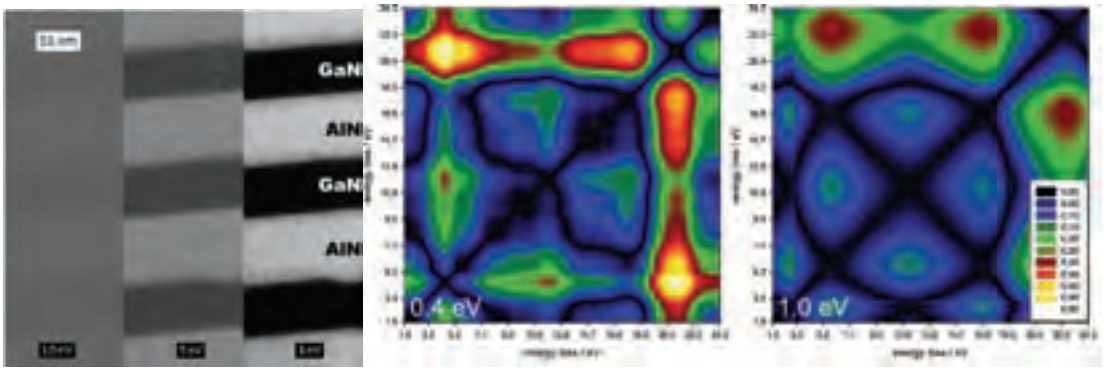


Fig.3 Material contrast between AlN and GaN in a plasmon-loss ratio image for different energy selecting slit widths; and material contrast as a function of energy-loss of the two images used for the ratio image, using a 0.4 eV and 1.0 eV slit width, respectively.

Precipitation in 9-12% Cr Steels

F. Méndez**, M. Albu*, B. Sonderegger**, S. Mitsche*
* Institute for Electron Microscopy and Fine Structure Research; Graz University of Technology & Zentrum für Elektronenmikroskopie Graz, Steyrergasse 17, 8010 Graz
** Institute for Materials Science, Welding and Forming Graz, University of Technology, Kopernikusgasse 24, 8010 Graz

Microstructure and precipitation in tempered martensitic 9-12% chromium steels for steam power plants are mainly determined by the chemical composition and heat treatment parameters. During the service life the microstructure of these materials changes: grains coarsen and precipitates form, grow or dissolve. These changes in the microstructure decrease the creep properties and limit the increase of the operating steam temperature. The microstructure evolution after long creep tests of new 9-12% chromium steel is the study subject of this paper. Misorientation of the boundaries has been measured using electron backscatter diffraction (EBSD) (fig.1). For precipitate studies transmission electron microscopy (TEM) was used. The microstructural information is complemented by compositional information obtained with integrated spectroscopic techniques such as energy dispersive X-ray spectroscopy (EDXS) (see fig. 2) and electron energy loss spectroscopy (EELS). Both EELS and EDXS spectra represent unique fingerprints of the precipitates being analysed, if their chemical composition significantly differ [1]. Using energy-filtered TEM, EELS and XEDS different phases present in this kind of steels: MX, M_2X, $M_{23}C_6$, BN, modified Z-phases and Laves phases, were identified [1](see figs. 3a, 3b and 3c).
Two different sample preparations have been applied: In order to analyse of the chemical composition, extraction replicas were prepared, where the particles are lying on an amorphous carbon film. Secondly, ion milled samples have been prepared for the study of the size and phase fraction of $M_{23}C_6$, MX and M_2X and the results were evaluated by stereological methods[2]. The investigated material shows a tempered martensite matrix structure. In the as-received condition MX [(V,Nb)(N,C)], M_2X [Cr_2N], $M_{23}C_6$ [$(Cr,Fe,Mo,W)_{23}C_6$] and BN phases have been identified at the boundaries and/or in the matrix. After thermally ageing and creep loading, two new phases form: laves phase (intermetallic phase A2B with high amount of W and Mo) and modified z-phase (complex nitride [(Cr,V,Nb)N]). The z-phase is associated with dissolution of MX carbonitrides and also the breakdown in long term strength in 9-12%Cr steels. The chemical composition of the different precipitates phases has been quantified via EDXS. These measurements have been carried out on the extraction replica specimens (where no matrix signal is present). $M_{23}C_6$ were found rich in Cr (58 at%), Fe (15 at%), W (5 at%) and traces of Mo and V. MX were found rich in V (38at%), Cr (10at%) and between 1-2 at% of Fe and Nb. M_2X were found rich in V (15 at%), Cr (43 at%) and Fe (6 at%). The modified Z-phase was found rich in V (29 at%), Cr (34 at%), Fe (3 at%) and Nb (1.5 at%). The size of the precipitates, $M_{23}C_6$, MX and M_2X, was increasing slightly during creep loading and thermally ageing. Furthermore, calculations for the phase fraction of the M_2X and MX have been done. While the phase fraction of the MX particles decrease in the specimens where the mod. Z-phase appears, the M_2X phase stays unaffected.
Although the present results already give much information on the microstructural evolution, more measurements have to be carried out. One specific point of interest is the interaction between the nitrides (MX, M_2X and mod. Z-phase): one of the main strengthening mechanisms in these materials is precipitate strengthening by MX particles; and the breakdown of creep strength could be associated with the formation of modified z-phase the cost of MX particles, which dissolve.

[1] Warbichler, P., Hofer, F., Hofer, P., Letofsky, E., Micron 29 (1998) 63.
[2] B. Sonderegger, Ultramicroscopy 106 (10) (2006) 941.
[3] H.K. Danielsen and J. Hald, Energy Materials 1 (2006) 49.
The authors wish to acknowledge Manuel Paller and the laboratory group for the specimen preparation.

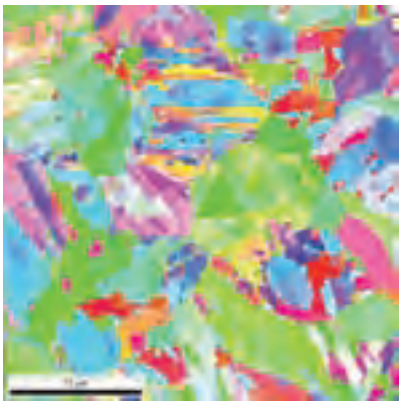


Fig.1 EBSD micrograph. The colours indicate the crystal orientations.

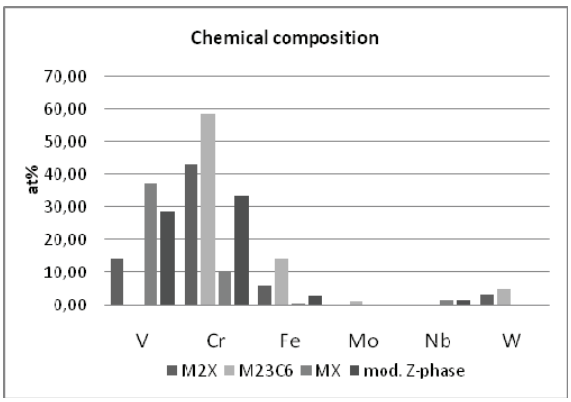


Fig.2 Chemical composition for the different precipitates phases present in the material.

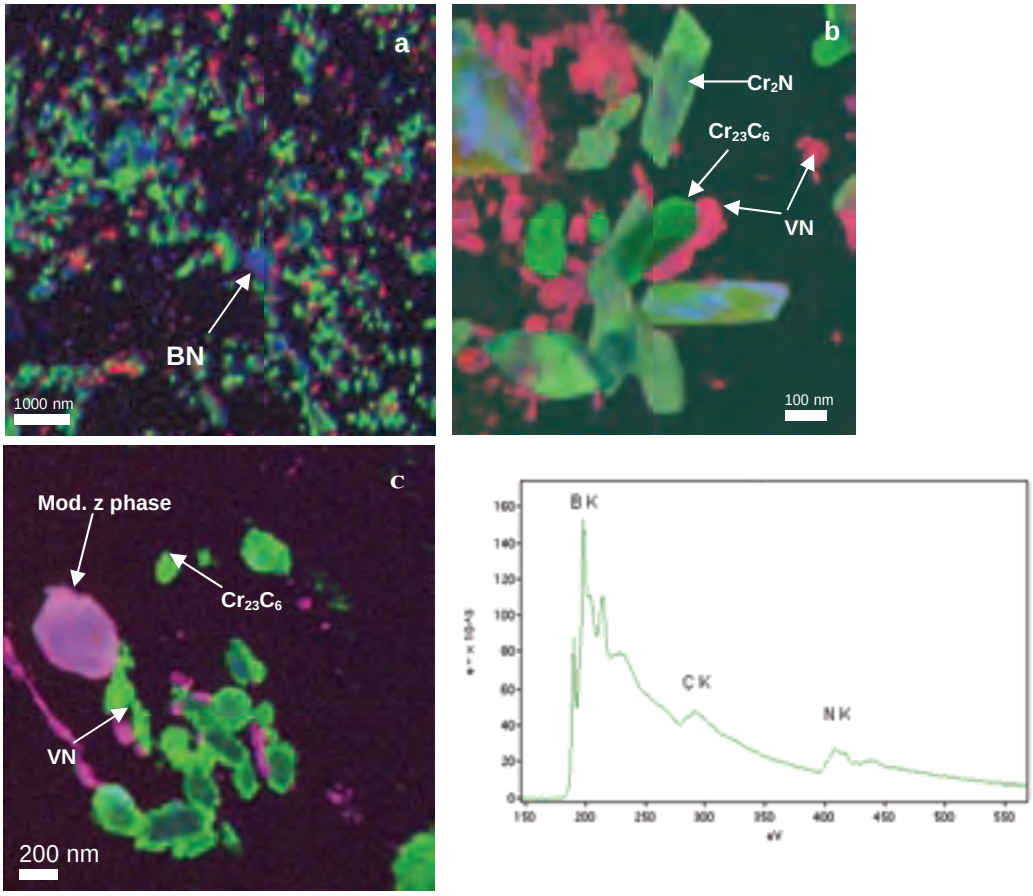


Fig.3 Elemental maps recorded with energy-filtered TEM imaging; a) RGB map for the as-received sample (green = Cr, blue = B, red = V), b) RGB map for a thermally aged specimen (green = Cr, blue = N, red = V), c) RGB map for a creep loaded specimen (green = Cr, blue = N, red = V), d) EELS of BN.

Superstructure and Domains in $La_{0.4}Sr_{0.6}CoO_{2.71}$

C.Gspan[a], W.Grogger[a], I. Letofsky-Papst[a], F. Hofer[a], E. Bucher[b] and W. Sitte[b]

[a] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology & Centre for Electron Microscopy Graz, Steyrergasse 17, 8010 Graz
[b] Department of Physical Chemistry, University of Leoben, 8700 Leoben, Austria

Due to their high ionic conductivity, strontium-substituted lanthanum cobaltites $La_{1-x}Sr_xCoO_{3-\delta}$ (LSCO) have potential applications as high temperature fuel cell cathodes, gas separation membranes or oxygen sensors. In the heavily Sr-doped materials the ionic conductivity obtained from galvanostatic polarisation experiments shows a distinct maximum at high oxygen deficiency ($3-\delta = 2.77$) [1]. This behaviour may be explained by oxygen vacancy ordering which will decrease the mobility of the vacancies.

In this study we have investigated $La_{0.4}Sr_{0.6}CoO_{3-\delta}$ powders, which were synthesised by the glycine nitrate process [2]. Two powders with different oxygen concentrations were compared: LSC ($3-\delta \approx 3$) and LSC+ ($3-\delta = 2.71$). X-ray diffraction (XRD), transmission electron microscopy (TEM) and electron diffraction (SAED) revealed significant structural differences between these two samples. In the case of LSC+ we found additional diffraction reflections in XRD and SAED which cannot be found in LSC.

Another difference between LSC and LSC+ is the occurrence of domains in LSC+ samples, which can be seen in TEM bright and dark field images (Fig.1). High resolution TEM of the boundary between two domains in LSC+ showed two possible configurations, e.g. one type of domain boundary with a superstructure in both domains rotated by 90° (Fig.2), which corresponds to the additional diffraction reflections in XRD and SAED in the [100] zone axis (Fig.3). Although several authors have tried to explain these superstructure reflections [3-5], it has not been possible to “visualise” these reflections in an electron diffraction simulation until now.

However, a detailed symmetry analysis of convergent beam electron diffractions (CBED) of LSC+ in various crystal orientations (Fig.4) showed that the point group is *4/mmm* with a tetragonal crystal system. This result leads to a perfect agreement between electron diffraction simulation and the experiment (superstructure), but can also explain the additional peaks in the XRD pattern. The structure model with the space group *I4*/mmm (No.139) is shown in Fig.5, which agrees with the superstructure proposed for $La_{0.4}Sr_{0.6}CoO_{3-\delta}$ by Van Doorn et al., where the mixing oxygen anions (vacancies) are only present in every second (001) (La/Sr)O plane.

References:

[1] E.Bucher, W.Sitte, I.Rom, W.Grogger, F.Hofer, Solid State Ionics 152-153 (2002) 417
[2] L.A.Chick, L.R.Pederson, G.D.Maupin, J.L.Bates L.E.Thomas, Mater. Lett. 10 (1990) 6
[3] Z.L.Wang, J.Zhang, Physical Review B54 (1996) 1153
[4] S.Malo, A.Maignan, Inorg. Chem. 43 (2004) 8169
[5] Z.L.Wang, J.Bentley, N.D.Evans, Micron 31 (2000) 355
[6] R.H.E van Doorn; A.J. Burggraaf, Solid State Ionics128 (2000) 65.

We gratefully acknowledge financial support by the *Fonds zur Förderung der wissenschaftlichen* Forschung, *Vienna, Austria* within the Special Research Program *Electroactive Materials.*

Fig.1 TEM bright field image of a LSCO+ crystal.

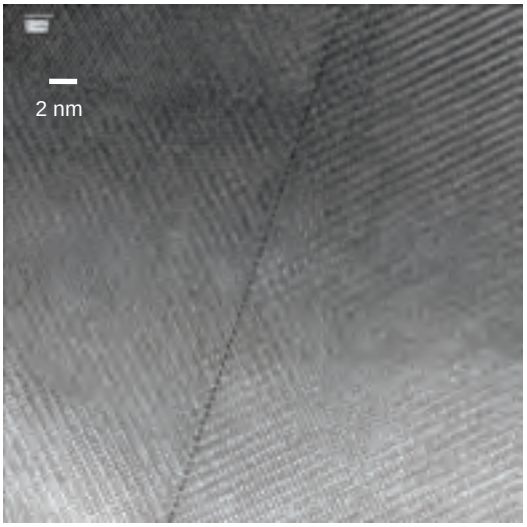


Fig.2 HRTEM image of a domain boundary in LSC+.

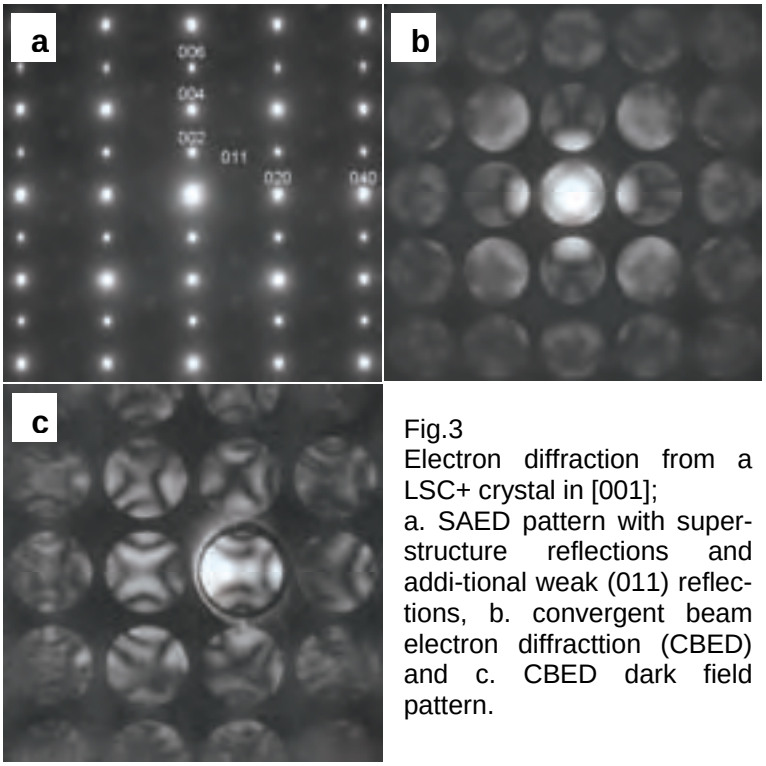


Fig.3
Electron diffraction from a LSC+ crystal in [001];
a. SAED pattern with super-structure reflections and addi-tional weak (011) reflec-tions, b. convergent beam electron diffracttion (CBED) and c. CBED dark field pattern.

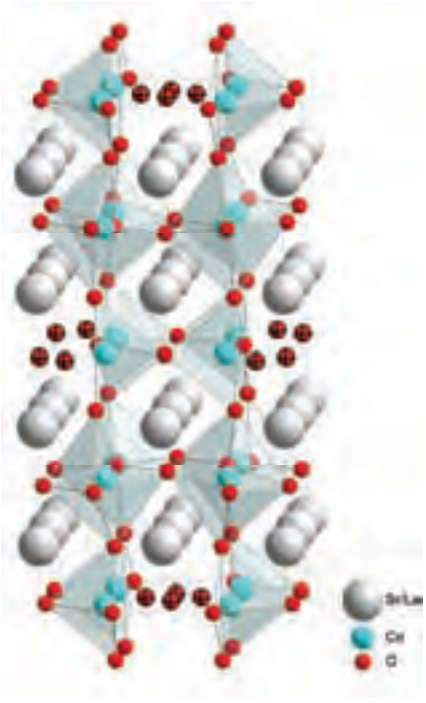


Fig.5 Structure model of LSC+ $La_{0.4}Sr_{0.6}CoO_{3-\delta}$ with (3-δ = 2.71), possible oxygen vacancies are shown with black crosses.

TEM Characterization of Optoelectronic Devices Based on Conjugated Polymers: Can FIB Specimen Preparation Help?

E. Tchernychova[1], W. Grogger[1], M. Sezen[1], P. Poelt[1], E. Fisslthaler[2], E.J.W. List[2]

[1] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology & Centre for Electron Microscopy Graz, Steyrergasse 17, 8010 Graz
[2] Institute of Solid State Physics, Graz University of Technology, Petersgasse 16, A-8010 Graz, Austria

The precise characterisation of novel material properties on nano- and atomic scales has always been of vast importance for the further refinement of the performance of optoelectronic devices based on organic semiconductors. The development of such multilayer thin-film devices consisting of materials with different properties requires a detailed understanding of the electronic properties and structure at the heterogeneous metal-contact/organic-layer interfaces. These issues are usually investigated by various experimental methods, among which transmission electron microscopy (TEM) is especially powerful for studying the morphology, crystallography, chemical composition and electronic properties at nanometer scale. In order to investigate the specific heterogeneous interfaces in the device, a focused ion beam (FIB) cut technique can be applied for TEM specimen preparation. However, specimen preparation might become a crucial issue, since a number of organic materials are highly sensitive to the heat and/or ion or electron beam treatment. The aim of the study was to optimise a specimen preparation method for organic optoelectronic devices in such a way as to be able to investigate the polymer's dielectric function in TEM directly.

In the present work, conjugated polymer based field effect transistors (OFET) and OFET-like test structures based on a poly(3-hexylthiophene) (P3HT) were investigated using conventional TEM (CTEM), high-resolution TEM (HRTEM), energy-filtered TEM (EFTEM), and electron energy-loss spectroscopy (EELS) techniques. TEM investigations were carried out using a Philips CM20 (LaB_6) microscope operated at 200 kV and equipped with Gatan Imaging Filter (GIF) and 1k×1k CCD camera, and an FEI Tecnai F20 (field emission gun) microscope operated at 200 kV and equipped with a monochromator, STEM unit, high-resolution GIF, 2k×2k CCD camera, and high-angular annular dark field (HAADF) detector (Fischione). The TEM specimens were prepared in the FIB by cutting out lamellae with thicknesses of about ~100 nm using different FIB current settings. The as-deposited P3HT reference specimens were spun over the NaCl crystal, which was later dissolved in water and the pieces of P3HT were caught onto regular TEM Cu grids.

Fig. 1 shows a CTEM micrograph of the OFET test structure. The Au/P3HT interface was observed to be diffuse due to the roughness of the polymer surface, on which Au was sputtered. It was found that the deposition of a protective Pt coverage using first electron-assisted deposition followed by general ion-assisted deposition prevents major damage to the polymer and the destruction of the Au layer. The complete picture of FIB specimen preparation damage was monitored by observing the π* peak in the C-K edge of EELS spectra. The comparison of the spectra, taken from the FIB lamella polymer to the as-deposited P3HT reference spectra acquired under the same irradiation conditions reveals the degraded π* peak (decreased intensity) in the FIB lamella spectrum (Fig. 2a,b). Such degradation occurs due to the destruction of the conjugated *sp2* bonds in polymers and changes in their electronic properties [1]. The polymer layer structure at the atomic level was additionally studied with HRTEM, however, no great differences between the FIB lamella and the P3HT standard specimens were observed. The results obtained have demonstrated that further optimisation of FIB specimen preparation is required prior the determination of the dielectric function of the polymer in TEM by means of valence EELS. Moreover, a fabrication of the OFET structure using soft substrates suitable for TEM specimen preparation with ultramicrotomy might be an alternative to overcome the observed degradation processes.

References
[1] Bruley J., Williams D.B., Cuomo J.J., Pappas D.P. *J. Microsc*. **180** (1995) 22-32
[2] B. Schaffer C. Mitterbauer, A. Schertel, A. Pongatsch, S. Rentenberger, E. Zojer, F. Hofer, Ultramicroscopy 101 (2004) 123-128.
This work was performed within the "ISOTEC" project funded by the Austrian Nanoinitiative. We kindly acknowledge Dipl. Ing. Andreas Klug for providing an OFET specimen for TEM investigations.

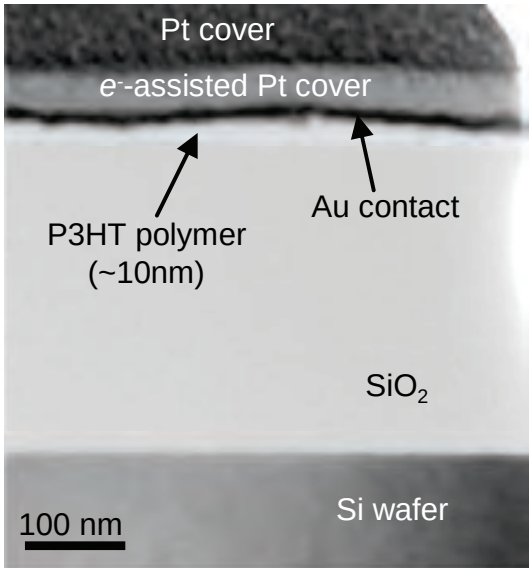


Fig.1 TEM image of FIB lamella, cut out of the OFET structure. Pt electron-assisted deposition performed prior to a regular ion-assisted deposition prevents the major damage of Au and P3HT layers.

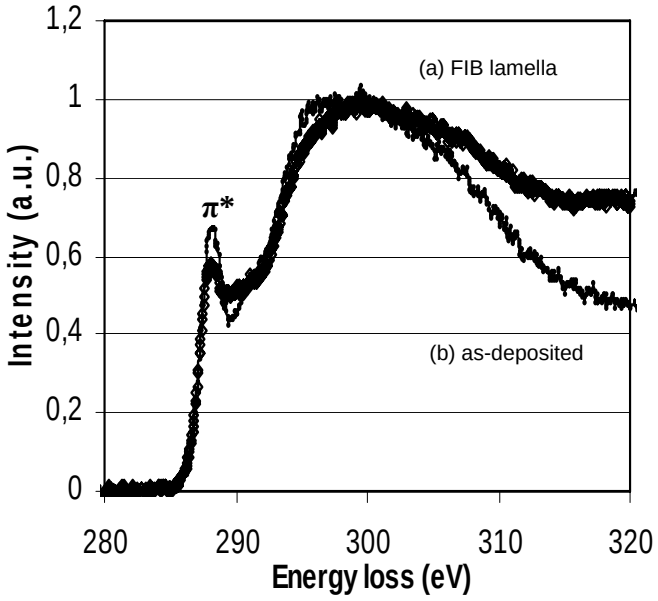


Fig.2 C K edge EELS spectra taken from the P3HT polymer: (a) from the FIB-prepared TEM lamella and (b) from as-deposited polymer film reference. A significant difference at the region of π* peak in both spectra can be observed.

Characterisation of fibrillar and pore structure in natural and man-made cellulose fibres by modern imaging techniques

K. Christian Schuster[1], Mohammad AbuRous[2], Uta Schädel[2], Peter Hosemann[2,3], Christian Teichert[3], Elisabeth Ingolic[4], Roger N. Ibbett[2], Thomas Bechtold[2]

[1] Lenzing AG, Textile Innovation, Lenzing, Austria
[2] Christian Doppler Laboratory for Textile Chemistry in Cellulosics, Inst. of Textile Chemistry and Textile Physics, Leopold Franzens Universität Innsbruck, Dornbirn, Austria
[3] Institute of Physics, Montanuniversität Leoben, Leoben, Austria
[4] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology & Centre for Electron Microscopy Graz

Textiles made from TENCEL® cellulosic fibres provide special functional and wellness properties: a cool and smooth surface which is gentle to the skin, improved wearing comfort properties due to excellent moisture transport and buffering, support to the body's temperature control system, good electrostatic properties and reduced odour formation and bacterial growth on the textile [1,2]. (TENCEL® is the brand name for fibres of the Lyocell type by Lenzing AG, Austria.) These properties are attributable to the special structure of Lyocell fibres – the high crystallinity and orientation [3], but especially the architecture of the fibre consisting of fibrils of various sizes, enclosing pores, and surrounded by a smooth skin [2,4,5,6], which is radically different from other man-made cellulosics.
Earlier studies have shown that the pore structure depends on the spinning and treatment parameters [6-8]. Following a crystallisation model of cellulose out of NMMO solution, the fibre cross-section contains a compact core, a porous middle zone and a semi-permeable fibre skin. Classical analysis methods such as scattering techniques deliver quantitative information about the pore structure and even the pore size distribution [4,7,8], but not much information on their spatial distribution.
This work is mainly focussed on visualising the Lyocell structure consisting of fibrils and pores, in relationship with the fibre physical and chemical properties. Scanning electron microscopy of broken fibres reveals the fibrillar structure in the dry state. Atomic force microscopy of fibre surfaces expands the resolution of imaging into the nanometer range [10].
In the wet state, we applied probe molecules and subsequent fluorescence microscopy to fibre cross-sections. Different porous zones were discriminated, partly confirming the model mentioned above [11]. Transmission electron microscopy (TEM) was applied to visualise the fine pore network structure in detail. It was necessary to overcome the problem of the low electron contrast of cellulose material, which does not accept common staining. Here we adapted a contrast-enhancing method to several cellulosics, and for the first time visualised the pore structure of Lyocell and Modal in overview and in fine detail down to the nanoscale.

References

[1] K.C. Schuster, H. Firgo , F. Haussmann , J. Männer, *Proc. of the Textile Institute 83rd World conference*, Shanghai, May 2004 , 422 – 426 (2004)
[2] J. Männer, K.C. Schuster, F. Suchomel, A.Gürtler, H.Firgo, *Lenzinger Berichte* 83 (2004), 99-110
[3] J. Lenz , J. Schurz , E. Wrentschur, *Acta Polymer.* 43, 307-312 (1992)
[4] K.C. Schuster, P. Aldred , M. Villa, M. Baron, R. Loidl, O. Biganska, S. Patlazhan, P. Navard, H. Rüf, E. Jericha , *Lenzinger Berichte* 82, 107-117 (2003)
[5] Schuster K.C. et al., Environmentally Friendly Lyocell Fibres, in: Natural Fibres, Plastics and Composits; ed.: F.T. Wallenberger and N.E. Weston, Kluwer Academic Publishers 2003; 123-146
[6] J. Zhang., H. Zhu, M. Shi, K. Lai.; *Chemical fibres international* 49, 494 (1999)

[7] M.E. Vickers, .N.P. Briggs, R.N. Ibbett, J.J. Payne, S.B. Smith, Polymer 42, 8241 (2001)
[8] K. Bredereck, F. Hermanutz, Rev. Prog. Color. 35, 59 (2005)
[9] O. Biganska; *Dissertation,* L'Ecole Nationale des Mines de Paris; France, December 2002
[10] P.Hosemann, C.Teichert, K.C.Schuster, Seeing at the Nanoscale, Santa Barbara, USA 13.-18.08.2005
[11] M. Abu Rous, K.C. Schuster, *Lenzinger Berichte*; 83, 92 - 98 (2004)

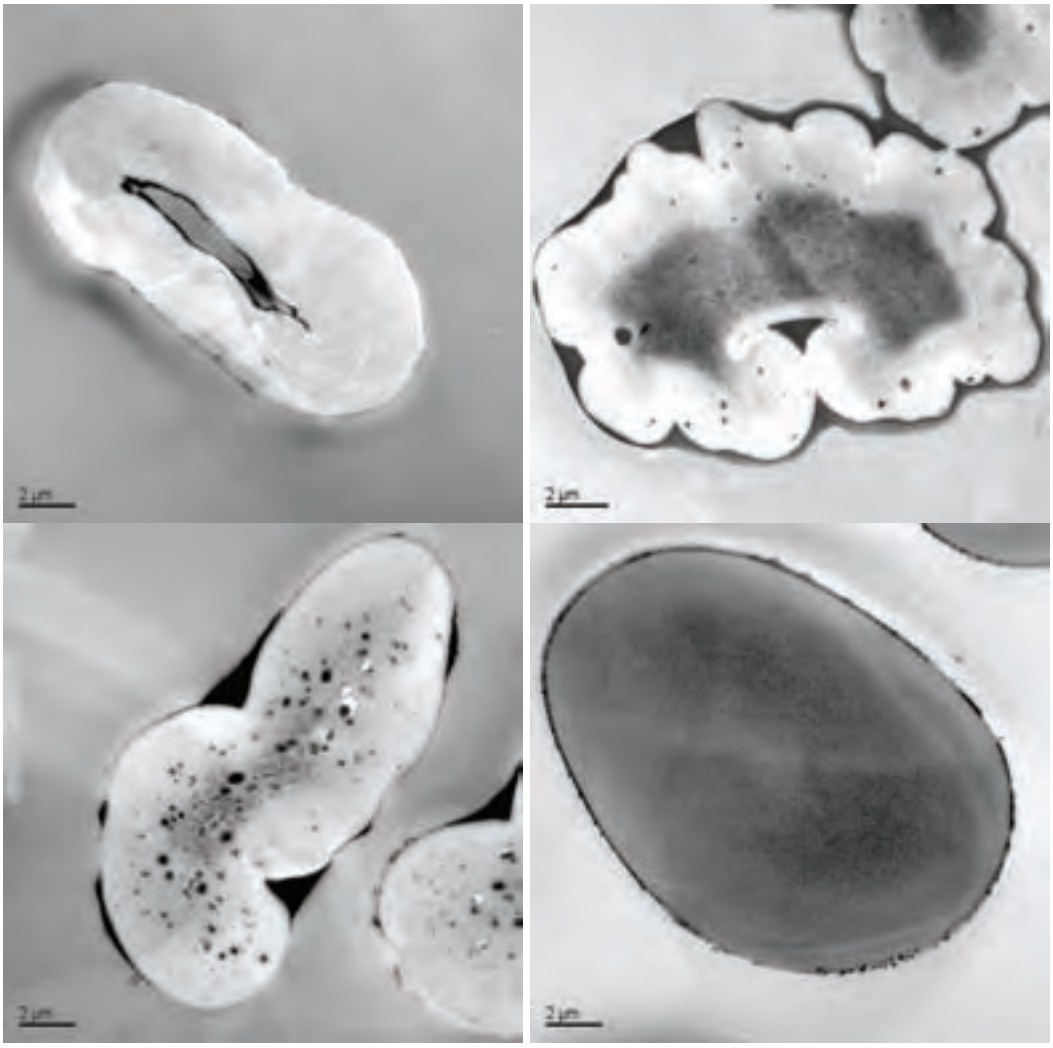


Fig.1 The amount and the distribution of water within the fibre structure can vary remarkably from one cellulose fibre type to another. Cotton (a), Lenzing Viscose® (b), Lenzing Modal® (c) and TENCEL® (d) show big differences in the amount of water, the distribution of water over the fibre cross section and the pore size as well as pore distribution.

Bacterial Spores in the Skin of a 1000-Year-Old Peruvian Mummy

M.A. Pabst[1], I. Letofsky-Papst[2], E. Bock[1], K.Spindler [3], S. Guillén[4], F. Hofer[2]

[1] Department for Cell Biology, Histology and Embryology, Medical University of Graz, A-8010 Graz
[2] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology, 8010 Graz
[3] Institute for Pre-and Protohistory, University of Innsbruck, A-6020 Innsbruck, Austria
[4] Centro Mallqui, Ilo, Peru

The skin of a 1000-year-old mummy, found in the necropolis of Chiribaya Alta in South Peru, was studied. The mummy was found unwrapped, lying in the sand of the desert. Because of the hot and dry climate the dermis was well preserved, where we found numerous bacterial spores (Fig.1). Bacterial spores are dormant stages of microorganisms, which are formed in response to starvation and can withstand extreme conditions of heat, radiation, toxic chemicals, dehydration and time [1].
For the transmission electron microscopy investigation, samples were rehydrated and fixed in a mixture of 3% glutaraldehyde and 3% paraformaldehyde in 0.1 M cacodylate buffer for 3 days at 4° C, followed by 2 hours fixation with 2% OsO_4 at room temperature before being embedded in TAAB epoxy resin. Ultrathin microtome sections, 30 to 60 nm thick, were mounted onto Cu grids. The sections were used for morphological investigations and for elemental analyses. The latter usually were not stained with heavy metal compounds because this staining could interfere with some elements in elemental analysis. The ultrathin sections were analysed in a Philips CM20 (TEM-STEM) using electron energy-loss spectrometry (EELS, Gatan Imaging Filter) and energy dispersive x-ray spectroscopy (EDXS, Noran HPGe).
The TEM investigation of these spores exhibits a core, a cortex, a lamellar inner and an outer spore coat (Fig.3a). At the border of the cortex and the inner spore coat, electron dense patches were observed frequently, which were analysed using EDXS (Fig.3b). These structures consist of carbon, oxygen, iron, aluminium, phosphorus, calcium and zinc, or in other patches silicon instead of zinc. In the dense core, EDX analysis revealed carbon, nitrogen, oxygen, silicon, phosphorus, calcium, and low concentrations of sulphur (Fig.3c). The EELS spectrum (Fig.3d) shows that the fine structure of the P-$L_{2,3}$ edge is typical of a phosphate [2]. Phosphate, as a constituent of DNA, is localised in this area of the spores as well as carbon, oxygen and nitrogen indicating the presence of proteins. Especially in the spore coat higher amounts of sulphur were found along with proteins. Here, a cystein rich protein is described, whose disulfide bonds are responsible for their radiation resistance. Calcium was detected by x-ray spectrometry (EDXS) in all three layers of the bacterial spores. Large amounts of dipicolinic acid are known to be present as Ca chelate in bacterial spores, which is thought to be the reason for heat resistance.
The bacterial spores found in the dermis seem to belong to the spore-forming genera Bacillus or Clostridium [3]. In addition we found spores of another bacterial species on the surface of the dermis which revealed a special surface structure. Closely packed ridges extend along the longitudinal axis of the spore and a kind of collar can be seen at one end (Fig.2).
Since we have only found spores on the mummy, one can assume that when the corpse dried out, the “nutritional source” for the bacteria was no longer present and as a result they formed spores, which were preserved for 1000 years along with the mummy.

References:
[1] Ingraham JL, Ingraham CA, Prentiss H., Introduction to Microbiology (1995) USA: Wadsworth Publishing Company
[2] Hofer F, Golob P, Ultramicroscopy 21 (1987) 379-384
[3] Perrin C, Nolly V, Mourer R, Schmitt D, Ann Dermatol Venerol, 121 (1994) 470-475

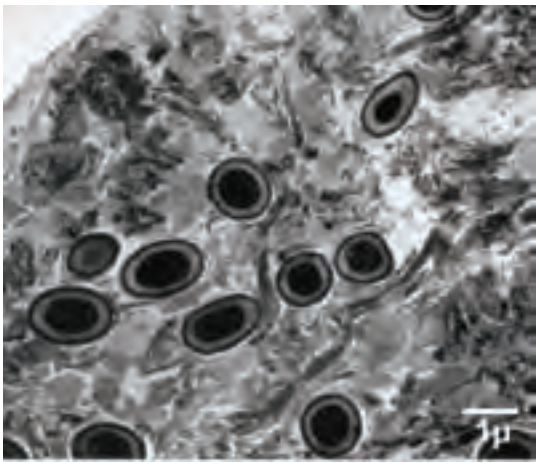


Fig.1 TEM image of bacterial spores, ultra-microtome section.

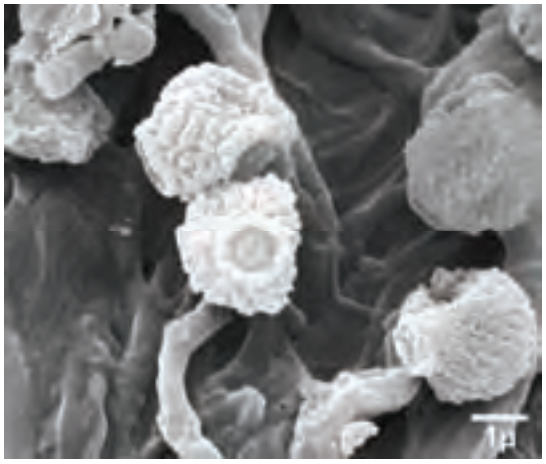


Fig.2 SEM image of bacterial spores showing a kind of collar on one end.

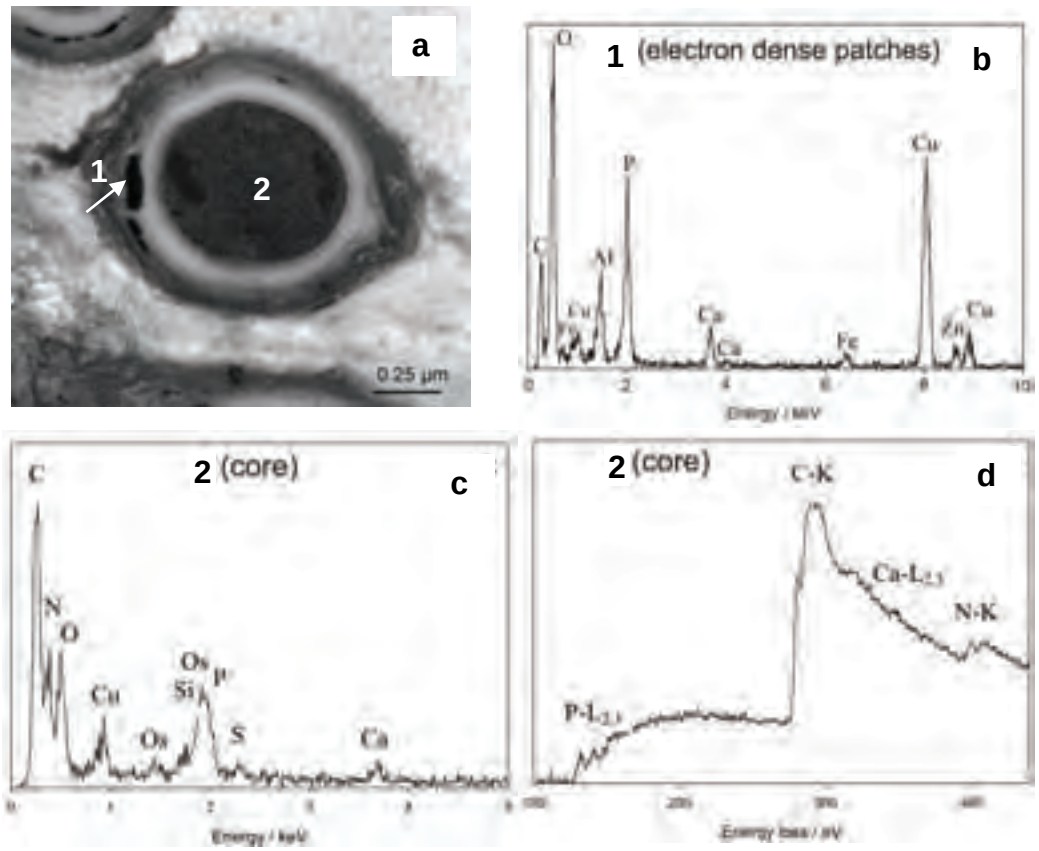


Fig.3 a) TEM image of a bacterial spore, b) EDX-spectrum from electron dense patch, c) EDX-spectrum from core, d) EELS-spectrum from core.

Quasicrystalline phase in melt-spun Al-Mn-Be ribbons

F. Zupanič[1], T. Bončina[1], W. Grogger[2], C. Gspan[2]

[1] University of Maribor, Faculty of Mechanical Engineering, Smetanova 17, SI-2000 Maribor
[2] Institute for Electron Microscopy and Fine Structure Research, Graz University of Technology, Steyrergasse 17, A-8010 Graz, Austria

Quasicrystals are solids with a highly ordered but non-periodic arrangement of atoms. They have been studied very intensively since their discovery, so that today many interesting properties of these materials are known. For example, quasicrystals can be used in the form of reinforcing particles in aluminium based alloys. Recently, it was found that beryllium strongly increased the quasicrystalline forming ability in well-known Al-Mn quasicrystalline alloys.
Based on this, we investigated the quasicrystalline phase in a melt-spun Al-Mn-Be alloy (Al - 6 wt% Mn – 1.5 wt. % Be) with the aim of determining the role of beryllium. Specimens for transmission electron microscopy (TEM) were cut out at specific sites of the alloy using the focused ion beam (FIB) in an FEI Nova 200 Nanolab. The TEM investigations were performed with an analytical high resolution TEM (FEI Tecnai F20), equipped with a high resolution energy filter (HR-GIF Gatan) and an energy dispersive x-ray spectrometer (EDXS, EDAX Si(Li)). Electron energy-loss spectroscopy (EELS), energy-filtering TEM (EFTEM) and high-resolution transmission electron microscopy (HRTEM) were done on an FEI Tecnai F20.
Figure 1 shows an individual quasicrystalline particle with corresponding electron diffraction patterns with twofold, threefold and fivefold symmetry axes. The position of the most important diffraction spot (211111) in the Elser's indexing scheme indicated that the quasicrystalline phase has a primitive icosahedral structure. The HRTEM image of a quasicrystalline particle taken in a fivefold symmetry axis is shown in Fig.2. It is possible to see spots of different brightness forming five sets of virtual lines with a mutual orientation of 72^{o}. By drawing an appropriately oriented pentagon, all virtual lines appeared parallel to the sides of the pentagon, which is completely consistent with the icosahedral fivefold symmetry and the Fast Fourier Transform (FFT).
The chemical composition of the matrix and particles of the quasicrystalline phase as well as elemental distributions were determined using EDXS, energy-filtering TEM (EFTEM), and Auger electron spectroscopy (AES). The results of EDXS and EFTEM showed that manganese is concentrated in the quasicrystalline phase (Fig. 3), whereas both methods failed to detect beryllium. We used AES analyse many particles of around 500 nm in diameter and detected beryllium in the quasicrystals with concentrations between 30 and 40 at%. EDXS in TEM revealed a Mn/Al weight ratio of around 0.4 in the particles and 0.02 in the matrix. The latter result indicates that in the melt-spun ribbons the Al-rich solid solution was supersaturated with Mn (its equilibrium maximum solubility is 1.25 %).
Both Be and Mn are enriched in the quasicrystalline particles, but it seemed that the electron-to-atom ratio remained close to that of the binary $Al_{80}Mn_{20}$ quasicrystal. This confirmed that the increased quasicrystal forming ability of Al-Mn alloys containing beryllium can be attributed to its presence in the quasicrystalline phase.

References

[1] A. Spaič, S. Markoli, Boštjan, I. Anžel, W. Grogger, F. Zupanič, Proceedings 7th Multinational Congress on Microscopy, June 26-30, 2005, Portorož, Slovenia, pp. 379-380.
[2] F. Zupanič, T. Bončina, A. Križman, W. Grogger, C. Gspan, B. Markoli, J. Alloys Compd. (in print).

Fig.1. a. TEM micrograph of an individual quasicrystalline particle in Al-rich matrix. SAED-patterns taken along b. two-, c. three- and d. fivefold axis of the quasicrystalline particle.

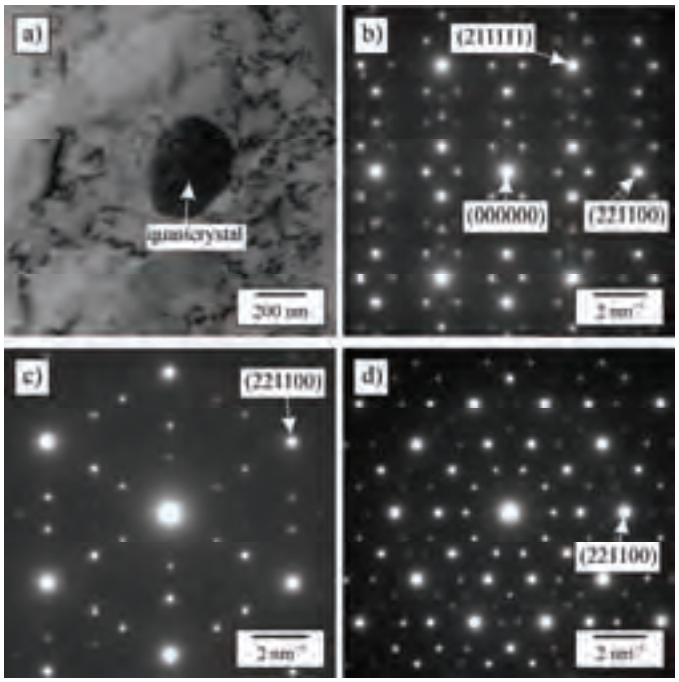


Fig.2 HRTEM of a quasicrystalline particle in a fivefold orientation with corresponding FFT insert (Fast Fourier Transform).

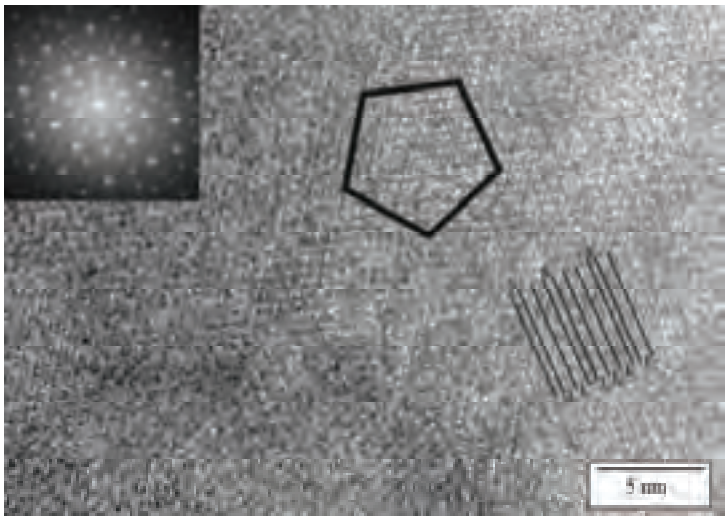
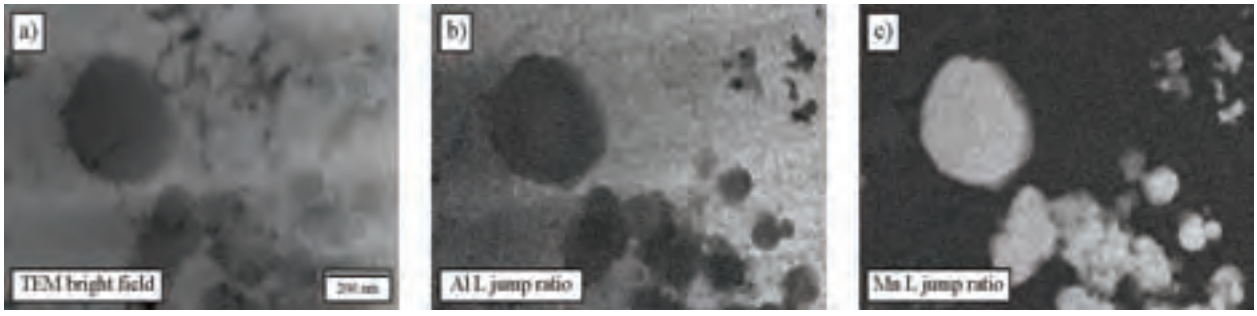


Fig.3 Distribution of elements in the alloy M5 by means of EFTEM; a) TEM bright-field image, zero-loss filtered image, b) elemental distribution of Al and c) elemental distribution of Mn (jump ratio images).



Gold Nanoparticles – Synthesis, Characterization and Influence of Additives

K. Riegler, M. Rogers, B. Schaffer, G. Kothleitner, F. Hofer

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

The synthesis of metal nanoparticles influences their shape and size, since almost every property within the nanometer range is size and shape dependent. Hence, particle formation control is the most important step in designing new materials with tailored properties.
This work focuses on the fabrication of gold nanoparticles (nanorods, nanoprisms, nanospheres) by wet-chemical synthesis. Special attention was paid to the influence of changes in the reaction conditions of common preparation methods, such as the reduction of $HAuCl_4$ in a boiling sodium citrate solution or the seed mediated growth method [1, 2].
In the seed mediated growth method, nanorods of uniform size and shape were synthesised in the presence of the rod-like micellar templates CTAB (cethyltrimethylammonium bromide) and DTAB (dodecyltrimethylammonium bromide). The dependence of the gold nanorod diameter on the nature of the surfactant used and the effect of the electrolyte sodium chloride on the aqueous micellar solution were investigated.

The morphology and structure of the various gold colloids were characterised via TEM, EFTEM, HRTEM and electron diffraction using the transmission electron microscope FEI Tecnai F20.
It was found that the alkyl chain length of the cationic surfactants had no noticeable effect on the diameter of the nanorods, but as a general trend, it was observed that the length of the nanorods increased with the increasing length of the alkyl chain. Consequently, higher aspect ratio nanorods were obtained with the surfactant CTAB, with the aspect ratio increasing with the amount of surfactant (Fig. 1a).
The presence of NaCl in the preparation of gold nanorods with the surfactant CTAB modified the self-assembly of the nanorods. At a concentration of 1M NaCl, the nanorods were not only aligned head-to-head with each other, but had indeed grown together and thus formed a network-like structure (Fig. 1b). It could be demonstrated with high-resolution transmission electron microscopy (HRTEM), that the rods had crystallographically grown together as shown in Fig.2. The clearly resolved interfringe distance of 0.24 coincides with the (110) lattice spacings. This nanorod network has been observed for the first time for gold particles. Similar observations have been reported with silver nanowires [5]. However, the reason why these particles are formed is not clear at this point.

Additional HRTEM studies were performed to investigate the crystallographic structure of the gold nanorods. It could be shown that the gold nanorods synthesised in a CTAB solution exhibit a penta-twinned structure, which confirms previous findings of several groups [3, 4]. These penta-twinned crystals consist of five distorted tetrahedral subunits arranged around the common, elongated [11}] fivefold axis, giving rise to five {111} twin boundaries.

References
[1] Turkevich J., Stevenson P.C., Hillier J., Discuss. Faraday Soc. 11 (1951) 55
[2] Murphy C.J., Jana N.R, Adv. Mater. 14 (2002) 80-82
[3] Johnson C.J., Dujardin E., Davis S.A., Murphy C.J., Mann S., J. Mater. Chem. 12 (2002) 1765
[4] Gai P.L., Harmer M.A., Nano Lett. 2 (2002) 2 771-774
[5] H. Sauer, L. Chen, X. Baoi, D.S. Su, in: Proceedings „Microscopy Conference 2005 – Dreiländer-tagung für Elektronenmikroskopie", Davos, Switzerland (2005), p.261.

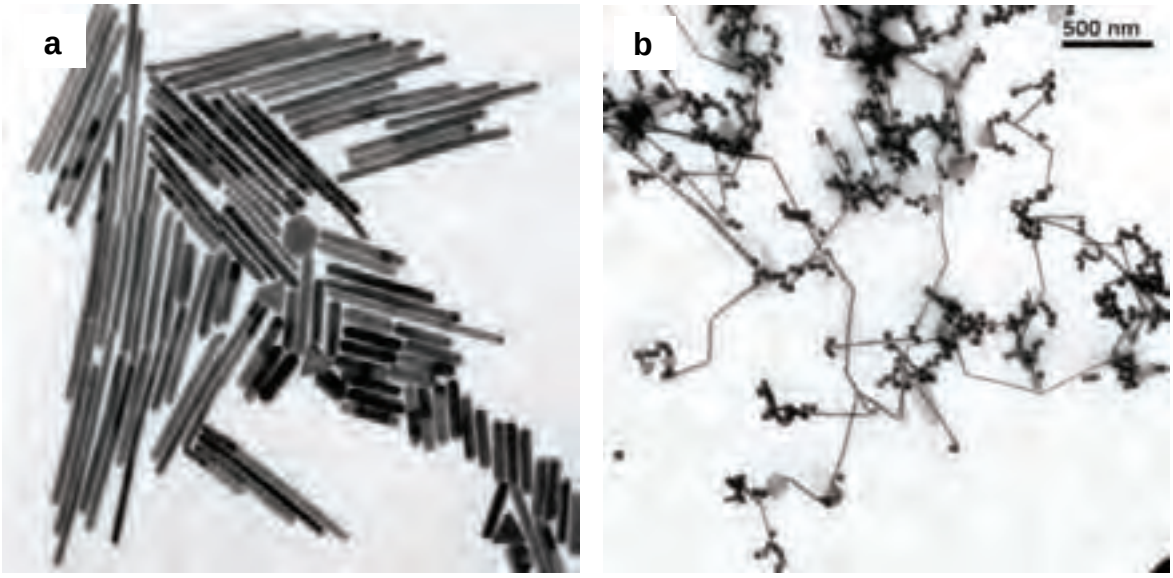


Fig.1 TEM images of the gold nanoparticles produced by the seed mediated growth in the presence of NaCl; a) self alignment of gold rods with 0.1 M NaCl; b) network like structure of gold nanorods with 1 M NaCl.

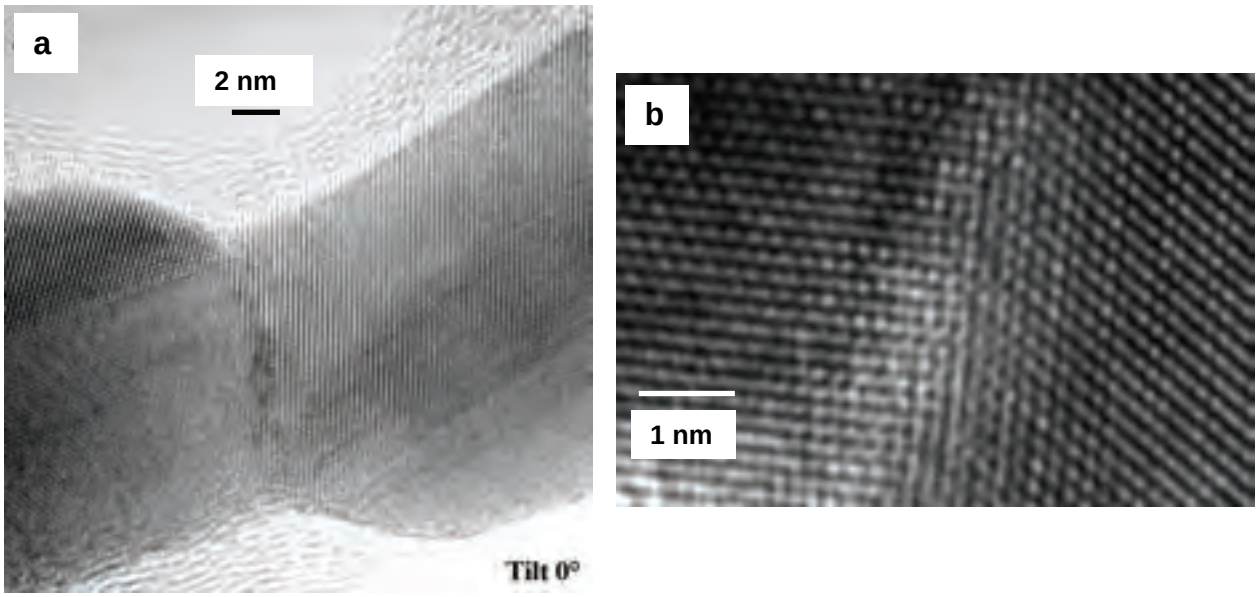


Fig.2a) TEM image of gold nanorods grown together from Fig.1b; b) HREM image of the interface between two gold nanorods.

Automated Particle Analysis of Aerosols formed during Biomass Combustion by SEM/EDX

S. Mitsche[1], P. Poelt[1], M. Schmied[1], T. Brunner[2], M. Jöller[2]

[1]Research Institute for Electron Microscopy, Graz University of Technology, Steyrergasse 17, A-8010 Graz, Austria
[2] Institute for Resource Efficient and Sustainable Systems, Graz University of Technology, Inffeldgasse 21 a, A-8010 Graz

The characterisation of particles with sizes of a few 100 nm or less is of great interest, for instance, to investigate the aerosol particles formed during the combustion process in heating plants. Automated analysis of particles by computer controlled scanning electron microscopy (CCSEM), coupled with image processing and energy dispersive x-ray spectrometry (EDXS), enables the unattended analysis of thousands of individual particles and, as a consequence, the correlation between their geometrical data and the chemical composition. Whereas this technique is well suited for supramicron particles, it is much more difficult to get sound results in the submicron range. Special care has to be taken in the preparation and the choice of the microscope parameters.
The investigations included K_2SO_4, $PbSO_4$ and PbCl particles, used as test particles, ground with a ball mill and filtered on a Nucleopore filter, as well as aerosol particles from biomass combustion plants (both uncoated and C-coated). The analyses were performed on a Zeiss DSM 982 Gemini scanning electron microscope attached to a Noran Voyager EDXS system. The automated particle analyses (APA) were controlled by an in-house developed Unix script.
Figure 1 shows a simplified flow diagram of an automated particle analysis program: First an image is recorded, subsequently the geometrical parameters of the particles are measured (including the coordinates of the particle centres), finally the beam is directed to the first particle and now successive x-ray spectra are recorded for all particles.
The measured sizes and size distributions of the particles in dependence on specimen preparation and on the detector used for imaging are displayed in Figure 2. The comparison of size measurements carried out both with APA and a 9-stage Berner type low-pressure impactor proved that the size distributions measured by APA are of the correct order of magnitude (Mitsche 2002).
The chemical analysis of the particles by EDXS is possible down to a minimum particle size of around 70 nm. For smaller particles the x-ray signal becomes too weak and noisy. Apart from the x-ray count rate, two obstacles in particular affect the accuracy of the analytical results: specimen damage and drift (Poelt 2000). Specimen damage is caused by the heating of the particles by electron irradiation and generally entails a loss of sulphur and chlorine in the particles (see Fig. 3). It can be substantially decreased by the choice of a suitable specimen coating, which is also necessary for preventing charging of the electrically non-conductive specimens (Schmied 2002). Fig. 4 demonstrates the accuracy of the analytical results in the submicron region by use of various test particles.
Specimen drift is around 10 nm/min (Vladar 1999), and originates from instabilities of the specimen stage and the lens currents of the microscope. As a consequence, the particles move away from their initially measured coordinates, hampering x-ray analysis of the particles.
But with a careful choice of all the parameters, reliable analytical results for aerosol particles formed during biomass combustion can be obtained. Two examples of correlations between morphology and chemistry are given in Figure 5.

References

Mitsche S, Poelt P and Brunner T, Proceedings ICEM 15, Durban, South Africa 1, 835-836 (2002)
Poelt P, Schmied S, Obernberger I, Brunner T and Dahl J, Scanning 24, 92-100 (2002)
Schmied S and Poelt P, Mikrochim. Acta 139, 171-177 (2002)
Vladár AE, Scanning 21, 191-196 (1999)

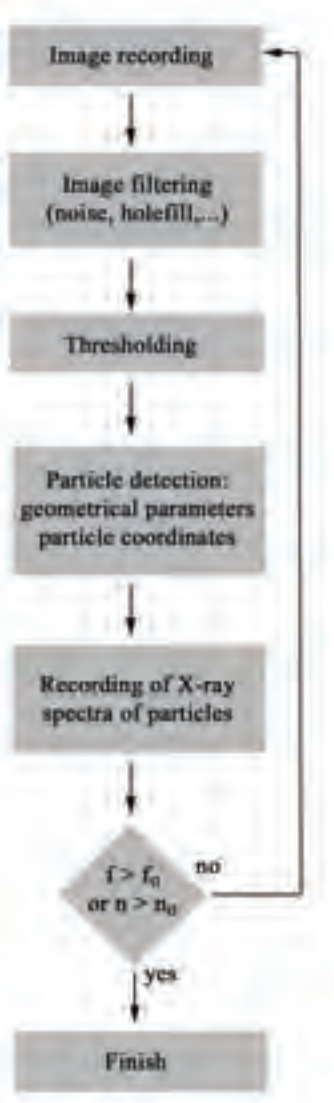


Fig.1 Flow diagram of an automated particle analysis program

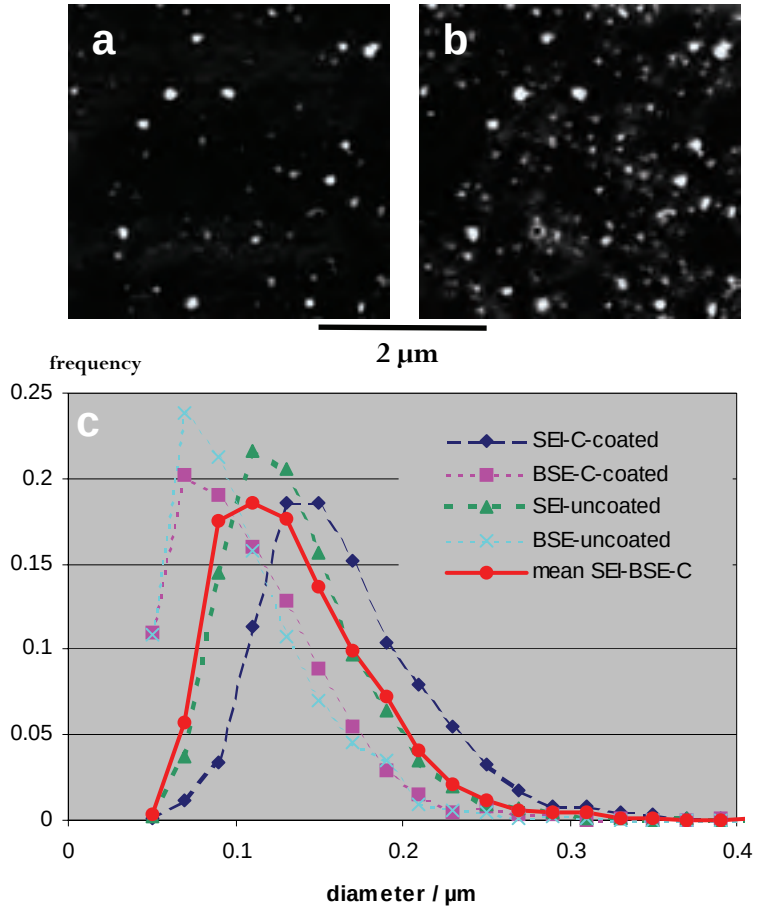


Fig.2 a) BSE-image and b) in-lens SEI-image of aerosol particles collected on a Nucleopore filter in a biomass heating plant; c) size distributions of the aerosol particles obtained with the in-lens detector (SEI) / 4 quadrant BSE-detector (BSE)) and from uncoated / coated particles. (fuel: bark; 1163 particles C-coated, 1438 particles uncoated, E_0 = 7 keV, I_p = 0.7 nA).

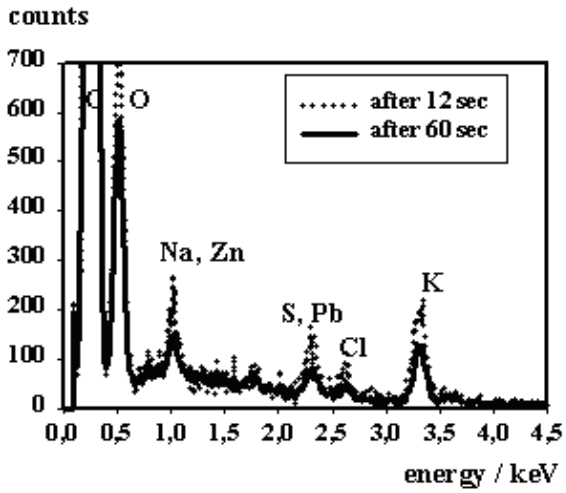


Fig. 3 EDX-spectra from aerosol particles from biomass combustion plant. The analysis was saved after 12 s whilst still accumulating; it was stopped after 60 sec and saved again (E_0 = 7 keV, I_p = 0.7 nA; mean particle diameter ~ 0.300 µm). Most of the elements are lost, but especially Na, S and Cl. C and O originate from the Nuclepore filter, as the analysis volume is greater than the particle size.

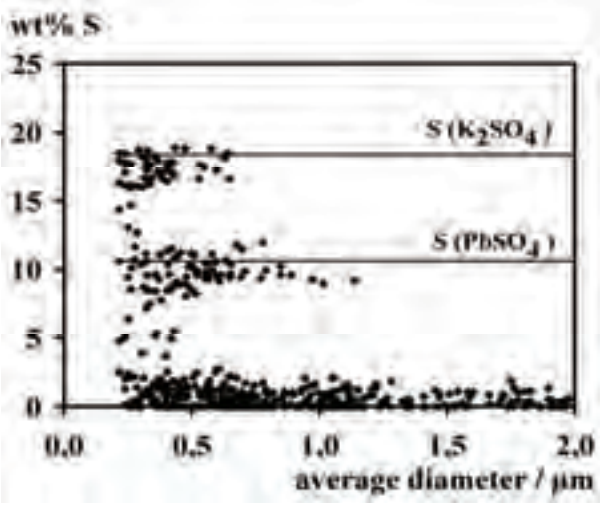
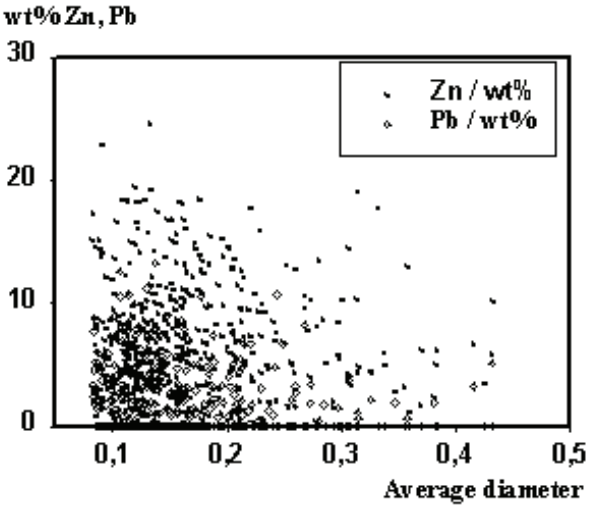


Fig. 4 Automated analysis of a standard particles: K_2SO_4 , $PbSO_4$ and $PbCl_2$ on a Nuclepore filter (carbon coated; approx. 550 particles, E_0 = 7 keV, I_p = 0.7 nA, oxygen by stoichiometry). The solid lines mark the exact sulphur concentration for the corresponding compound. The statistical spread increases with the decrease of the particle diameter.



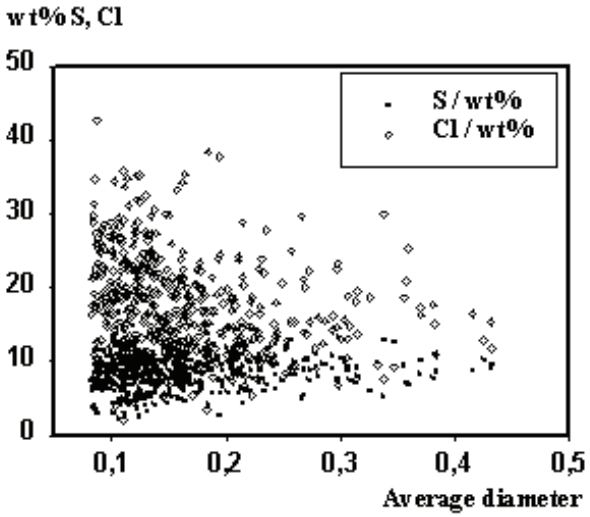


Fig. 5 Automated analysis of fly ash particles collected on a Nuclepore filter (carbon coated, approx. 500 particles, E_0 = 7 keV, I_p ~ 0.7 nA, calculation of oxygen by stoichiometry); dependence of S- and Cl-concentration (left) and Zn- and Pb-concentration (right) on the average diameter.

A Reference Library for Fracture Analysis of Materials (Fractography)

H. Schröttner

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

Materials produced in industry – and the constructions, devices and other products made from them – are all subject to fracture. Further to their great economic impact, some of the failures are of special concern because of safety, environmental or legal considerations.
It is well known that a detailed fracture analysis of materials is central to avoid further damage and helps to improve the quality of future materials and constructions**.** The basis for fracture analysis is the investigation of macroscopic and microscopic features of the fracture surface, which in combination with the facts and circumstances of a particular failure case usually allow finding the reason of the evidence.
The interpretation of features observed on fracture surfaces is simple in many cases, but it can prove to be fairly difficult in practice. This is particularly the case on high strength quenched and tempered steels, or in alloys (such as cast irons and pearlitic steels) where the microstructure affects the crack path. Although for metallic alloys many significant reference sources already exist in the form of atlases of fractographs [1], it is necessary to increase the knowledge about typical failures of new materials and constructions.

The cooperation partners provide selected specimens which have been broken under well defined conditions. The fracture surfaces are studied with a special light microscope (Infinite Focus, Alicona Imaging, Grambach Austria) thus revealing macroscopic features and the 3D reconstruction of the surface topography. Every specimen is also investigated in the scanning electron microscope (SEM) including x-ray spectrometry data. A main advantage of the new library lies in the fact that the SEM images are recorded by means of high resolution SEMs and are combined with different imaging modes of the scanning force microscope (AFM). Additional investigations of the crystal structure on polished and etched cros-sections complete the results.
The database is made up of separate materials sections and contains light microscope, SEM, AFM and TEM fractographs. In combination with mechanical data these illustrations are invaluable to an understanding of the causes, surface features, and mechanisms of fracture of a wide range of ferrous and nonferrous industrial alloys. Although special emphasis lies on metallic materials (cast alloys, steels), ceramics, fibers, polymers and polymer composites are increasingly included in the library.

References
[1] ASM Handbook Volume 12: Fractography, ASM International (1987) Cleveland, USA.

The cooperation with Dr. Rudolf Vallant (Institute for Materials Science, Welding and Forming, TU Graz) and with the Austrian Foundry Institute in Leoben is gratefully acknowledged. The project is financially supported by the Austrian Research Promotion Agency (FFG, Vienna) in the programme PROKIS04.

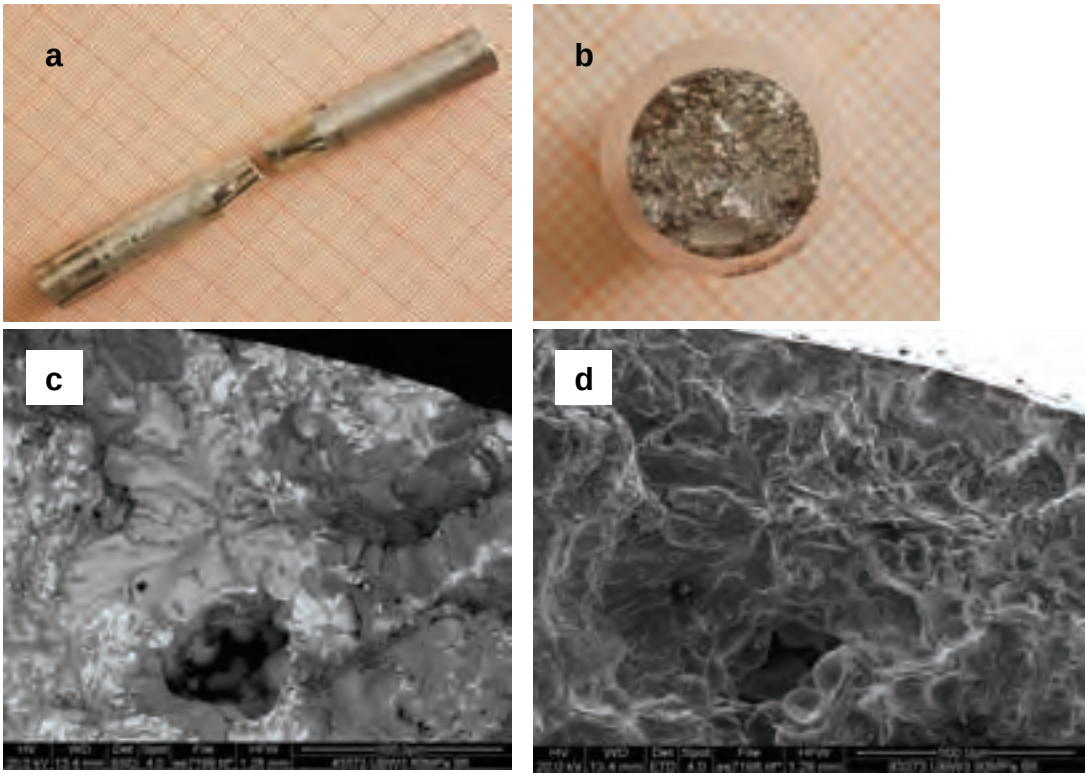


Fig.1 Fracture investigation of a cast aluminium alloy; a.-b. macroscopic images; SEM images c. with backscattered electrons and d.with secondary electrons. The SEM images exhibit large precipitates consisting of alloying elements and cavities.

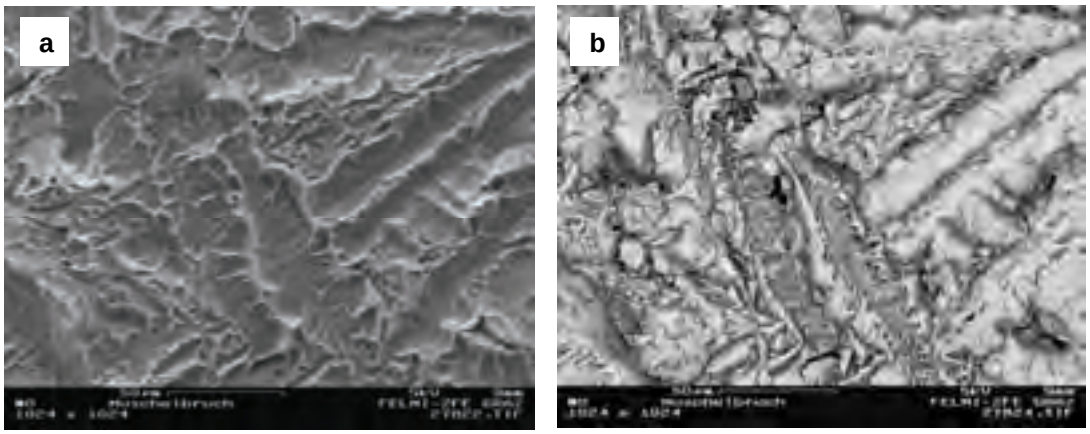


Fig.2 Rock candy fracture in steel casting with aluminium nitride precipitates; a. SE image, b. BSE image.

16. Author Index

Performance Report 2005/06

Technische
Universtät Graz
Steyrergasse 17
8010 Graz, Austria
Tel.:++43 316 873 8320
Fax: ++43 316 811 596
e-mail: sekretariat@felmi-zfe.at
www.felmi-zfe.at

ISBN-10: 3-902465-85-9
ISBN-13: 978-3-902465-85-6