Performance Report 2003/2004

Forschungsinstitut für Elektronenmikroskopie
und Feinstrukturforschung
Technische Universität Graz

Zentrum für Elektronenmikroskopie Graz

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Vorwort

Dieser Zweijahresbericht umfasst einen Überblick über die neuesten Errungenschaften des Forschungsinstitutes für Elektronenmikroskopie (FELMI) der Technischen Universität Graz. Darüber hinaus beinhaltet er die Aktivitäten des Zentrums für Elektronenmikroskopie Graz (ZFE Graz), das vom "Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung" betrieben wird. Beide Institutionen arbeiten eng miteinander zusammen.
Die beiden vergangenen Jahre waren durch die Inbetriebnahme von neuen Spitzengeräten geprägt, die es dem Institut ermöglichen, eine maßgebliche Rolle in der österreichischen Nanotechnologieszene zu spielen, sodass ein Kapitel dieses Berichtes dem derzeitigen Stand und diversen Zukunftsperspektiven gewidmet ist. Wir sind allen unseren Fördergebern für ihre stete Hilfe und Unterstützung zu Dank verpflichtet. Der wissenschaftliche Teil des Berichtes ist ein klarer Beleg dafür, dass alle Gelder zweckmäßig verwendet wurden.
In das letzte Kapitel wurden einige Abstracts aufgenommen, die die breitgefächerte Palette unserer Kooperationen mit anderen Universitätsinstituten und Unternehmen über die vergangenen beiden Jahre hervorheben sollen.

Schlussendlich sollen die Begeisterung und das Engagement unserer Mitarbeiter am Institut gewürdigt werden, die die Basis für unseren Erfolg bilden. Der Schlüssel zum Erfolg wird auch in der Zukunft ihre Bereitschaft, neue Ideen zu entwickeln und hart zu arbeiten, sein.

Jänner 2005

Preface

This biannual report contains an overview of the latest accomplishments of the Research Institute for Electron Microscopy (FELMI) of the Graz University of Technology. In addition it 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.
The last two years were marked by the launch of new leading-edge instrumentation, which enables the institute to become an essential player in the Austrian nanotechnology scene, worthwhile to dedicate a section of this report to the present status and some future prospects.

We are very grateful to all our funding agencies for their continuous help and encouragement. The scientific part of the report will certainly provide evidence that all the money was well used.

In the final chapter we have included a few abstracts, which shall highlight the broad range of our collaborations with other university institutes and companies over the last two years.

Finally, the enthusiasm and the dedication of our collaborators at the institute should be honoured, being fundamental to our success. The key element for future progress continues to be their motivation to generate new ideas and to work hard.

January 2005

Ferdinand Hofer

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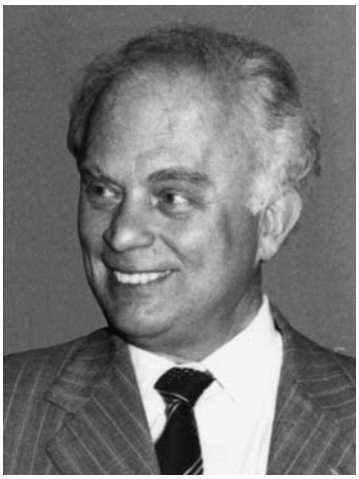
1. Fritz Grasenick verstorben / In memorian of Fritz Grasenick

Hofrat Baurat h.c. Dipl.-Ing. Dr.techn.
Am 28. Februar 2003 ist Herr Hofrat Dr. Fritz Grasenick im Alter von 87 Jahren gestorben. Er gilt als einer der wesentlichen Pioniere der Elektronenmikroskopie in Österreich und hat seit 1951 (bis zu seiner Pensionierung im Jahr 1981) an der Technischen Universität Graz das international renommierte Forschungsinstitut für Elektronenmikroskopie und Feinstrukturforschung aufgebaut.
An der Technischen Hochschule in Graz erwarb er sich mit dem Studium der Technischen Chemie eine gediegene wissenschaftliche Ausbildung, die er an der Universität Heidelberg fortsetzte. Das Rigorosum legte er dann an der TH Graz erfolgreich ab. Nach einigen Jahren in der Privatwirtschaft wurde er von Professor Jantsch an die Tech-nische Hochschule zurückgeholt und 1950 mit dem Aufbau einer elektronenmikroskopischen Forschungsstelle beauftragt.
Herr Grasenick war einer der ersten im deutschsprachigen Raum, der das Fachgebiet der Elektronenmikroskopie nicht nur methodisch weiterentwickelte, sondern auch erfolgreich versuchte, die Elektronenmikroskopie für Probleme der angewandten Forschung einzusetzen. So sind durch ihn zahlreiche Untersuchungsmethoden geschaffen worden, wodurch bestimmte für die österreichische Wirtschaft wichtige Stoffgruppen wie z.B. Zement, Papier, Stahl und Kunststoffe erstmals einer eingehenden mikroskopischen Charakterisierung zugänglich gemacht werden konnten. Herr Grasenick konnte mit tatkräftiger Unterstützung des damaligen Landeshauptmannes Josef Krainer sen. den Verein zur Förderung der Elektronenmikroskopie gründen, in dem heute bedeutende steirische Betriebe unter der Leitung von Professor Helmut List vertreten sind.
Mit seinen außergewöhnlichen, von Ideenreichtum und Weitblick getragenen wissenschaftlichen Leistungen hat Fritz Grasenick die Entwicklung der Elektronenmikroskopie wesentlich geprägt und dafür auch europaweit Anerkennung gefunden. Fritz Grasenick sind zahlreiche Ehrungen und Auszeichnungen zuteil geworden.

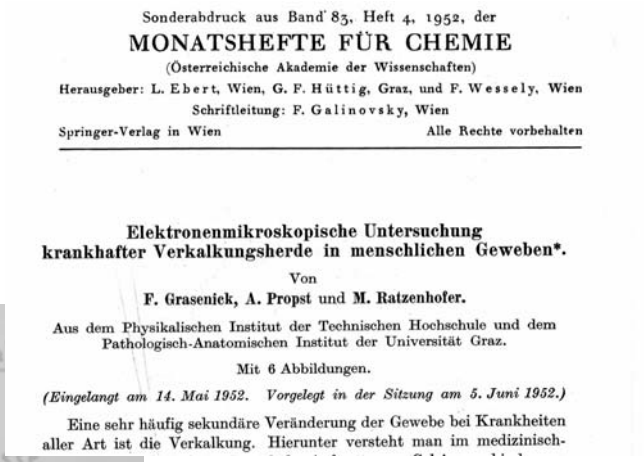
Hofrat Baurat h.c. Dipl.-Ing. Dr.techn.
Hofrat Dr. Fritz Grasenick has died at the age of 87 years on February 28th, 2003. He is regarded as one of the key pioneers of electron microscopy in Austria and as such, beginning in 1951, he has built up the internationally renowned research institute for electron microscopy and fine structure research at the Technical University in Graz, which he conducted until his retirement in the year 1981.
With the study of chemistry at the Technical University Graz he gained a thorough scientific education, which he then consolidated at the University of Heidelberg. Back at the TU Graz, he received his doctorate. In 1950, after some years in industry, he got a call from the university by Professor Jantsch to build up a research institute for electron microscopy.
Dr. Grasenick was one of the first within the German speaking countries, who not only tried to develop new methods and techniques in the area of electron microscopy but who also emphasized its application for material science problems. Numerous examination methods, developed by him, made it possible to characterize some important types of materials relevant for the Austrian economy for the first time by means of electron microscopy, such as cement, paper, steel and polymers. With the active support of the provincial governor Josef Krainer senior, Mr Grasenick was also able to found an organization for the promotion of electron microscopy, which today is managed by Professor Helmut List, and nowadays represents numerous Styrian companies.
Driven by his outstanding creativity, his long-term vision and due to his extraordinary scientific performance, Mr. Grasenick has significantly influenced the development of electron microscopy, for which he received Europe-wide recognition, numerous honors and countless awards.

Fritz-Grasenick-Gedenksysmposium am 27.November 2003 / Fritz-Grasenick Memory Symposium on November 27th, 2003



Fritz Grasenick

First paper of Fritz Grasenick



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Elektronenmikroskopische Untersuchung krankhafter Verkalkungsherde in menschlichen Geweben*.

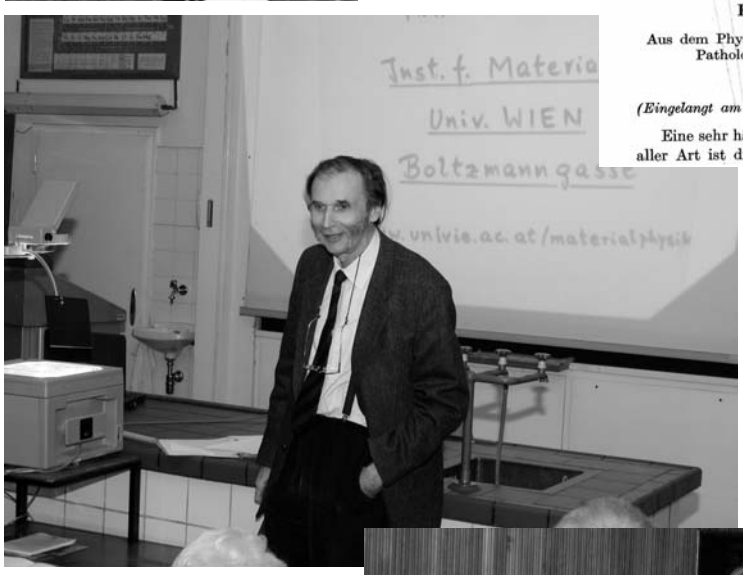
Von
F. Grasenick, A. Propst und **M. Ratzenhofer.**

Aus dem Physikalischen Institut der Technischen Hochschule und dem Pathologisch-Anatomischen Institut der Universität Graz.

Mit 6 Abbildungen.

(Eingelangt am 14. Mai 1952. Vorgelegt in der Sitzung am 5. Juni 1952.)

Eine sehr häufig sekundäre Veränderung der Gewebe bei Krankheiten aller Art ist die Verkalkung. Hierunter versteht man im medizinisch-



Plenarvortrag präsentiert von Prof. H. Karnthaler, Universität Wien
Plenary lecture by Prof. Dr. Hans-Peter Karnthaler, University of Vienna

Family Grasenick, on the right Helga Grasenick (Widow)

2. Das Institut / The Institute

2.1. Forschungsziele

Sowohl für die Entwicklung als auch für die Anwendung moderner Technologien wird es immer wichtiger, die Struktur von Festkörpern und funktionellen Bauelementen im Mikro- und Nanobereich zu charakterisieren. Die Interessen reichen von der Diagnostik im Bereich der Produktentwicklung bis hin zur angewandten Materialforschung, wo die Kenntnis der Mikro- und Nanostruktur und deren Zusammenhang mit technologischen Eigenschaften von grundlegender Bedeutung sind.
Das FELMI der TU Graz ist gemeinsam mit dem ZFE Graz eine der führenden Einrichtungen für modernste Mikro- und Nanostrukturanalysen von Werkstoffen mit atomarer Auflösung. Das vorhandene Spektrum unterschiedlichster mikroskopischer Untersuchungsmethoden bietet einen umfassenden Fundus an hochentwickelten bildgebenden und spektroskopischen Verfahren für das Studium von technologisch relevanten Materialien und der entsprechenden Probleme.
Das Institut sucht Vorteile aus den Synergien zwischen den verschiedenen Forschungsrichtungen, zwischen verschiedenen, anspruchsvollen experimentellen Einrichtungen sowie zwischen Grundlagenforschung und Anwendung derartiger Verfahren für die industrielle Forschung zu ziehen. Dementsprechend versucht das FELMI-ZfE die bestehenden mikroskopischen Präparations- und Charakterisierungstechniken und/oder neue Techniken insbesondere im Bereich der Materialwissenschaften zu entwickeln. Gleichzeitig wendet es diese Techniken zur Charakterisierung aller Arten von Materialien und Werkstoffen an, um effiziente Antworten auf und Lösungen für material- und werkstoffwissenschaftliche Problemstellungen anbieten zu können.
Das Institut kooperierte mit etwa 60 Universitätsinstituten und 100 Industriebetrieben (vor allem aus Österreich, aber auch aus Europa und Übersee). In diesem Zeitraum wurden 70 Diplomanden und Dissertanten (hauptsächlich Studenten der TU Graz) betreut.
Schlussendlich wurde mit der Einführung neuer Forschungsfelder die Organisationsstruktur entsprechend angepasst, wobei der Fokus auf

2.1. Research Objectives

For both the development and application of advanced technologies it is becoming increasingly important to characterize 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 FELMI of the TU Graz in co-operation with the ZFE Graz is one of the leading facilities in Europe for state-of-the-art atomic resolution characterization 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 attempts to take advantage from the synergies that emerge from the various fields of research interests, from different, sophisticated experimental tools and from fundamental research and applications of these techniques performed in conjunction with companies.
Consequently, the FELMI-ZFE tries to improve existing microscopy preparation and characterization techniques and/or to develop new techniques especially in the field of materials science. At the same time it applies these techniques to the characterization of all kinds of materials, providing efficient answers and solutions to materials science problems.

There have been co-operations with about 60 university institutes and 100 companies (mainly from Austria but also from Europe and overseas).

During this period 70 graduate and PhD students (mainly from the TU Graz) benefited from the support of the institute.
Finally the organisational structure has been changed with the introduction of new research areas, each of them focussing on a specific aspect

jeweils einen spezifischen Mikroskopie- oder Materialaspekt gelegt wurde.

- Rasterelektronemikroskopie und verwandte physikalische Untersuchungsmethoden (Peter Pölt)
- Analytische Rasterelektronenmikroskopie (Mario Schmied)
- Analytische Transmissionselektronenmikroskopie and Probenpräparation (Gerald Kotheitner)
- Hochauflösungselektronenmikroskopie (Werner Grogger)
- Mikroskopie von Polymeren und biologischem Gewebe (Elisabeth Ingolic)
- FTIR- und Raman-Mikrospektrometrie (Peter Wilhelm)

Gewisse tiefgreifende Änderungen im wissenschaftlichen Umfeld haben zu einer Reorganisation und zu einer Verschiebung zu nachfolgenden Tätigkeitsschwerpunkte geführt:

- Entwicklung von Nanostrukturen und neuen Materialien.
- Kombination mit theoretischen Verfahren (z.B. Simulation von EELS-Nahkanten-Feinstrukturen und TEM-Hochauflösungsbildern, Finite-Elemente-Simulationen)
- Einführung neuer innovativer Charakterisierungsmethoden (neuartige Lichtmikroskopie, Kombination Elektronenmikroskopie mit physikalischen Eigenschaften).

of microscopy or materials.

- Scanning electron microscopy and related physical methods (PeterPölt)
- Analytical scanning electron microscopy (Mario Schmied)
- 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)

Certain profound changes in the scientific environment have caused a reorganization and shift of our activities towards:

- Development of nanostructures and new materials.
- Combination with theoretical approaches, e.g. simulation of EELS near edge fine structures and high resolution TEM images, finite element simulations.
- Introduction of new innovative characterization methods (new light microscopy, combination of electron microscopy with physical properties).

2.2. Forschungsgeräte und neue Entwicklungen

Die Forschung basiert auf einer exzellenten Geräteausstattung, die durch Spezialistenteams betreut wird. Ziel ist es, derartige Team-Aktivitäten im Rahmen von bereits vorhandenen oder neuen Kooperationen zu entwickeln und dabei sicherzustellen, dass andere Institute davon gleichfalls profitieren können.
Forschung, die auf mehrere Teams verteilt ist, fördert erstens den Austausch von Wissen, von wissenschaftlichen Ergebnissen und verbessert den Bekanntheitsgrad unserer Arbeit, und reduziert zweitens die Gesamtbetriebskosten für die Forschungsgeräte, die ansonsten bei weitem zu hoch für ein einzelnes Institut wären.
Ein wesentlicher Teil der Elektronenmikroskope der TU Graz befindet sich am Institut. Wir konnten mit Erfolg unsere Instrumentierung stetig aufrüsten, aber auch die Ausbildung unserer WissenschafterInnen und des Mikroskop-Bedienungspersonals verbessern, um hochmoderne Untersuchungen sowohl für Universitätsinstitute als auch für Unternehmen anbieten zu können.
Unser Labor beherbergt nun ein analytisches Hochauflösungs-Elektronenmikroskop, zwei analytische Transmissionselektronenmikroskope, ein Environmental Rasterelektronenmikroskop, mehrere Rasterelektronenmikroskope sowie FTIR-, Raman- und fortgeschrittene Lichtmikroskopie. Damit verbundene Methoden umfassen energiedispersive Röntgenanalytik (EDX), wellenlängendispersive Röntgenanalytik, Elemental Mapping, Elektronenverlust-Spektrometrie, energiefilternde Mikroskopie, Niedrigdosis-Abbildungsverfahren, Elektronenbeugung und zahlreiche andere Spezialmethoden.
Die letzte Errungenschaft ist die Einführung eines Focused-Ion-Beam-Mikroskops im Herbst 2003.

- **Focused-Ion-Beam-Anlage (FIB)**

In den Jahren 2001 bis 2002 bemühten wir uns intensiv um den Ankauf einer dringend benötigten FIB-Anlage. Schlussendlich war unser Projekt von Erfolg gekrönt, da der Rat für Forschung und Technologieentwicklung“ (RFT) im Wien das Projekt zur Förderung empfahl. Nach einer langen Testphase wurde beschlossen, ein NOVA200 NanoLab der Fa. FEI (Eindhoven,

2.2. Research Tools and New Developments

The research is based on sophisticated equipment operated by specialized groups. The goal is to develop group activities in the framework of already existing or new collaborations ensuring other institutes can benefit as well. Firstly, research, spread out over several groups, favours the exchange of knowledge, of scientific results and improves the publicity of our work, but secondly reduces the total cost of ownership for instrumentation, which otherwise would be far too high for just one institute.
A significant portion of electron microscopes of the Graz University of Technology are kept in the institute. 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, one environmental scanning electron microscope, several scanning electron microscopes as well as FTIR-, Raman and advanced light microscopy. 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.

The latest achievement is the introduction of a focused ion beam microscope in autumn 2003.

- **Focused Ion Beam instrument (FIB)**

From 2001 to 2002 we intensively strove for the purchase of an urgently needed FIB machine. Our constant efforts have been successful, because finally the „Rat für Forschung und Technologieentwicklung” (RFT) in Vienna recommended the project. After a long testing period we decided to order a NANOLAB Nova 200 manufactured by FEI Company (Eindhoven,

Niederlande) zu bestellen. Das Gerät wurde im Oktober 2003 installiert und auf die spezifischen Anforderungen unseres Instituts adaptiert. Das Gerät gestattet es Querschnitte von Oberflächen, Defekten und nanoskopische Strukturen zu schneiden und zu dünnen und Nanostrukturen und dünne Filme mit Hilfe eines Gaseinlass-Systems abzuscheiden. Für hochaufgelöste Bildgebung ist ein Rasterelektronenmikroskop mit Feldemissionsquelle im System inkludiert.

- **Grobdruck-Rasterelektronenmikroskop (Environmental SEM = ESEM)**

Das Grobvakuum-Rasterelektronenmikroskop, das ursprünglich im Juni 2002 installiert wurde, ermöglicht eine breite Palette an neuen Charakterisierungsmethoden. Das neue Mikroskop wird für die Untersuchung von „feuchten" Proben bei hoher räumlicher Auflösung verwendet und ist mit einem Heiz/Kühltisch sowie mit einer Zugbühne ausgestattet. Mit dieser Ausrüstung ist das Mikroskop besonders für in-situ-Untersuchungen von Phasenübergängen, Korrosionseigenschaften, Hochtemperatur-Verhalten, Trocknungsprozessen usw. geeignet. Das Mikroskop wird vor allem zur Problemlösung in den Bereichen Technische Chemie, Biochemie, Biologie und Biotechnologie verwendet. Das ESEM wurde durch die Fa. FEI (Eindhoven, Holland) geliefert und wurde im Mai 2003 mit einer Feldemissionsquelle und einer größeren Probenkammer aufgerüstet.
Das Projekt wurde durch den Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung mit Unterstützung der Steirischen Wirtschaftsförderung (sfg) in Graz und der Wachstumsförderung des Bundesministeriums für Wirtschaft und Arbeit (BMWA) in Wien aus der Taufe gehoben. Ein Teil wurde durch die Technische Universität Graz im Rahmen eines speziellen vom Rat für Forschung und Technologieentwicklung (RFT) in Wien finanzierten Projektes unterstützt.

- **Ein Monochromator für die analytische Hochauflösungs-Elektronenmikroskopie (HR-AEM)**

Das erste analytische Hochauflösungs-Elektronenmikroskop in Österreich (installiert im Jahre 2001) wurde im Februar 2003 mit einem

the Netherlands). The instrument was installed during October 2003 and is specifically tailored to the needs of our institute. The instrument allows to cut and mill cross-sections of surfaces, defects and nanosized structures and to deposit nanostructures and thin films by using the gas injection system. In order to enable high resolution imaging a scanning electron microscope equipped with a field emission gun is included in the system.

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

The low-vacuum scanning electron microscope which was originally installed during June 2002 enables a broad range of new characterization techniques. The new microscope is used for the studying of "wet" specimens at high spatial resolution and is equipped with a cooling and heating stage as well as a strain and tensile stage. In this outfit, the microscope is particularly suited for in-situ studies such as phase transitions, corrosion properties, high temperature behaviour, drying processes and so on. The microscope is mainly used for problem solving in the fields of technical chemistry, biochemistry, biology and biotechnology. The ESEM was delivered by FEI (Eindhoven, The Netherlands) and was upgraded with a field emission gun and a bigger specimen chamber in May 2003.
The project was launched by the „Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung" with the support of the "Steirische Wirtschaftsförderung" (SFG) in Graz and the „Wachstumsförderung" of the Federal Ministry of Economical Affairs (BMWA) in Vienna. Part of it was supported by the TU Graz within a special project financed by the „Rat für Forschung und Technologieentwicklung" (RFT) in Vienna.

- **A monochromator for the analytical high resolution electron microscope (HR-AEM)**

The first analytical high resolution electron microscope in Austria (installed in 2001) was upgraded with a monochromator during

Monochromator aufgerüstet. Zu diesem Zeitpunkt war dies das zweite Gerät auf der Welt.
Die Grundversion des Mikroskops ist mit einer Feldemissionsquelle, einer hochauflösenden Objektivlinse, einem energiedispersiven Röntgendetektor und einem Imaging-Filter ausgestattet. Mit diesem Gerät kann die Auflösung für eine Vielzahl von wichtigen Charakterisierungsmethoden wie HREM, EELS, EFTEM und STEM verbessert werden. Daher wird es zur Materialcharakterisierung nahe an der atomaren Auflösung verwendet und soll helfen die Herausforderungen neuer Materialien und Bauelemente wie nanostrukturierte Materialien, Nanopartikel, Halbleiterbauelemente oder funktionelle Materialen zu bewältigen.
Im zweiten Schritt wurde das Auflösungsvermögen des Mikroskops durch die Installation eines hochauflösenden Energiefilters (2002) und eines Monochromators für die Elektronenquelle (Februar 2003) gesteigert. Damit kann die Energieauflösung von etwa 0,6 eV auf etwa 0,18 eV verbessert werden, was Information über die chemischen Bindungsverhältnisse in nanometerkleinen Probenbereichen zugänglich macht, wobei die Nahkanten-Feinstruktur von EELS-Spektren genützt wird. Dies wird für die Untersuchung von inneren Oberflächen in Materialien und von einzelnen Nanopartikeln besonders hilfreich sein. Die Erweiterung wurde durch ein spezielles Kooperationsprojekt mit FEI (Eindhoven, Niederlande) und Gatan (Pleasanton, USA) ermöglicht, und durch den Forschungsförderungsfonds der Gewerblichen Wirtschaft (FFF) in Wien und durch die Steiermärkische Landesregierung in Graz gefördert.

February 2003. At this time it was the second unit in the world.
The basic microscope is equipped with a field emission gun, a high resolution objective lens, an energy-dispersive x-ray detector and an imaging filter. With the instrument we could improve the resolution of a variety of important characterization techniques such as HREM, EELS, EFTEM and STEM. Consequently, it is used for characterizing materials at nearly atomic resolution, and it shall help to meet the challenges of new classes of materials and devices, like nanostructured materials, nanoparticles, semiconductor devices and functional materials.
In the second step the resolving power of the microscope was increased by installing a high resolution energy-filter (2002) and a monochromator for the electron gun (February 2003). Energy resolution can now be improved from about 0.6 eV to about 0.18 eV which gives access to chemical bonding information in nanometer small specimen areas, harnessing the near edge-fine structures in EELS spectra. This will be especially helpful for studying inner boundaries in materials and single nanoparticles. The extension was enabled by a special cooperation project with FEI (Eindhoven, The Netherlands) and Gatan (Pleasanton, USA), which was financially co-funded by the „Forschungsförderungsfonds der Gewerblichen Wirtschaft“ (FFF) in Vienna and by the “Steiermärkische Landesregierung” in Graz.

- **FPA-Detektor für das FT-Infrarot-Mikroskop**

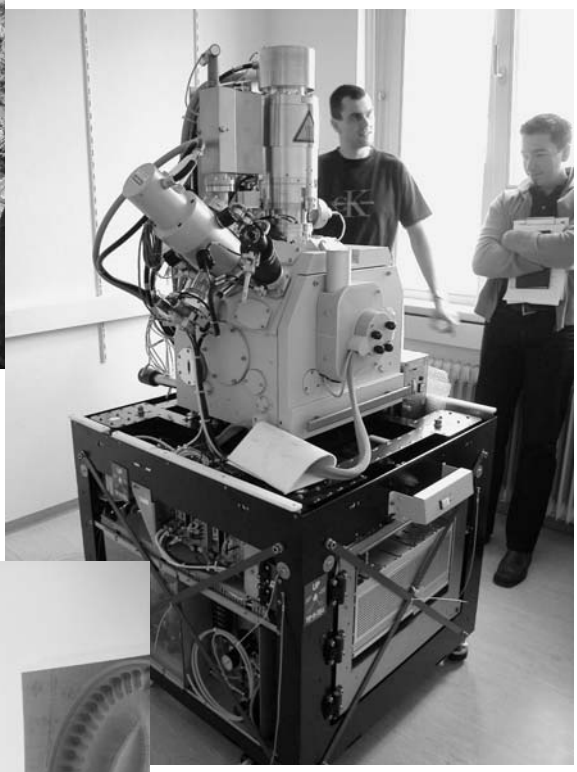
Das FT-Infrarot-Mikroskop Bruker Equinox 55 Spektrometer mit einem Hyperion 3000 Mikroskop wurde im Herbst 2002 installiert. Es ist mit Spezialobjektiven (Oberflächenanalyse mit Glanzwinkel-Objektiv ATR, Dünnschichtuntersuchung mit einem GIR) ausgestattet und wurde mit einem Abbildungs-Zusatz (FPA Imaging Detector) aufgerüstet. Das Mikroskop wurde im Rahmen eines Projektes der TU Graz angeschafft, das vom Rat für Forschung und Technologieentwicklung (RFT) in Wien genehmigt wurde.

- **FPA-detector for the FT-infrared microscope**

The FT-infrared microscope Bruker Equinox 55 spectrometer with a Hyperion 3000 microscope was installed during autumn 2002. It is equipped with special objectives (surface analysis with a grazing angle objective ATR, thin films studies with a GIR), and was upgraded with an imaging device (FPA imaging detector). The basic instrument was aquired with a project of the TU Graz, which has passed the “Rat für Forschung und Technologieentwicklung (RFT)”, Vienna.

Installation der Focused Ion Beam Anlage Nova 200 NANOLAB von FEI im Herbst 2003
Installation of the Focused Ion Beam Nova 200 NANOLAB (FEI) during autumn 2003

Ing. Hartmuth Schröttner, Dr. Mario Schmied



Dr. Mario Schmied, Martina Dienstleder
after the installation of the FIB

Dipl.-Ing. Michael Rogers
working with the FIB

2.3. Das „Leben“ im Labor

Während der vergangenen beiden Jahre hat sich die Lage des Institutes aufgrund der Einführung neuer Mikroskope und neuer faszinierender Methoden verbessert. Dank der breitgefächerten Vielfalt an Projekten und Partnerschaften konnte die Zahl der Beschäftigten und Doktoranden vergrößert werden.

Personal:
Wie in den Jahren zuvor war die Fluktuationsrate bei den Mitarbeitern beträchtlich und aus gegebenen Gründen wird sich dies auch in Zukunft fortsetzen.

- Dipl.-Ing. Dr. techn. Peter WARBICHLER trat am 30. September 2003 in den Ruhestand. Dr. Warbichler leitete die Arbeitsgruppe „Transmission Electron Microscopy of Materials“. Er gehörte dem Institut seit 1972 an.
- Dipl.-Ing. Harald ZEDLACHER trat am 30. November 2003 in den Ruhestand. Dipl.-Ing. Zedlacher leitete die Arbeitsgruppe Administration und gehörte seit 1970 dem Institut an.
- Helmut MARKOVIC trat am 1. Mai 2003 in den Ruhestand. Er gehörte dem Institut seit 1970 an.
- Dipl.-Ing. Siegfried KALTMANN ist seit 1. März 2003 am Institut als Maschinbauingenieur beschäftigt.
- Dipl.-Ing. Christian GSPAN ist seit 20. November 2003 am Institut beschäftigt und arbeitet in der Gruppe von Dr. Grogger.
- Dipl.-Ing. Dr. Ilse LETOFSKY-PAPST ist seit März 2003 in Karenz.
- Dipl.-Ing. Dr. Andreas GUPPER verließ das Institut am 15. September 2003 und wechselte an das Imperial College, London, UK (Schrödinger-Stipendium).
- Mag. Boril CHERNEV ist seit 1. Oktober 2003 am Institut beschäftigt und arbeitet in der Gruppe von Dr. Wilhelm.
- Dipl.-Ing. Dr.techn. Karin WEWERKA ist seit 1. Oktober 2003 am Institut in der Gruppe von Dr. Kothleitner beschäftigt. Sie ist seit Juli 2004 auf Karenz.
- Ing. Claudia MAYRHOFER ist seit 1. Oktober 2003 am Institut in der Gruppe von

2.3. The “life” in the laboratory

During the last two years, the situation of the institute improved due to the introduction of new microscopes and new challenging methods. Thanks to the broad diversity of contracts and partnerships, we could further increase the number of employees and PhD students.

Staff
Again change of staff has been considerable and due to several reasons we expect this situation to continue in the future.

- Dipl.-Ing. Dr. techn. Peter WARBICHLER retired on September 30, 2003. Dr. Warbichler was heading the workgroup „Transmission Electron Microscopy of Materials”. He has been a member of the institute since 1972.
- Dipl.-Ing. Harald ZEDLACHER retired on November 30th, 2003. Dipl.-Ing. Zedlacher was heading the administrative workgroup and has been a member of the institute since 1970.
- Helmut MARKOVIC retired on May 1st, 2003. He has been a member of the institute since 1970.
- Dipl.-Ing. Siegfried KALTMANN entered the institute on March, 1st, 2003 and is working in the technical supporting group.
- Dipl.-Ing. Christian GSPAN entered the institute on November, 20th 2003 is now working in the group of Dr. Grogger.
- Dipl.-Ing. Dr. Ilse LETOFSKY-PAPST is on maternity leave since March 2003.
- Dipl.-Ing. Dr. Andreas GUPPER left the institute for a Schrödinger scholarship (Imperial College, London, U.K.) on September, 15th, 2003.
- Mag. Boril CHERNEV entered the institute on October, 1st, 2003 and is working in the group of Dr. Wilhelm.
- Dipl.-Ing. Dr.techn. Karin WEWERKA entered the institute on October 1st, 2003 working in the group of Dr. Kothleitner. She is on maternity leave since July 2004.
- Ing. Claudia MAYRHOFER entered the institute on November, 1st 2003 working in the group of Dr. Kothleitner.

Dr. Kothleitner beschäftigt.

- Sanja SIMIC ist seit August 2004 in Karenz.
- Dipl.-Ing. Dr. Christoph MITTERBAUER verließ das Institut am 31. Dezember 2004 und wechselte auf eine Post-Doc-Stelle an der Universität von Kalifornien, Davis, USA.
- Manuel PALLER ist seit 1. Juli 2003 am Institut (ZFE) beschäftigt und arbeitet nun in der Gruppe von Dr. Grogger.
- Mag. Dr. Werner ROM ist seit 1. Jänner 2005 am Institut beschäftigt.
- Dipl.-Ing. Dr. Werner GROGGER habilierte sich im Juni 2004 im Fach „Angewandte Festkörperphysik".
- Dipl.-Ing. Dr. Gerald KOTHLEITNER habilitierte sich im Juni 2004 im Fach „Angewandte physikalische Chemie".

- Sanja SIMIC is on maternity leave since August 2004.
- Dipl.-Ing. Dr. Christoph MITTERBAUER left the institute on December 31st, 2004 for a Postdoc position at the University of California, Davis, USA.
- Manuel PALLER entered the institute on July 1st, 2003 (ZFE Graz) and is now working in the group of Dr. Grogger.
- Mag. Dr. Werner ROM entered the institute on January, 1st 2005.
- Dipl.-Ing. Dr. Werner GROGGER received his „Habilitation" in the topic „Applied solid state physics" during June 2004.
- Dipl.-Ing. Dr. Gerald KOTHLEITNER received his „Habilitation" in the topic „Applied physical chemistry" during June 2004.

Auszeichnungen:

- Auszeichnung der Austrian Cooperative Research für den ZFE-Businessplan am 4. November 2003.
- "Best Advanced Scientist Award" für Dipl.-Ing. Stefan Mitsche für seine Posterpräsentation bei der MICROSCIENCE 2004, London .
- "Best New Scientist Award" für Dipl.-Ing. Michael Rogers für seine Posterpräsentation bei der MICROSCIENCE 2004, London .

Awards:

- Award for ZFE Business Plan of the Austrian Co-operative Research, November, 4th, 2003.
- "Best Advanced Scientist Award" for Dipl.-Ing. Stefan Mitsche for his poster presentation at the MICROSCIENCE 2004, London, July 2004.
- "Best New Scientist Award" for Dipl.-Ing. Michael Rogers for his poster presentation at the MICROSCIENCE 2004, London, July 2004.

Einbindung in die wissenschaftliche Gemeinschaft

- F. Hofer ist Mitglied des Redaktionsstabes von MICRON, Vorstandsmitglied der Austrian Cooperative Research (ACR), der Österreichischen Gesellschaft für Elektronenmikroskopie (ASEM) und stellvertretender Aufsichts-ratsvorsitzender von Anton Paar AG.
- F. Hofer ist Mitglieder des inter-nationalen Beirats der „Autumn School for Electron Microscopy and Advanced Materials“ an der Humboldt-Universität Berlin (BRD).
- G. Kothleitner ist seit 2002 Mitglied des Beirates der Arbeitsgruppe „EFTEM & EELS“ in der Deutschen Gesellschaft für Elektronenmikroskopie (DGE).
- Mitarbeiter des Institutes waren als Gutachter für folgende wissenschaftliche Institutionen und wissenschaftliche Zeitschriften tätig: Netherlands Organization for Scientific Research (NOW), Holland; National Science and Research Council (NSERC), Kanada; Engineering & Physical Sciences Research Council (EPRSC), Großbritannien; Universität Bremen, BRD; Universität Sevilla, Spanien; Universität Leipzig (BRD).
  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.
- Organisation des Internationalen Symposiums „EELS of Steels“ in Bruck an der Mur, vom 12.-14.Juni 2003 (40 Teilnehmer aus 6 Ländern).
- Organisation des Fritz-Grasenick-Gedenksymposiums an der TU Graz am 12. Novermber 2003 im Gedenken an den Gründer des Institutes.
- Die Zusammenarbeit mit Forschungsgruppen der Steirischen Universitäten im Spezial-Forschungsbereich „Elektroaktive Stoffe“ wird bis April 2006 fortgeführt.
- Start des neuen Universitätslehrganges der TU Graz „Nanotechnologie und Nanoanalytik“, mit Beiträgen des FELMI, Oktober 2004.
- Die Kooperation mit Kompetenzzentren wie

Acceptance in the scientific community

- F. Hofer is member of the editorial board of MICRON, board member of the Austrian Cooperative Research (ACR), of the Austrian Society for Electron Microscopy (ASEM) and co-chairman of the supervisory board of the company ANTON PAAR in Graz.
- F. Hofer is member of the international advisory board of the Autumn School for “Electron Microscopy and Advanced Materials” at the Humboldt University, Berlin, Germany.
- G. Kothleitner is board member of the Working Group “EFTEM & EELS” within the German Electron Microscopy Society (DGE), since 2002.
- Staff members were active as referees both for scientific institutions and scientific journals: Netherlands Organization for Scientific Research (NOW), The Netherlands; National Science and Research Council (NSERC), Canada; Engineering and Physical Sciences Research Council (EPRSC), UK; University of Bremen, Germany; University of Sevilla, Spain; University of Leipzig, Germany.
  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.
- Organisation of the Workshop “EELS of Steels” in Bruck an der Mur, June, 12-14, 2003 (40 participants from 6 countries).
- “Fitz-Grasenick-Gedenksymposium” organized at the TU Graz on November 12th, 2003 in memoriam of the founder of the institute.
- Collaboration with research groups from the Styrian Universities in the Special Research Programme “Electroactive Materials” will continue until April 2006.
- Launch of the university course of the TU Graz “Nanotechnology and Nanoanalysis”, with contributions from the FELMI, October 2004.
- Cooperation with competence centres such

dem Materials Center Leoben und dem an der TU Graz angesiedelten ABC-Kompetenzzentrum wurde ausgeweitet.

- Bauliche Sanierung und energetische Optimierung des Gebäudes Steyrergasse 17-19 mittels Contracting, Zusammenarbeit mit Joanneum Research GmbH, Graz.
- Erfolgreiche Teilnahme des Institutes an der MATERIALICA in München im September 2004 mit Unterstützung der Steirischen Wirtschaftsförderung (SFG).
- Die Zahl der durch Institutsmitglieder gehaltenen eingeladenen Vorträge im Ausland konnte gesteigert werden, die erweiterten Aktivitäten erstreckten sich von Amerika über Europa bis nach Australien und Japan.
- Das ZFE Graz ist ein Mitglied der Austrian Cooperative Research (ACR) und kooperiert

as the Materials Center Leoben and the ABC Center at the TU Graz is extended.

- Renovation and energy optimisation of the building Steyrergasse 17-19 with Contracting, cooperation with Joanneum Research GmbH Graz.
- The institute successfully participated in the MATERIALICA fair in Munich, Germany during September 2004; in co-operation with the "Steirische Wirtschaftsförderung" (SFG).
- The number of invited talks given by members of the institute abroad increased and the activities were spread out ranging from America over Europe to Australia and Japan.
- ZFE Graz is a member of the Austrian Cooperative Research (ACR) with several scientific co-operations with other ACR institutes.

Das Institut in den Medien / The institute in the news

- *innovatives-oesterreich.at Jänner 2003*, „TU Graz: Analytik von Nanoschichten"
- *TUG Print* 5 WS02/03, S.19 „Eine Kalendergeschichte"
- *innovatives-oesterreich.at Dezember 2003*, „Nanostrukturforschung an der TU Graz"
- *science.ORF.at* – News; Jänner 2004 „TU Graz: Nanostrukturen im Fokus"
- *Mitteilungsblatt der Österr. Physikal. Gessellschaft,* März 2004 „TU Graz: Nanostrukturen im Fokus"
- *TUG Print* 6 SS2003, S.19 „Fritz Grasenick verstorben"
- *TUG Print 6* SS2003, S.19 „Monochromator für Supermikroskop an der TU Graz"
- *Forschungsjournal der TU Graz WS 03/04:* „RFT-Projekt: Neue Untersuchungsmethoden für Mikrosystemtechnik und Nanotechnologie"
- *ORF 1* 28.7.2004, Radio Interview, "Nanotechnologie"
- *TUG Print 11 SS2004,* Juni 2004, „Nanoschrift: 120 Bücher auf einen Stecknadelkopf"
- *online2infozine*, Online Zeitung des Österr. Verbandes f. Elektrotechnik, 27.7.2004, „Nanolabor an der TU Graz"
- *science.orf.at*, 28.7.2004, „Nanoschrift-kleiner als die Wellenlänge des Lichtes"
- *steiermark.orf.at,* 27.7.2004, „Neuartige Minischrift"
- *Kurier,* 27.7.2004 „Ein Buchstabe hat Platz auf 0,04 Millionstel Quadradmillimeter"
- *APA-Online,* 29.7.2004, „Grazer Nano-Keilschrift kann Diamanten unsichtbar codieren"
- *Der Standard*, 2.8.2004, „Ionenstrahlen beschriften Diamanten"
- *Kleine Zeitung-online* 30.7.2004, „Nanokeilschrift kann Diamanten un-sichtbar codieren"
- *steiermark.orf.at* 2.8.2004, „Die Nationalbibliothek auf einem Blatt"
- *Salzburger Nachrichten* 30.7.2004, „Nano-Keilschrift"
- *Wiener Zeitung*, 4.8.2004, „Nano-Keilschrift am FELMI: Unsichtbar codieren"
- *APA-Journal Forschung* 31.7.2004, „Nano-Keilschrift"
- *Grazer Woche*, 5.9.2004, „Das wohl kleinste ABC der Welt"
- *derstandard.at*, 4.8.2004, „Nano-Beschriftung für Edelsteine"

2.4. Pläne für die Zukunft

Die umfassende Verwertung der bestehenden wissnschaftlichen Einrichtungen sowie der Aufbau neuer zukunftsweisender Verfahren sind für das Institut von zentraler Bedeutung. Folgende instrumentelle Erweiterungen werden für 2005 geplant:

- Aufrüsten des Hochauflösungs-TEM Tecnai F20 mit einem 2kx2k-CCD-Detektor
- Hochauflösungs-Rasterelektronenmikroskop
- Rasterkraftmikroskop
- Ersatz von veralteten Mikroskopen und Geräten.
- CNC-System für Materialbearbeitung

Neben der Intensivierung von Kooperationen mit Instituten der anderen steirischen Universitäten in dem unlängst genehmigten ISOTEC-Projekt der Österreichischen NANO Initiative bemühen wir uns über Kooperationen mit namhaften Forschungsinstituten aus aller Welt den wissenschaftlichen Austausch zu verstärken. Für die kommenden Jahre sollen auch internationale wissenschaftliche Kongresse und Workshops nach Österreich geholt werden. 2005 wird der International EELS Workshop EDGE2005 am Grundlsee organisiert.

2.4. Plans for the future

A major point of focus is to fully exploit existing instrumentation and to introduce new leading edge instrumentation. We consider the introduction of the following instruments during 2005:

- Upgrade of high resolution TEM Tecnai F20 with a 2kx2k CCD detector
- High resolution scanning electron microscope
- Atomic force microscope
- Replacement of out-dated equipment
- CNC system for materials machining

Besides enforced co-operations with groups from other Styrian Universities in the recently accepted ISOTEC-Project of the Austrian NANO Initiative, we hope to increase scientific exchange through cooperation with well known research institutes throughout the world. For the next years we also plan to bring international scientific congresses and workshops to Austria: 2005 we will organize the International EELS Workshop EDGE2005 in Grundlsee, Austria.

Stefan Mitsche trägt beim 2. Institutsseminar am 11.November 2003 vor.
Stefan Mitsche presents his lecture at the 2. institute seminar on November 11th , 2003.

Danksagung

Die experimentelle Arbeit auf dem Gebiet der Elektronenmikroskopie benötigt neben der vollen Motivation aller Mitarbeiter auch beträchtliche finanzielle Unterstützung. Ohne die Hilfe vieler Institutionen wäre es nicht möglich gewesen, das Niveau der Gerätschaften, der Kooperationen und der Resultate, wie es in diesem Leistungsbericht präsentiert wird, zu halten und zur Verfügung zu stellen. Im Berichtszeitraum schulden wir daher den Repräsentanten unserer Universität besonderen Dank:
Rektor O.Univ.-Prof. Dr. Erich HÖDL,
Rektor O.Univ.-Prof. Dr. Hans SÜNKEL,
Vizerektor Hofrat Dr. Johann THEURL,
Dekan O.Univ.-Prof. Dr. Robert TICHY und der zentralen Universitätsverwaltung.

Die finanzielle Unterstützung unserer Arbeit wurde hauptsächlich durch zahlreiche Förderorganisationen gewährt. Dies sind im Detail:

- Fonds zur Förderung der wissenschaftlichen Forschung (FWF)
- Forschungsförderungsfonds der gewerb-lichen Wirtschaft (FFF)
- Austrian Cooperative Research (ACR)
- Bundesministerium für Wirtschaft und Arbeit (BMWA) in Wien,
- Steirische Wirtschaftsförderung (SFG)
- Steiermärkische Landesregierun (Lan-deshauptmannstellvertreter Dipl.-Ing. Leopold SCHÖGGL) .

Ausdrücklich danken wir auch Professor Dipl.-Ing. Dr. h.c. Helmut LIST, Komm. Rat. Dipl.-Ing. Ulrich SANTNER, Komm.Rat Dr. DDipl.-Ing. Gerhard KATZENBERGER, die viel von ihrer kostbaren Zeit dafür aufgewendet haben, unsere Arbeit im Verein zur Förderung der Elektronenmikroskopie zu unterstützen.

Acknowledgements

The experimental work in electron microscopy not only needs full motivation of all collaborators, but also significant financial support. Without the help of many institutions it would not be possible to maintain and to provide this level of the instrumentation, the co-operations and the quality of the results presented in this performance report. We are especially grateful to the officials of our university:
Rector O.Univ.-Prof.Dr. Erich HÖDL,
Rector O.Univ.-Prof. Dr. Hans SÜNKEL,
Vice Rector Hofrat Dr. Johann THEURL ,
Dean O.Univ.-Prof. Dr. Robert TICHY
as well as the central administration of the university.

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

- Austrian Science Fund (FWF) in Vienna,
- Austrian Industrial Research Promotion Fund (FFF) in Vienna,
- Austrian Cooperative Research (ACR),
- Federal Ministry of Economics and Labour in Vienna,
- Styrian Business Promotion Agency (SFG),
- Government of Styria in Graz (Landeshauptmannstellvertreter Dipl.-Ing. Leopold SCHÖGGL).

Last but not least we must particularly thank Professor Dipl.-Ing. Dr.h.c. Helmut LIST, Komm.Rat Dipl.-Ing. Ulrich SANTNER and Komm.Rat Dipl.-Ing. Gerhard KATZEN-BERGER, who spend much of their valuable time supporting our work in the “Verein zur Förderung der Elektro-nenmikroskopie”.

Symposium "Neues aus der Welt der Mikro- und Nanowelt", 28.10.2004
„News from the Micro- and Nanoworld", October 28th 2004

Landeshauptmannstellvertreter
Dipl.-Ing. Leopold Schöggl

Rektor Univ.-Prof. Dr,
Dipl.-Ing.Hans Sünkel

From left:
Univ.-Prof. Dr. Wolfgang Ernst
Univ.-Prof. Dr. Helmut Jäger
KR Dipl.-Ing. Ulrich Santner
Hofrat Dr. Herwig Horn

3. Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung

Die Industriepartner-Organisation wurde 1959 gegründet, um das Institut zu unterstützen und eine stärkere Wechsel-wirkung zwischen industriellen und uni-versitären Forschern zu ermöglichen. Auf der einen Seite unterstützte der Verein das Institut bezüglich der Verbesserung der Gerätschaften, was (zumindest zum Teil) eine hochmoderne Geräteausstattung er-möglichte. Dies war nicht zuletzt wegen der begrenzten universitären Mittel im von zentraler Bedeutung. Auf der anderen Seite konnte durch ihn ein hochqualifiziertes und gut ausgebildetes Stammpersonal am Zentrum für Elektronenmikroskopie (ZFE Graz) unterhalten werden. Das ZFE Graz ist Mitglied der „Austrian Cooperative Research (ACR)"-Organisation.
Das Programm des ZFE Graz ist dahingehend angelegt, der Industrie nützliche Ergebnisse von etablierten oder neu auftauchenden Mikroskopiemethoden zur Verfügung zu stellen und die unternehmensinternen Spezialisten in der Industrie auf dem allerneuesten Stand der Entwicklung in diesem Gebiet zu halten.

The industrial associates' organisation was established in 1959 to support the institute and to facilitate greater interaction between industrial and academic scientists. On the one hand the "Verein" supported the institute in terms of improvement of instrumentation thus enabling state-of-the-art instrumentation (at least partly) which was always very important because of the limited resources of the university. On the other hand it allowed maintaining a high-skilled and well-trained permanent staff in the Center for Electron Microscopy Graz (ZFE Graz). The ZFE Graz is member of the "Austrian Cooperative Research" (ACR) organisation.
The program of the ZFE Graz 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.

Die Politik und die Vorgangsweise der Vereins und des ZFE Graz werden durch einen Lenkungsausschuss aus universitären und industriellen Wissenschaftern festgelegt. Seit 1995 steht Prof. Dipl.-Ing. Helmut LIST (AVL Graz) dem Verein vor. Der Verein hat gegenwärtig 28 Mitglieder primär österreichischer Provenienz. Seit der letzten Hauptversammlung am 28. Februar 2002 setzt sich das Verwaltungsgremium für die nächsten sechs Jahre folgendermaßen zusammen:

Policies and procedures of the "Verein" and the ZFE Graz are established by a steering committee consisting of academic and industrial scientists. Since 1995 the "Verein" is headed by Prof. Dipl.-Ing. Helmut LIST (AVL Graz) and presently the "Verein" has 28 members mainly from Austria. Since the last general business meeting on February 28, 2002 the administrative body for the next six years is given as follows:

Präsidium / Presidential Committee:

Präsident / President:	Prof. Dipl.-Ing. Dr-Ing.h.c. Helmut LIST
1. Stellvertreter des Präsidenten / 1. Vice president:	Komm. Rat Dipl.-Ing. Ulrich SANTNER
2. Stellvertreter des Präsidenten / 2. Vice president:	Komm. Rat DDipl.-Ing. Dr. Gerhard KATZENBERGER

Vorstand / Managing Committee:

Vorsitzender / Head:	Komm. Rat Dipl.-Ing. Ulrich SANTNER
Finanzreferent / Financial referee:	DDr. Wilfried SCHÖNAUER
Vertreter der TU Graz/ Representative of TU Graz:	O. Univ.-Prof. Dr. Hartmut KAHLERT
Leiter des / Head of ZFE Graz:	Ao. Univ.-Prof. Dipl.-Ing. Dr. Ferdinand HOFER

Rechnungsprüfer / Controller:

1. Rechnungsprüfer:	Dr. Hermann PUCHER
2. Rechnungsprüfer:	Mag. Andrea DOLLESCHAL

Wissenschaftlicher Beirat / Scientific Board:

Univ.-Prof. Dipl.-Chem. Dr. Jürgen BESENHARD, TU Graz
Univ.-Prof. Dipl.-Ing. Dr. Horst CERJAK, TU Graz
Univ.-Prof. Dipl.-Ing. Dr. Harald P. FRITZER, TU Graz
Univ.-Prof. Dr. Georg HOINKES, Universität Graz
Univ.-Prof. Dipl.-Ing. Dr. Franz JEGLITSCH, Montanuniversität Leoben
Univ.-Prof. Dr. Hartmuth KAHLERT, TU Graz
Univ.-Prof. Dr. Klaus LEDERER, Montanuniversität Leoben
Univ.-Prof. Dr. Maria-Anna PABST, Universität Graz
Univ.-Prof. Dipl.-Ing. Dr. Franz STELZER, TU Graz

Wirtschaftlicher Beirat / Economic advisory Board

Komm. Rat Dr. Theo GUMPELMAYER, ACR Wien
Dipl.-Ing. Dr. Armin HOLZNER, Semperit Technische Produkte, Wimpassing
Dkfm. R. KOLITSCH, Graz
Dipl.-Ing. Dr. Wolfgang NEISSL, Borealis AG, Linz
Prof. Dr. Bernhard PELZL, Joanneum Research Forschungsgesellschaft mbH, Graz
Min. Rat Dipl.-Ing. Dr. Stefan Kolarsky, Bundesministerium für Bildung, Wissenschaft und Kultur, Wien
Dipl.-Ing. Christian RAINER, Omya, Gummern
Vizerektor Dipl.-Ing. Dr. Johann THEURL, TU Graz

Mitglieder des „Vereins zur Förderung der Elektronenmikroskopie“
Members of the “Verein zur Förderung der Elektronenmikroskopie”

austriamicrosystems Austria Micro Systems AG Unterpremstätten	Omya AG OMYA AG Oftringen, Schweiz
AT&S Austria Technologie & Systemtechnik Leoben	Omya AG OMYA GmbH Gummern
AVL AVL List GmbH Graz	PLANSEE Plansee AG Reutte
BC COMPONENTS BC Components Austria GmbH Klagenfurt	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 Surface Specialties Austria GmbH
B&B Birgl & Bergmeister Papierfabrik GmbH Niklasdorf	SOLVAY Solvay Österreich GmbH Ebensee
EPCOS EPCOS OHG Deutschlandsberg	TANN PAPERS Tann Papier GmbH Traun
Chemson Gruppe Chemson Polymer Additive AG	TUG Technische Universität Graz
IB STEINER Ingenieurbüro Steiner Leoben	Teich Teich AG Weinburg
iv Industriellenvereinigung Graz	TREIBACHER AUERMET Treibacher Auermet GmbH Treibach-Althofen
 Fritz-Haber-Institut der Max-Plank-Gesellschaft	TÜV ÖSTERREICH TÜV Österreich Wien
JOANNEUM RESEARCH Joanneum Research Graz	UNI GRAZ Karl-Franzens-Universität Graz
WKO Wirtschaftskammer Steiermark, Graz	VOITH PAPER Voith Paper Service GmbH Wimpassing
m m m WWW.UNILEOBEN.AC.AT Montanuniversität Leoben	

4. Personalstand / Institute Representatives and Staff

Institutsvorstand / Head of Institute

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

Wissenschaftliche Mitarbeiter / Scientific Staff

ALDRIAN Adolf, Ing. *
BRUNEGGER Albert, Ing.
CHERNEV Boril, Mag. Dr.rer.nat. * (seit / since Okt. / Oct. 2003)
GROGGER Werner, Dipl.-Ing. Dr.techn. ao.Univ.-Prof.
GUPPER Andreas, Dipl.-Ing. Dr.techn. (bis / until Sept. 2003)
INGOLIC Elisabeth, Dr.phil.
KALTMANN Siegfried, Dipl.-Ing.
LETOFSKY-PAPST Ilse, Dipl.-Ing. Dr.mont. (Karenz / Maternity leave)
MAYRHOFER Claudia, Ing. * (seit / since Nov. 2003)
MITSCHE Stefan, Dipl.-Ing.
MITTERBAUER Christoph, Dipl.-Ing. Dr.techn.
PÖLT Peter, Dipl.-Ing. Dr.techn.
REICHMANN Angelika, Dipl.-Ing. Dr.techn. * (seit / since Feb. 2004)
SCHMIED Mario, Dipl.-Ing. Dr.techn.
SCHRÖTTNER Hartmuth, Ing.
WAGNER Julian, Dipl.-Ing. Dr.techn.
WEWERKA Karin, Dipl.-Ing. Dr.techn. (seit / since Oct. 2003)
WARBICHLER Peter, Dipl.-Ing. Dr.techn. (Ruhestand / retired Okt. / Oct. 2003)
WILHELM Peter, Dr.phil.
ZEDLACHER Harald, Dipl.-Ing. *

Doktoranden / PhD students

GSPAN Christian, Dipl.-Ing. ** (seit / since Okt. / Oct. 2003)
RECHBERGER Werner, Mag.Mag.
ROGERS Michael, Dipl.-Ing.
SCHAFFER Bernhard, Dipl.-Ing. **
ZANKEL Armin, Dipl.-Ing.

Allgemeine Mitarbeiter / General Staff

ARNUSCH Denise, Lehrling Bildlabor / Image lab-apprentice
BAHR Peter, Operator Elektronenmikroskopie / EM Operator
BIRNSTINGL Gerhard, Mechaniker / Mechanic
BRANDL Christian, Operator Elektronenmikroskopie/ EM Operator *
BRUNEGGER Margit, Assistent Chemielabor / Chem.lab-assistant *
CZAPEK Wolfgang, Mechaniker / Mechanic *
DIENSTLEDER Martina, Assistent Chemielabor / Chem.lab-assistant *
ELIS Christof, Operator Elektronenmikroskopie / EM Operator
FREUND Angela, Reinigungskraft / Cleaner
GOGER Sabine, Sekretariat / Secretariat *
GUSMAGG Anneliese, Sekretariat / Secretariat *
KORTSCHAK Elisabeth, Chemisch-technische Assistentin / Chem.tech. assistant * (maternity leave until May 2003)

KRANZELBINDER Elke, Reinigungskraft / Cleaner
PALLER Manuel, Assistent Chemielabor / Chem.lab assistant *
ROßMANN Anita, Lehrling Chemielabor / Chem.lab apprentice
SEIDL Emanuel, Lehrling Mechanik / Mech. apprentice
SIMIC Sanja, Operator Elektronenmikroskopie / EM Operator *
STREUßNIG Fatima, Sekretariat / Secretariat *
VEIT Melanie, Lehrling Chemielabor / Chem.lab-apprentice
WALLNER Margit, Bildlabor / Image lab
WINDISCH Gerhard, Design, PC- & LAN-Admin.

* Mitarbeiter des ZFE / ZFE staff, ** Projektmitarbeiter / supported by projects

Gastforscher / Guest scientists

Dr. Joachim LOOS, Eindhoven University of Technology, Eindhoven, Holland / The Netherlands, Februar – März 2003 / February –March 2003
Dr. Koji KIMOTO, Avanced Materials Laboratory, Tsukuba, Japan, März 2004 / March 2004
Dr. Olga VOLOBUJEVA, Technical University Tallinn, August 2004
Dr. Vesna SROT, Jozef Stefan Institut, Ljubljana, Slovenia, November 2004
Dr. Thomas MOSKALEWICZ (AGH University of Science and Technology, Krakow, Poland, Dezember 2004 / December 2004.

Back row from left: G.Birnstingl, H.Schröttner, C.Brandl, W.Rom, A.Zankel, M.Rogers, S.Mitsche, P.Bahr, C. Elis, W.Czapek, C.Gspan, H.Zedlacher, P.Pölt, G.Windisch, J.Rattenberger, E.Seidl,
Middle row from left: S.Kaltmann, E.Kranzelbinder, S.Goger, P.Wilhelm, A.Roßmann, M.Sezen, M.Brunegger, A.Freund, M.Albu, W.Grogger, F.Streussnig, A.Gusmagg, A.Reichmann, C.Mayerhofer, M.Paller, U.Stürzenbecher, G.Kothleitner, E.Ingolic
Front row from left: D.Arnusch, M.Dienstleder, E.Tchernykova, W.Rechberger, F.Hofer, A.Brunegger, B.Schaffer, B.Chernev, M.Wallner, J.Wagner

5. Laboreinrichtungen und wichtigste Instrumente / Laboratory Facilities and Highlights of Equipment

Rasterelektronenmikroskope (REM)

- Analytisches Hochauflösungs-REM: LEO Gemini DSM986
  0.1-30 kV, Feldemissionsquelle, mit EDX-Detektor Noran Voyager 3105A, mit TSL-EBSD-Detektor, Mikrohärte-tester (Anton Paar) und Tieftemperatur-Probentransfersystem (entwickelt am FELMI-ZFE)
- Niederdruck-Rasterelektronenmikroskop (ESEM): FEI Quanta 200 mit Noran Vantage EDX-System, Heiz-bühne (bis 1500°C), Peltier-Kühltisch
- Analytisches REM: Philips SEM505
  1-30 kV, LaB_6-Kathode, EDX-Detektor DX4 (EDAX) mit ultradünnem Fenster und Microspec WDX-System 2A mit Mehrschichtkristallen
- Probenbühnen für Tieftemperaturuntersuchungen, Mikro-Röntgenfluoreszenz und Röntgenmikroskopie.

Scanning Electron Microscopes (SEM)

- 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 200 equipped with Noran Vantage EDX system, heating stage (up to 1500°C), Peltier cooling-stage
- Analytical SEM: Philips SEM505
  1-30 kV, LaB_6 cathode, EDX detector DX4 (EDAX) with ultra-thin window and Micro-spec WDX system 2A with multilayer crystals
- 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 TM FIB/SEM ausgerüstet mit einem Omniprobe™-Manipulator, verschiedenen Gasinjektionssystemen (Pt-Abscheidung, I_2 für erhöhte Metallätzung, XeF_2 für erhöhte Isolatorätzung), EDAX Genesis EDX system, Ionendetektor (CDEM).

Focused Ion Beam Instrument (FIB)

- FEI Nova™ 200 NanoLab: DualBeamTM FIB/SEM equipped with Omniprobe™ manipulator, various Gas injection systems (Pt deposition, I_2 enhanced metal etch, XeF_2 insulator enhanced etch), EDAX Genesis EDX system, Direct Ion Detector (CDEM)

Transmissionselektronenmikroskope (TEM)

- Analytisches Hochauflösungs-TEM: FEI TECNAI F20
  200 kV, Feldemissionsquelle, Super-Zwillings-Objektivlinse, STEM (Sonde 0.2 nm) mit HAADF-Detektor, with EDX-Si(Li)-Leichtelement-Detektor (EDAX) und High Resolution Gatan Imaging Filter (HRGIF) mit 1kx1k-CCD-Kamera
- Analytisches TEM: Philips CM20
  200 kV, LaB_6-Kathode, Zwillingslinse, STEM mit SE-Detektor und Gatan BF/DF-

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 (HRGIF) with 1kx1k CCD camera
- Analytical TEM: Philips CM20
  200kV, LaB_6 cathode, twin lens, STEM with SE detector and Gatan BF/DF detector,

Detektor, EDX-Detektor (HPGe, Noran) und Gatan Imaging Filter (inkl. 1kx1k-CCD-Kamera)

- Analytisches TEM: FEI Tecnai F12
  120 kV, LaB_6-Kathode, Zwillingslinse, and EDX-Si(Li)-Detektor mit ultradünnem Fenster (EDAX), CCD-Kamera
- Probenhalter: Philips 2-Achsen-Kipphalter, Philips Kryotransfer- und Kühlhalter
  Gatan 2-Achsen-Kipp-Kühlhalter für analytische Arbeiten, „Low-background"-Halter, Dreh- und Heizhalter

Weitere Gerätschaften

- FT-IR-Mikroskope
  1) Bruker Equinox 55 Spektrometer mit einem Hyperion 3000 Mikroskop, ATR-Objektive (Ge-Kristall), Glanzwinkel-Objektiv, MIRacle Einzelreflex-Horizontal-ATR-Einheit (Diamant- und Ge-Kristall), Sadtler KnowItAll Datenbanken und Suchsoftware.
  2) Modernes analytisches Mikroskop von Spectra-Tech mit ATR-Objektiv (ZnSe- and Ge-Kristalle), angeschlossen an ein Bomem-MB-Spektrometer.
- Raman-Mikroskop: Renishaw System 2000, mit Leica DMLM Forschungsmikroskop, duales Lasersystem: Diodenlaser (782 nm, 25 mW) und HeNe-Laser (633 nm, 17 mW), holographische Kerbfilter, CCD-Detektor, motorgesteuerte xyz-Bühne für Mapping- und Konfokalexperimente, Raman-Imaging, elektrochemische Zelle, elektrochemische Zelle, Heiz-Kühltisch
- Lichtmikroskop: Zeiss Axioplan zur Beobachtung in Durch- und Auflicht mit Hellfeld, Dunkelfeld, mit Polarisation, Interferenzkontrast (DIC), Phasenkontrast und einer Polaroid DMC Digital Microscope Camera.
- Stereo-Lichtmikroskop Leica M Z 6 zur Probenpräparation
- Lichtmikroskop METAVAR (Reichert)
- Modernes Lichtmikroskop „Alicona Infinite Focus" für die 3D-Topographie (Alicona, Grambach)

EDX detector (HPGe, Noran) and Gatan Imaging Filter (including a 1kx1k CCD camera)

- Analytical TEM: FEI Tecnai F12
  120 kV, LaB_6 cathode, twin lens, and EDX Si(Li) detector with ultrathin window (EDAX), 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

Further Instrumentation

- 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.
- Stereo light microscope Leica M Z 6 for sample- preparation
- Light microscope METAVAR (Reichert)
- Advanced light microscope: "Alicona Infinite Focus" for 3D topography (Alicona, Grambach)

Präparative Gerätschaften für die Elektronenmikroskopie

- Diamantsäge (Well)
- Diamantsäge ISOMET 1000 (Buehler)
- Minimet Polisher (Buehler)
- Ultraschall-Kernschneider (Gatan)
- Muldenschleifgerät (Gatan)
- Dreibein-Poliergerät (Southbay Technology)
- Elektrolytische Dünnungsapparatur (Struers TenuPol 5)
- Kleinwinkel-Ionendünnungsapparat (entwickelt am FELMI-ZFE), ausgerüstet mit Niedrigenergie-Ionenquellen (Technoorg Linda)
- Ionendünnungs- und Poliersystem PIPS mit digitaler Zoom-Kamera (Gatan)
- Kryopräparationssystem EPA 101 mit Quadrupol-Massenspektrometer QMG311 (Entwicklung des ZFE)
- Verdampfer- und Sputteranlage (Entwicklung des ZFE)
- Präparationssystem EPA101 (Entwicklung des ZFE)
- Elektronenstrahlverdampfer (Leybold, Balzers)
- Experimentelles Elektronenmikroskopie-Präparationssystem basierend auf einem EPA101 (Entwicklung des ZFE)

- Computer-Netzwerk mit 95 Computern, 3 Switches (PC, Mac, Unix)
- Canon Farblaserdrucker und qualitative hochwertige Drucker für Photographien

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)
- Electrolytic thinning device (Struers TenuPol 5)
- 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 EPA101 (ZFE development)
- Electron beam evaporators (Leybold, Balzers)
- Experimental electron microscopical preparation system based on EPA101 (ZFE development)

- Computer network with 95 computers, 3 switches (PC, Mac, Unix)
- Canon Color Laser Printer and High quality printers for photographs

6. Lehrveranstaltungen / Academic Education

Wir bieten moderne und flexible Lehrveranstaltungen inkl. Diplomarbeiten und Dissertationen an. Die Schulung der Mitarbeiter und Studenten durch Einbindung in die Forschung spielt eine wesentliche Rolle. Der Mitarbeiterstab des FELMI bietet nachfolgende Lehrveranstaltungen in Physik, Chemie und Materialwissenschaften an der Technischen Universität Graz an.

We offer modern and flexible courses including Diploma 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 the Graz University of Technology.

No.	Sem.	Title	Type	Lecturer
519.001	SS	Electron Microscopy in Solid State Physics I	2 Vo	W. Grogger
519.002	WS	Electron Microscopy in Solid State Physics II	2 Vo	W. Grogger
513.010/11	WS/SS	Applied Physics Laboratory Course	5 Pr	J. Wagner
511.	WS/SS	Experimental Physics Course	6 Pr	J. Wagner
519.007	WS	Electron Microscopy in Materials Science	2 Vo	G. Kothleitner
519.008	SS	Materials Characterization by Electron Microscopy	2 Pr	G. Kothleitner
519.013	WS	Analytical Electron Microscopy	2 Vo	F. Hofer
519.014	SS	Structure Analysis by High Resolution Electron Microscopy	3 Se	F. Hofer
519.015	WS	Special Aspects of Analytical Electron Microscopy	3 Se	F. Hofer
519.016	SS	Structure Analysis by High Resolution Electron Microscopy	2 Vo	F. Hofer
519.065	WS	Structure Analysis by High Resolution Electron Microscopy	2 Vo	F. Hofer
539.211	WS	High Performance Polymers	1 Vo	K. Wewerka

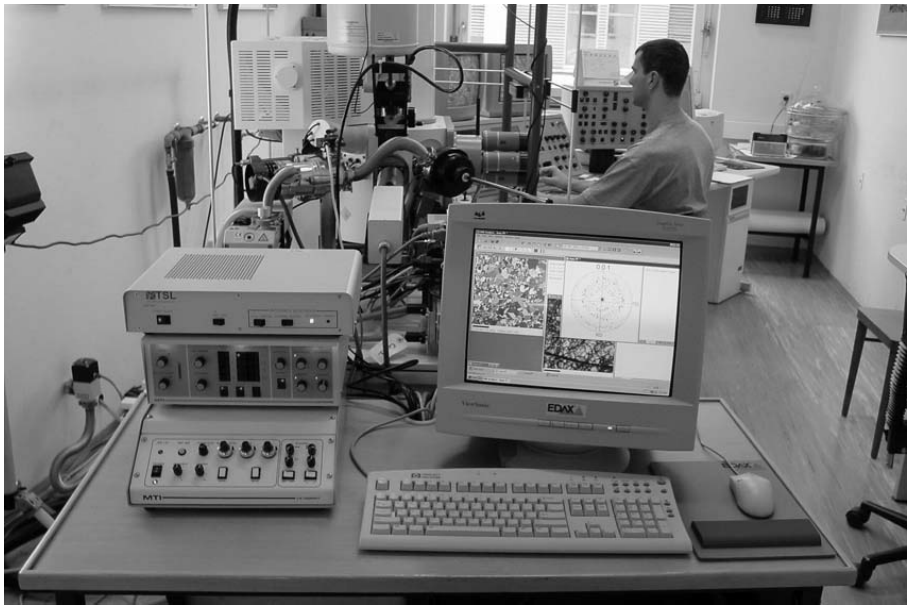
Der Mitarbeiterstab des FELMI ist intensiv in das Programm des **Universitätslehrganges "Nanotechnologie und Nanoanalytik"** (Abschluss: Master of Advanced Studies) eingebunden.

FELMI staff is heavily engaged in the programme of the **University Course "Nanotechnology and Nanoanalysis"** (Degree: Master of Advanced Studies).

No.	Sem.	Title	Type	Lecturer
ULG 003	WS	Nanoanalysis and Structural Investigations with Microscopical Methods	2 Vo	F. Hofer
ULG 004	SS	Micro- and Nanoanalysis in the Electron Microscope	2 Pr	W. Grogger,M. Schmied
ULG 120	WS	High Resolution Elctron Microscopy	2 Vo	F. Hofer
ULG 125	SS	New methods in Light Microscopy	2 Pr	P. Wilhelm
ULG 126	SS	Nanostructuring of Materials with Ion- and Electron Beams	2 Pr	P. Pölt

Zusätzlich wurden Institutsführungen mit Vorlesungen und Demonstrationen für Physik- und Chemielehrerexkursionen sowie für Studierende der TU Graz, von Schulen und lokalen Universitäten angeboten. Des Weiteren haben wir im November 2003 für interessierte Personen außerhalb der Universität eine URANIA-Vorlesungsreihe zum Thema „Elektronen-mikroskopie“ organisiert.

Tours of the institute including lectures and demonstrations have additionally been offered for excursions of physics and chemistry teachers and for students from the TUG, schools and local universities. Moreover, we have organised URANIA-lectures in November 2003 presenting the achievements of “Electron Microscopy” to interested people from outside of the universities.



Ing. H. Schröttner at the scanning electron microscope Gemini / LEO equipped with a FEG, EDX-spectrometer, cryo-system and EBSD system

KR Dipl.-Ing. Ulrich Santner and Ferdinand Hofer in the ZFE booth at the MATERIALICA 2004, Munich

7. Dissertationen und Diplomarbeiten / Diploma Theses and Doctoral Theses

7.1. Abgeschlossene Habilitationen / Finished Habilitations

Ao.Univ.-Prof. Dr. Dipl.-Ing. Werner GROGGER
„Quantitative nanoanalysis at high spatial resolution using energy-filtering transmission electron microscopy“
Ao.Univ.-Prof. Dr. Dipl.-Ing. Gerald KOTHLEITNER
“Considerations regarding the performance, operation and data analysis with electron energy-loss spectrometers and energy-filtering transmission electron microscopes“

7.2. Dissertationen und Diplomarbeiten am FELMI / Doctoral Theses and Diploma Theses at the FELMI

- Abgeschlossene Dissertationen / Finished doctoral thesis:
  Dipl.-Ing.Andreas GUPPER: „Enhanced applications of microscopic characterisation techniques in materials science”
  Dipl.-Ing. Christoph MITTERBAUER: „Investigations of nanostructured materials by using electron energy loss spectroscopy in the transmission electron microscope“
- Derzeit laufende Dissertationen / Doctoral theses in progress:
  Dipl.-Ing. Andreas DITTMANN: “Microscopical characterization of tribological systems”
  Dipl.-Ing. Christian GSPAN: “Electron microscopical investigation of La(Sr,Co)O3 perowskites”
  Dipl.-Ing. Stefan MITSCHE: „Chemical and crystallographical characterization of submicron particles and thin layers in the scanning electron microscope “
  Dipl.-Ing. Bernhard SCHAFFER: „Characterisation of nanocrystalline, electro active materials by means of high resolution electron microscopy and energy filtering microscopy”
  MMag. Werner RECHBERGER: „Characterising crystalline materials with high resolution scanning transmission electron microscopy and electron energy loss spectroscopy“
  Dipl.-Ing. Michael ROGERS: „Electron microscopical investigation of nanoparticles and functional nanostructures“
  Dipl.-Ing. Armin ZANKEL: „In-situ experiments in the environmental scanning electron microscope“
- Derzeit laufende Diplomarbeit / Diploma thesis in progress:
  Katharina RIEGLER: “TEM-investigation of noble metal nanoparticles”

7.3. Unterstützung von Dissertationen und Diplomarbeiten von anderen Universitätsinstituten / Doctoral Theses and Diploma Theses in other university institutes

Graz University of Technology

Fakultät für Technische Mathematik und Technische Physik / Faculty for Physics

- Institut für Festkörperphysik / Institute of Solid State Physics
  Dipl.-Ing. Stefan MÜLLEGGER, Doctoral Thesis
  Dipl.-Ing. Alexander POGANTSCH, Doctoral Thesis
  Dipl.-Ing. Horst SCHEIBER
  Dipl.-Ing. Gernot PAUER, Doctoral Thesis

Dipl.-Ing. Christian SÜSS, Doctoral Thesis
Dipl.-Ing. Franz-Peter WENZL, Doctoral Thesis

- Institut für Experimentalphysik / Institute of Experimental Physics
  Mag.Dieter SOMITSCH, Doctoral Thesis
  Dipl.-Ing. Boris WILTHAN, Doctoral Thesis
  Julia GREINER, Diploma Thesis
  Ilse KRISCHE, Diploma Thesis

Fakultät für Technische Chemie, Verfahrenstechnik und Biotechnologie / Faculty of Chemistry, Engineering and Biotechnology

- Institut für chemische Technologie anorganischer Stoffe / Institute for Chemical Technology of Inorganic Materials
  Dipl.-Ing. Hosseinmardi AZARNOUSH, Doctoral Thesis
  Josef HARING, Diploma Thesis
  Dipl.-Ing. Tiziana PIPOLI, Doctoral Thesis
  Dipl.-Ing. Atanaska TRIFONOVA, Doctoral Thesis
  Joong-Hee HAN, Doctoral Thesis
  Dipl.-Ing. Markus R. WAGNER, Doctoral Thesis
  Dipl.-Ing. HEIJZE, Doctoral Thesis
  Dipl.-Ing. Angelika BASCH, Doctoral Thesis
  Dipl.-Ing. Markus THALER, Doctoral Thesis
  Dipl.-Ing. Peter RAIMANN, Doctoral Thesis
  Dipl.-Ing. Christiane KOREPP, Doctoral Thesis
  Anilkumar METTU, Doctoral Thesis
  Dipl.-Ing. Eva WALLNÖFER, Doctoral Thesis
  Dipl.-Ing. Hosseinmardi AZARNOUSH, Doctoral Thesis
  Dipl.-Ing. Klaus LEITNER, Doctoral Thesis
  Dipl.-Ing. R. DORNER, Doctoral Thesis
- Institut für chemische Technologie organischer Stoffe / Institute for Chemical Technology of Organic Materials
  Dipl.-Ing. Gernot LANGER, Doctoral Thesis
  Martin TSCHERNER, Diploma Thesis
  Dipl.-Ing. Sonja SMOLAK, Doctoral Thesis
  Dipl.-Ing. Thomas E. HAMEDINGER, Doctoral Thesis
  Dipl.-Ing. Martin KNIENDL, Doctoral Thesis
  Dipl.-Ing. Stefan KONTUR, Doctoral Thesis
  Monika PIBER, Diploma Thesis
- Institut für Anorganische Chemie / Institute of Inorganic Chemistry
  Dipl.-Ing. Janette STEPHAN, Doctoral Thesis
- Institut für Ressourcenschonende und Nachhaltige Systeme / Institute of Ressource Efficient and Sustainable Systems
  Dipl.-Ing. Markus JÖLLER, Doctoral Thesis
- Institut für Apparatebau, Mechanische Verfahrentechnik und Feuerungstechnik / Institute of Chemical Apparatus design, Particle technoogy and Combusdion
  Dipl.-Ing. Andreas ANZEL, Doctoral Thesis
  Dipl.-Ing. Stephen SACHER, Doctoral Thesis
- Institut für Biochemie / Institute for Biochemistry
  Dipl.-Ing. Alice LOIDL, Doctoral Thesis
  Heidemarie MÜLLNER, Doctoral Thesis
  Tamara WRIESNEGGER, Diploma Thesis

- Institut für Thermische Verfahrenstechnik und Umwelttechnik / Department of Chemical Engineering and Environmental Technology
  Dipl.-Ing. Martin ERNST, Doctoral Thesis
- Institut für Physikalische und Theoretische Chemie / Institute of Physical and Theoretical Chemistry
  Dipl.-Ing. Edith BUCHER, Doctoral Thesis

Fakultät für Bauingenieurwissenschaften / Faculty of Civil Engineering

- Institut für Angewandte Geowissenschaften / Institute of Applied Geosciences
  Dipl.-Ing. Barbara KOSEDNAR, Doctoral Thesis
  Dipl.-Ing. Jens HILLEBRECHT, Doctoral Thesis
- Institut für Baustatik / Institute for Structural Analysis
  Dipl.-Ing. Gerhard SOMMER, Doctoral Thesis

Fakultät für Informatik / Faculty of Computer Science

- Institut für Maschinelles Sehen und Darstellen / Institute for Computer Graphics and Vision
  Dipl.-Ing. Pierre ELBISCHGER, Doctoral Thesis

Fakultät für Maschinenbau und Wirtschaftswissenschaften / Faculty of Mechanical Engineering and Economics

- Institut für Werkstoffkunde, Schweißtechnik und Spanlose Formgebungsverfahren / Institute of material science, welding and forming
  Dipl.-Ing. Martin STOCKINGER, Doctoral Thesis
  Dipl.-Ing. Rudolf VALLANT, Doctoral Thesis
  Dipl.-Ing. Bernhard SONDEREGGER, Doctoral Thesis
- Institut für Strömungslehre und Wärmeübertragung / Institute of Fluid Mechanics and Heat Transfer
  Dipl.-Ing. Vladimir KOLOBARIC, Doctoral Thesis

Karl-Franzens-Universität Graz / University of Graz

- Institut für Pathophysiologie / Institute of Pathophysiology
  Claudia MAYER, Diploma Thesis
- Institut für Zoologie / Institute of Zoology
  Monika SCHMIKL, Diploma Thesis
  Markus PROKSCH, Diploma Thesis
  Tobias PFINGSTL, Diploma Thesis
  Sylvia SCHÄFFER, Diploma Thesis
- Institut für Chemie / Institute of Chemistry
  Dipl.-Chem. Jörg ENGSTLER, Doctoral Thesis
  Sylvia SCHÄFFER, Diploma Thesis
- Institut für Allgemeine und Experimentelle Pathologie / Institute of General and Experimental Pathology
  Mag. Nicole SCHEER, Doctoral Thesis
- Institut für Experimentalphysik / Institute for Experimental Physics
  Mag. Petra GRANITZER, Doctoral Thesis
  Dipl.-Ing.Mag. Christina GATSCHELHOFER / Inst. für Medizinische Systemtechnik und Gesundheitsmanagement / Institute for Medical technique and health management, JOANNEUM RESEARCH, Graz

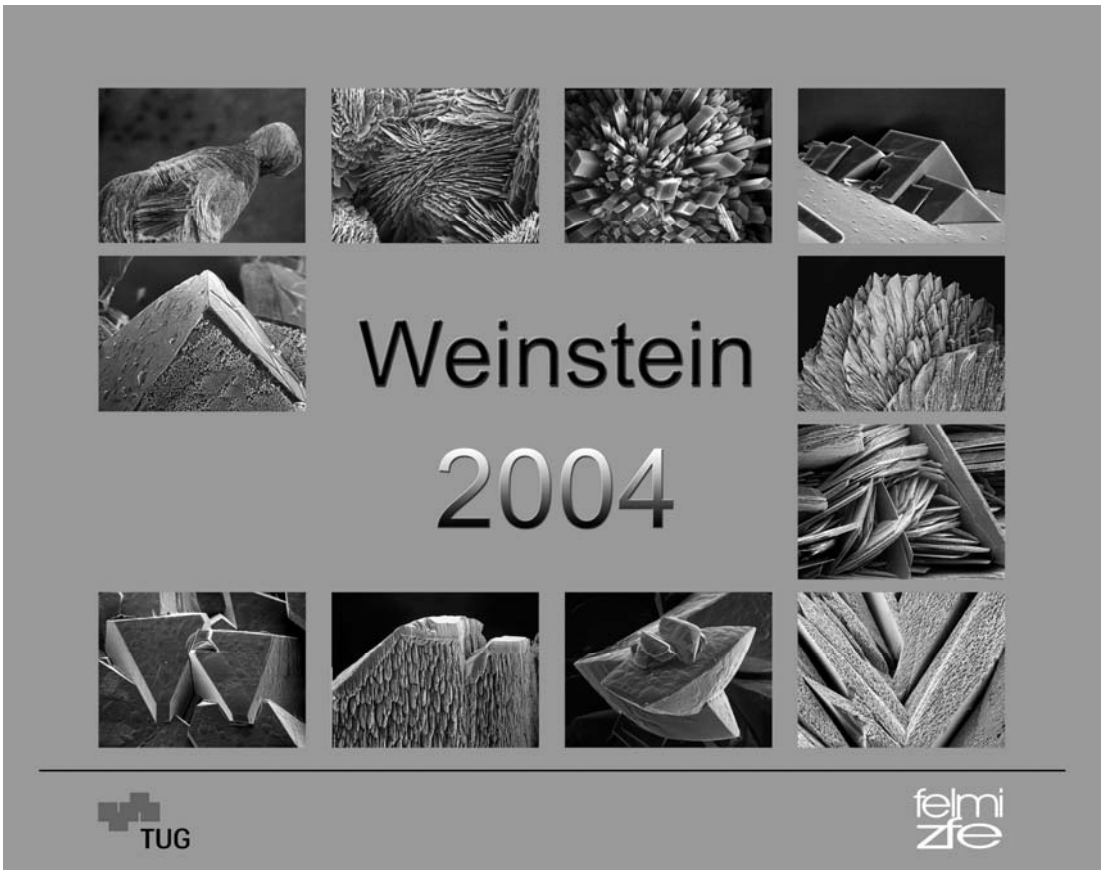
Montanuniversität Leoben / University of Leoben

- Institut für Physik / Institute of Physics
  Dipl.-Ing. Sascha KREMMER, Doctoral Thesis

- Department Allgemeine, Analytische und Physikalische Chemie / Department General, Analytical and Physical Chemistry
  Doris SCHERER, Diploma Thesis
  Christian GROGER, Diploma Thesis
- Werkstoff-Kompetenzzentrum-Leoben Forschungsgesellschaft mbH / Materials Center Leoben
  Dipl.-Ing. Ingo SILLER, Doctoral Thesis
- Polymer Competence Center Leoben GmbH / Polymer Competence Center Leoben
  Dipl.-Ing. Armin TEMEL, Doctoral Thesis
- Department Metallkunde und Werkstoffprüfung / Department of Physical Metallurgy and Materials Testing
  Dipl.-Ing. Michael BISCHOF, Doctoral Thesis

Technische Universität Wien / Vienna University of Technology

- Institut für Straßenbau und Straßenerhaltung
  Dipl.-Ing. Klaus STANGL, Doctoral Thesis
- Institut für Angewandte Physik
  Dipl.-Ing. Julian WAGNER, Doctoral Thesis
- Institut für Angewandte Synthesechemie
  Dipl.-Ing. Michael KUNZ, Doctoral Thesis
  Mag. Doris MACHL, Doctoral Thesis



FELMI-ZFE Kalender für das Jahr 2004 / Calendar for 2004

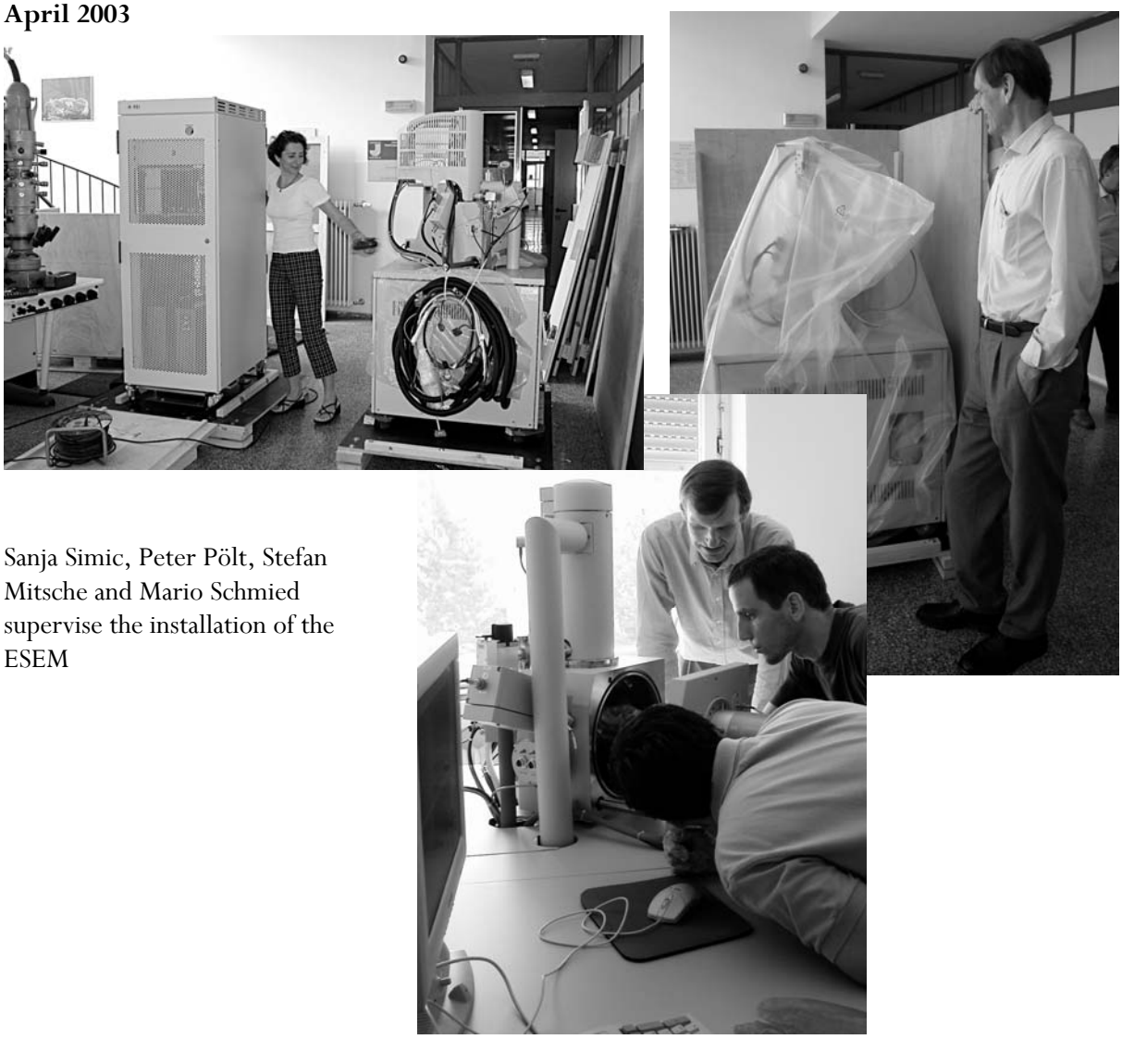
8. Forschungsaktivitäten der Arbeitsgruppen / Main Research Areas of the Workgroups

Die Forschungsaktivitäten des Institutes konzentrieren sich auf die Entwicklung neuer mikroskopischer Charakterisierungsmethoden, die für die Untersuchung von Mikrostrukturen in unterschiedlichsten Festkörpern, Werkstoffen und biologischen Proben eingesetzt werden: z.B. Legierungen, Stähle, Metalle, Keramiken, Verbundwerkstoffe, Mineralien, Polymere und Biomaterialien.
2003 und 2004 wurden Fördermittel durch den FFF, den FWF im Rahmen des Sonderforschungsbereiches "Elektroaktive Stoffe" und den Zukunftsfonds Steiermark zugeteilt.

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 2003 and 2004 research funds were allocated by the FFF, the FWF within the Special Research program "Electroactive materials" and the Zukunftsfonds Steiermark.

Installation des Grobvakuum-Rasterelektronenmikroskops FEI Quanta 600 im April 2003
Installation of the Environmental Scanning Electron Microscope FEI Quanta 600 during April 2003



Sanja Simic, Peter Pölt, Stefan Mitsche and Mario Schmied supervise the installation of the ESEM

8.1. Mikroanalytik mittels Rasterelektronenmikroskopie

Die Rasterelektronenmikroskopie ermöglicht eine nahezu vollständige Charakterisierung von Bulkproben: Oberflächentopographie (auch quantitativ, inkl. Ermittelung der Oberflächenrauhigkeit mittels Stereobildern); chemische Zusammensetzung (EDXS, WDXS) und kristallographische Mikrostruktur (EBSD).
Ein computergesteuertes REM in Kombination mit einer Bildverarbeitung ermöglicht die automatische Analyse von geometrischen als auch chemischen Parametern tausender Einzelteilchen bis hinab zu einer Größe von etwa 100 nm. Dies wird für die Analyse von Feinstaub in Umweltproben, z.B. Aerosole und Flugascheteilchen in Biomasse-Verbrennungsanlagen genutzt. Zahlreiche Proben, insbesondere kleine Partikel aus der Umwelt, erfahren durch den Elektronenbeschuss eine starke Schädigung. Sie ändern ihre Topographie als auch ihre chemische Zusammensetzung. Die grundlegenden Mechanismen sind noch nicht hinreichend geklärt. weshalb die grundlegenden Reaktionen, die zur Probenschädigung führen, studiert werden.
EBSD (Elektronenrückstreubeugung) im Rasterelektronenmikroskop ermöglicht die Identifizierung der Kristallphase der Mikrostrukturen und die Bestimmung der Orientierung der einzelnen Kristallite. Durch das Rastern der Probe über ausgewählte Probenbereiche erhält man einen vollständigen Datensatz zur Mikrostruktur dieser Bereiche. Danach können Korngrößenverteilungen errechnet werden, eine Texturanalyse und eine quantitative Bestimmung von teilweise rekristallisierten Polykristallen vorgenommen werden.
Die konventionelle REM ist auf den Hochvakuumbereich beschränkt. Daher stellt das Grobvakuum REM (ESEM) eine extrem wichtige Erweiterung dar, da damit in der Probenkammer Drücke bis hinab zu etwa 10 Torr und unterschiedlichste Gasatmosphären möcglich sind. Dies ermöglicht die direkte Untersuchung von feuchten Proben. Zusätzlich kann durch die Verwendung einer Heizbühne (bis zu 1500°C) die Probentemperatur variiert

8.1. Microanalysis using scanning electron microscopy

Scanning electron microscopy enables a nearly complete characterisation of a bulk specimen: surface topography (also quantitative, including surface roughness by use of stereo images); chemical composition (EDXS, WDXS); and the crystallographic microstructure (EBSD).

Computer controlled SEM in combination with image processing enables the unattended analysis of both the geometrical and chemical parameters of thousands of individual particles down to a size of around 100 nm. This is used for the analysis of aerosols and fly ash particles e.g. in biomass heating plants.

Many specimens, especially small environmental particles, experience strong specimen damage by electron irradiation. They change both their topography and their chemical composition. The basic mechanisms are not completely revealed now. Work is in progress to elucidate the fundamental reactions causing the damage.

EBSD (electron backscatter diffraction) in the scanning electron microscope enables the identification of the crystal phase of microstructures and the determination of the orientation of individual crystallites.
By scanning the specimen across selected areas of the specimen one obtains a full record of the microstructure of these areas. Subsequently grain size distributions can be calculated, texture analysis and quantification of partially re-crystallised polycrystals performed, etc.

The conventional SEM operates in the high vacuum range. On the other hand the Environmental SEM (ESEM) permits pressures down to around 10 Torr, and the use of different chamber gases. This makes the investigation of wet specimens possible. Additionally, by use of a heating stage (up to 1500°C), the specimen temperature can be varied.

Thereby chemical reactions like corrosion of metals, electrolyte – solid interactions, but also the formation of alloys can be studied on a

werden. Dadurch können chemische Reaktionen wie die Korrosion von Metallen, Elektrolyt-Festkörper-Wechselwirkungen, aber auch die Legierungsbildung im Mikrometer-Maßstab beobachtet werden. In naher Zukunft werden sich unsere Forschungsarbeiten auf die Weiterentwicklung von in-situ-Experimenten im ESEM konzentrieren. Ein Meilentein auf diesem Weg ist die Entwicklung einer neuen Methode für die Aufnahme dreidimensionaler Element-Verteilungsbilder.

micrometer scale. Thus in the near future developments of the in-situ experiments in the ESEM will form an essential part of the research activities.
Presently, we plan a project for developing a new method for recording 3D elemental maps.

8.2. Nanoanalytik mittels analytischer Transmissionselektronenmikroskopie

Die mechanischen, chemischen und elektrischen Eigenschaften von Festkörpern werden nicht nur durch die Kristallstruktur des perfekten Materials bestimmt sondern auch durch ihre Defektstruktur. So hängen beispielsweise die mechanischen Eigenschaften von Stählen von der Art, der Größe und der Verteilung der Ausscheidungen ab, die Festigkeit von Keramiken wird durch das Vorhandensein von Sekundärphasen kontrolliert, und die Qualität von Halbleiter-Bauelementen hängt von der Kontrolle der chemischen und strukturellen Defekte ab. Ein bedeutendes Forschungsziel der Gruppe liegt daher in der Korrelation dieser physikalischen Eigenschaften mit der Mikrostruktur von Festkörpern.
Die Transmissionselektronenmikroskopie (TEM) gestattet es, strukturelle Informationen über die meisten Feststoffe (inkl. Polymere und biologische Gewebe) bis in den atomaren Maßstab zu erlangen. Infolge ihres nahezu stetigen Vergrößerungsbereiches von 20x bis 1000000x können Defekte leicht identifiziert und untersucht werden. Diese Untersuchungen werden durch analytische Methoden wie Elektronenenergieverlustspektrometrie (EELS), energiefilternde TEM (EFTEM) und energiedispersive Röntgenspektrometrie (EDXS) unterstützt und ergänzt, woduch die Analyse der chemischen Zusammensetzung von dünnen Proben bis in den Nanometerbereich möglich wird.
Die Forschung innerhalb der Gruppe konzentriert sich auf die Verbesserung der Methoden zur Erfassung von EELS- und

8.2. Nanoanalysis using analytical transmission electron microscopy

The mechanical, chemical or electrical properties of materials are not only determined by the crystal structure of the perfect material, but also by their defect structure. For example, the mechanical properties of steels depend on the type, size and distribution of precipitates, the strength of ceramics is monitored by the presence of impurity phases and the quality of semiconductor devices depends on the control of chemical and structural defects. A major research objective of the group is to correlate these physical properties to the microstructure of materials.

Transmission electron microscopy (TEM) allows obtaining structural information of most solid state materials (including polymers and biological tissue) down to an atomic scale. Due to its almost continuous magnification range between 20x and 1.000.000x defects can be easily identified and studied. These investigations are supported or supplemented by analytical techniques such as electron energy-loss spectrometry (EELS), energy-filtering TEM (EFTEM) and energy-dispersive x-ray spectrometry (EDXS) which allow analysing the elemental and chemical composition of thin samples down to a nanometre scale.

Research within the group is concentrated on improving techniques for recording EELS- or EFTEM data at high spatial resolution such as elemental mapping with EFTEM, and EFTEM spectrum imaging. In a second step we focus on the development or improvement of new or

EFTEM-Daten mit hoher räumlicher Auflösung wie "Elemental mapping" mit EFTEM und „EFTEM spectrum imaging". Im zweiten Schritt fokussieren wir uns auf die Entwicklung und Verbesserung von neuen oder vorhandenen Auswertungsmethoden, z.B. die Quantifizierung von EELS-Spektren und / oder EFTEM-Elementverteilungen, die Interpretation und Simulation von Nahkanten-Feinstrukturen (ELNES) und erweiterte Energieverlust-Feinstrukturen (EXELFS) um chemische Bindungsinformation aus nanometergroßen Probenbereichen offen zu legen. Erste Schritte zu einer verbesserten Interpretation dieser Phänomene wurden mit der Einführung von ELNES-Simulationen gesetzt.
Der Aufbau des weltweit zweiten Monochromator-TEM-Systems (Februar 2003), das in der Lage ist EELS-Spektren mit hoher Energieauflösung aufzunehmen und daher chemische Bindungsinformation aus nanometer kleinen Probenbereichen liefert, wird unsere Forschungsbemühungen auf dem Gebiet der Nanoanalytik massiven Auftrieb verleihen.
Wir versuchen weiters unser Verständnis der Wechselwirkung zuwischen hochenergetischen Elektronen mit Materie zu verbessern, da dies eine Voraussetzung für die quantitative Analyse mittels EELS und EFTEM darstellt respektive dem Verständnis der ultimativen Grenzen in der Charakterisierung von Chemie auf dem atomaren Maßstab dient.

existing data reduction methods, e.g. quantitative analysis of EELS spectra and/or EFTEM elemental maps, interpretation and simulation of near edge fine structures (ELNES) and extended energy-loss fine structures (EXELFS) for revealing chemical bonding information from nanometre sized specimen regions.
In collaboration with groups from the University of Leeds (Rik Brydson) and the Vienna University of Technology (Peter Schattschneider) we have successfully started to simulate the ELNES structures.

The introduction of the second monochromated TEM system in the world which is able to record EELS spectra with high energy resolution thus offering chemical bonding information from nanometre sized specimen regions, will help to boost our research efforts in the field of nanoanalysis.

Furthermore, we try to increase the understanding of the interaction of high energy electrons with matter which is prerequisite for quantitative analysis using EELS and EFTEM or for understanding the ultimate limits for the characterization of chemistry at near atomic scale.

8.3. Raman- und FTIR-Mikrospektroskopie

Infrarot- und Raman-Spektroskopie sind leistungsfähige analytische Methoden zur Identifi-kation von organischen und anorganischen Substanzen, basierend auf der Anregung von Schwingungen innerhalb des Moleküls. In Verbindung mit einem Lichtmikroskop können kleine und komplexe Proben mit hoher lateraler Auflösung untersucht werden. (IR: ca. 10 µm, Raman: ca. 1-2 µm), wobei die Probenpräparation - wenngleich sie nicht unterbewertet werden soll – in den meisten Fällen minimal gehalten werden kann. Umfassenden Spektren-Datenbanken ermöglichen die Charakterisierung von

8.3. Raman- and FTIR microspectroscopy

Infrared and Raman spectroscopy are powerful analytical techniques for the identification of organic and inorganic substances, based on the excitation of vibrations within the molecule. Coupled to a light microscope, small and complex samples can be analysed with high lateral resolution (IR: ca. 10 µm, Raman: ca. 1-2 µm), whereby sample preparation – though not to be underestimated - in most cases can be kept to a minimum. Comprehensive spectral libraries enable the characterisation of polymers, fillers, gels, contaminations etc., thus complementing the results of X-ray spectroscopy (elemental composition) obtained

Polymeren, Füllstoffen, Defekten etc. und ergänzen sich so mit der Röntgenspektroskopie (Elementzusammensetzung) aus dem Elektronenmikroskop.
Die Forschung innerhalb der Gruppe konzentriert sich auf die Untersuchung von Polymeren mittels Mikro-Raman- und Infrarot-Spektroskopie und bildgebenden Verfahren. Die Kontrolle der Polymerorientierung und -kristallinität sind von hohem industriellem Interesse um die Eigenschaften von Kunststoffen zu verstehen und zu verbessern. Weitere Untersuchungen befassen sich mit der Morphologie von Polymergemischen und der thermischen Degradation von Polyvinylchlorid.
Da das neue IR-Mikroskop mit einem FPA-Detektor ausgerüstet wurde, können jetzt auch IR-Bilder aufgenommen werden. Aus einem Gesichtsfeld von 270 µm * 270 µm im Transmissionsmodus respektive 50 µm * 50 µm in ATR-Modus können 64*64 Spektren gleichzeitig aufgenommen werden, um die Verteilung der einzelnen Komponenten mit höchstmöglicher lateraler Auflösung anzuzeigen. Dünne organische Filme (monomolekulare Lagen) auf reflektierenden Substraten können mittels eines GIR-(grazing incidence reflection)-Objektivs untersucht werden.

8.4. Probenpräparation und Geräteentwicklung

Das Institut kann auf eine langjährige Tradition in der Entwicklung von elektronenmikroskopischen Präparationsapparaturen zurückblicken. Infolge dieser Bemühungen haben wir einen hohen Standard bei der Präparation von elektronenmikroskopischen Proben, was eine wesentliche Voraussetzung für den erfolgreichen Einsatz der Elektronenmikroskopie darstellt. Derzeit konzentrieren wir uns auf die Verbesserung von Zuguntersuchungen im ESEM und die Verbesserung der Präparation von TEM-Proben (Kombination der FIB-präparierten Proben mit der Niederenergie-Ionendünnung).
Aufgrund des langjährigen Know-hows auf dem Gebiet der Kryo-Elektronenmikroskopie sehen wir auch gute Entwicklungsmöglichkeiten für die Untersuchung von „weichen Materialien".

in the electron microscope.

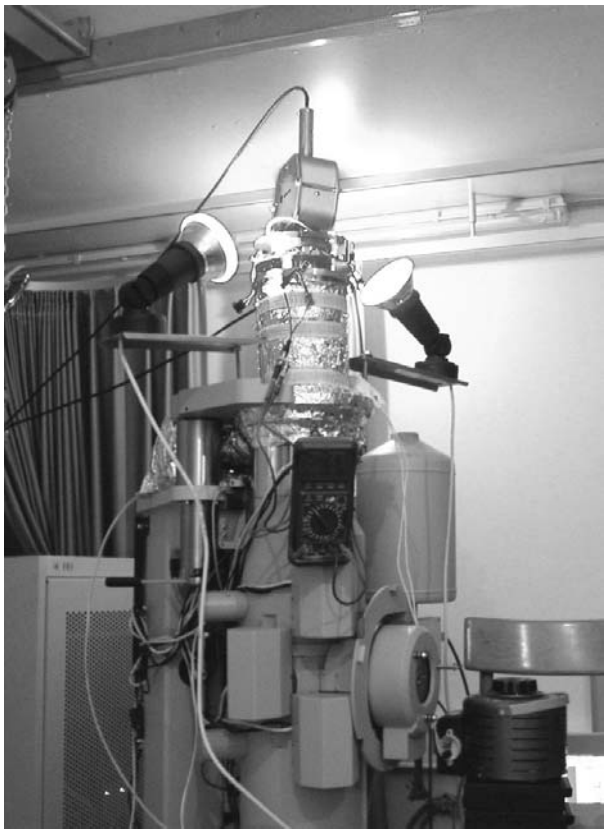
Scientific research within the group is concentrated on the studies of polymers using micro Raman and infrared spectroscopy and imaging techniques. Control of polymer orientation and crystallinity is of high industrial interest in order to understand and improve the properties of plastic materials. Further studies are done on the morphology of polymer blends, and the thermal degradation of polyvinyl chloride.

New IR instrumentation allows IR imaging with an FPA detector. From an area of 270 µm * 270 µm in transmission mode or 50 µm * 50 µm in ATR mode, respectively, 64*64 spectra can be taken simultaneously, to display the distribution of individual components with the highest possible lateral resolution. Thin organic films (monomolecular layers) on reflecting substrates can be analysed by a GIR (grazing incidence reflection) objective.

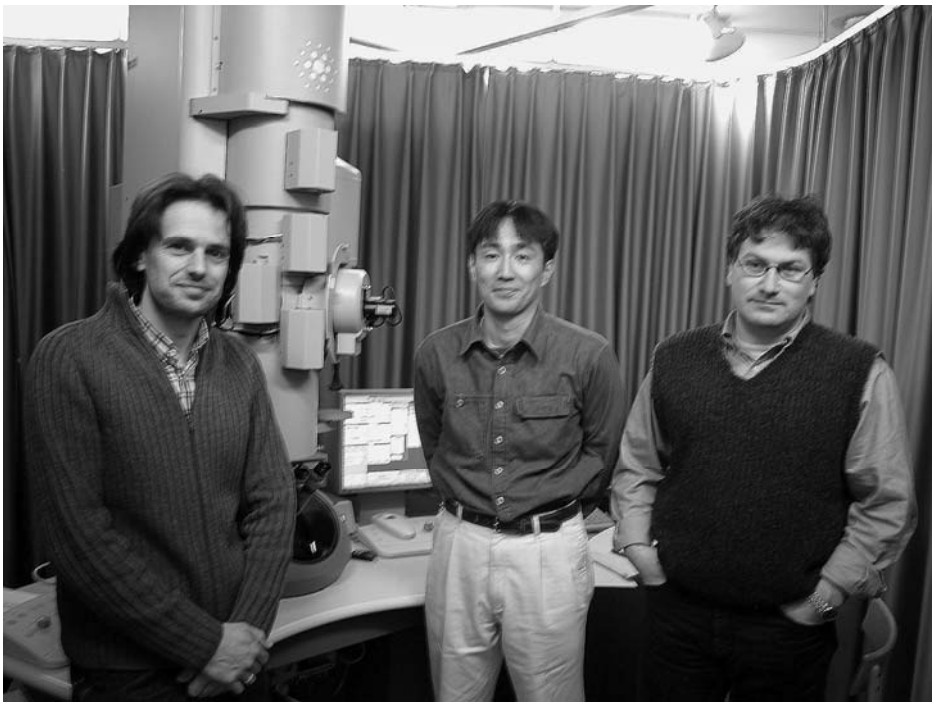
8.4. Specimen preparation and apparatus development

The institute has a long and well known tradition in the development of electron microscopical preparation equipment. Due to these efforts we have a high standard for the preparation which is prerequisite for the successful application of electron microscopy. During the last years we concentrated on the construction of auxiliary tools, such as improving a tensile stage for investigations in the ESEM. Additionally, the preparation of TEM specimens by polishing FIB prepared samples with low energy ion milling is established.
The institute has a well known expertise in the field of cryo electron microscopy which will be enforced in near future. In this respect we will concentrate our research activities on the characterisation of all kinds of soft matter.

Einbau des Monochromators in das Hochauflösungselektronenmikroskops Tecnai F20 im Februar 2003
Installation of a monochromator on the high resolution electron microscope Tecnai F20 during February 2003



Ausheizen des Kathodenraums
Heating of the gun chamber



Dr. Werner Grogger, Dr. Koji Kimoto (Tsukuba, Japan) and Dr. Gerald Kothleitner in front of the monochromated Tecnai F20

9. FELMI-ZFE Projekte / 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, May 1, 1999 – April 30, 2006.
- „Nanoanalytical characterization of multilayered biomaterials", Wissenschaftlich-Technische Zusammenarbeit Österreich-Polen, ÖAD Project 9/03, 1.1.2004 – 31.12.2005.
- „Energy-filtered transmission electron microscopy (EFTEM) and high resolution scanning transmission electron microscopy (HR-STEM) of nanoparticles and interfaces in materials", Wissenschaftlich-Technisches Abkommen Österreich-Slowenien, ÖAD Project SI-A19/0405, 1.1.2004-31.12.2005.
- "Improvement of nanoanalytical characterization techniques for the study of inner boundaries in materials", supported by FFF, Vienna, January 1, 2000 – December 31, 2003.
- „Study of polymer orientation with Raman microscopy", supported by FFF, Vienna, September 1, 2000 – Mai 30, 2003.
- "Orientation imaging microscopy and phase analysis of materials", supported by FFF, Vienna, November 1st, 2000 – October 31st, 2003.
- Project „Wachstumsförderung", arranged by ACR and supported by Federal Ministry for Economic Affairs and Labour (BMWA), Vienna, January 1, 1999 – December 31, 2003.
- „Multimethodenanalytik von Nanoteilchen und Nanoteilchenverbunden", supported by "Steiermärkischer Zukunftsfonds", in cooperation with University of Leoben and Karl-Franzens University Graz, July, 1, 2003 – June, 30, 2006.
- „Neue Untersuchungsmethoden für Mikrosystemtechnik und Nanotechnologie", supported by Federal Ministry for Education, Science and Culture (BMBWK), Vienna, recommended by the Rat für Forschung und Technologieentwicklung (RFT), June 1, 2002 – June 30, 2003.
- "Nanoanalysis and Nanostructuring for Organic Optoelectronic Devices" within the ISOTEC-Project (FWF and Austrian Nanotechnology Initiative, Austrain Space Agency, Vienna) March 1st, 2005 – February 28th, 2007.
- PROKIS-Project "Kompetenzerweiterung ZFE Graz" arranged by ACR and supported by Federal Ministry for Economic Affairs and Labour (BMWA), Vienna, January 1, 2003 – December 31, 2006.
- 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), October 1, 2004 – December 31, 2006.

10. Vorträge am Institut / Presentations at the Institute

25.2.2003: Dr. Joachim **LOOS** (Technical University of Eindhoven, Eindhoven, The Netherlands): „Struktur-Eigenschaft Relationen in Polymer Systemen"

28.4.2003: Dr. Klaus-Jochen **EICHHORN** (Inst. f. Polymerforschung Dresden, Germany): „Ellipsometrie - eine optische Methode zur Charakterisierung komplexer Polymerstrukturen auf Festkörperoberflächen"

27.5.2003: Dr. Christina **SCHEU** (Max-Planck-Institut für Metallforschung, Stuttgart, Germany): „TEM Investigations of Thin Metal Films on $SrTi0_3$ Substrates"

12.-14.6.2003: Interntional Workshop "EELS of Steels", Bruck an der Mur, Austria
Dr. Jim **BENTLEY** (Oak Ridge National Laboratory, USA) "EFTEM & Spectrum Imaging of Segregation and Phase Transformations in Steels and Alloys"
Dr. Rik **BRYDSON** (University of Leeds, U.K.) "EELS Studies of the Dissolution of Cementite during the Tempering of Carbon Steels"
Prof. Dr. Alan **CRAVEN** (University of Glasgow, U.K.) "The Use of ELNES in the Study of Precipitate Evolution in Low Alloy Steels"
Prof. Dr. Joachim **MAYER** (RWTH Aachen, Germany) EFTEM and Electron Diffraction Investigations of Nb-doped and TRIP Steels
Prof. Dr.Mark **RAINFORTH** (University of Sheffield, U.K.) "Spectroscopic Imaging of NbC Precipitates on Dislocation Cores in a Model Fe-30Ni Microalloyed Steel"
Dr. Colin **SCOTT** (CMC IRSID, Maizieres-les-Metz, France) "PEELS and the Design of New Steels for the Automotive Sector"

24.6.2003: Dr. Ursula **DIEFENBACH** (Freie Universität Berlin, Institut f. Chemie, Berlin, Germany) am Institut: „Multifunktionelle Phosphazene - Struktur, Eigenschaften, Anwendungsgebiete"

2.10.2003: Dr. Piet **LEEMSTRA** (Dutch Polymer Institute, Technical University of Eindhoven, Eindhoven, The Netherlands): „Future of Polymer Science and Technology in Europe"

22.10.2003: Dr. David **Mc COMB** (Department of Materials Imperial College London, U.K.): „Electron energy-loss spectroscopy: A sub-nanometre probe of chemistry, structure and bonding"

12.11.2003: Dr. Ken **WILLIAMS** (Renishaw PLC, Wotton-under-Edge Gloucestershire, U.K.): „The Structural and Chemical Analyzer – A succesful marriage between sanning electron microscopy and Raman-spectroscopy"

27.11.2003: Prof.Dr.H.-P. **KARNTHALER** (Inst. f. Materialphysik, Univ. Wien, Austria): „Atome sehen im Transmissionselektronenmikroskop"

23.3.2004: Dr. Koji **KIMOTO** (Advanced Materials Laboratory, National Institute for Materials Science, Japan): „Improvement and application of TEM-EELS for Materials Science"

30.3.2004:Dr. Michael **KUNDMANN** (Gatan Inc., Pleasanton, USA): „Current State of Post-Column Energy Filtering for Analytical TEM

8.5.2004: Dr. Petr **SKOCOVSKY** (FEI Company Brno, Czechia): „New Aspects from ESEM Research and Development"

15.6.2004: Dr. Bradley **THIEL** (University of Cambridge, Cavendish Laboratory, Great Britain): " Secondary Electron Imaging of Dielectric Materials in Low Vacuum SEM"

15.6.2004: Prof. Dr. Wolfgang **NEUMANN** (Department of Physics, Humboldt University, Berlin, Germany): "Strukturelle und chemische Charakterisierung nanostrukturierter Halbleitermaterialien mittels Transmissionselektronenmikroskopie".

16.8.2004: Dr. Helmuth **MEISSNER** (Onyx Optics, Pleasanton, USA): „Composite crystals for solid state lasers"

11.11.2004: Prof. Dr. Vesna **VOLOVSEK** (Department of Physics, Univ. of Zagreb, Croatia): „Vibrational Analysis of Polymerization of Aminopropylsilanetriol“

9.12.2004: Dr. Francis **MORRISSEY** (FEI Company, Eindhoven, The Netherlands): “An overview of FIB and dualbeam applications”

Preis für ZFE Graz beim Businessplan-Wettbewerb der Austrian Cooperative Research (ACR), Wien am 4. November 2003

Award for the business plan of the ZFE Graz in the busisnnes plan competion of the Austrian Cooperative Research (ACR), Vienna November 4th 2003

Dr. Michael Binder (RFT), Prof. Dr. Theo Gumpelmayr (ACR), Dr. Ferdinand Hofer, Dipl.-Ing. Harald Zedlacher, Dr. Gerald Kothleitner, Karl Wizany (BMWA)

11. Publikationen von Institutsmitarbeitern / Publications of Institute Staff

2003

C. Mitterbauer, G. Kothleitner, W. Grogger, H. Zandbergen, B. Freitag, P. Tiemeijer, F. Hofer
"Electron Energy-Loss Near Edge Structures of 3d Transition Metal Oxides Recorded at High Energy Resolution", Ultramicroscopy 96, (2003) 469-480.

G. Kothleitner, C. Mitterbauer, W. Grogger, H. Zandbergen, P. Tiemeijer, B. Freitag, M. Barfels, F. Hofer
"First Applications of Electron Energy-Loss Spectroscopy with High Energy Resolution", Mat. Res. Soc. Symp. Proc. Vol. 738, (2003) 27-32.

W. Sitte, E. Bucher, W. Preis, I. Papst, W. Grogger, F. Hofer
"Microstructural Aspects of the Ionic Transport Properties of Strontium-Substituted Lanthanum Cobaltites", Mat. Res. Soc. Symp. Proc. Vol. 756, (2003) 527-532.

W. Kohs, H. J. Santner, F. Hofer, H. Schröttner, J. Doninger, I. Barsukov, J. H. Albering, K.-C. Möller, J. O. Besenhard, M. Winter
"A Study on Electrolyte Interactions with Graphite Anodes Exhibiting Structures with Various Amounts of Rhombohedral Phase", J. Power Sources 199-121, (2003) 528-538.

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"EELS Performance Measurements on a New High Energy Resolution Imaging Filter", Micron 34, (2003) 211-218.

J. H. Obenholzner, H. Schröttner, H. Delgado
"Barite aerosol particles from volcanic plumes and fumaroles - FESEM/EDS analysis", EGS-AGU-EUG Joint Assembly, Nice, France, April 2003; Abstract CD-ROM "Geophysical Research Abstracts", Vol. 5, (2003) EAE03-A-08119.

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"Glass shards, pumice fragments and volcanic aerosol particles - diagenesis a recorder of volcanic activity?", EGS-AGU-EUG Joint Assembly, Nice, France, April 2003; Abstract CD-ROM "Geophysical Research Abstracts", Vol. 5, (2003) EAE03-A-08137.

A. Gupper, P. Wilhelm
"Spectroscopic characterisation of different commercial polypropylene products", 15th European Symposium on Polymer Spectroscopy, Heraklion, Crete, June 2003, Book of Abstracts (2003) p.82.

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"Spectroscopic imaging techniques for characterisation of polymer morphologies: a combination of infrared and electron microscopy", 15th European Symposium on Polymer Spectroscopy, Heraklion, Crete, June 2003, Book of Abstracts (2003) p.66.

W. Rechberger, P. Warbichler, G. Kothleitner, F. Hofer
"Effects of Sample Preparation on Cr-Steels: An EFTEM Study", Microsc. Microanal. Vol. 9 (Suppl. 3), (2003) 90-91.

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"Energy-filtering TEM at the frontier of spatial and energy resolution", "Advanced Materials 2003", Proc. 10th Int. Symp. Adv. Mater. (ISAM 2003), eds. Y. Bando et al., Tsukuba, Japan, (2003) pp. 41-42.

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"High Energy Resolution EELS with a Monochromated (S)TEM", 6. Multinat. Congr. Microsc. - Europe. Extens., Pula (2003) pp. 175-176.

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A. Gupper, P. Wilhelm, M. Schiller

"Degradation of Poly(vinyl Chloride) with Different Additives, Studied by Micro Raman Spectroscopy", Polymers & Polymer Composites, 11 (2003) 123-132.

J. Obenholzner, P. Pölt, H. Schröttner, H. Delgado

"Barite aerosol particles from volcanic plumes and fumaroles - FESEM/EDS analysis", Workshop des Osservatorio Vesuviano: "Vesuvio: dentro il vulcano", Ercolano, Napoli (8.-10. Mai 2003), Abstract book (2003) p. 48.

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"Energiefilternde Elektronenmikroskopie am Beispiel eines Cr-Stahles", Prakt. Met. Sonderband 34 (2003) 229-234.

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"Width determination of SiO_2 films in Si-based devices using low-loss EFTEM: image contrast as a function of sample thickness", Micron, 34 (2003) 1-7.

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"Imaging of ultrathin silicon oxide layers in semiconducting devices by means of energy filtering transmission electron microscopy (EFTEM)", 13th Int. Conf. on Microscopy of Semiconducting Materials (MSM13), Cambridge (2003).

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F. Hofer

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C. Mitterbauer

"Elektronenmikroskopische Untersuchungen von nanostrukturierten elektroaktiven Materialien", SFB-Seminar, Reinischkogel, Austria, February 2 to 4, 2003.

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G. Kothleitner

Seminar "Grundlage von Mapping und Quantifizierung mittels EELS und die praktische Umsetzung", "Quantitative EELS", "Quantitative EFTEM", "EELS Spectrum Imaging", "Filter Performance Tuning", Fitz-Haber-Institut Berlin (Max-Planck-Society), Germany, March 24 to 30, 2003.

F. Hofer (invited)

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G. Kothleitner (invited)

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"Synthesis of Multicomponent Nano-Particles with an Aerosol Reactor – Reference Materials for SEM/EDXS Analyses", SCANNING 2003, San Diego, CA, USA, Mai 5, 2003.

F. Hofer

"Neue Methoden der Festkörpercharakterisierung am FELMI", Forschungstag „Advanced Materials", TU Graz, Austria, Mai 8, 2003.

C. Mitterbauer

"High Energy Resolution EELS with a Monochromated (S)TEM", Multinational Congress on Electron Microscopy, Pula, Croatia, June 1 to 5, 2003.

P. Wilhelm

"Spectroscopic Imaging Techniques for Characterization of Polymer Morphologies: A Combination of Infrared and Electron Microscopy", 15th European Symposium on Polymer Spectroscopy (ESOPS 15); Crete, Greece, June 8 to 12, 2003.

A. Gupper

"Spectroscopic Characterization of Different Commercial Polypropylene Products", 15th European Symposium on Polymer Spectroscopy (ESOPS 15); Crete, Greece, June 8 to 12, 2003.

F. Hofer

"EFTEM jump ratio imaging for the detection of precipitates in steels and alloys", Workshop on "EELS of Steels & Alloys", Bruck an der Mur, Austria, June 12 to 14, 2003.

G. Kothleitner

"Elemental Occurrence Maps and MLS-fitting to EELS Spectrum Images: Novel Approaches for the Compositional Analysis of Steels and Alloys", Workshop on "EELS of Steels & Alloys", Bruck an der Mur, Austria, June 12 to 14, 2003.

C. Mitterbauer

"ELNES as a Fingerprint for Identifying Transition Metal Nitrides", Workshop on "EELS of Steels & Alloys", Bruck an der Mur, Austria, June 12 to 14, 2003.

W. Grogger

"EFTEM Investigation of a Ferritic ODS Alloy: From Spectra to Chemical Phase Maps", Workshop on "EELS of Steels & Alloys", Bruck an der Mur, Austria, June 12 to 14, 2003.

F. Hofer

"Elektronenmikroskopie in den Technischen Biowissenschaften", Workshop über "Forschungsschwerpunkt Technische Biowissenschaften", TU Graz, Austria, June 25, 2003.

P. Pölt, M. Schmied

"Materialcharakterisierung mittels Elektronenmikroskopie", Fa. Magna, Graz, Austria, July 2, 2003.

F. Hofer (invited)

"Quantitative nanoanalysis by electron energy-loss spectrometry and energy filtering",1st Stanislaw Gorczyca Summer School on Advanced TEM: "Diffraction and analytical methods for phase identification", Krakow, Poland, July 2 to 5, 2003.

C. Mitterbauer

"Comparative EELS near-edge fine structure investigations of titanium oxides", Microscopy and Microanalysis 2003, San Antonio, Texas, August 3 to 7, 2003.

F. Hofer (invited)

"Energy-filtering TEM at the frontier of spatial and energy resolution", EUROMAT 2003, Lausanne, Switzerland, September 1 to 9, 2003.

P. Pölt

"SEM/EDXS and Polymers – A sociable couple?", Electron Microscopy and Analysis Group (EMAG) 2003, Oxford, U.K., September 2 to 5, 2003.

F. Hofer (invited)

"Analytical Electron Microscopy in Materials and Biological Sciences", Microscopy Conference MC2003, Dresden, Germany, September 7 to 12, 2003.

B. Schaffer

"TEM Investigations of Cross-Sectional Prepared Organic Light Emitting Devices",Microscopy Conference MC2003, Dresden, Germany, September 7 to 12, 2003.

W. Grogger (invited)

"EFTEM and EELS at the Frontiers of Spatial and Energy Resolution: What does "Resolution" mean in Practice?",Microscopy Conference MC2003, Dresden, Germany, September 7 to 12, 2003.

G. Kothleitner

"Experiencies and Possibilities with a 200kV Monochromated (S)TEM", Microscopy Conference MC2003, Dresden, Germany, September 7 to 12, 2003.

P. Wilhelm (invited)

„The Power of FT-IR Imaging in Materials Characterization", The fifth National FTIR Symposium, Vanajanlinna, Finnland, September 10 to 14, 2003.

B. Schaffer

"EFTEM and EELS at the Frontiers of Spatial and Energy Resolution: What does "Resolution" mean in Practice?", 12. Tagung Festkörperanalytik, TU Wien, Austria, September 22 to 24, 2003.

G. Kothleitner

"Performance Tests and Possibilities with a New 200 kV Monochromated (S)TEM",12. Tagung Festkörperanalytik, TU Wien, Austria, September 22 to 24, 2003.

B. Chernev

"Site-resolved X-ray investigations on injection-molded polypropylene filled with magnesium hydroxide", Conference on Interfaces and Interphases in Multicomponent Materials, Balatonfüred, Hungary, October 6 to 9, 2003.

P. Wilhelm (invited)

"Imaging methods applied to polymer blends", The Federation of Analytical Chemistry and Spectroscopy Societies (FACSS) 2003; Lauderdale, USA, October 19 to 23, 2003.

F. Hofer

"Synthesis and electron microscopical characterization of noble metal nanoparticles of various morphologies", Graz – Mainz Joint Seminar on Nanoscience and –technology, Joaneum Reseach, Graz, Austria, October 20, 2003.

W. Grogger

"Nanoanalysis of organic light emitting diodes in the analytical electron microscope", Symposium „Nanotechnology and LEDS", Loipersdorf, Austria, November 18, 2003.

2004

G. Kothleitner (invited)

"Experiencies with a 200 kV monochromated (S)TEM", Australian Conference on Microscopy and Microanalysis 2004, Geelong, Australia, January 28 to February 8, 2004.

G. Kothleitner (invited)

"EELS Quantification and Detection Limits", Australian Conference Workshop on Microscopy and Microanalysis 2004, Geelong, Australia, January 28 to February 8, 2004.

M. Schmied

"Real Sample Features VS. Imaging Artifacts in an ESEM", Australian Conference on Microscopy and Microanalysis 2004, Geelong, Australia, January 28 to February 8, 2004.

F. Hofer (invited)

"Electron Energy-loss Spectrometry in the Electron Microscope", Leitung des Fachsymposiums „Analytische Elektronenmikroskopie an dünnen Schichten", Frühjahrstagung Festkörperphysik 2004, Regensburg, Germany, March 8 to 10, 2004.

G. Kothleitner (invited)

"Monochromated STEM EELS on FIB Samples" at Gatan, Pleasanton, CA, USA, April 26 to May 3, 2004.

F. Hofer (invited)

"Principles of EELS and EFTEM in the transmission electron microscope" and „Application of EELS and EFTEM in materials science and biology", European Microbeam Analysis Society (EMAS) 2004, Bled, Slovenia, May 8 to 11, 2004.

G. Kothleitner

"FIB-Zielpräparation kombiniert mit hochauflösender TEM-EELS-Analytik", Workshop: „Präparative Aspekte der TEM", Fraunhofer-Institut für Werkstoffmechanik, Halle/Saale, Germany, May 12, 2004.

W. Grogger (invited)

"Elektronensondenmethoden – Nanoanalytik im Elektronenmikroskop", Symposium Materialanalytik: Lady or Kitchenmaid? „Elektronensondenmethoden", Analytica Conference, München, Germany, May 11 to14, 2004.

P.Pölt

"Moderne Elektronenmikroskopie in der Papierforschung", WFT Research, Traun, Austria, May 27, 2004.

M. Schmied (invited)

"3D-Surface reconstruction" 3D Microscopy Workshop 2004, Technologiezentrum Grambach, Austria June 21, 2004.

F. Hofer (invited)

"Electron Energy-loss Spectrometry at the Frontiers of Spatial and Energy Resolution", 49th International Field Emission Symposium (IFES04),Seggauberg, Austria, July 12 to 16, 2004.

F. Hofer (invited)

"Electron Energy-Loss Spectrometry at High Energy Resolution for Materials Research",13th European Microscopy Congress, Antwerp, Belgium, August 22 to 27, 2004.

G. Kothleitner (invited)

"Hochauflösende Elektronenenergieverlustspektrometrie an FIB präparierten Lamellen", 13. Arbeitstagung „Angewandte Oberflächenanalytik AOFA 13, Dresden, Germany, Sepember 15, 2004.

F. Hofer

"Nanoanalytik – Methoden - Verfahren - Herausforderungen", Institute for International Research, „Nanotechnologie", Wien, Austria, September 29, 2004.

M.Schmied (invited)

"Advanced Methods for the Study of Microsctructures and Fracture Surfaces", Microstructure Analysis and Fractographie of Ceramics, Montanuniversität Leoben, Austria, September 30,.2004.

W. Grogger (invited)

"EFTEM and EELS at the Frontiers of spatial and energy resolution", Autumn School 2004 on Advanced Materials Science and Electron Microscopy", Humboldt Universität Berlin, October 3rd-7th 2004.

F. Hofer (invited)

"Introduction to Analytical Transmission Electron Microscopy", Autumn School 2004 on Advanced Materials Science and Electron Microscopy", Humboldt Universität Berlin, October 3rd-7th 2004.

G. Kothleitner (invited)

"About the Concept, the Operation and Uses for TEM Gun Monochromators",
Arbeitskreistreffen EELS-EFTEM, Lausanne, October 1st, 2004.

P. Wilhelm (invited)

„Polymer Characterisation with Raman Microscopy", Raman Day, St.Avold, France October 13, 2004.

P.Pölt

"Dynamische Mikroskopie mittels Environmental Scanning Electron Microscopy" Symposium "News from the Micro- and Nanoworld", TU Graz, October 28, 2004.

G.Kothleitner

„Materialcharakterisierung und Nanostrukturierung mit der NANOLAB-Anlage", Symposium "News from the Micro- and Nanoworld", TU Graz, October 28, 2004.

W.Grogger

„Über den Einsatz des Analytischen Hochauflösungselektronenmikroskops" Symposium "News from the Micro- and Nanoworld", TU Graz, October 28, 2004.

P.Wilhelm

„Bildgebende Verfahren in der Mikrosprektrometrie mittels Infrarot- und Raman-Mikroskopie", Symposium "News from the Micro- and Nanoworld", TU Graz, October 28, 2004.

G. Kothleitner

„Seeing the Invisible / Neues aus der Mikro- und Nanowelt", Tagung des Forums Technik u. Gesellschaft, Graz University of Technology, Austria, November 25, 2004.

P.Wilhelm

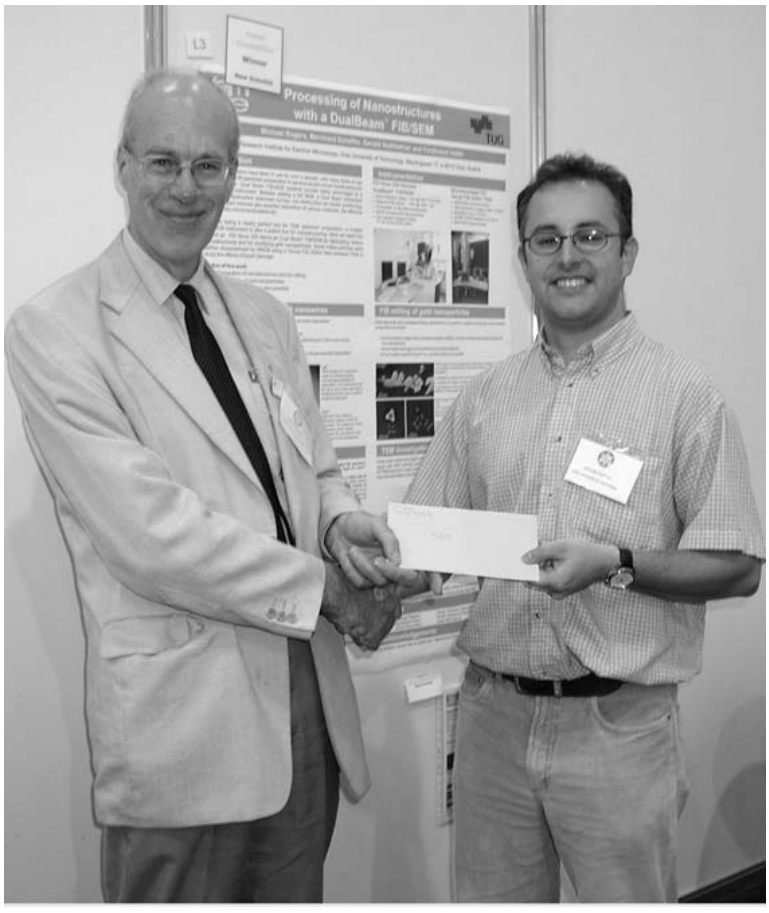
„Exciting new applications of imaging techniques for polymer characterisation", Bruker Anwendertreffen, Ettlingen, BRD, November 16, 2004.

P.Pölt

„Das ESEM-Anwendungen in den Material- und Biowissenschaften", ASEM-Hauptversammlung, University of Salzburg, Austria, December 3, 2004.

F. Hofer

„Neues aus der Mikro- und Nanowelt", EPCOS OHG, Deutschlandsberg, Austria, December 17, (2004).



Professor John Hutchison überreicht Herrn Dipl.-Ing. Michael Rogers den Preis „MicroScience 2004" für das beste Poster bei der MicroScience 2004 in London.

Professor John Hutchison (President Royal Microscopical Society) awards Dipl.-Ing. Michael Rogers for his poster presentation with the prize "MicroScience 2004" at MicroScience 2004 in London.

13. Posterpräsentationen von Institutsmitarbeitern / Poster Presentations by Institute Staff

2003

B. Schaffer

"Imaging of ultrathin silicon dioxide layers in semiconducting devices by means of energy filtering transmission electron microscopy (EFTEM)", XIII. International Conference on Microscopy of Semiconducting Materials, Cambridge, U.K., March 31 to April 3, 2003.

G. Kothleitner

"Digital Filters Applied to EELS Spectrum Images" and "Factors Affecting the Performance of EELS Energy-Filters", Forschungstag "Advanced Materials", "Analytische Transmissionselektronenmikroskopie", TU Graz, Austria, Mai 8, 2003.

P. Pölt

"Analytische Rasterelektronenmikroskopie", Forschungstag "Advanced Materials", TU Graz, Austria, Mai 8, 2003.

P. Wilhelm

"Imaging Methods for Morphological Investigations on a Polymer Blend", Forschungstag „Advanced Materials", "IR- und Ramanmikrospektrometrie", TU Graz, Austria, Mai 8, 2003.

M. Schmied

"Production and Characterization of Ternary $Si0_2/Ti0_2/Fe_20_3$ Nano-Particle Standards for SEM/EDXS Analyses", European Microbeam Analysis Society (EMAS) 2003, Cadiz, Spain, May 18 to 22, 2003.

P. Pölt

"EBSD-Contamination, Calibration and Specimen Damage", European Microbeam Analysis Society (EMAS) 2003, Cadiz, Spain, May 18 to 22, 2003.

J. Wagner

"Measuring the Angular Dependent Energy Distribution of Backscattered Electrons at Variable Geometry", European Microbeam Analysis Society (EMAS) 2003, Cadiz, Spain, May 18 to 22, 2003.

A. Gupper

"Raman Spectroscopy: Quantitative Spectra Interpretation of Oriented Polypropylene Products", 2nd International Conference on Advanced Vibrational Spectroscopy, Nottingham, U.K., August 24 to 29, 2003.

M. Rogers

"Synthesis and characterization of noble metal nanoparticles of various shapes", EUROMAT 2003, Lausanne, Switzerland, September 1 to 9, 2003.

B. Schaffer

"HREM Study of Hexagonal and Rhombohedral Graphites for use as Anodes in Lithium Ion Batteries, Deutsche Gesellschaft für Elektronenmikroskopie (DGE) - Tagung: " Microscopy Conference MC2003", Dresden, Germany, September 7 to 12, 2003.

W. Rechberger

"Effects of samle preparation on Cr-steels: An EFTEM study", Deutsche Gesellschaft für Elektronenmikroskopie (DGE) - Tagung: " Microscopy Conference MC2003", Dresden, Germany, September 7 to 12, 2003.

S. Mitsche

"Quantification of the Recrystallized Fraction in a Nickel-Base-Alloy from EBSD-Data", Deutsche Gesellschaft für Elektronenmikroskopie (DGE) - Tagung: "Microscopy Conference MC2003", Dresden, Germany, September 7 to 12, 2003.

C. Mitterbauer

"High energy resolution EELS using a monochromized 200 kV TEM: Comparative investigation of the Ti $L_{2,3}$ and 0 K edge of titanium oxides", Deutsche Gesellschaft für Elektronenmikroskopie (DGE) - Tagung: "Microscopy Conference MC2003", Dresden, Germany, September 7 to 12, 2003.

P. Wilhelm

"Raman Microscopy: A Convenient Method for the Characterization of Orientation and Crystallinity of Polypropylene", Polymer Crystallization (PC) 2003, Linz, Austria, September 18 to 21, 2003.

M. Rogers

"Synthesis and Characterization of Noble Metal Nanoparticles", Autumn School on Advanced Materials Science and Electron Microscopy 2003, Humboldt University Berlin, Germany, September 27 to October 1, 2003.

B. Chernev

"Effect of compatibilizing agents on the morphology of recycled polymer blends", Conference on Interfaces and Interphases in Multicomponent Materials, Balatonfüred, Hungary, October 6 to 9, 2003.

2004

P. Wilhelm

"Spectroscopic Imaging Techniques for Polymer Characterisation: Infrared and Electron Microscopy – A Good Match", 17th International Symposium on Polymer Analysis and Characterization, Heidelberg, Germany, June 7 to 9, 2004.

S. Mitsche, P. Pölt, C. Sommitsch, M. Walter

"An EBSD-study of recrystallisation behaviour of a nickel base alloy caused by hot compression", Microscience 2004, London, U.K., July 6, 2004.

A. Zankel, P. Pölt, E. Ingolic

"Polymer fracture - in situ observations in the ESEM", Microscience 2004, London, London, U.K. July 6, 2004.

M. Schmied, A. Zankel, H. Schröttner

"Contrast formation in a low vacuum or environmental SEM: a time dependent process", Microscience 2004, London, U.K., July 6, 2004.

M. Rogers, **B.Schaffer, G.Kothleitner, F.Hofer**

"Processing of nanostructures with a DualBeam FIB/SEM", Microscience 2004, July 6 2004.

H. Schröttner, M. Schmied, S. Scherer

"Robust, dense and accurate 3D surface reconstruction in SEM through automatic calibration data calculation from multiple images",13th European Microscopy Congress 2004, Antwerp, Belgium, August 2004.

J. Wagner, M. Schmied, S. Mitsche

"Advanced investigations of the composition of submicron particles by using EDXS and Monte Carlo methods", 13th European Microscopy Congress 2004, Antwerp, Belgium, August 2004.

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

"Characterization and compensation of environmental magnetic fields for a monochromatized TEM", 13th European Microscopy Congress 2004, Antwerp, Belgium, August 2004.

F. Hofer, G. Kothleitner, W. Grogger

"Electron energy-loss spectrometry at high energy resolution for materials research", 13th European Microscopy Congress 2004, Antwerp, Belgium, August 2004.

P. Pölt, S. Mitsche, C. Sommitsch, M. Walter

"EBSD - and the recrystallisation of Ni-base alloys", 13th European Microscopy Congress 2004, Antwerp, Belgium, August 2004.

W. Rechberger, G. Kothleitner, W. Grogger, F. Hofer

"STEM performance on a monochromated TEM", 13th European Microscopy Congress 2004, Antwerp, Belgium, August 2004.

M. Rogers, B. Schaffer, F. Hofer

"Synthesis, size control and electron microscopic characterization of lamellar gold nanoparticles", 13th European Microscopy Congress 2004, Antwerp, Belgium, August 2004.

A. Zankel, P. Pölt, M. Gahleitner, C. Grein, E. Ingolic

"The fracture behaviour of polymers - In-situ investigations in the ESEM",European Microscopy Congress 2004, Antwerp, Belgium, August 2004.

M. Schmied, S. Mitsche

"Channeling contrast by Focused Ion Beam and EBSD - two complementary techniques", 13th European Microscopy Congress 2004, Antwerp, Belgium, August 2004.

B. Schaffer

„High energy resolution EFTEM image series of graphite in the plasmon energy-loss region", Autumn School on Advanced Materials Science and Electron Microscopy 2004, Humboldt University Berlin, Germany, October 1, 2003.

C. Gspan, W.Grogger, C. Mitterbauer, F. Hofer, E. Bucher, W.Sitte

„Superstructure and Domains in $La_{0.4}Sr_{0.6}CoO_{3-\delta}$", Autumn School on Advanced Materials Science and Electron Microscopy 2004, Humboldt University Berlin, Germany, October 1, 2003.

B. Chernev

"Comparative Imaging Studies of Polymer Blends", 6th IPF Colloquium, Dresden, Germany, November 21, 2004.

14. Fortbildungsaktivitäten für Institutsmitarbeiter / Research Visits of Staff Members in other Institutions

2003

April 2003	Dipl.-Ing. J. Wagner, Ing. H. Schröttner at 4th Annual UK-Nordic ESEM Userclub Meeting in Eindhoven, The Netherlands, Work plan: ESEM Facilities
May 2003	Dr. P. Wilhelm, Dipl.-Ing. A. Gupper, Ch. Brandl at Bruker-Seminar in Graz, Austria, Work plan: FT-NIR/TD-NMR Spektroscopy
July 2003	Dr. P. Wilhelm at Bruker in Ettlingen, Germany, Work plan: FPA-Introduction
Oct. 2003	Dr. P. Pölt, Dipl.-Ing.. S. Mitsche at DGM-advanced training course in Aachen, Germany, Work plan: "Deformation, Recrystallisation, Texture"
Oct. 2003	Dipl.-Ing. B. Schaffer at FEI in Eindhoven, The Netherlands, Work plan: Tecnai Advanced MS-Course
Nov. 2003	MMag. W. Rechberger at FEI-User-Meeting in Aachen, Germany, Work plan: Tecnai FEG-Course

2004

April 2004	Dipl.-Ing. S. Mitsche at 11th Conference and Workshop on EBSD, Sheffield, U.K., Work plan: Electron Back Scattered Diffaction
April 2004	Dr. P. Wilhelm at the TU Wien, Austria, Work plan: High-resolution microscopy
June 2004	Dipl.-Ing. B. Schaffer at 36th Course for Electron Crystallography in Enrice, Sicily, Italy, Work plan: Novel Approaches for Structure Determination of Nanosized Materials

Renovierung des Institutsgebäudes Steyrergasse 17 im Sommer 2003 (Kooperationsprojekt „Bauliche Sanierung und energetische Optimierung“ mit Joanneum Research, Graz)
Renovation of the building Steyrergasse 17 in cooperation with Joanneum Research Graz during summer 2003 (building renovation and energy otpimisation project)

Austausch der Fenster (Juni 2003) / Exchange of the windows

Aussenrenovierung des Hauses Steyrergasse 17

Renovation of the front of the building Steyrergasse 17

15. Abstracts of Scientific Main Results 2003-2004 / Kurzbeschreibungen wichtiger Ergebnisse 2003-2004

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.

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Advanced Investigations of the Composition of Submicron Particles by using EDXS and Monte Carlo Methods 70
Robust, Dense and Accurate 3D Surface Reconstruction in SEM through Automatic Calibration Data Calculation from Multiple Images 72
Contrast Formation in a Low Vacuum or Environmental SEM: a Time Dependent Process 74
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Worldwide Database of free Scripting Resources in Digital Micrograph™ 114

EBSD – and the Recrystallisation of Ni-base Alloys

P. Poelt[1], S. Mitsche[1], C. Sommitsch[2] and M. Walter[2]

[1] Research Institute for Electron Microscopy, Graz University of Technology, Steyrergasse 17, 8010 Graz
[2] Böhler Edelstahl GmbH, Postfach 96, A-8605 Kapfenberg, Austria

Recrystallisation of a material is a process that is often used to tune the mechanical properties of materials. Parameters like the volume fraction of the recrystallised grains, the size and distribution of the recrystallised and deformed grains and possible textures influence the behaviour of the material. Electron Backscatter diffraction (EBSD) has emerged as a very useful technique for the measurement of these parameters, not at least because of its ability to elucidate direct neighbourhood relationships between the grains.

EBSD was used to study the recrystallisation of a Ni-base-alloy (Böhler L306 VMR [Alloy 80A]). The specimens were compressed at a temperature of 1120° C and a constant strain rate of 0.1/s to different strains. Subsequently they were cooled down at air. Finally the cylindrical compression samples were cut to transversal cross sections at a quarter of the specimen height, were the local and the global strain rate correspond each other. A more detailed description of the whole procedure can be found in Mitsche et al. [1]. The subsequent EBSD - measurements were performed by a TSL – system equipped with a SIT camera and attached to a Zeiss DSM 982 Gemini FEG – SEM.

Several methods have been proposed for the determination of the recrystallised fraction, but the grain orientation spread has proven most useful [1-3] and was used in this work. Figure 1 demonstrates clearly, that with progressive recrystallisation the recrystallised grains, which form a closed network, encircle every deformed grain. No big clusters of deformed grains could be found any longer. Starting from the grain boundaries, the deformed grains are gradually transformed into recrystallised grains, giving the deformed grains their "frayed" appearance. Therefore, corresponding to the increase of the fraction of recrystallised material, the mean diameter of the deformed grains is decreasing. As a consequence, nucleation for the recrystallisation process does seem to occur only at the grain boundaries of the deformed grains (necklace structure), but not within these grains themselves, which would cause a break-up of the grains. On the other hand, the mean diameter of the recrystallised grains is approximately independent of the size of the recrystallised fraction (Table 1).

Figure 2 (top left) shows, that no distinct texture is visible for the deformed grains for small strains (with only minor recrystallisation). But with the increase of the fraction of recrystallised grains, a pronounced texture does appear (Figure 2, top). It is caused most likely by the deformation of the material itself. Another possibility would be a preference for recrystallisation of grains with an orientation in the [111] direction. No texture was observed for the recrystallised fraction itself (Figure 2, bottom).

References

[1] S. Mitsche, P. Poelt, C. Sommitsch, M. Walther, Proc. Micr. Conf. MC 2003, Microsc. Microanal. **9** (Suppl. 3) (2003), p. 344 – 345.
[2] S. I. Wright, Proc. 12th Intern. Conf. on Textures (1999), p. 104 – 109.
[3] J. Tarasiuk, Ph. Gerber, B. Bacriox, Acta Materialica **50** (2002), p. 1467 – 1477.

This work was sponsored by the Austrian Industrial Research Promotion Fund (FFF – project 803060)

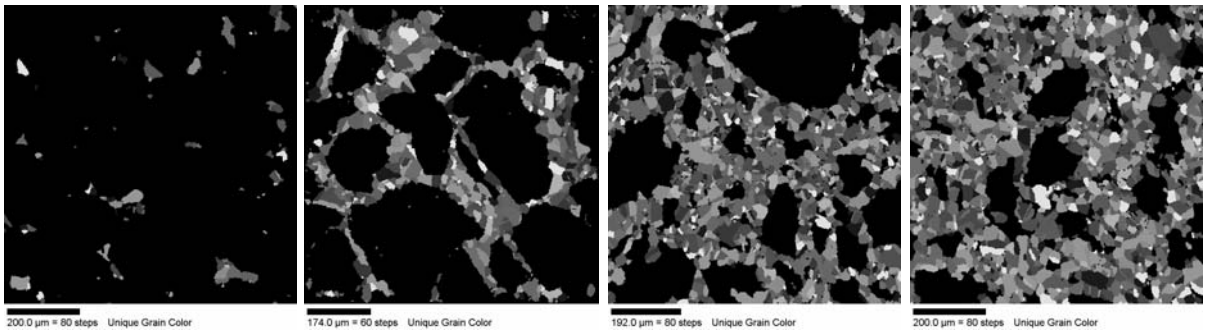

Fig.1 Grain map of the deformed (top) and recrystallised (below) fraction of a Ni-base-alloy, with the strain increasing from left to right (strain values as given in Table 1).

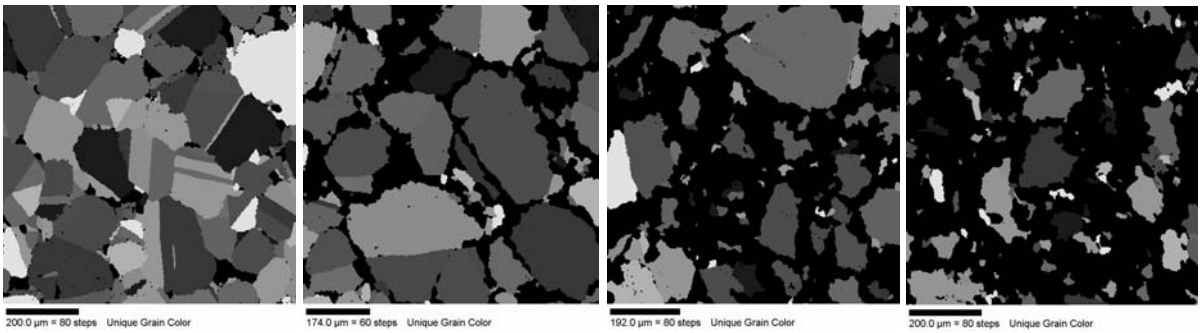

Fig.2 Inverse pole figure maps to the grain maps of Fig. 1 (top: deformed fraction, bottom: recrystallised fraction).

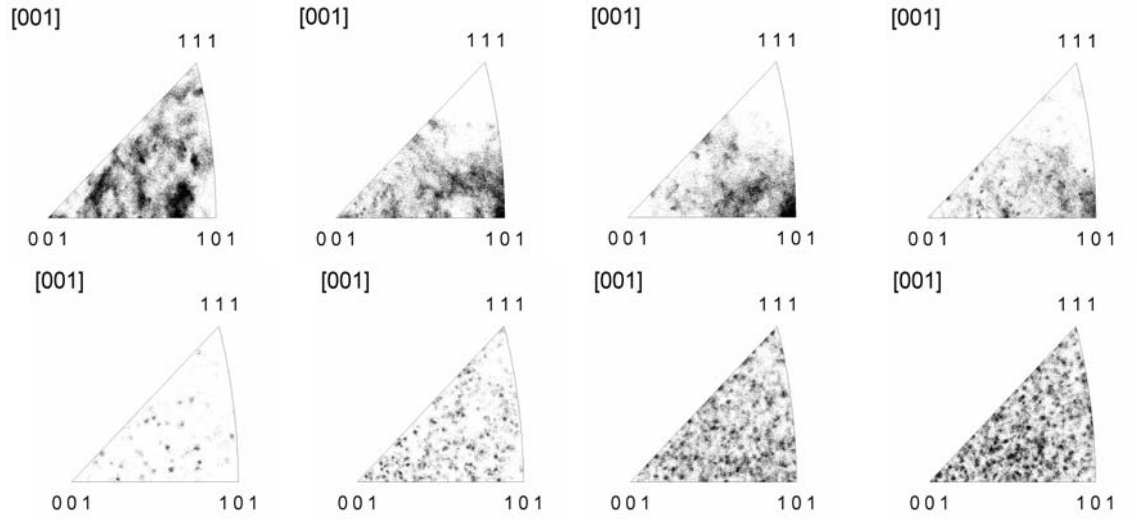

Table1 Grain sizes of the grains of the deformed and recrystallised fractions (mean diameter in µm).

strain	grain size / µm deformed	grain size / µm recrystallised
0.105	46.2	17.6
0.303	33.7	17.5
0.500	33.6	16.8
0.700	30.8	17.8

Channelling Contrast by Focused Ion Beam and EBSD – Two Complementary Techniques

M. Schmied, S. Mitsche

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

Focused Ion Beam (FIB) systems in combination with a Scanning Electron Microscope (SEM) offer many new possibilities for material analysis. Besides the unchallenged features of the FIB, such as material removal and deposition, the interaction of ions with the sample also generates secondary electrons (ion induced secondary electrons, SE_{Ion}) and ions, which can be used for excellent imaging modes [1]. Due to the strong dependence of the secondary electron- and ion yield on the crystallographic orientation of grains, images with high crystallographic contrast can be obtained. This so called ion channelling effect arises from collisions of ions and atoms in the direction of a low indexed axis [2], which results in low secondary and ion yield und thus dark regions in the image. Another well established method to gain crystallographic information from samples is Electron Backscatter Diffraction (EBSD) analysis, where electrons are impinging the sample under 70° and thus create pattern to determine the crystalline structure and orientation of the single grains.

It has been shown [2,3,4] that there exists a strong dependence of the ion channelling effect on the tilting angle of the sample, which leads to crystal orientation and structure dependent changes in the contrast of SE_{Ion} images. Therefore, it is a challenge to combine these two methods to increase the level of information of SE_{Ion} images by using EBSD data [5].

EBSD analyses on a nickel base alloy were performed with a TSL OIM system attached to a Gemini 982 (LEO). The ion channelling experiments were done with the Nanolab Nova200 Dualbeam (FEI). Figure 1a shows the inverse pole figure (IPF) of the nickel base alloy, where the grain size and orientation can be determined. By using the orientation information from EBSD data the image contrast in Fig.1c (dark regions = ion channelling) can nicely correlated to the 101 orientated grains, which are marked (black) in the IPF image in Fig.1b. More than that the critical angle for channelling can be evaluated for nickel by using the crystal orientation (see Fig.1d).

The combination of EBSD data and ion channelling data reveals that SE_{Ion} images contain a lot more information than grain size. The image contrast is strongly affected by the grain structure and orientation. Additionally the comparison of the both techniques allows estimating the influence of surface defects (e.g. oxide layer) due to their differential penetration depth. The main aim of this fusion of techniques will be the possibility of gathering three-dimensional crystal information, by applying the FIB as a milling machine and imaging source, valorised by once taken EBSD data.

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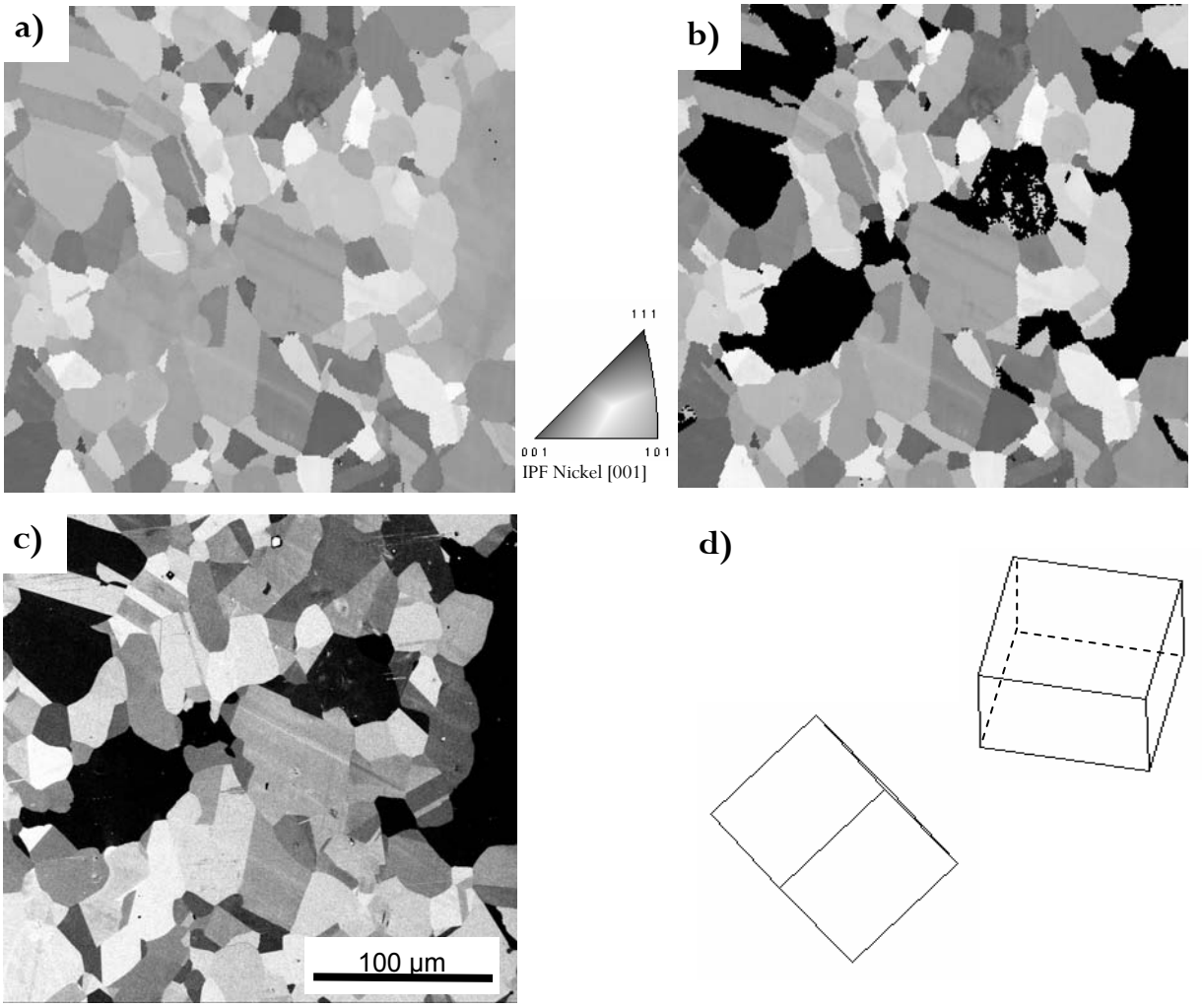

Fig.1 a) Inverse pole figure (IPF) of nickel base alloy (fcc); b) IPF nickel base alloy with black marked 101 orientated grains; c) ion induced secondary electron image (channelling contrast, 30kV Ga^+ ions) of the same area; d) schematic crystal orientation corresponding to dark and bright grains in Fig.1c.

Advanced Investigations of the Composition of Submicron Particles by using EDXS and Monte Carlo Methods

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It is a well known problem for electron microscopists, that the possible origin of the generated X-rays takes a volume which is sometimes larger than the size of the particles being analysed (dependent on the mean atomic number and the operating parameters of the scanning microscope).
Standard correction procedures like $\Phi(\rho z)$ or ZAF developed for bulk samples do not consider the geometrically caused deviation in the calculation of the fluorescence and absorption effects. Additionally the mass size effect [1], which describes the mass and size related loss of electrons through the side walls of the particles, has to be considered. With reference to the described methods [1] for submicron particle analyses the MC simulation seems to be the most promising approach.

The evaluation of the accuracy of standardless EDXS-analysis of submicron particles was performed by using the CASINO-program written by the group of Gauvin [2] and modified by Ro and Osan et al. [3]. Standard SiTiFe-particles [4] with diameter from 50nm to 1500nm and a defined composition (Si[wt%] to Ti[wt%] = 3, Si[wt%] to Fe[wt%] = 6) were used for the investigations. These particles were produced at 800°C in an aerosol reactor. As a precursor a three component system consisting of an alcoholic tetraethylorthosilicate solution (TEOS), a titaniumtetraisopropoxide (TTIP) in an alcoholic solution and an ion salt ($Fe(NO_3)_3 \cdot 9H_2O$) was used. TEM investigations showed that the particles are homogeneous within the scope of presently available characterisation methods (electron diffraction and EFTEM). Additionally a particle bulk sample was also investigated by ICP (inductively coupled plasma) measurements, where the elemental ratio could be confirmed (Si / Ti = 3.14 and Si / Fe = 5.95).

The simulated spectrum of a 100nm standard particle fits nicely to the measured spectrum as shown in Fig. 1. The correlation between the simulated and measured spectrum implies a general correctness of the theoretical principles implemented in the modified CASINO program. Size dependent quantitative analyses were taken with (modified CASINO) and without (NORAN Voyager – standard $\Phi(\rho z)$ correction for bulk samples) geometrical correction. As shown in Fig. 2 and 3 the conventional procedure yields to an underestimation of the expected elemental ratio between Si/Fe and Si/Ti, whilst the Monte Carlo based approach generates an overestimation.
The gathered results will be used for a detailed discussion of the advantages, possible errors and sources of systematic deviations of the mentioned correction procedure. Further particles with other compositions will be investigated to explore the suitability of this method for a general purpose.

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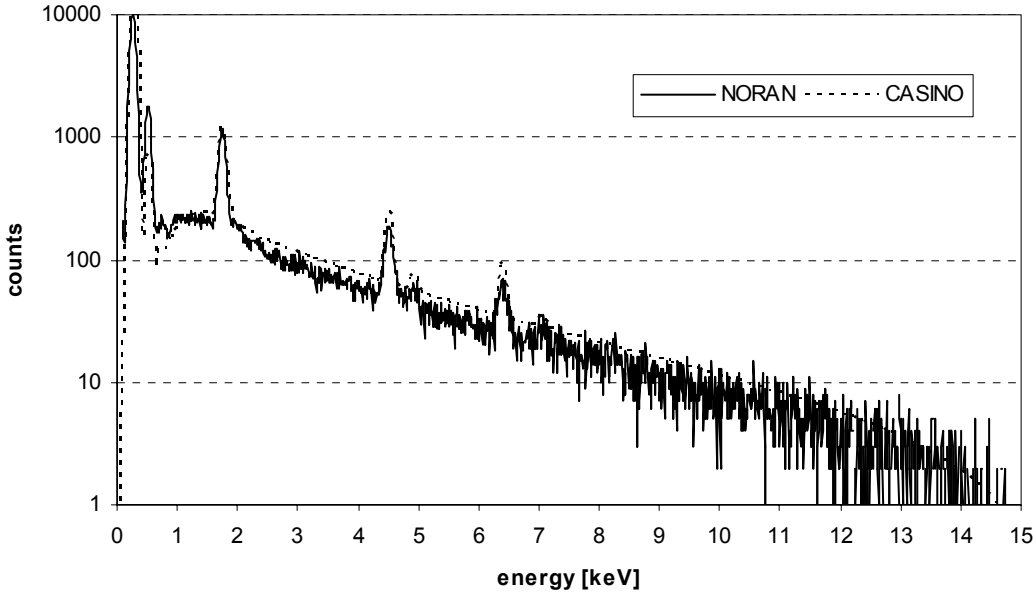


Fig.1 Comparison of measured and simulated EDX-spectra of a 100nm standard particle

Fig.2 Elemental ratio of Si/Ti [wt%] at 60s acquisition time and at 15kV

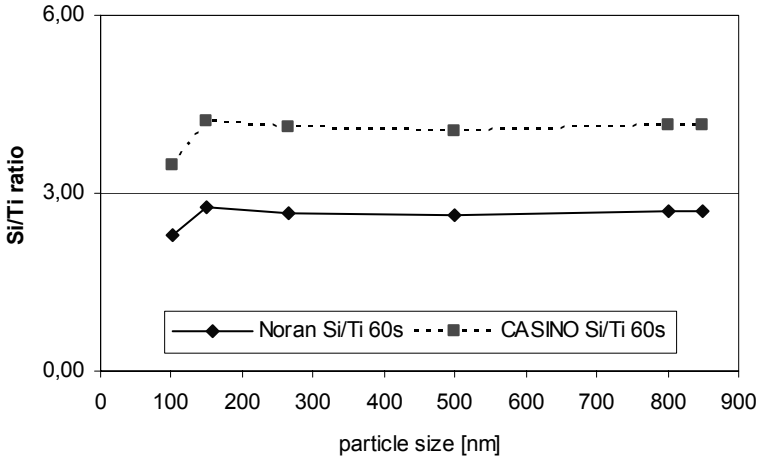
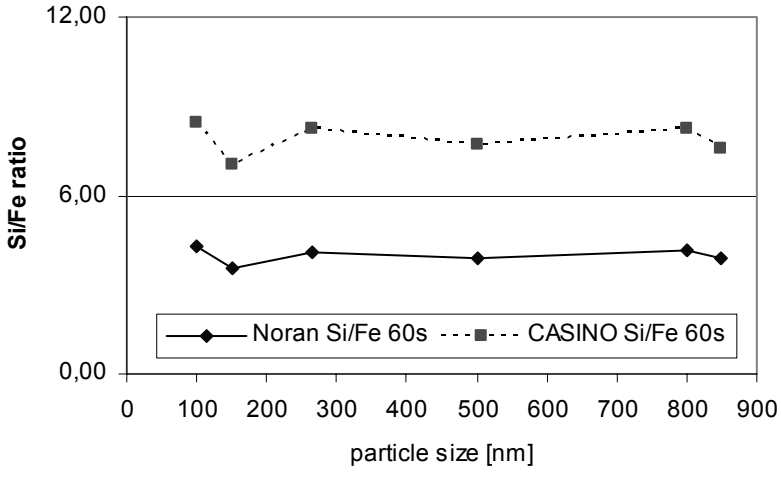


Fig.3 Elemental ratio of Si/Fe [wt%] at 60s acquisition time and at 15kV



Robust, Dense and Accurate 3D Surface Reconstruction in SEM through Automatic Calibration Data Calculation from Multiple Images

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The scanning electron microscope (SEM) is an excellent tool for the characterisation of surfaces. It offers a large depth-of-focus and high contrast images with a lateral resolution up to 1nm or even below. The scanning process of a surface leads to a two-dimensional image of a three-dimensional surface. Nevertheless SEM images reveal also 3D information. In order to access this information conventional approaches analyze stereoscopic images. The major drawback of this approach is found in the sensibility to the accurate determination of the calibration data, namely the tilt angle. The user is faced with the almost unsolvable task of determine the accurate tilt angle, as most stages do not offer the desired angle resolution of 0.1 degree [1,2].
In this paper we present a new approach that solves this problem. The operator is capturing three images at different tilt angles. The only restriction the operator is faced is a nearly eucentric tilting and a rough estimate of the tilt angle with the given order. The presented method automatically determines the true tilt angle up to 0.1 degrees accuracy based on image processing algorithms. After calculation of the accurate calibration data a dense 3D reconstruction can be calculated.

Experiments were performed with images of the size 1024 x 768 pixel resolution, captured at a pixel size of 0.3µm at a working distance of 5mm. In order to evaluate the accuracy of the system a known depth standard (Fig.1a) for tactile depth measurement devices was observed. The estimated tilt angles were 5 and -10 degrees. The automatically determined tilt angles lead to 4.49 and -9.53 degrees. The known depth of the height step is 92 µm. The reconstruction based on the classical stereo approach leads to a height measurement of 79.1µm (Fig.2). The difference to the true height is found in the wrong tilt angles. The automatically determined angles lead to a height measurement of 93.2µm (Fig. 2).
The resulting digital elevation model (DEM, Fig.1b) can then be used for consecutive height analysis as profile, roughness, area and even volume measurements [3]. The great benefit of the (E)SEM technique is that the topography of almost any specimen can be imaged, reconstructed and analysed provided that the surface is visible the images without touching the surface or destroy sensitive samples or surface details by contact with the stylus. It doesn't matter if the surface is conductive or not, biological, made of plastic or rubber or even a transparent paint. The presented approach does allow to directly measure depth in the digital image (*topomicroscopy*) at highest accuracy.

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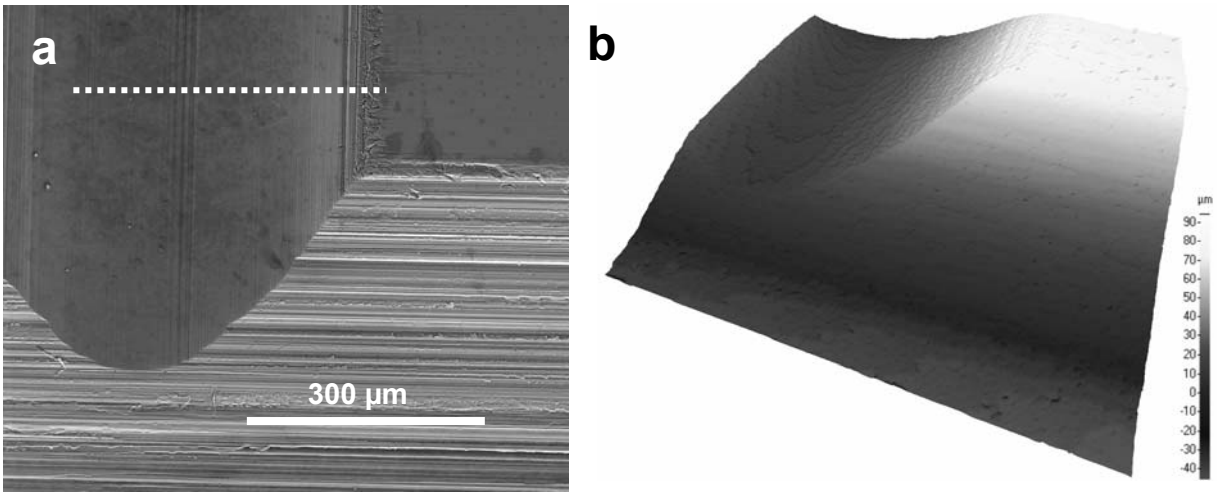

Fig.1 a) Secondary electron (SE) image of the depth standard with marked line (dashed) of profile measurement, b) calculated digital elevation model (DEM)

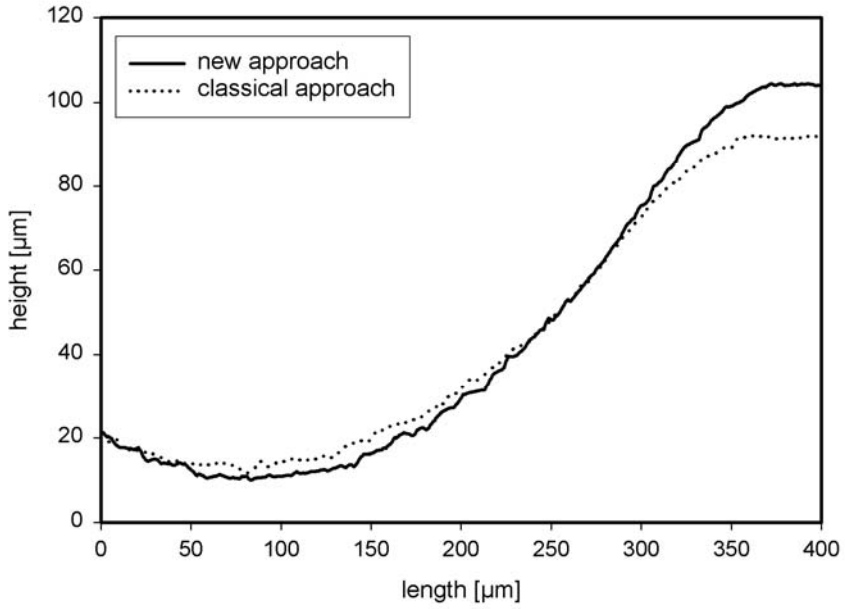

Fig.2 Comparison of the two depth profiles obtained with and without automatically determined tilt angles (classical approach gives a depth of 79.1 µm; new approach gives a depth of 93.2 µm).

Contrast Formation in a Low Vacuum or Environmental SEM: a Time Dependent Process

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The image contrast formation in a low vacuum or environmental SEM is governed by number of phenomena, which result from the interaction of gas molecules with electrons and thus the change in electric fields between detector and sample. The performance of the different detectors (GBSD, GSE, LFD) is enormously effected by the electron-ion recombination rate or the space charge, which may cause imaging artefacts, such as contrast inversion.

Ion current profiles (see Fig.1) were measured with the stated formula to obtain amplification and scavenging profiles for the GSE- and LF-detector. Differences in these profiles are used to explain the influence of the sample – detector geometry on the image quality and the amount of disturbing artefacts. Also the influence of scan rates, working distance and all the other operation parameters on contrast formation is discussed within the aspects of a time dependent process. Therefore time dependent measurements of the ion current were performed with different sets of acquisition parameters. As a consequence general aspects of the contrast formation are discussed with special focus on avoiding the misinterpretation (see Fig.2) of low vacuum or environmental SEM images.

As shown in Fig.1a there is a much higher ion-beam current obtained with the LFD as for the GSED, which is also resembled in the higher gas-gain curve in Fig.1b. This can be addressed to the different geometric positions in the chamber, where the LFD exhibits a longer distance to create the electron amplification. On the other hand also the electron-ion recombination rate (scavenging) is increased using the LFD, which implies that more electrons are captured using the LFD and thus, cannot contribute to the SE signal. All this experiments have been performed at the fixed time (10s) after switching on the electron beam, whereas differences in the I_{ion} beam current response could be observed using other measuring times. This reveals the importance of the distribution of the field lines between detector and sample and shows that these processes have strong time dependence. This difference has to be taken into account, when contrast inversion (Fig.2) appears as a function of scan speed (or pixel dwell time)

$$I_{ion} = \Gamma I_{PE} e^{-\Psi I_{PE}}$$

Γ.....amplification rate without scavenging
Ψ.....electron - ion recombination rate
I_{ion}.....ion induced current
I_{PE}.....primary beam current

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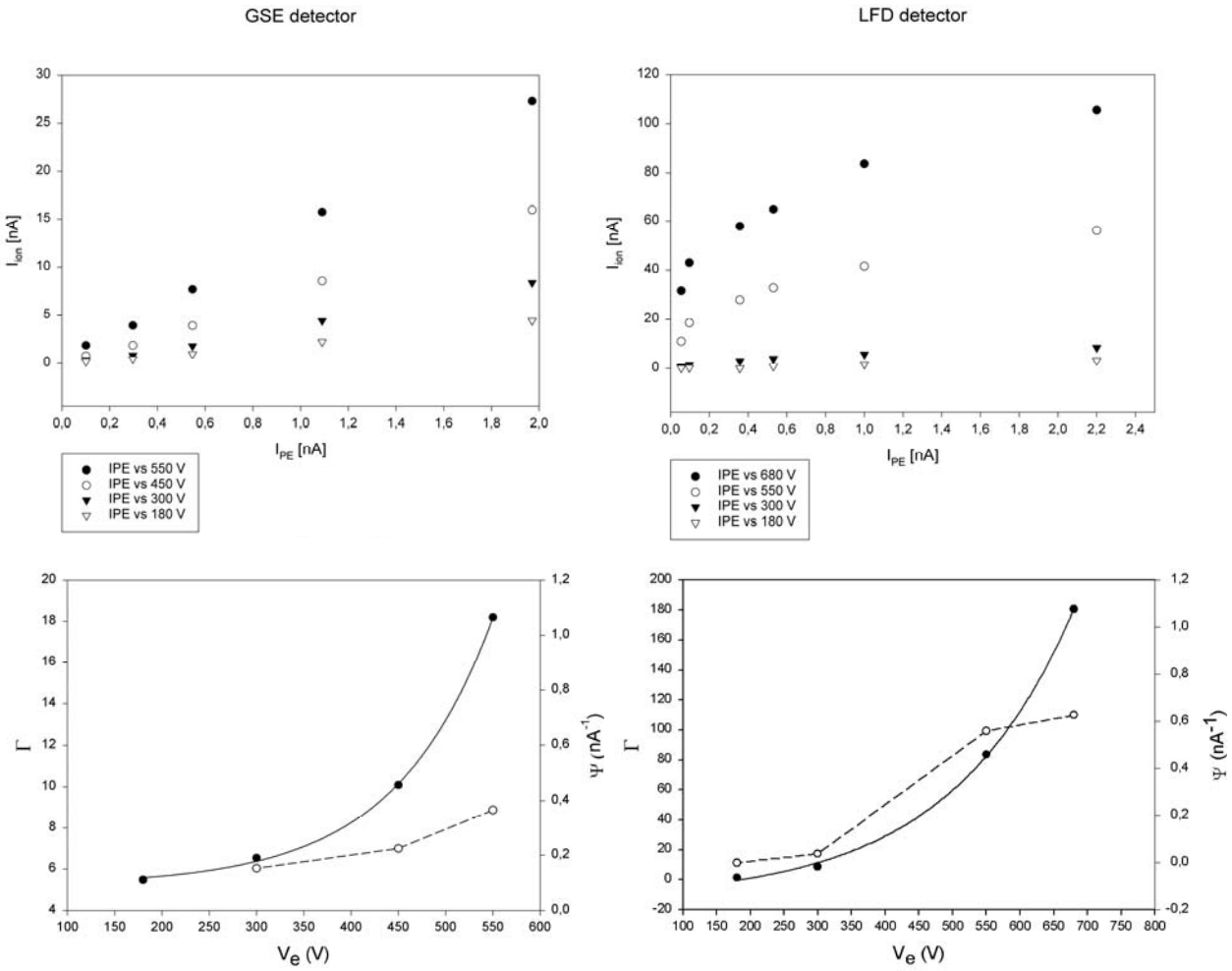

Fig.1 a) I_{ion} current response in relation to increasing beam current and detector voltage; b) gas amplification (Γ) and ion-electron recombination rates (Ψ) as a function of detector voltage

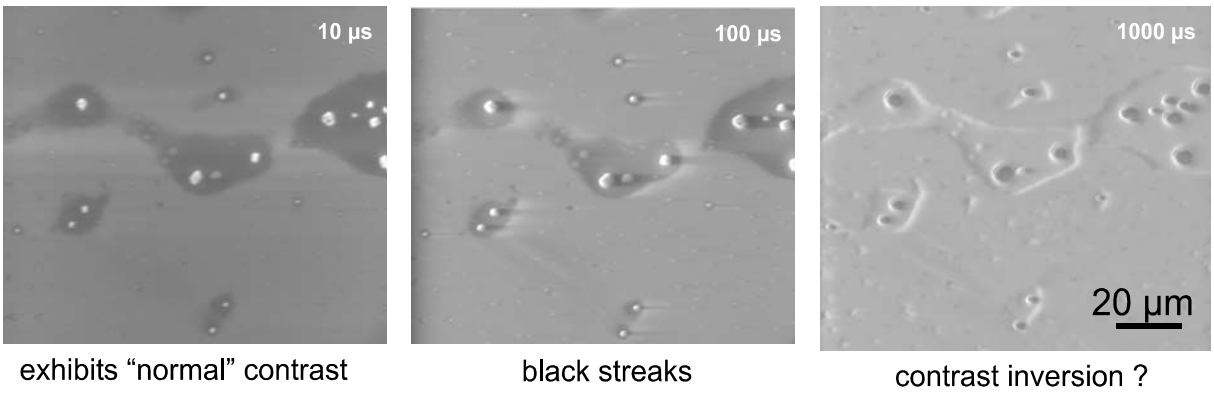

Fig.2 Scan speed dependence of contrast formation using the LFD in a low vacuum electron microscope

Spectroscopic Imaging Techniques for Polymer Characterisation: Infrared and Electron Microscopy – A Good Match

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The morphology of high-tech polymeric materials plays an important role in determining the final materials properties. It is controlled during the various processing steps and strongly influenced by the basic materials. Enhanced knowledge on the structure of the final product could help to understand its limitations and support a further development.

In this work a new reactive Poly(tetrafluoroethylene) / Polyamide (PTFE/PA) compound used as wear resistant maintenance–free slide bearing material was under investigation. Upon conventional mixing of PTFE and PA6 at about 250°C relatively large particles occur in the final product because of their immiscibility. This is detrimental for mechanical stability and the formation of a homogeneous PTFE distribution in the PA matrix. By irradiation with electrons reactive groups in the PTFE powder are produced. As result of their reaction with PA above 200°C in the extruder block copolymers are formed which act as compatibiliser. A decrease in PTFE particle size proportional to the irradiation dose could be observed.

Reactive PTFE/PA compounds were characterised by scanning and transmission electron microscopy, and FTIR microscopy (transmission and ATR imaging). Scanning electron microscopy revealed the morphological and transmission electron microscopy in combination with a high-resolution filter additionally provided chemical information via electron energy loss spectrometry (EELS). Due to the combination of infrared and electron microscopy these polymer blends could be examined with excellent spectral and spatial resolution.

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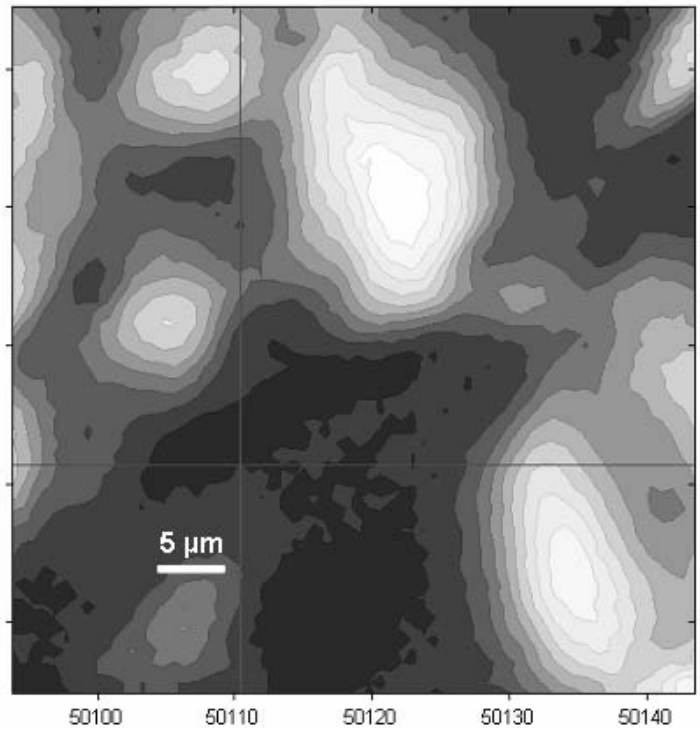
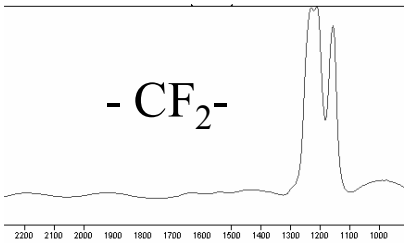
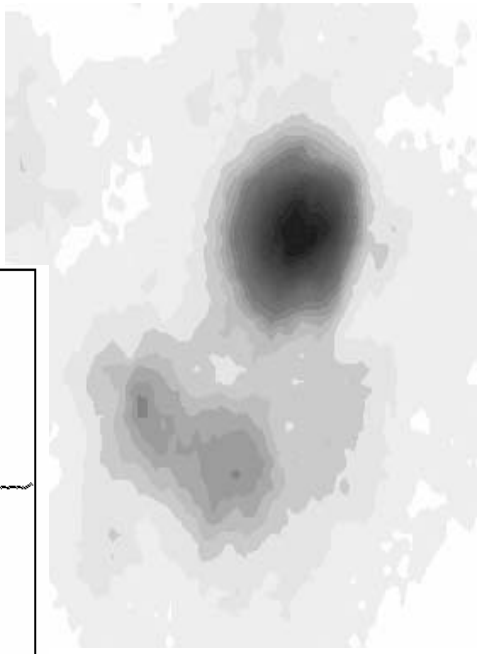


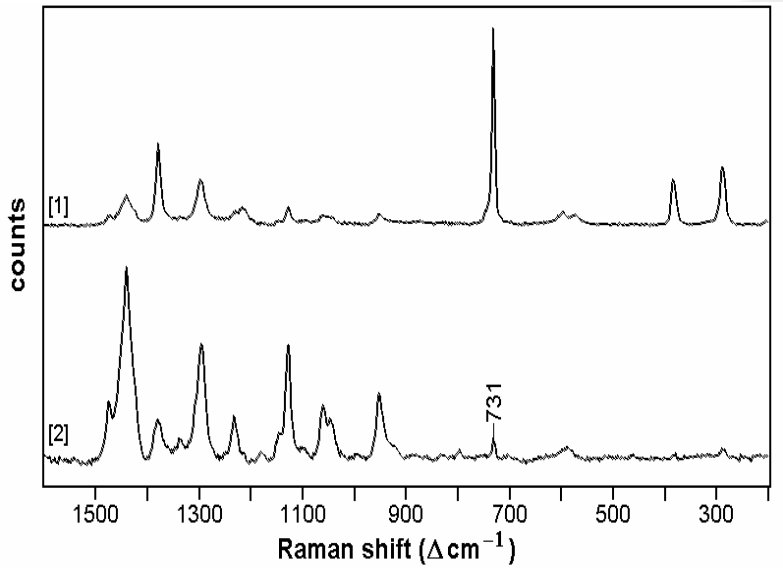
Fig.1 Infrared ATR image (area 50 µm x 50 µm) of a PA – PTFE blend: PTFE clusters shown in bright colour (intensity distribution of the C-F absorption bands).



Raman image created with 731 cm^{-1} line of PTFE (image size: 20 µm * 20 µm).

Intensity distribution (PTFE clusters) shown in dark colour.





Investigations of Thin Polymer Films with Vibrational Spectroscopic Techniques

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For more and more applications thin organic films are necessary to fulfil a variety of demands. The thicknesses of such polymer films may be in the range of a few nanometers or even below. Homogeneous, non-destructive and controlled material deposition is an important technological aspect that has to be verified before the method can be implemented into a commercial production process.

In these studies thin polymer films deposited on reflecting metal surfaces by atmospheric pressure ion deposition (APID) have been characterised by grazing incidence FT-IR spectroscopy. Such spectra are compared to transmission spectra of a reference material in order to prove the intact nature of the polymer film and to get an idea of the film thickness. Additionally to FT-IR spectroscopy Raman spectroscopy was employed to characterise these thin films. The conjugated sequences in organic polymers, as they are used in light emitting diodes, give rise to resonance enhancement and enable detection of small polymer amounts with a lateral resolution in the range of a micrometer, which is sufficient for the creation of mappings and images based on vibrational spectroscopic data. Furthermore, application of well defined gold nanoparticles onto the polymer surface lead to an increased sensitivity due to surface enhancement such that the presented Raman spectra based either on the SERRS technique or on resonance enhancement. Determination of film thicknesses was done by electron microscopy.

The obtained results show that infrared spectroscopy is a very well suited technique to study thin films and that application of grazing incidence objectives brings an enormous advantage compared to the conventional IR reflection mode. Spectra are of excellent signal to noise ratio and acquisition time remains also moderate. But most importantly the rich chemical information of the infrared spectra allows statements about the chemical composition of these thin polymer films or degradation processes occurring during the deposition processes.

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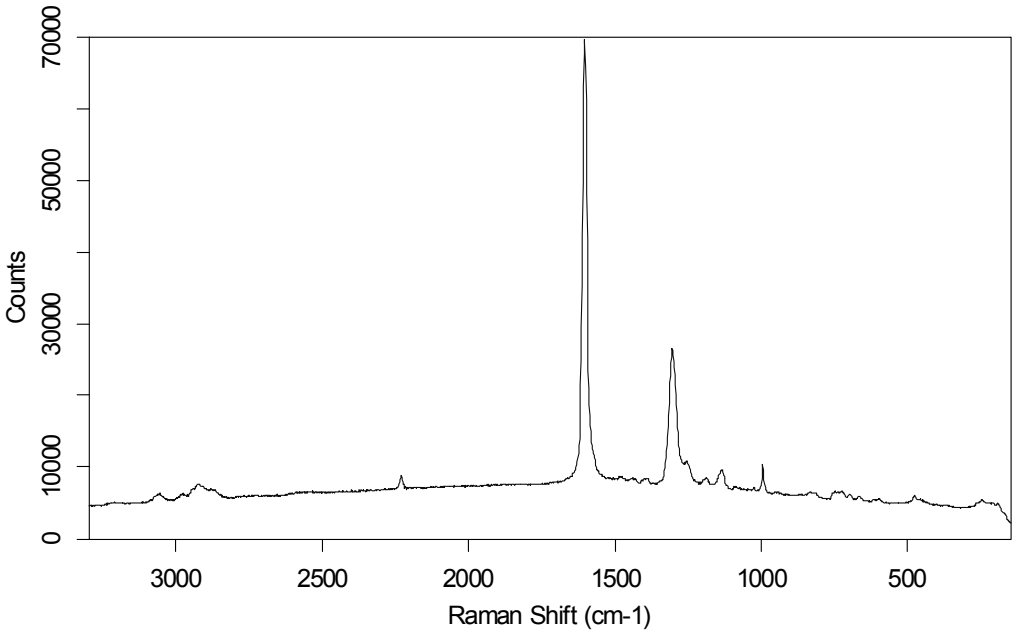

Fig.1 The Raman spectrum features a spectrum of an organic dye for light emitting diodes. The strong band at about 1600 cm-1 is due to the conjugated system in the molecule and the signal can also be detected at very low dye concentrations or film thicknesses.

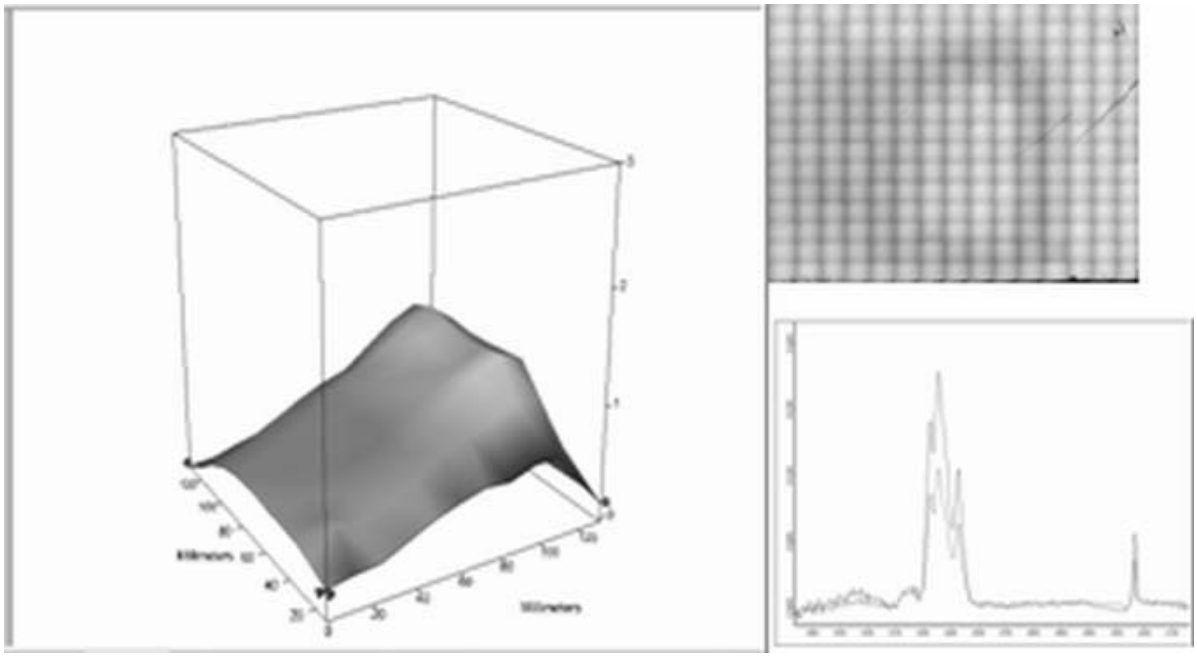

Fig.2 The high sensitivity of the IR grazing angle incidence technique allowed us to proof identity between feedstock and thin film material. By using the infrared data of point spectra measured on a grid pattern a mapping of the film - showing integrated the nCHx intensity and hence thickness of the film - could be created, which is in good agreement with the visible image (top right picture). Differences in absorbance due to varying film thicknesses are shown in the spectra below (estimated film thicknesses between 20 and 40 nanometers)

Analytical Electron Microscopy in Materials and Biological Sciences

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During the last two decades we have seen an almost exponential growth in the coupling of microscopic imaging techniques with spectroscopic methods. This occurred in all forms of microscopy, from optical, to electron, ion and x-ray microscopy, and more recently to scanned tip imaging microscopies. In particular, the combination of a transmission electron microscope with an x-ray spectrometer and an electron energy-loss spectrometer - now commonly called analytical electron microscopy (AEM) – turned out to be a very powerful means for providing physical and chemical information in the form of spectra and images. Due to the introduction of high brightness electron sources (Schottky emitter, cold field emitter) combined with improvements in the design of spectrometers and detectors it is now feasible to obtain useful data with spatial resolution close to atomic dimensions, given the right kind of instrumentation and specimen.

In analytical electron microscopy two different instrumental approaches have been followed: Firstly, the scanning TEM (STEM) equipped with an EELS spectrometer and a cold field emitter offers ultimate performance concerning spatial resolution and detection limits [1]. Although it is mainly applied to solving materials science problems [2,3], there are a few promising applications in biology [4], too. With the correction of spherical aberration aiming at sub-Ångström probes and true atomic resolution, the STEM enables the acquisition of EELS-spectra from single atom columns [5]. Correction of the relevant aberrations has already been demonstrated, but the real challenge is to achieve the necessary stability and low levels of interference in the microscope system and its environment.

Secondly, the energy-filtering TEM (EFTEM) became extremely popular both in materials and biological sciences, largely due to the availability of commercial high performance energy-filters [6,7,8]. The ability to select any spectral feature from an EELS-spectrum opens up a variety of new powerful imaging techniques. The most obvious advantage of EFTEM imaging is the measurement of compositional information in the form of elemental maps which can easily be quantified in order to yield concentration and chemical phase maps [9,10]. The elemental mapping approach can be the best way to analyse nano-scale features in materials such as fine precipitates and interfaces/boundaries, since two dimensional fluctuations in composition around such small features, which may be easily missed by point or line-scale analyses, can be revealed in images of elemental distributions [11]. EFTEM allows to record elemental maps at sub-nanometre resolution, being mainly limited by chromatic and spherical aberration of the objective lens and by delocalisation of the inelastic scattering process [12,13,14].

Both STEM/EELS and EFTEM may be used to acquire complete 3D data sets containing energy-loss information for all pixels. This technique is called STEM or EFTEM spectrum imaging. It is the starting point for automatic detection, identification and compositional quantification of chemical elements [10,15]. On the STEM the recent availability of CCD detectors optimised for spectroscopy allows a 512x512 EELS spectrum image with a 1340 channel EELS spectrum at each point to be recorded in less than 6 minutes. EFTEM spectrum imaging is ideal for mapping high local concentrations distributed over large specimen areas, but recent instrumental developments such as better correction of spectral aberrations up to the 4^{th} order will improve the isochromaticity of the filter thus enabling the acquisition of EFTEM spectrum images in 1 eV steps [16]. It will facilitate to derive even chemical bonding information via the near edge fine structure from EFTEM spectrum images.

Recent developments in AEM focus on a further improvement of the instrumental energy resolution by using monochromators for the electron source thus making it possible to record EELS spectra with an energy-resolution in the range of 0.1 eV [17,18]. Since this is well below the natural line widths of EELS near-edge fine structures (ELNES), monochromated TEMs provide ultimate chemical bonding

information from specimen areas as small as several nanometers [19]. Another application of a monochromated EELS/TEM system may be for valence-electron spectroscopy, when measuring the water content of tissue or distinguishing peaks due to chromophore dyes for example [20]. Structures below 2 eV loss e.g. the band gap in semiconductors has been particularly hard to measure because it lies within the tail of the zero-loss peak. High energy resolution will therefore benefit EELS studies of the local electronic structure at defects and interfaces in semiconductors and of the electronic structure of nanotubes or small particles.

Although EELS and EFTEM have excellent spatial resolution in relation to other techniques, they have not been so successful for measuring very low elemental concentrations which is mostly caused by the high background underlying ionisation edges. Here, due to its better signal-to-noise ratio energy-dispersive x-ray spectroscopy in the AEM may present some advantages over EELS and EFTEM, especially for mapping of heavier elements [21].

It is well known that the ultimate limit to spatial resolution and detection limits is set by radiation damage to the specimen. Consequently, the expected enhancements in energy and spatial resolution will be compared with fundamental limitations that arise from the natural widths of spectral peaks, the delocalisation of inelastic scattering and the problem of electron irradiation damage.

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Energy-filtering TEM at the Frontier of Spatial and Energy Resolution

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Electron energy-loss spectroscopy (EELS) in the transmission electron microscope (TEM) is now used routinely as a means of measuring chemical and structural properties of very small regions of a thin specimen. The power of this technique depends significantly on two parameters: its spatial resolution and the energy resolution available in the spectrum and in the energy-filtered TEM (EFTEM) image.
The field emission source has made an energy resolution below 1 eV possible and it is now feasible to obtain data with a spatial resolution close to atomic dimensions, given the right instrumentation and specimen. EFTEM allows to record elemental maps at sub-nanometre resolution, being mainly limited by chromatic and spherical aberration of the objective lens and by delocalisation of inelastic scattering [1,2]. Recently the possibility of correcting spherical and even chromatic aberrations of electron lenses has become a practical reality thus improving the point resolution of the TEM to below 0.1 nm [3]. The other limiting factor for EFTEM resolution is delocalisation. However, recent measurements show that resolution values in the range of 1 nm and below can be achieved, even for energy-losses of only a few eV [4,5,6]. This is demonstrated in the study of a 1.5 nm thin oxide layer in silicon (Fig.1), where the resolution degradation due to delocalisation of the inelastic signal is below 1 nm. Since EFTEM using the low-loss electrons of the EELS spectrum offers very high detection sensitivity, t is perfectly suited for the measurement of such very thin layers in semiconductors and magnetic recording media.
In terms of energy-resolution, EELS and EFTEM compare less favourably with other spectroscopies. For common TEMs, the overall energy-resolution is mainly determined by the energy width of the electron source, typically between 0.5 and 1.5 eV. For comparison, synchrotron x-ray sources and beam line spectrometers provide a resolution well below 0.1 eV for absorption spectroscopy. During the early sixties, the energy spread of an electron beam could be reduced by incorporating an energy-filter into the illumination system [7], but the system lacked spatial resolution. Later developments combined high energy resolution in the range of 0.1 eV with improved spatial resolution [8]. Recently, Tiemeijer introduced a new high resolution EELS system based on a FEI Tecnai F20 [9]. Its energy resolution is essentially improved due to the following factors: (a) the Wien filter monochromator below the Schottky emitter [9]; (b) the improved stabilisation of the high voltage tank; and (c) a new high resolution imaging filter [10]. The overall energy resolution is in the range of 0.1 eV and opens up the possibility to fully exploit the chemical bonding information contained in the near edge fine structure of ionisation edges (ELNES).
In order to demonstrate the advantages of the monochromised TEM, we have investigated near-edge fine structures of the metal $L_{2,3}$ and O K edges in transition metal-oxides with an energy resolution of 0.15 eV [11]. For example, metal $L_{2,3}$ edges in EELS-spectra of 3d transition-metal oxides display a great variety of near edge fine structures (Fig.2), which can only be observed in full detail in spectra with an energy resolution of better than 0.2 eV. However, the situation is different for oxygen K-edges (Fig.3). Here, the monochromized TEM with an energy-resolution of 0.15 eV does not improve the information content, because the natural line width of the O K-edge is in the range of 1.0 eV which can be mainly attributed to solid state effects. Consequently, the advantage of 0.1 eV resolution will be limited to certain ionisation edges (below 1500 eV) and will likely be confined to a region within a few eV of the ionisation threshold.
Although the energy resolution of 0.1 eV is not sufficient for vibrational and phonon spectroscopy, the improved resolution is clearly advantageous for studying electronic structure and bonding effects derived from near edge fine structures, and dielectric function features (band gaps, inter- and intra-band transitions) evaluated from the low-loss region of the spectrum.

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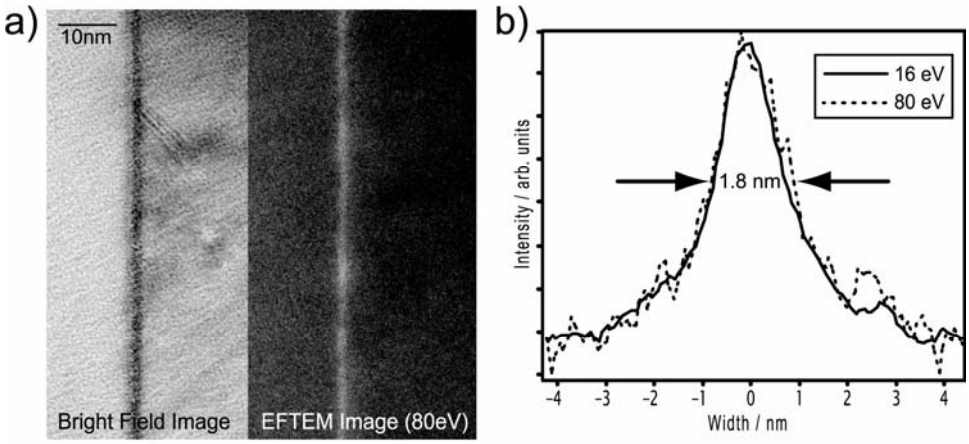

Fig.1 EFTEM investigation of a thin silicon oxide layer in silicon, a) TEM image and energy-filtered image recorded with inelastic electrons of 80 eV; b) the width of the line profile closely corresponds to the nominal

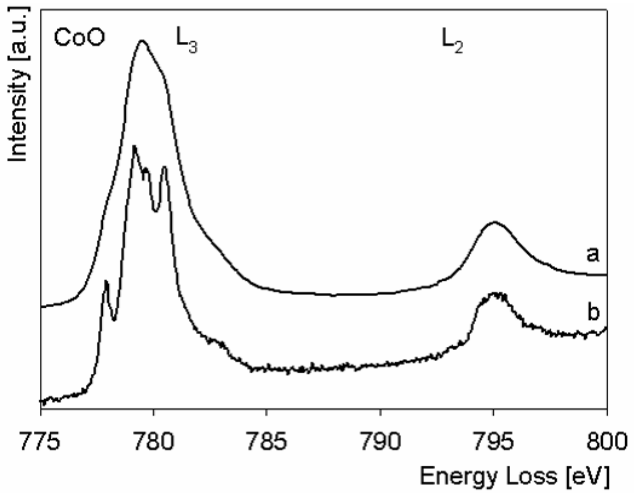

Fig.2 Co $L_{2,3}$ edges from CoO recorded with a) a FEI Tecnai F20 equipped with a Schottky emitter and a Gatan imaging filter, b) a Tecnai F20 equipped with a Schottky emitter, a monochromator and a HR-Gatan imaging filter.

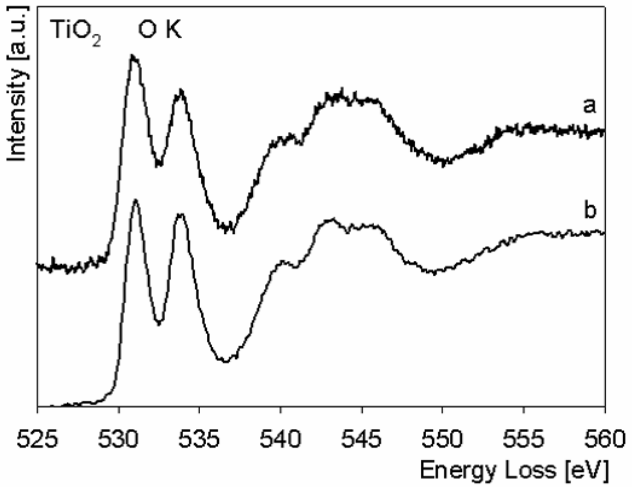

Fig.3 Oxygen K edge from TiO_2 (rutile), recorded with a) a Philips CM20 equipped with a LaB_6-cathode and a Gatan imaging filter; b) a Tecnai F20 equipped with a Schottky emitter, a monochromator and a HR-Gatan imaging filter.

Experiencies and Possibilities with a 200 kV Monochromated (S)TEM

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The energy resolution in electron energy-loss spectra attainable with present-day analytical TEMs is typically limited to around 1 eV, preventing detailed information about the electronic structure of materials to be obtained. For comparison, synchrotron x-ray sources and beam line spectrometers provide a resolution well below 0.1 eV for absorption spectroscopy. The main limitations in TEM EELS stem from the instability in the high tension, the insufficient aberration correction of the spectrometer and the natural energy width of the electron source. To go beyond, FEI / Gatan have recently commercially introduced a monochromated 200 kV (S)TEM aiming at an energy resolution level for EELS which is roughly a factor of 5-10 better [1,2]. The much better energy resolution in this instrument was in part due to improvements in the high voltage supply, the more stable electronics and improved electron optics of the spectrometer but mostly because of the incorporation of a Wien filter monochromator positioned directly after the field emission gun. Improved spectral resolution reveals many more effects that cannot be predicted as precisely as they can be measured and opens up new possibilities in both the analysis of low loss and core loss spectra.

The smaller energy spread of the zero-loss peak is particularly significant for measurements in the low loss region. One problem for thin insulators or semiconductor samples in the TEM is the fact that the dominating elastic signal strongly extends into the energy region of the bandgap (1-10 eV), forming a background that can hardly be estimated and extracted. Furthermore the reliability in the determination of optical properties via Kramers-Kronig analysis can be improved when the intensities are more accurately known. Figure 1 (left) displays two area normalized zero-loss peaks with the monochromator switched on and off, showing the advantage of a narrower zero-loss profile when subtracting low loss intensities.

In core-loss spectroscopy it is well known that the line width of ELNES features are further influenced by intrinsic effects like core hole lifetime broadening and the broadening caused by the finite lifetime of the excited state. First monochromator measurements on selected transition metal oxides [3] confirmed that O-K edges are intrinsically broader compared to L_3-edges, for which the core level widths lie in the range of 0.2-0.4 eV (cf fig. 1 right). The observed $2p_{1/2}$ level (L_2) widths on the other hand are significantly larger, which is likely to be due to rapid $L_2L_3M_{45}$ decay processes (Coster-Kronig broadening), rising from Ti to Co. The final state lifetime is less well characterized, but various approaches [4,5,6] predict the damping of the DOS to be within 2 and 6 eV for the first 20 eV. The model followed by Muller et al. is based on experimental measurements of the dielectric function determined from the EELS valence spectra whereas Schattschneider et al [priv. comm.] make use of momentum resolved measurements of the loss function.

First experiences, tests and measurements on material science specimens will be presented to illustrate that with the improved EELS resolution a better understanding of the chemical and electronic properties can be obtained.

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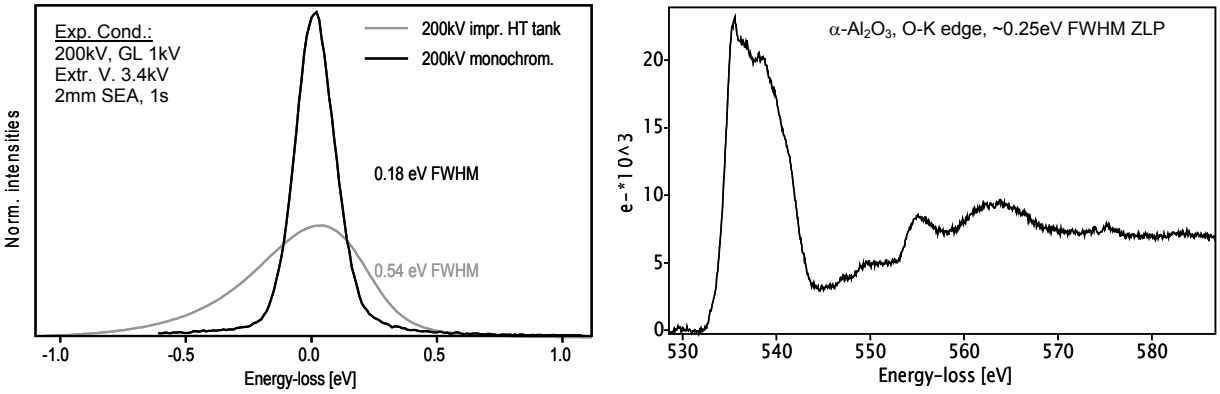

Fig.1 Left) Comparison of two zero-loss peaks (area normalized) taken with the WIEN filter monochromator switched on and off on a Tecnai T20F as installed on the FELMI. Right) Oxygen K edge of α-Al_2O_3 acquired with an exposure time of 30s. The splitting of the main peak (as well as an extra shoulder) and several other features as predicted by theory are clearly visible.

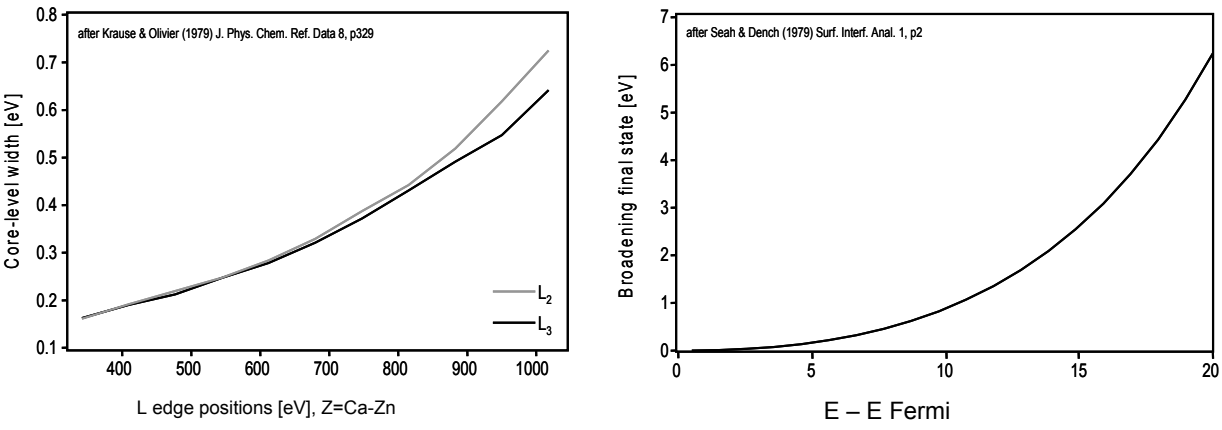

Fig.2 Left) Core level widths as a function of L edge positions for the transition metals after Krause & Olivier (1979). Preliminary measurements seem to indicate that L_2 widths are largely underestimated. Right) Final state broadening estimation based on inelastic mean free paths from Seah and Dench (1979).

Electron Energy-Loss Spectrometry at High Energy Resolution for Materials Research

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Electron energy-loss spectroscopy (EELS) in the TEM is one of the key tools allowing retrieving essential information on chemical bonding and electronic structure of materials at the nanoscale thus opening up the way for solving advanced materials science problems.
Since the energy resolution in EELS spectra attainable with present-day analytical TEMs is limited to around 1.0 eV, important spectral features cannot be resolved. For comparison, synchrotron x-ray sources and beam line spectrometers can provide a resolution well below 0.1 eV for absorption spectroscopy since a decade. The main limitations in energy resolution in EELS in the TEM stem from the instability in the high tension, the natural energy width of the electron source and the insufficient aberration correction and stability of the spectrometer. To go well beyond 0.7 eV with a Schottky emitter TEM, FEI and Gatan have recently introduced a Tecnai F20 which is equipped with a pre-specimen monochromator, a high resolution imaging filter and a better stabilized high voltage supply [1]. With this setup it is principally possible to obtain less than 0.1 eV in a 2 nm probe. However, stray fields may limit the attainable energy resolution and therefore the system installed in Graz presently yields about 0.18 eV (Figure1).
The improved energy-resolution on a TEM opens new possibilities for studying detailed electronic structure and bonding effects evaluated from near edge fine structures and dielectric function measurements via the low-loss part of the spectrum. For the latter, especially for the direct measurement of band gaps in nanoscale structures an energy resolution in the range of 0.10 eV can be mandatory [2]. However, in core-loss spectroscopy it is well known that the line widths of ELNES features are further influenced by intrinsic effects like core-hole and excited state lifetime broadening and by the broadening introduced by solid state effects [3]. First HR-EELS measurements on transition metal oxides [4] showed that oxygen K edges are intrinsically broader compared to $L_{2,3}$ edges, for which the theoretical predictions [3] lie in the range of 0.2 to 0.4 eV (Figure 2). It was found that the natural line width for the first peak in the ELNES of the $L_{2,3}$ edges is in the range of 0.3 to 0.6 eV which is in close agreement with x-ray absorption measurements. Consequently, an energy reso-lution of about 0.2 eV is sufficient for most ELNES studies.

A typical example for demonstrating the power of the monochromated TEM for studying materials is shown in Figure 3. The Al $L_{2,3}$ edge of α-Al_2O_3 was recorded with the monochromated Tecnai F20 (~0.25 eV) and with a CM20 equipped with a LaB_6 cathode (~ 0.8 eV). The $L_{2,3}$ peak at the threshold shows a faint shoulder which can be clearly seen in Figure 4 and fitting Gaussians to the raw data reveals two peaks separated by 0.49 eV which can be attributed to spin-orbit splitting (L_3 and L_2). The spin-orbit splitting agrees with results from x-ray absorption spectroscopy [5] and with EELS data which have been obtained from maximum entropy deconvolution of the Al $L_{2,3}$ edge recorded with an energy resolution of 0.35 eV [6].

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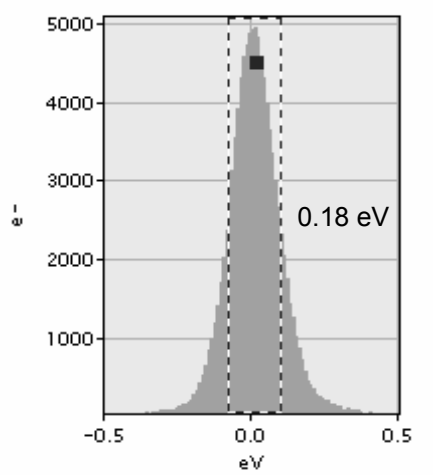

Fig.1 Zero-loss peak recorded with the monochromated Tecnai F20 as installed on the FELMI in Graz, acquisition time 1 s.

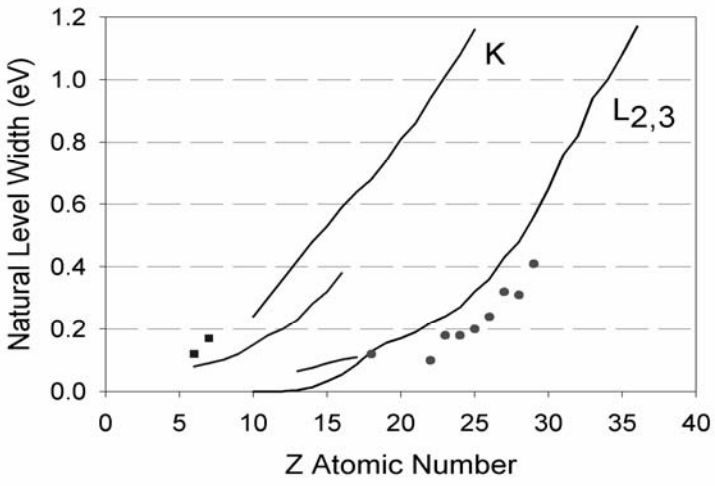

Fig.2 Natural line widths of K and L core levels vs. atomic number Z; solid lines show calculated data [3,4], rectangles and circles show experimental core level widths from gaseous molecules.

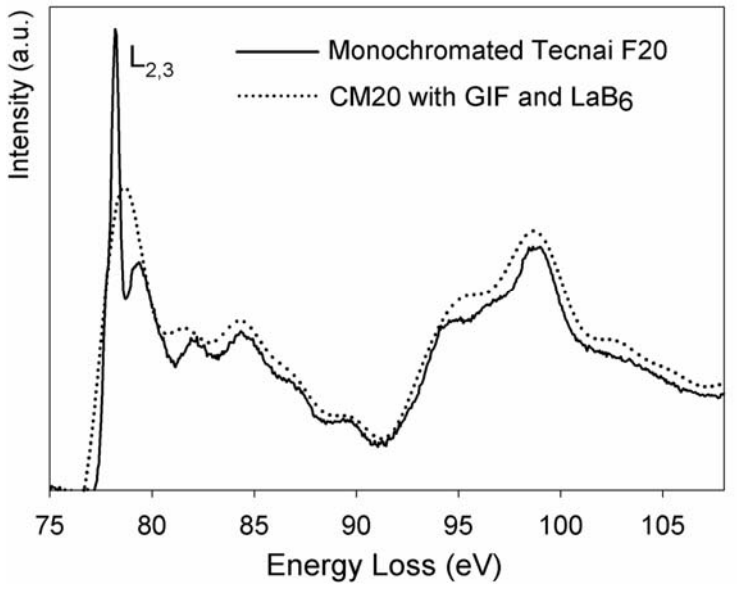

Fig.3 Al $L_{2,3}$ ELNES of α-Al_2O_3, raw data from the monochromated Tecnai F20 (background subtracted) are compared with data recorded at 0.8 eV energy resolution.

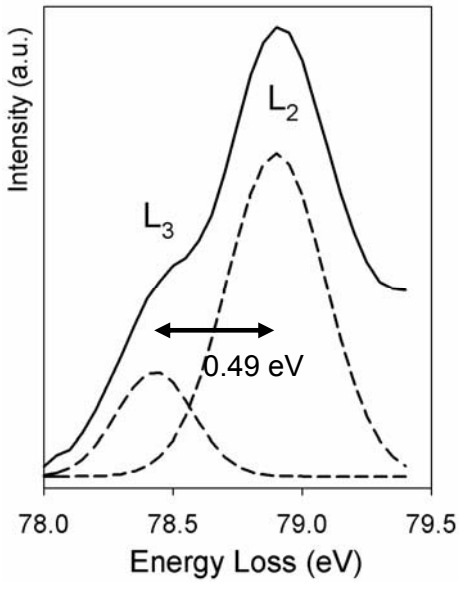

Fig.4 Al $L_{2,3}$ ELNES of α-Al_2O_3 fitted with Gaussians revealing the spin-orbit splitting; the separation of the L_3 and L_2.

Characterization and Compensation of Environmental Magnetic Fields for a Monochromized TEM

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High energy resolution in electron energy-loss spectrometry (EELS) has turned out to be an important prerequisite for the exploration of chemical bonding and electronic structure information by investigating the near-edge fine structure (ELNES) of an ionization edge. Subtle differences and small details of core-loss edges can only be detected, if the energy resolution is in the 100 – 200 meV range [1].

Several approaches have been tried to get access to this regime using software (deconvolution techniques) and hardware improvements, like monochromator devices and high energy resolution spectrometers. Since lately, transmission electron microscopes (TEM) with a Wien filter monochromator are commercially available from FEI [2]. These instruments in combination with high-resolution imaging filters from Gatan (HR-GIF) [3] offer energy resolution values of 100 – 200 meV measured as the full width at half maximum (FWHM) of the zero-loss peak.

However, the factors influencing energy resolution not only depend on the instrument, like energy spread of the electron beam, acceleration voltage stability, and monochromator and spectrometer performance, but also on the environment of the microscope in terms of magnetic fields. Simultaneously with the improvement in energy resolution of such instruments, their sensitivity to external stray fields has increased, too. Special care must be taken to ensure minimum influence and effect on the overall energy resolution.

In this work we characterized the influence of external magnetic fields on the energy resolution of the system in a time-resolved way. This was accomplished by a special readout mode of the CCD camera of the HR-GIF in which the position of the zero-loss peak is measured against time (figure 1a). Since the time resolution in these "streak images" is rather high (time/pixel in the horizontal direction of figure 1a; typically in the 1 ms range), a frequency evaluation can be performed showing the contributions of the individual frequencies (figure 1b). The resulting zero-loss peak for an acquisition time of 0.5 s is displayed in figure 2; its FWHM is less than 150 meV.

In a first step, the impact of external magnetic fields on the EELS performance was determined experimentally by using streak images. For that purpose, magnetic fields of defined field strength and frequency were artificially created by three perpendicular pairs of coils. Under these conditions streak images were acquired and subsequently analyzed in terms of energy-resolution (broadening of the zero-loss peak). Figure 3 shows the frequency response to a 600 nT external magnetic field at different frequencies. It can be seen from the graph that the sensitivity of the system to external fields increases with increasing frequency. On the other hand, this experiment also allows an estimation of the maximum allowed fields in order to reach a certain energy resolution: For instance it must be less than 20 nT at 40 Hz to reach better than 250 meV energy resolution, if the only limiting factors were external magnetic fields. With this setup we also found that the system seems to be most sensitive to fields perpendicular to the microscope-GIF plane.

In order to minimize the influence of external magnetic fields a field compensation system was installed (MR-3 Triaxial Magnetic Field Compensation System from Stefan Mayer instruments [4].This system is able to reduce magnetic fields to a few nT and helps to establish a stable situation as far as magnetic fields are concerned. Experiments are in progress to evaluate its effect on the measured energy resolution.

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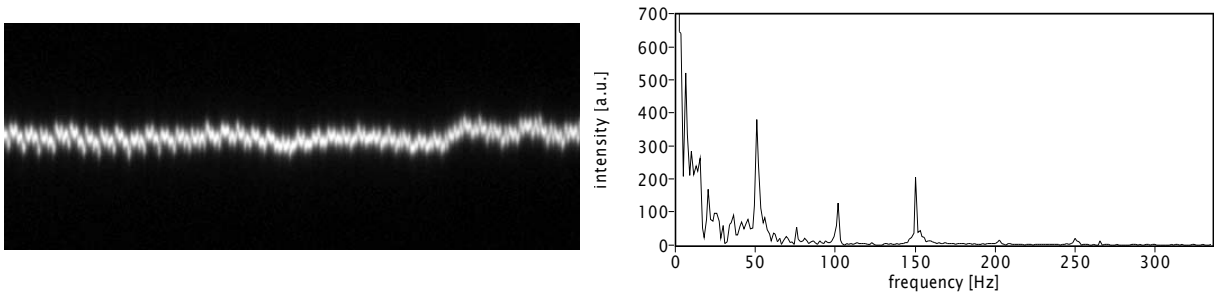

Fig.1 Streak image (a) and frequency evaluation of the zero-loss movements (b). The main contributions to the instabilities arise from 50 Hz, 150 Hz, and low frequency portions. The image height in (a) corresponds to 2 eV; its width to 0.76 s.

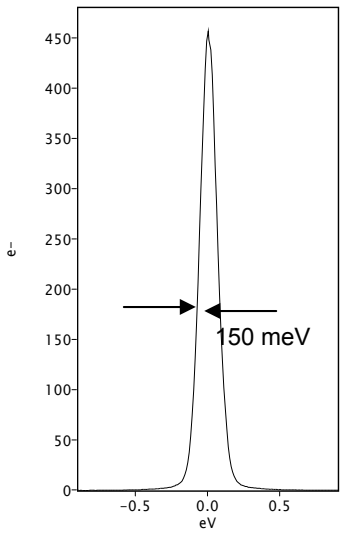

Fig.2 Zero-loss profile extracted from a 0.5 s portion of figure 1a.

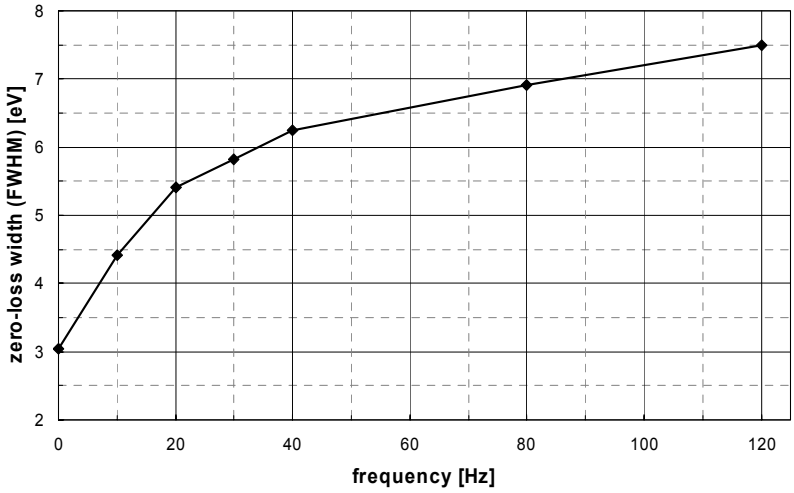

Fig.3 Frequency dependency of the high energy resolution EELS system to a 600 nT (peak-to-peak) external magnetic field.

High Resolution STEM EELS and EFTEM Investigations on FIB Samples with a Monochromator Microscope

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Materials characterization using analytical transmission electron microscopy currently makes enormous progress, on one hand due to the advances in site specific specimen preparation by means of focused ion beam instruments (FIB) [1] and on the other hand due to the development of more powerful microscopes and better analytical accessories [2].
In addition to its capabilities as a micro/nanostructuring or modification tool, as a deposition apparatus, and as an implantation instrument, the FIB provides the ability to precisely remove material via physical (and chemically assisted) sputtering while imaging the sample with secondary electrons or ions, making it ideally suitable for efficient TEM sample preparation. In this paper we used the FEI Nova200 NanoLab™ dual beam FIB for preparing thin foils of Vanadiumnitride films, deposited on silicon substrates via electron beam evaporation. Transition metal nitrides offer an attractive variety of physical and chemical properties which make them potential candidates for many technological applications, for instance as barrier material in Cu metallization [3,4]. The specimen lift-out was accomplished in-situ by fastening a 1.5 µm thick lamella to an Omniprobe™ tungsten needle using Pt deposition, detaching it from the substrate, lifting it out and attaching it to a TEM grid, again by means of Pt deposition. The specimen was then milled to a thickness of about 70 nm and was finally polished with 5 kV Ga ions at 10° incident angle to further reduce amorphization. The endpoint of sample thinning with the FIB was monitored by observing contrast changes of the lamella using a 1 pA (30 kV) ion beam. That way relative sample thicknesses in the range of t/λ=0.3-0.4 could be achieved, which translates into an absolute value of about 50 nm, assuming an inelastic mean free path of 130 nm for the parameters employed during the EFTEM/EELS work.
For the analytical investigation of the samples we used our 200 kV monochromated FEG-TEM equipped with a high resolution imaging filter [5]. The instrument is capable of resolving 0.2-0.3 eV, while providing 2 nm STEM probes. This performance was achieved by operating the monochromator in an accelerating gun lens mode at a potential of 0.8 kV and an excitation of 0.4 [6]. Although in this mode the total beam current is less, the smaller aberrations gave better EELS and STEM performance. Figures 1a and b show two high-angle annular dark field images taken with a monochromated STEM beam. In the EFTEM jump ratio image (figure 2a) different VN zones are visible giving rise to distinct differences in the N-K ELNES, as revealed by the monochromated spectra in figure 2b.

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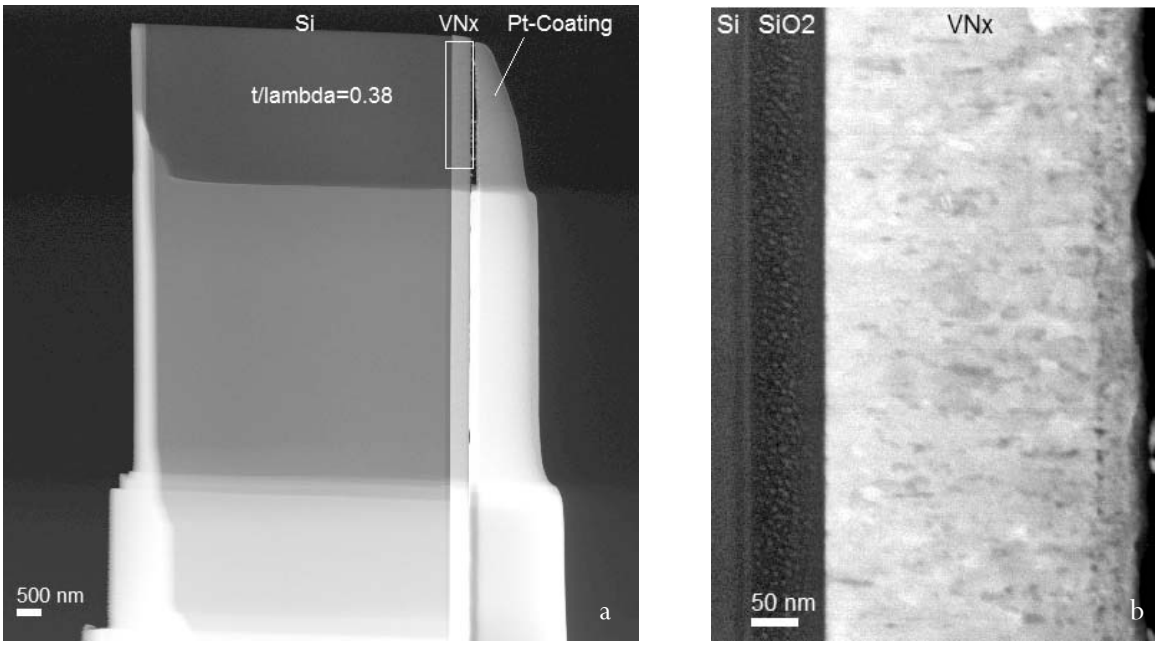

Fig.1 a.) High-angle annular dark-field image of a FIB milled VN_x coating on silicon. The thickness in the substrate amounts to ~50 nm. b.) HAADF image taken in the monochromator ON mode using an accelerating gun lens setup. The fine-grained crystallites of the sample stayed largely intact after milling.

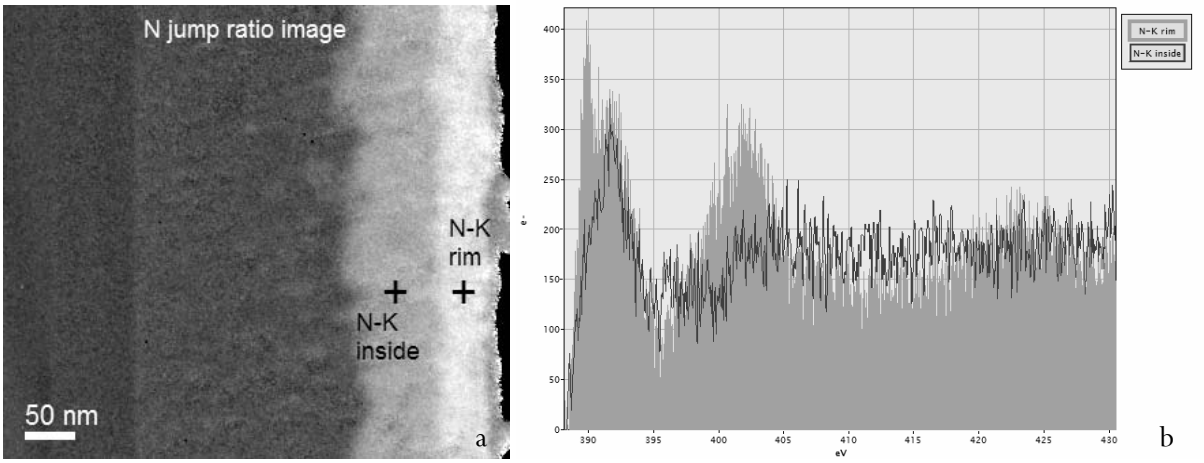

Fig.2 a.) EFTEM image of the nitrogen distribution, revealing two zones with different concentrations of nitrogen. b.) Monochromated STEM EELS spectra featuring deviating ELNES at these locations.

HREM Study of Hexagonal and Rhombohedral Graphites for Use as Anodes in Lithium Ion Batteries

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It is well known that the performance of graphite for use as anode material in lithium ion batteries largely depends on its bulk and surface structure [1]. It was, for instance, observed that the electrochemical behaviour is influenced by the graphite crystal modification. Layered graphite exits in two crystallographically different forms, the hexagonal form (α- or 2H-phase) with a AB… graphene layer stacking sequence, and the rhombohedral modification (β- or 3R phase) with an ABC… stacking sequence [2].

In order to study the influence of the respective phases on the electrochemical behaviour, we have prepared and investigated two graphite samples. The first sample was prepared by heat-treatment of graphite (courtesy of Superior Graphite Co.) at 2000°C under nitrogen gas to create 100 % α-phase. The second sample was made from the same graphite but mechanically processed by impact milling to increase the extent of the β-phase to about 25 %. The relative phase contents could be characterized by X-ray diffraction [3].

HREM investigations in an FEI Tecnai F20 equipped with a field emission gun and a supertwin lens revealed very different surface and "sub-surface" morphologies for the two graphites. In case of the hexagonal graphite, unique convoluted graphene layers could be determined at the prismatic surfaces. If the crystal is viewed along the (001) zone axis, these layers are visible at the surface in the form of "lines" running parallel to the crystal edges (Fig.2a). A view along the (110) zone axis (Fig.2b) makes the convoluted "onion-like"-oriented graphene layers at the prismatic surface more clearly visible. An additional characteristic feature of this sample is the high stacking order in the sub-surface and bulk region. At contrast, the HREM images of the rhombohedral graphite reveal disordered surface layers and a sub-surface region with many stacking faults and slight graphene layer bendings (Fig.3).

The anode performance of these two graphite samples depends strongly on the electrolyte. It was found that differences in the sub-surface layer structure have a most significant influence on the performance in an ethylene carbonate/dimethyl carbonate based electrolyte. The differences in surface structure and morphology are considered to have the highest impact in a propylene carbonate/ethylene sulfite based electrolyte. Only for an ethylene carbonate/diethyl carbonate based electrolyte, the performance differences are small [3]. Put it simply, the structural features of the respective graphites can be detected with a proper electrolyte.

As a simple rule, a high number of structural defects is beneficial to the performance; e.g. the discharge capacities are higher than those with hexagonal graphite. Additionally, these defects create hindrances inside the graphite, which just allow the small unsolvated lithium cations to penetrate into the bulk graphite. Therefore, the rhombohedral graphite is less vulnerable to co-intercalation of solvents, a reaction being detrimental to practical graphite anode performance [4].

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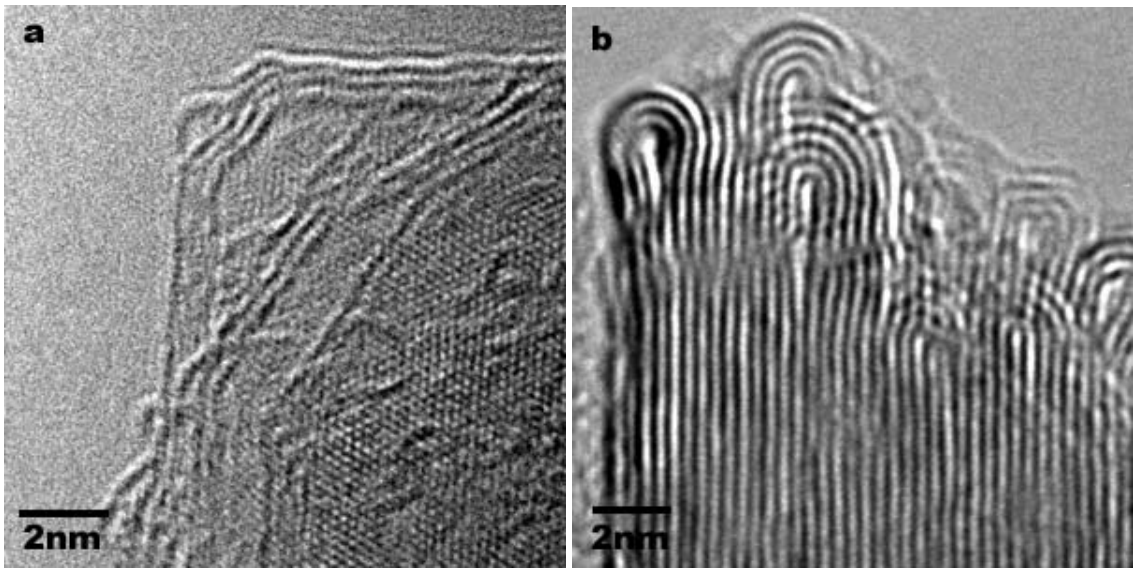

Fig.1 High resolution TEM image of hexagonal graphite heat treated at 2000°C under N_2; a. crystal viewed along [001] zone axis; b. crystal viewed along [110] zone axis revealing the convoluted graphene layers at the termination of the prismatic layers.

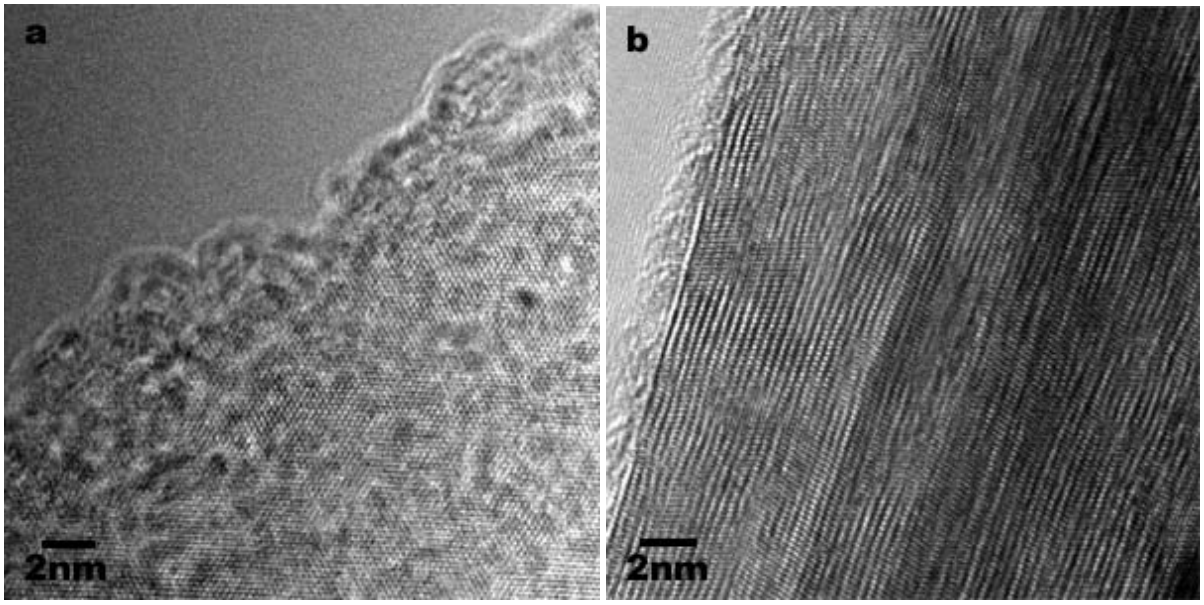

Fig.2 High resolution TEM image of rhombohedral graphite mechanically processed; a. crystal viewed along [001] zone axis; b. crystal viewed along [110] zone axis revealing the distorted graphene layers.

Synthesis, Size Control and Electron Microscopic Characterization of Lamellar Gold Nanoparticles

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We report the synthesis of gold nanotriangles and their characterization in the electron microscope. We show for the first time how the triangular particles can be selectively grown up to large lateral sizes with high edge-length/thickness ratios.

The particles were synthesized by reduction of tetrachloroauric acid in aqueous solution at room temperature. Sodium citrate was used as both reduction- and capping agent. The usage of the seeding method where 5 nm gold seeds were added to the reaction solution yielded a higher amount of triangular particles. Size control could be achieved by varying the pH of the solution by adding various amounts of hydrochloric acid. A lower pH resulted in selective growth of the nanotriangles and in increase of the mass fraction of the triangles to over 95%.

The particles were characterized by SEM (Leo Gemini FEG-SEM and FEI Nova200 DualBeam FIB/SEM) and TEM (FEI Tecnai F20 and Philips CM20, both equipped with high resolution GIFs). The thickness of the particles was determined by recording EFTEM thickness maps.

Figure 1 shows particles from solutions with various final pH values. Edge-length of the synthesized particles ranged from 25 nm up to 50 microns. Interestingly the edge-length/thickness ratio of the particles increased dramatically with increasing particle size, e.g. for triangles with 50 nm edge length an average thickness of about 8 nm (edge-length/thickness ratio of 6.25) was found whereas for particles with edge lengths of 20-40 microns the average edge-length/thickness ratio was 411. Furthermore we have found the particle thickness to depend on the degree of crystal truncation which will be subject of further research.

Figure 2a shows the HREM image of a nanotriangle viewed along the [111] zone axis. The lattice fringes have a spacing of 0.25 nm that can be explained by electron diffraction.

Figure 2b shows the electron diffraction pattern of a gold nanotriangles also viewed along the [111] crystallographic zone axis. The 1/3(422) reflections with a spacing of 0,25 nm indicate parallel twin planes which confirms the results obtained by Kirkland et al. [1] who examined gold particles synthesized following the method of Chiang & Turkevich [2]. However, the different intensities of the 1/3(422) reflections of various nanotriangles indicate different relative amounts of (111) twin planes. We assume that nanotriangles below 50 nm edge length are usually truncated bitetrahedral crystals with few parallel central twin planes whereas prismatic particles are common with increasing particle edge-lengths.

Figure 2c, the HREM micrograph of a gold nanotriangle typical for a sample as shown in figure 1a viewed side-on, shows a twinned truncated bitetrahedral crystal shape.

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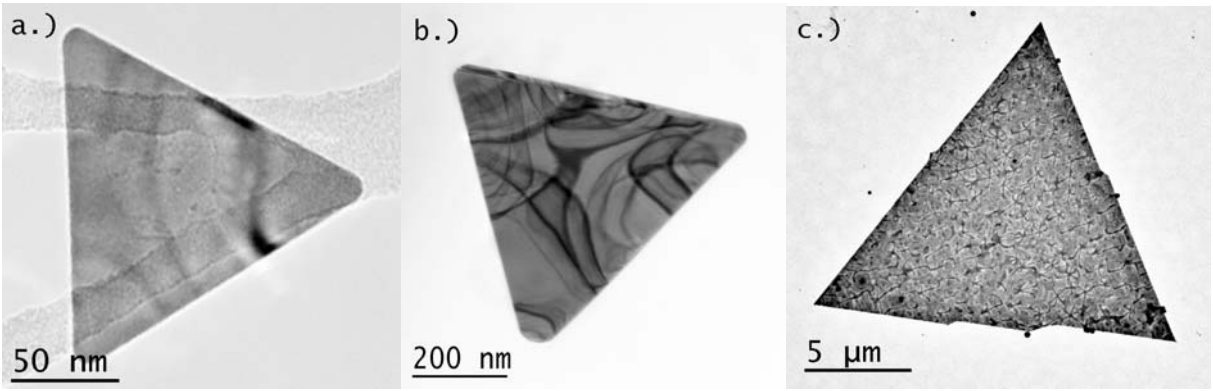
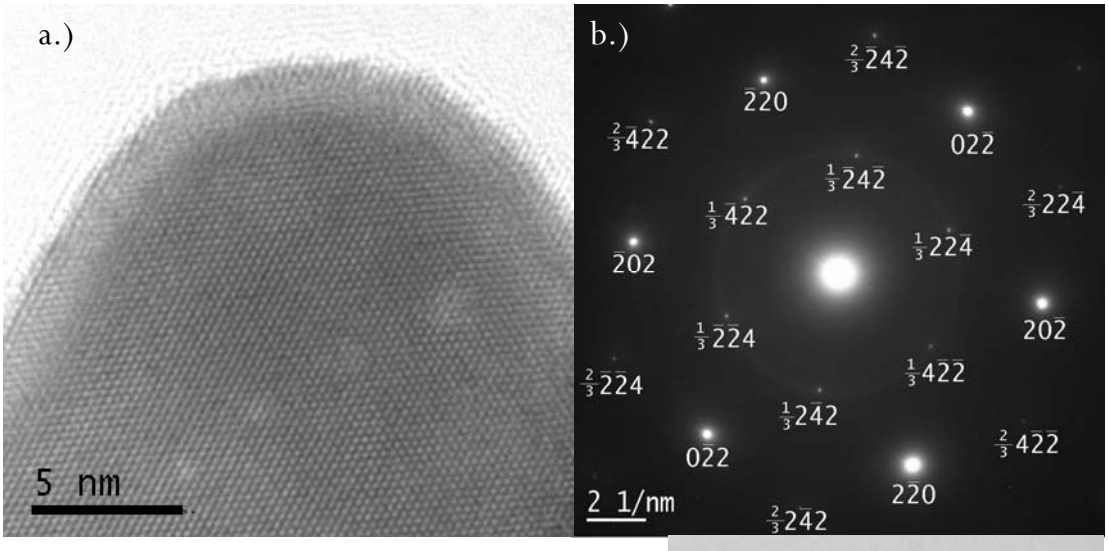


Fig.1 TEM bright field images of trigonal gold particles. a) 155 nm edge length grown in solution with final pH of 5.55; b) 630 nm edge length grown in solution with final pH of 3.01; c) 17 microns edge length grown in solution with final pH of 2.63



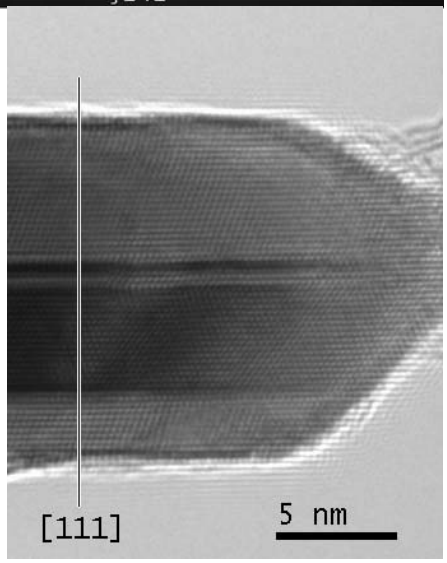


Fig.2 HRTEM images and diffraction pattern of a twinned truncated bitetrahedral gold nanotriangle. Figure 2a shows the particle viewed along the [111] crystallographic zone axis. No crystal defects can be seen from this perspective but an unusual lattice spacing of 0.25 nm is observed. Figure 2b is the selected area electron diffraction pattern of a particle similar to the particle shown in figure 1a. The additional 1/3(422) spots are due to (111) twin planes. Figure 2c shows the same particle viewed side-on. There are several central (111) twin planes that are responsible for the truncated bitetrahedral shape of the nanoparticle.

Effects of Sample Preparation on Cr-Steels: An EFTEM Study

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Structure-property correlation plays an important role in the design of advanced materials which are increasingly based on sub-micrometer and sometimes even nanometer grains. Especially energy-filtering transmission electron microscopy (EFTEM) in combination with electron energy-loss spectroscopy (EELS) has many advantages for characterising metals and alloys that no other technique can quite match [1]. It combines the advantage of large area view (some 10 μm) with high spatial resolution (down to a few nm).

The mechanical properties of steels are determined predominantly by secondary phases, i.e. impurities, precipitates and grain boundary phases on a microscopic scale. Consequently, in the development of steel technology, much attention has been paid to the investigation of steel microstructure. In many systems (e.g. steels) it is essential to obtain both precipitate distribution and shapes and also the distribution of the elements. Owing to the contribution of the steel matrix precipitates are sometimes difficult to detect, e.g. very small particles are coherent with the lattice of the matrix. With the advent of EFTEM a powerful method for detecting precipitates and grain boundary phases in materials has become accessible [2,3,4,5].

In this study we applied the EFTEM technique to the investigation of Chromium steels. The instrument used was a Philips CM20 operated at 200 kV with a LaB_6-cathode, equipped with a Gatan Imaging Filter (GIF). Specimens were prepared in two different ways, employing low-angle ion milling (Ar^+, 6 h, 4° resp. 6°, 4 kV) as well as electropolishing (5% $HClO_4$ + 95% CH_3COOH, 50 V, 15°C). To check the quality of the thinning, thickness maps were acquired and in addition elemental maps for Fe, Cr, Ni, V, N, C were recorded. Figure 1a, b contains large field of view bright field images of the ion milled (a) and the electropolished specimen (b) recorded under rocking beam illumination employing a rocking angle of 70mrad in order to reduce diffraction effects. Depending on the type of preparation, morphological differences between the specimens become visible. A more detailed view on the electropolished specimen at higher magnification is shown in Fig.1c (elastic image taken with a rocking beam) and in the thickness map (fig.1d), which shows areas of thicknesses ranging from 70 nm (bright) to 110 nm (dark zones). Preliminary estimations of the inelastic mean free path for the bright and the dark zones based on EDX analysis seem to indicate that the contrast is primarily due to differences in thickness rather than to the differences in the mean free path of inelastic scattering. The dark structures in the bright field image (fig.1c) correspond to the thicker areas in the thickness map (fig.1d). A comparsion of elemental jump ratio images with the thickness map reveals that zones of different chemistry correlate to different thickness. Besides showing Chromiumcarbides and Vanadiumnitrides collectively in black, Fig.1e (Fe M_{23} jump ratio image) also reveals thicker platelet-like structures corresponding to Ni and Cr enrichments, as can be seen on the Ni jump ratio image [6].

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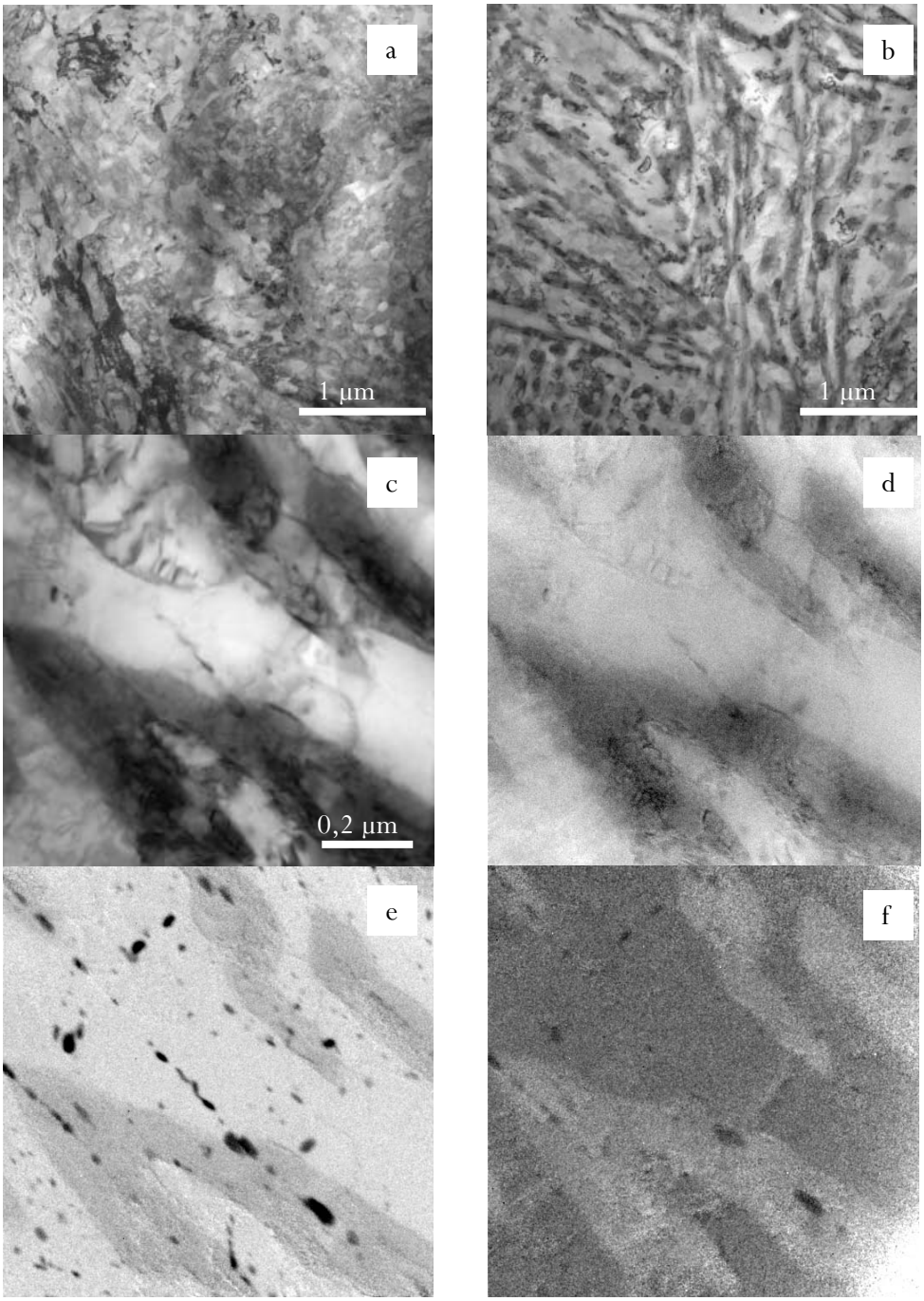

Fig.1 TEM bright field image of a.) the ion milled and b.) the electropolished Cr-steel specimen recorded under rocking beam illumination; c.) Zero-loss filtered bright field image of the electropolished specimen at higher magnification using RB d.) Absolute thickness map: 70 nm (bright) to 110 nm (dark); e.) Fe M_{23} jump ratio image; f. Ni L_{23} jump ratio image.

The Formation of Molecular Nitrogen in Chromium Nitrides Monitored by EELS

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The ability to damage inorganic materials by electron irradiation in a Transmission Electron Microscope (TEM) is well known [1]. Electron energy-loss spectrometry (EELS) is an appropriate tool to investigate the damage effect by means of the ELNES (energy-loss near-edge structure), which contains information about the local bonding and environment of atoms in solids. ELNES arises because the final states of the excitation process are unoccupied states above the Fermi level which may be appreciably modified by chemical bonding [2]. The aim of this work is to monitor changes in the N K ELNES of chromium nitride (CrN). Today CrN hard coatings are widely used in decorative application as a substitute for electroplated hard Cr or as chemically and temperature resistant hard coating on forming tools, moulds and dies.

CrN crystallises in the rock salt structure (fcc) and is antiferromagnetic with a Néel point of about 0°C. The CrN samples (CrN and $Cr_{0.47}N_{0.53}$) were prepared by reactive magnetron sputtering of a metallic chromium target in a nitrogen plasma. For the oxidised CrN sample ($Cr_{0.5}O_{0.2}N_{0.3}$), a controlled amount of oxygen was added to the plasma gas. Silicon wafers were selected as the proper substrate material for subsequent analysis. The quantitative composition of each film was determined by Electron Probe Micro Analysis (EPMA) and Rutherford Backscattering (RBS). Cross-sectional TEM specimens were prepared by standard metallographical methods and final Ar ion milling under a low angle. Experimental work was carried out on a 200 kV Philips CM20 (S)TEM with a LaB_6-cathode, equipped with a Gatan Imaging Filter (GIF). The measurements were taken with an energy resolution of 0.80 eV full width at half maximum of the zero loss peak and a chosen dispersion of 0.05 eV/channel. All spectra were corrected for dark current and intrinsic channel to channel gain variations of the CCD. The background below the ionisation edges was subtracted by using the power-law model and the spectra were corrected for the effect of thickness by means of the Fourier-ratio deconvolution [3]. The acquisition times for recording the spectra were chosen with 30 seconds. The experimental setup (microscope and GIF) was done at a different sample-area in order to minimise radiation damage at the point of interest.

Fig. 1 shows the N K ELNES of the not irradiated samples. The N K edge of CrN exhibits two features at 399.0 and 401.1 eV, respectively. The octahedral coordination of the N surrounding the Cr ions affects the crystal field splitting and this results in a separation of the N K edge into two features. The first peak corresponds to the electronic transitions to unoccupied N 2p orbitals hybridised to Cr 3d-t_{2g} orbitals while the second results from hybridised N 2p and Cr 3d-e_g states [4]. However, the intensity of the latter is much lower so that in most cases it is hardly visible.

Fig.2 shows the N K ELNES after 150 s irradiation with a high-current-density electron beam. The N K ELNES of CrN (cf. fig. 2A) remains nearly unaffected. By changing the composition, however, a sharp feature emerges at 401.4 eV. The relative intensity of this feature further increases from $Cr_{0.47}N_{0.53}$ to $Cr_{0.5}O_{0.2}N_{0.3}$. The feature in question presumably arises from a 1s-π^* transition of molecular nitrogen located in the interstitial position of the oxide layers [4]. The O K ELNES of the $Cr_{0.5}O_{0.2}N_{0.3}$ sample remains unaffected of the structural changes [5].

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We kindly acknowledge M. Dienstleder and A. Brunegger (both FELMI) for sample preparation.

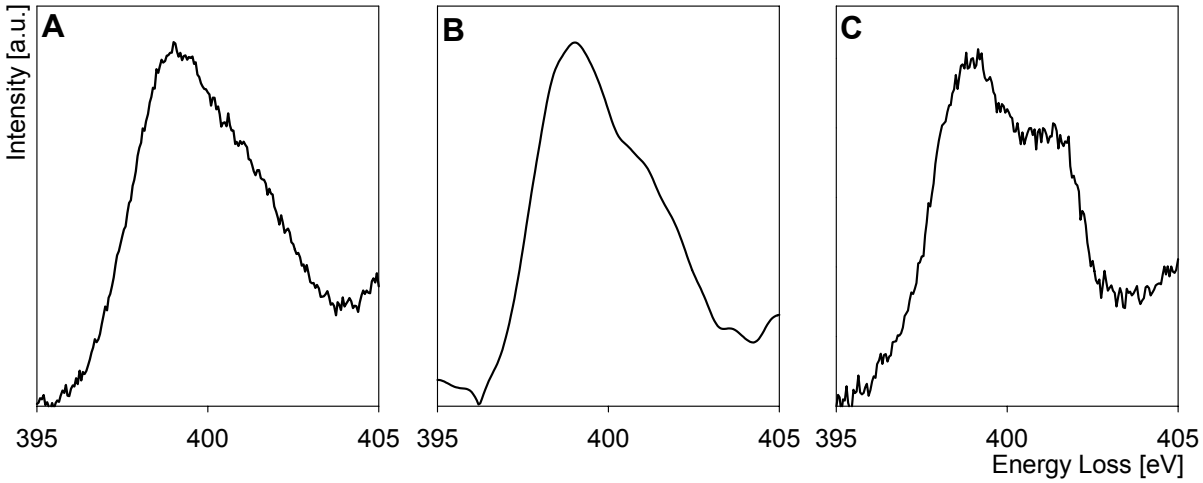

Fig.1 N K ELNES before radiation showing (A) CrN, (B) $Cr_{0.47}N_{0.53}$ and (C) $Cr_{0.5}O_{0.2}N_{0.3}$.

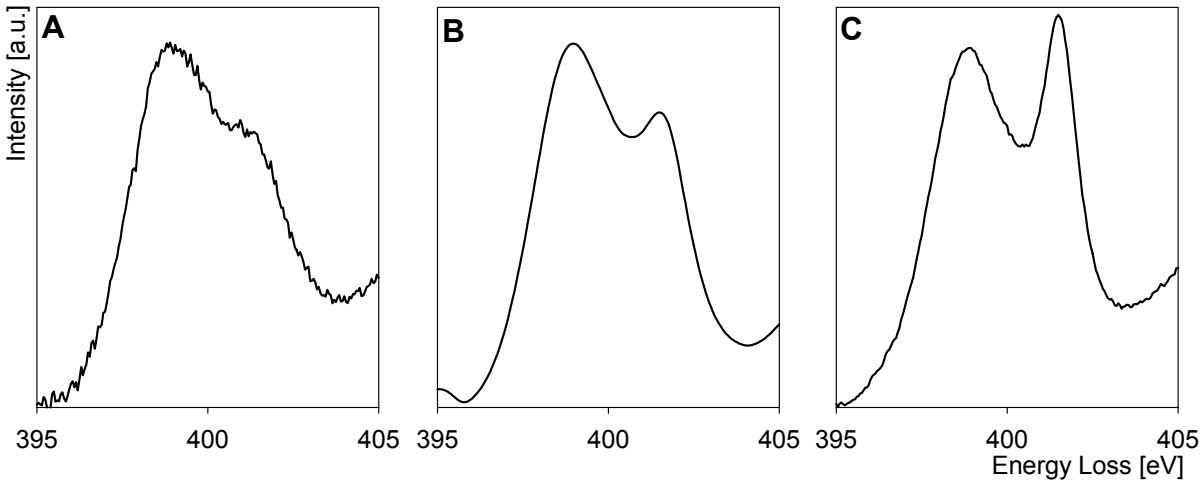

Fig.2 N K ELNES after a 150 seconds radiation showing (A) CrN, (B) $Cr_{0.47}N_{0.53}$ and (C) $Cr_{0.5}O_{0.2}N_{0.3}$.

STEM Performance on a Monochromated TEM

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Electron energy-loss spectroscopy (EELS) has developed into an established technique for chemical and structural analysis of thin specimens in the transmission electron microscope (TEM) [1]. The energy resolution in 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 (S)TEMs [2,3], offering the advantage to better resolve EELS fine structures, which contain valuable bonding information.

The method of Z-contrast imaging within an (S)TEM, utilizing a high-angle annular dark-field (HAADF) detector can perfectly complement the better energy resolution, since EELS spectra can be collected simultaneously [4]. In combination with a monochromator microscope not only high spatial resolution images can be recorded but also high energy resolution EELS spectra are attainable.
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 requirements for good test samples on one hand were large enough Z differences and on the other hand small enough features to evaluate a spatial resolution limit. Titanium oxide particles embedded in a Niobium matrix seemed to fulfil this requirement [5].

In STEM the spatial resolution of experimental images and spectra is determined by the size of the electron probe at the specimen. In unfiltered 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, however, (Monochromator on, dispersion of the beam, 0,25 eV energy resolution) the probe size is enlarged to approximately 2 nm as there is not enough demagnification of the source. In terms of energy resolution the performance depends also on the setup of the monochromator, since it can be operated in either a decelerating or an accelerating gun lens mode. Because aberrations (monochromator and spatial) are different in these modes, the quality of the images also vary. In this work we optimize the STEM probe for both gun lens modes to get the best energy resolution and the best spatial resolution.
Figure 1 contains two HAADF images generated with a monochromated STEM probe that was about 2 nm in size. The dark spots correspond to the Titaniumoxide particles which are surrounded by the brighter niobium matrix. The marked titaniumoxide particle has a size of 3 nm which gives an idea of the spatial resolution. The energy resolution of this probe, being 0,3 eV, is shown in Figure 2 as the FWHM of the zero loss peak.

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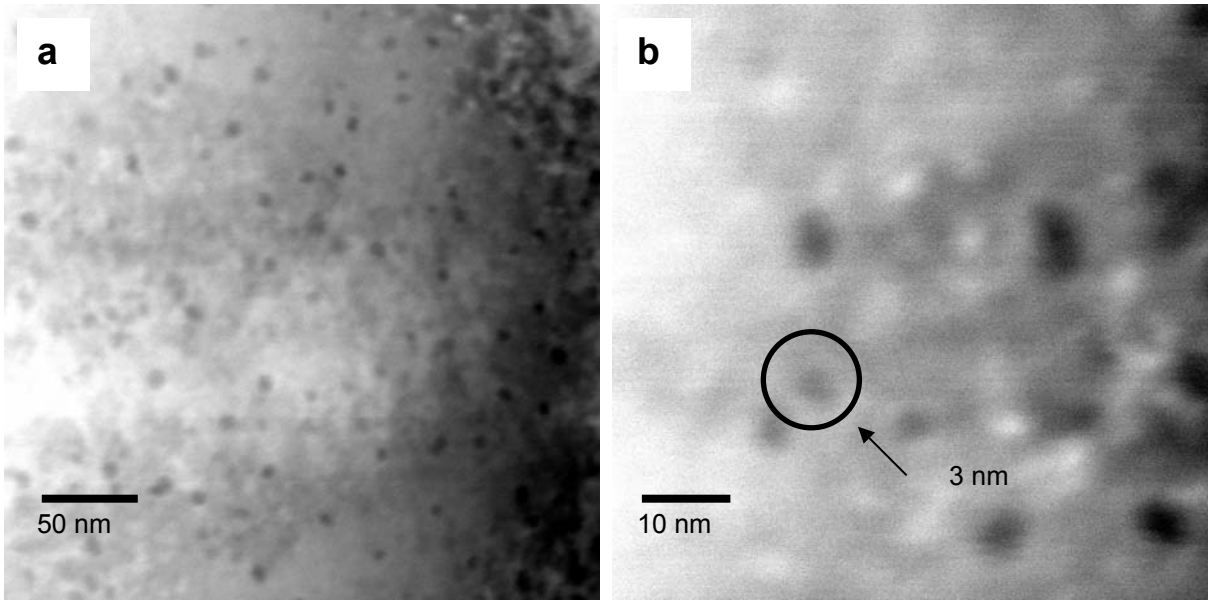

Fig.1 HAADF image generated with a monochromated STEM probe of titaniumoxide precipitates in a niobium alloy at a. low magnification and b. high magnification.

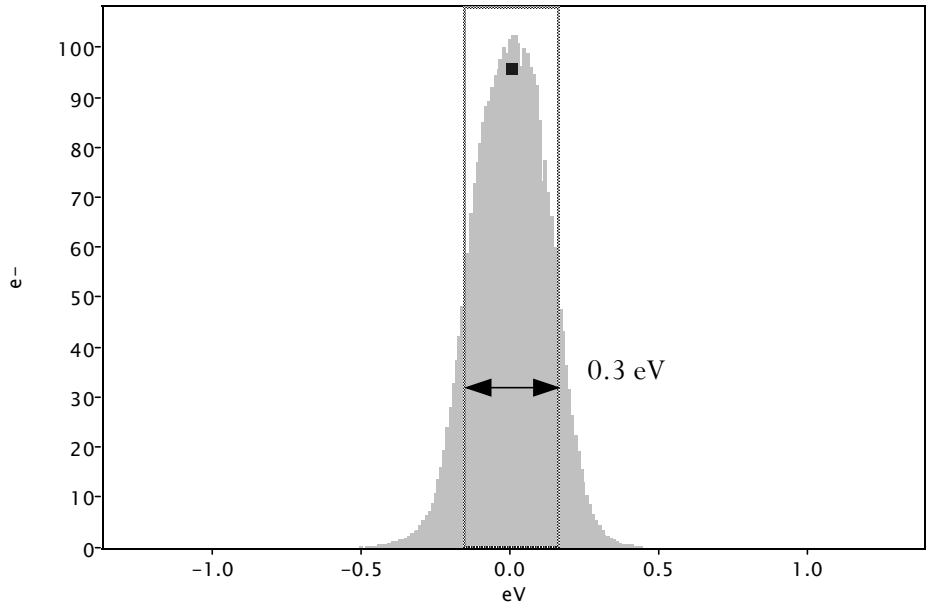

Fig.2 Zero loss peak of monochromated STEM probe. FWHM = 0.3eV

Identification and Characterization of a Novel Enzyme Involved in Steryl Ester Mobilization from Lipid Depots of the Yeast *Saccharomyces cerevisiae*

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Most eukaryotic cells contain sterols which are essential lipid components of membranes. Sterol biosynthesis, uptake, transport, storage, utilization and efflux are important and fundamental processes. To address these problems, we use the baker's yeast *Saccharomyces cerevisiae* as an experimental model system. Recent work in our laboratory was focused on hydrolytic enzymes involved in the mobilization of the storage form of sterols, the steryl esters (STE), from lipid depots, the so-called lipid particles. As a result of these efforts, a novel steryl ester hydrolase named Yeh2p (Yeast Steryl Ester Hydrolase 2) was identified and partially characterized. A manuscript describing these studies entitles "*YEH2/YLR020c* encodes a novel steryl ester hydrolase of the yeast *Saccharomyces cerevisiae*" has most recently been accepted for publication in the Journal of Biological Chemistry and will appear in spring 2005.
In mammalian cells, esterification of sterols is catalyzed either by lecithin:cholesteryl acyltransferase or by the two acyl-CoA:cholesteryl acyltransferases ACAT1 and ACAT2. In the yeast *Saccharomyces cerevisiae,* sterols which are not immediately needed as membrane components can be esterified in the endoplasmic reticulum by the STE synthases Are1p and Are2p (1-4). Together with triacylglycerols (TAG), STE are stored in lipid particles and form the hydrophobic core of this compartment (reviewed in ref. 5).
To identify protein(s) involved in yeast STE hydrolysis we decided to screen the *Saccharomyces cerevisiae* genome for ORFs containing sequences which are homologous to known mammalian STE hydrolases from various organisms. This approach identified the gene product of *YEH2/ YLR020c* as a yeast plasma membrane located STE hydrolase and the first enzyme of this kind characterized in yeast. Deletion of yeast *YEH2* led to complete loss of plasma membrane STE hydrolase activity, whereas overexpression of the gene resulted in a significant elevation of the activity. Purification of enzymatically active Yeh2p close to homogeneity unambiguously identified this polypeptide as a STE hydrolase. In addition to evidence obtained *in vitro* experiments *in vivo* contributed to the characterization of this novel enzyme. Sterol analysis of *yeh2Δ* unveiled a slightly elevated level of zymosterol suggesting that the esterified form of this sterol precursor is a preferred substrate of Yeh2p. However, in strains bearing hybrid proteins with strongly enhanced Yeh2p activity decreased levels of all STE were observed. Thus, it appears that Yeh2p activity is not restricted to distinct STE but rather has broad substrate specificity.

To test possible effects of imbalanced sterol/STE levels on the cellular structure of the yeast we performed microscopic inspection of the respective strains with special emphasis on the two compartments which are mainly involved in sterol homeostasis, lipid particles and plasma membrane. Whereas number and structure of lipid particles were not changed in strains bearing a highly active his-*YEH2* hybrid protein, significant changes of the plasma membrane structure were observed when a his-*YEH2* mutant was subjected to electron microscopic inspection (Figure 1). The membranous structures clearly identified as plasma membrane and peripheral endoplasmic reticulum (6) in wild-type appeared to fuse in his-*YEH2* resulting in punctuate areas. In contrast to the continuous membrane system seen in wild-type the plasma membrane of his-*YEH2* looked fuzzy and rigid. This structural feature may also be the reason why several attempts failed to isolate plasma membrane from his-*YEH2* by standard procedures. Changes in the structure of the yeast plasma membrane often result in altered sensitivity of cells to drugs or other stress inducing agents present in the environment. Indeed, mutants harboring his-

YEH2 were sensitive to nystatin, a polyene component which preferentially binds to sterols in the plasma membrane. Thus, changes in nystatin sensitivity as well suggested a disturbed structure of the plasma membrane in particular due to alterations of sterol incorporation into this compartment.

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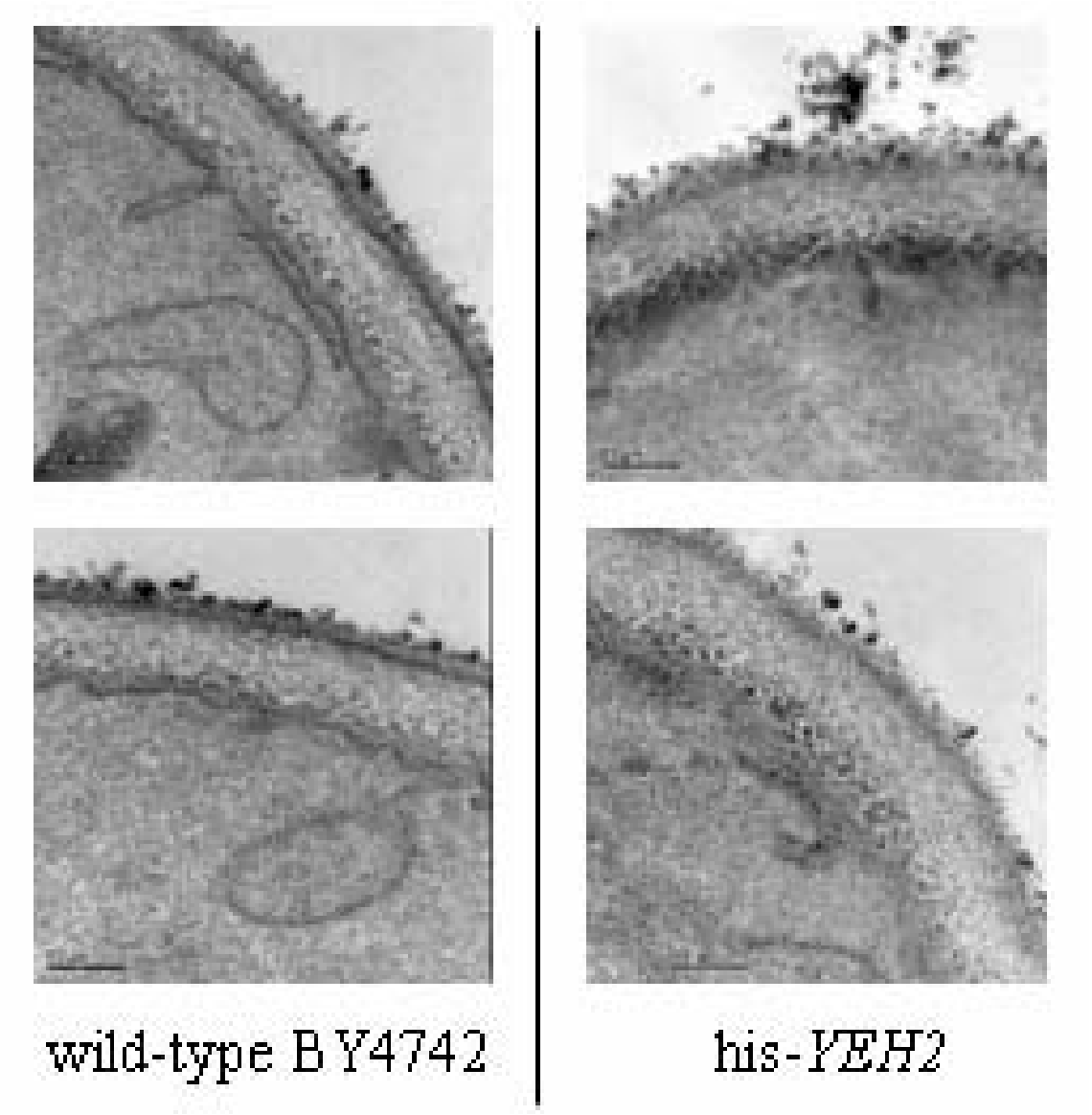

Fig.1 Highly active his-Yeh2p affects the structure of the plasma membrane. Noteworthy, the structure of the plasma membrane is significantly disturbed in the mutant. Electron microscopy of the wild-type BY4742 (left) and the mutant his-*YEH2* (right) was performed using standard methods employing staining with uranyl acetate. Images were produced on a Tecnai G^2 12 (FEI Company) equipped with a CCD camera (Gatan Bioscan) at 100kV.

Isolation and Characterization of Peroxisomes from the Yeast *Pichia pastoris*

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The methylotrophic yeast *Pichia pastoris* is a highly successful system for the production of heterologous proteins (1, 2). One advantage of *Pichia pastoris* as expression system is its efficient and tightly regulated *AOX1* (alcohol oxidase) promoter (3) Besides its role as an expression system, *Pichia pastoris* is also of interest for cell biologists, because of its advantages for studying peroxisome relating topics, e.g., peroxisome biogenesis (4-7). Many studies have been published about *Pichia pastoris,* but hardly any information about cell biological features of this microorganism can be found in the literature. Surprisingly little is known about biochemical and cell biological properties of peroxisomes from *Pichia pastoris*. For this reason, the Diploma Thesis by T. Wriessnegger was initiated aimed at the development of a method for the isolation of this organelle at high purity, establishing appropriate tests for measuring the enrichment and contamination with other cellular fractions, and analyzing peroxisomes with special emphasis on peroxisomal membrane components.
This Diploma Thesis contributed to the systematic analysis of *Pichia pastoris* peroxisomes by addressing the following tasks:

- The growth kinetics of *Pichia pastoris* after shift to methanol and oleic acid medium, respectively, were investigated.
- Structural differences of peroxisomes when grown on different carbon sources (methanol, glucose, glycerol, oleic acid) were visualized by electron microscopy.
- Since results obtained with previous protocols for the isolation of peroxisomes from *Pichia pastoris* were not very satisfying, a major aim of this study was to develop a more efficient and reliable isolation protocol.
- The quality of the obtained peroxisomal fractions was tested by Western Blot analysis using various antibodies against marker proteins.
- The protein composition of the peroxisomal fraction was analyzed.
- Analysis of the lipid composition of peroxisomal membranes from *Pichia pastoris* was performed including quantification of phospholipids, sterols and fatty acids.

When *Pichia pastoris* was grown on methanol (Fig. 1A and B) or oleic acid (Fig. 1G and H) containing media, peroxisomes were fully developed as confirmed by electron microscopic inspection. Compared to glucose as a standard medium (C and D); growth on glycerol (E and F) stimulated formation of mitochondria. With methanol as the carbon source, alcohol oxidase (AOX) was synthesized as the predominant protein in peroxisomes, whereas induction of peroxisome proliferation by oleic acid changed the peroxisomal polypeptide pattern, and enzymes involved in fatty acid β-oxidation were expressed at high amounts. Major phospholipids of *Pichia pastoris* peroxisomes are phosphatidylcholine followed by phosphatidylethanolamine, phosphatidylinositol and phosphatidylserine, and major fatty acids in peroxisomes are oleic acid, stearic acid, palmitic acid and linoleic acid. Ergosterol is the most prominent sterol of *Pichia pastoris* peroxisomes. In summary, this investigation provided a useful basis for further studies of *Pichia pastoris* organelles and the manipulation of these organelles for protein expression studies.

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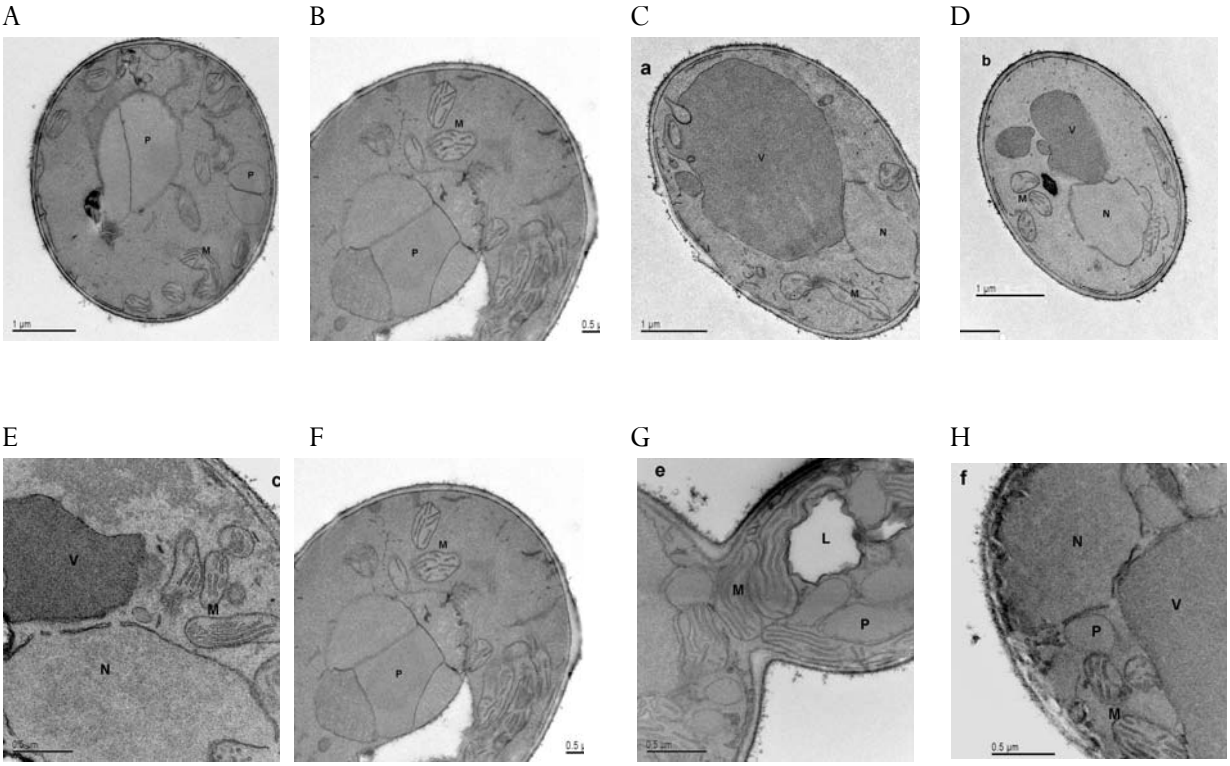

Fig.1 Electron micrographs showing subcellular morphology of *Pichia pastoris* X33A when grown on methanol (A and B); glucose medium (C and D); glycerol (E and F) and oleic acid medium (G and H); P, peroxisomes; N, nucleus; M, mitochondria; V, vacuole; L, lipid droplets.

Human Brain Investigations in an ESEM – a New Approach to Clinical Inspection ?

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Biological samples, such as human tissues, are and have been in special focus in different research areas since a long time. Scanning electron microscopy, from a traditional point of view, did not have that impact as an analysis tool for biological samples as e.g. light microscopy, which was due to the fact that these materials are not prone to the high vacuum and normally are not conductive. The up-streaming ESEM technique has totally changed the possible applications of SEMs in that field.

Human brain and its fracture behaviour is of special interest for biomechanical simulations, which may give a more detailed view on the impact of surgery devices on this special tissue. In this study human brain was investigated (for the first time) in an ESEM, whereas different preparation steps before the investigation has been evaluated. The first human brain samples were investigated without any sample preparation (fixation) and analysed in the ESEM in low vacuum mode at 100Pa without cooling. A degradation process at the surface of the human brain could be observed, whilst formalin fixed tissue (Fig.1) was relatively stable at the same conditions (see Fig.2). Mechanical fractures were made by just disrupting the white matter structures with some tweezers. Fig.2c clearly shows the end of a disrupted single neuronal axon, whereas the myelin sheath can be visualized. Further investigations were made at the interface between the external meningeal and the grey matter. Arterioles between the meningeal and grey matter are shown in Fig.2b. Fig.2c gives an impression on the structure of the white matter, showing a high depth of view, as one of the major advantages using the ESEM technique.

The investigations on untreated and fixated human brain tissues showed that the ESEM technology allows the inspection of these biological materials at very high magnifications. Due to the advantages of the ESEM technique (high depth of view, high resolution, inspection of wet samples etc.) it may become an additional method for clinical tissue screenings. Future activities will cover the in-situ inspection of these tissues, with the objective to get more data on the fracture behaviour of tissues.

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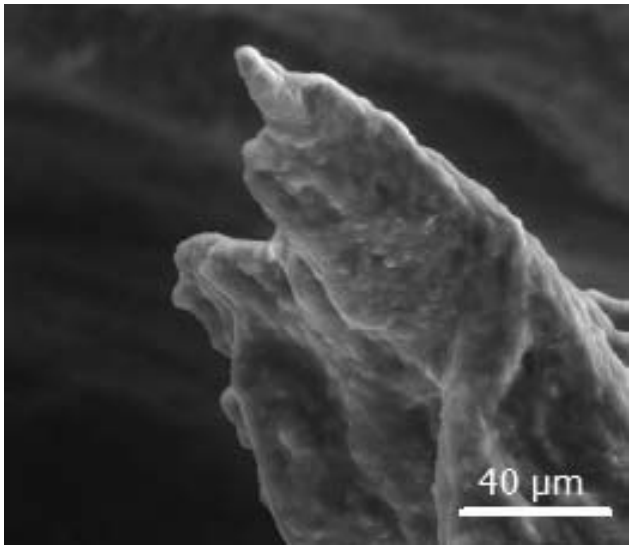
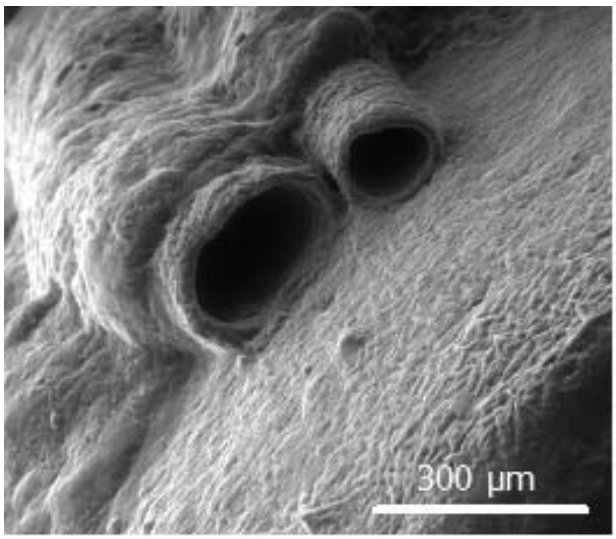
Fig.1 formalin-fixed and sliced human brain showing white and grey matter





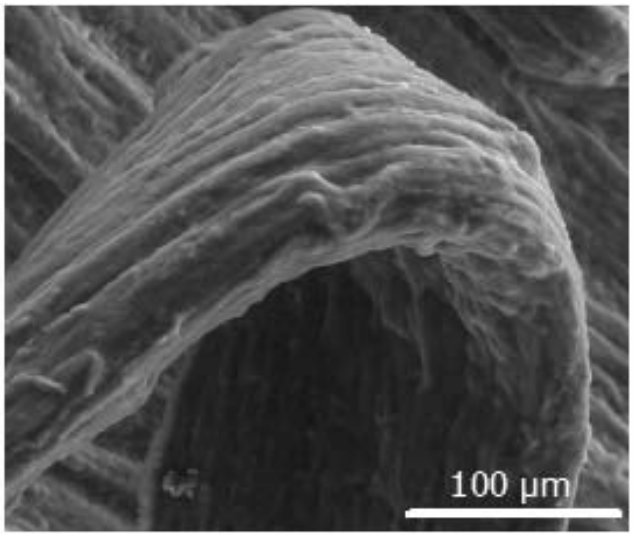


Fig.2 ESEM images taken at 100Pa; a) single neuronal axon with myelin sheath, b) arterioles between grey matter and external meningeal; c) white matter

ESEM – In-situ Heating Stage Experiments on the Sintering Behaviour of "Greenbody" Ceramics

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The investigation of non-conductive, wet and dirty samples is one of the main advantages of the ESEM technology. More than that, an ESEM enables the microscopical investigation of dynamic experiments, such as tensile tests or heating stage experiments. The heating stages can be used at temperatures up to 1500°C, which allow the in-situ observation of phase transitions, melting points and morphological changes at very high magnification. The sintering behaviour of "green-bodies" determines enormously the properties of the produced ceramic components. Therefore, it is essential to understand this sintering processes in detail, which determine the crystal-formation, crystal-size and –orientation.

Based on DTA (differential thermoanalysis) data of a ceramic green-body, which shows the phase transitions within the sintering process at certain temperatures (~900°C and ~1200°C), the heating stage experiment was performed with the same temperature profile. Water vapour was used as the imaging gas at the fixed value of 400Pa. During the heating stage experiment (total 9 hrs) the sample and heating shield bias was adjusted to optimum contrast and brightness. The phase transition at 900°C could not be detected, whereas at 1200°C and above the morphological changes nicely correspond to the stated phase transitions in the DTA results. Crystal growth could be observed during the cooling down procedure, where two separate forms of crystallites with different size distributions were formed. The total sintering shrinkage was determined to be approximately 25% (vol.), which also corresponds to the conventional sintering data.

From the produced ceramic compounds (spheres) TEM samples were prepared using the Nanolab Nova200 (Dualbeam from FEI). TEM investigations of conventional- and heating stage produced ceramics were performed, to evaluate the differences in the two "manufacturing" processes. The differences in crystal size distribution from conventional- and heating stage sintering may be caused by different cooling down rates and different environments. In general the in-situ heating stage experiments help enormously to understand such temperature dependent processes on a microscopic basis. Further experiments will cover other phase transitions from different materials and will study the influence of the applied conditions, such as the kind of imaging gas and pressure.

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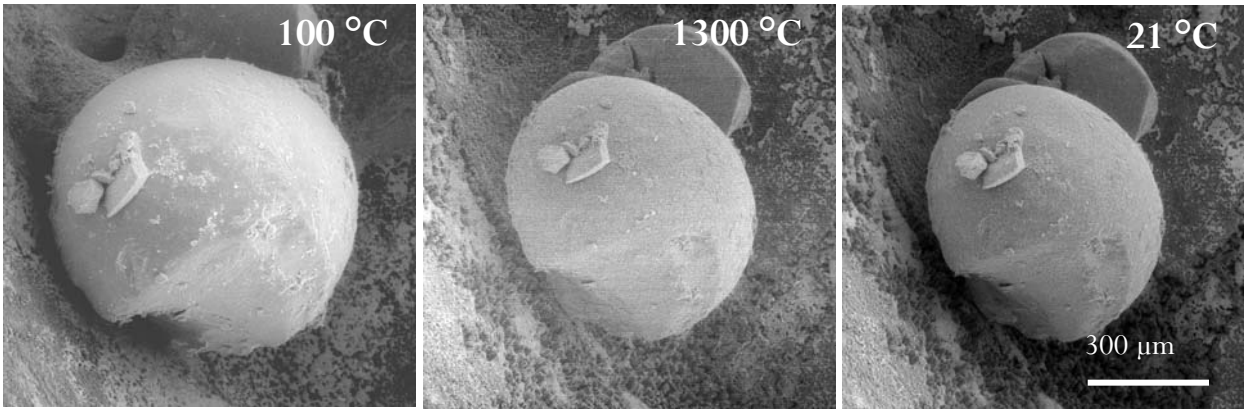

Fig.1 Shrinkage of the ceramic green-body after in-situ heating stage experiment

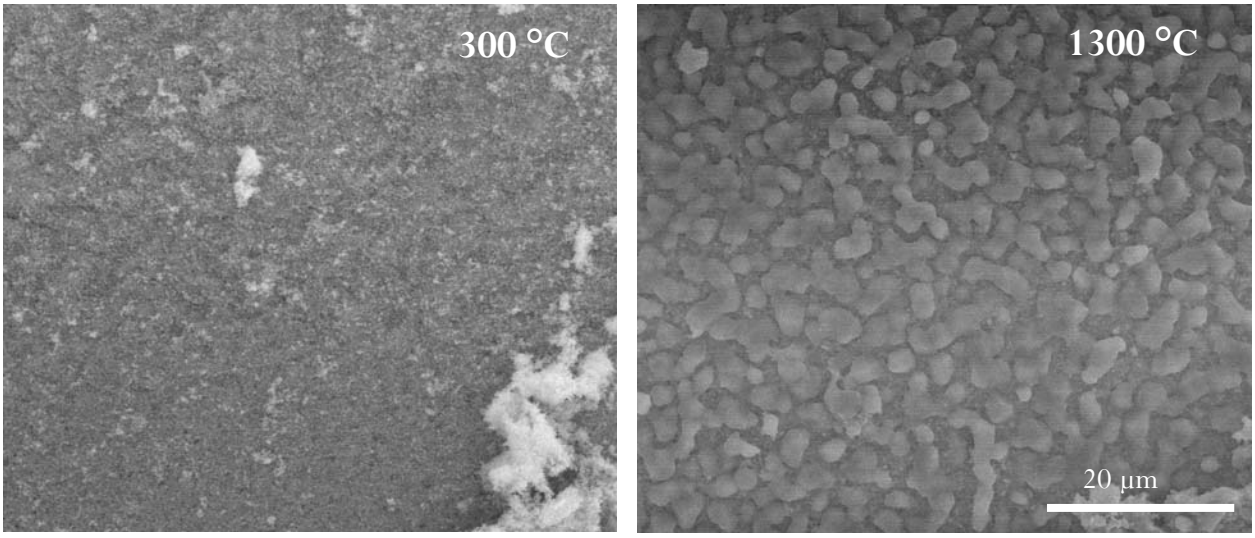

Fig.2 Phase transitions at the surface of the ceramic green-body

TEM Investigations of Cross-Sectional Prepared Organic Light Emitting Devices

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Light emitting devices (LEDs) based on conjugated organic molecules (oLEDs) or polymers (pLEDS) have been a focus of academic and industrial research for the past decade. The devices usually consist of several layers in a sandwich type structure with layer thicknesses around or below a hundred nanometers. A typical device consists of a transparent electrode (e.g. indium doped tin-oxid, ITO) followed by one or more organic layers and a cathode of a low work-function metal or metal alloy (e.g. Ca, Ba, Mg:Ag or Li:Al). Device properties such as injection of charge carriers from the electrodes, charge carrier transport within the device, and the localization of the recombination zone are often optimized by introducing additional layers and are to a great extend determined by the interfaces between these layers (organic/metal, organic/organic).

In the last years several attempts have been made to prepare cross-sectional samples of organic LEDs for transmission electron microscopy (TEM) investigations. However, differences in hardness of the organic and inorganic compounds turned out to be a major obstacle for common preparation methods such as ion milling and ultramicrotomy. Ion milling preferably thinned and destroyed the relatively soft organic layers while ultramicrotomy introduced artifacts into the electrode layers of harder inorganic materials. A third method using a focused ion beam (FIB) microscope [1] now allowed us to overcome these problems [2].

We prepared two cross-sectional samples of different organic LEDs consisting of the layers ITO/para-sexiphenyl/Al and ITO/PEDOT:PSS/alkoxy-substituted poly(para-phenylene)/Ca/ Al/Au, respectively (FIG 1). The so called 'lift-out' technique using a FIB was applied and the samples were subsequently investigated with a Philips CM20/STEM microscope. Investigations showed that this preparation method is very successful and allows for a lot of detailed studies on layer morphology not available before. Though preparation artifacts due to gallium implantation from the ion beam can not be completely neglected, the quality of the specimen allows accurately measuring layer thicknesses and characterizing interfaces. Furthermore, analytical methods using electron energy loss spectroscopy (EELS) or energy filtered TEM (FIG 2) can be applied to gain detailed, spatially resolved knowledge on the chemical composition of devices e.g. layers and interfaces.
To the best of our knowledge, this is the first report on cross-sectional TEM images of organic LEDs. The preparation method, however, has already been successfully applied to organic solar cell devices [3,4].

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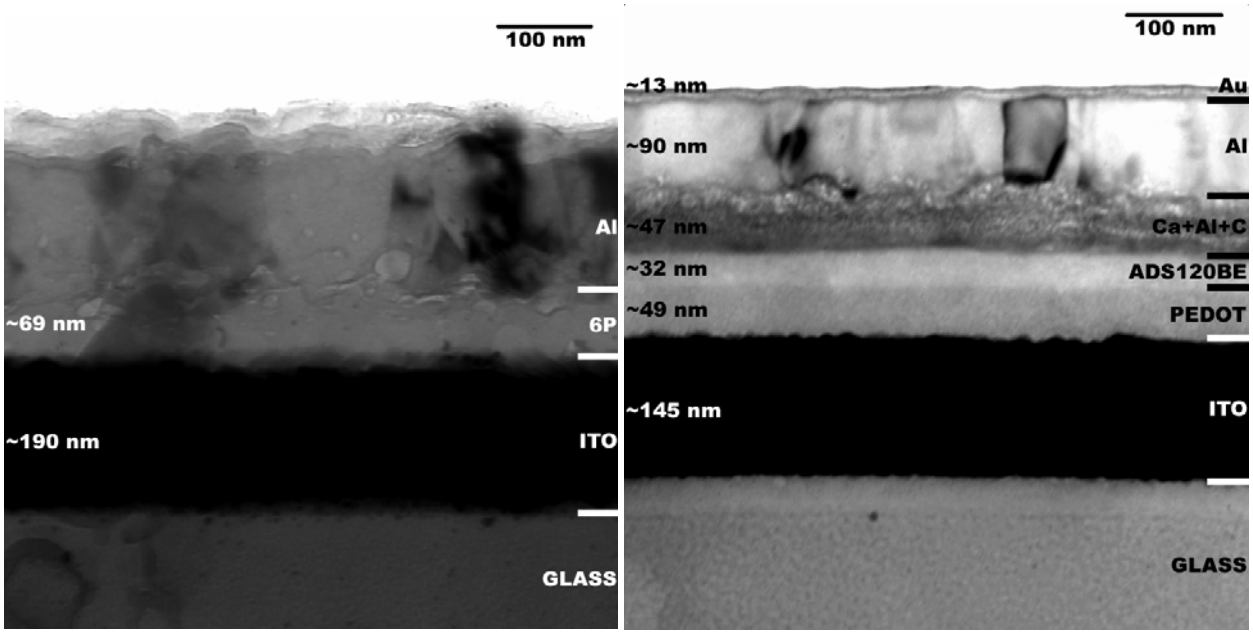

Fig.1 TEM bright field images of two different cross-sectional prepared organic LED devices.

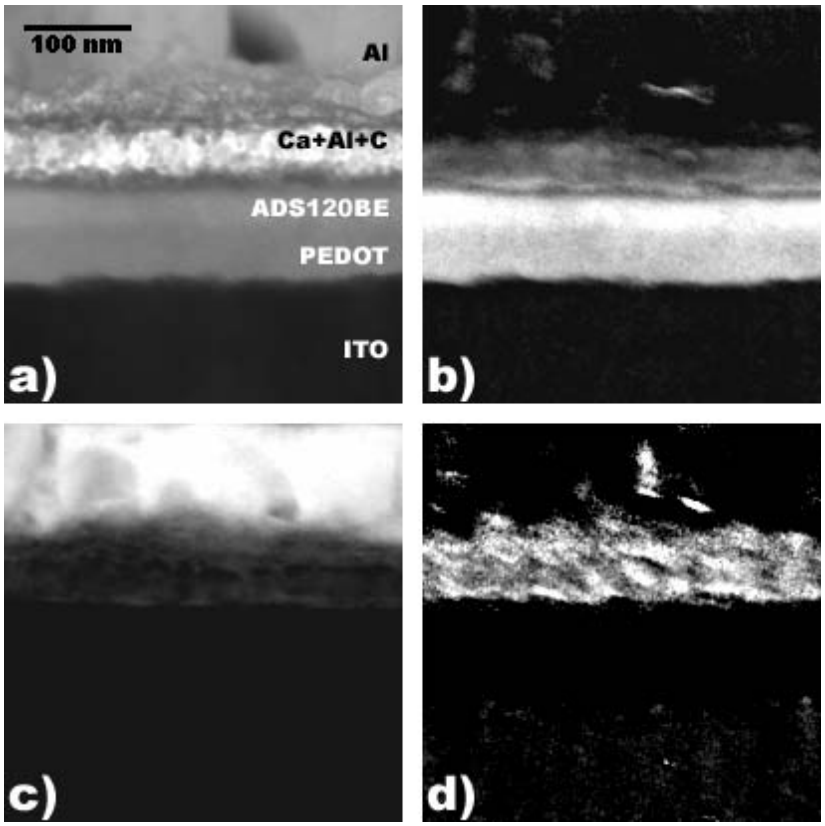

Fig.2 a) TEM bright field image of a cross-sectional prepared LED device; b-d) elemental maps showing carbon, aluminum and calcium distribution of the same specimen position, respectively.

Advantages of a Monochromator for Bandgap Measurements using Electron Energy-loss Spectroscopy

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The bandgap is one of the principal properties of electronic materials. Electron energy-loss spectroscopy (EELS) in transmission electron microscopy (TEM) gives the information on the direct and indirect transitions with high spatial resolution [1]. However, a bandgap measurement requires high energy resolution, and therefore most measurements had to be performed with cold field emission guns whose energy spread (0.35 eV) is smaller than that of Schottky emitters (0.5eV) and thermionic electron sources (~1 eV). Recently, a monochromator for a TEM has been developed for reducing the energy spread of the electrons to 0.1-0.2 eV [2]. Several groups have demonstrated the efficiency of the monochromated TEM for core-loss spectroscopy and valence-band spectroscopy [3,4].

In order to evaluate the use of a monochromated TEM for bandgap measurements the zero-loss peaks of a monochromator and a cold field emission gun are compared in this work [5]. A Schottky emission TEM (FEI TECNAI F20) equipped with a monochromator and a high resolution imaging filter (Gatan) was used to record the zero-loss peak in the EELS spectrum at 200 kV. The total energy spread of the system can be as low as 0.17 eV. For comparison, we also used a conventional 300 kV microscope (Hitachi HF-3000) equipped with a cold field emission source and a Gatan imaging filter 2002. The energy-spread shows a narrow energy spread of 0.27 FWHM (fig.1) which is nearly the best performance of cold field emission systems. In the present experiment the energy spread of the monochromated system is 0.23 eV. Both zero-loss peaks are similar on the left side, but are significantly different on the right side. This asymmetrical intensity distribution of the cold field emission system is caused by wave-mechanical tunneling which is unavoidable in field emission and covers the spectral region most relevant for bandgap measurements.

Therfore we can conclude bandgap measurements the intensity at the bandgap energy is more important than the full-width at half maximum of the zero-loss peak and a monochromated TEM is preferable to other TEMs. To demonstrate this, we have measured the bandgap of silicon (1.1 eV) with the monochromated TEM which can be evaluated from the onset of the EELS spectrum (fig.2).

Our results suggest that the HR-EELS approach will be essential to study the detailed electronic structure of nanoscale materials as a function of momentum transfer and energy loss directly in the small areas of interest.

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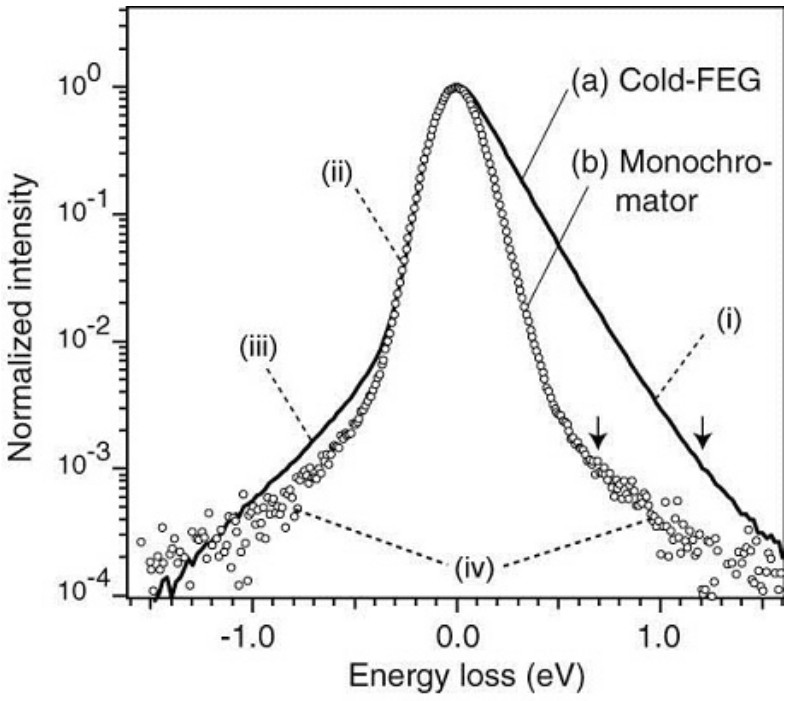

Fig.1 Zero-loss peaks recorded with a (a) cold field-emission gun (CFEG) and a (b) monochromated TEM. Normalized intensities are shown on a logarithmic scale.

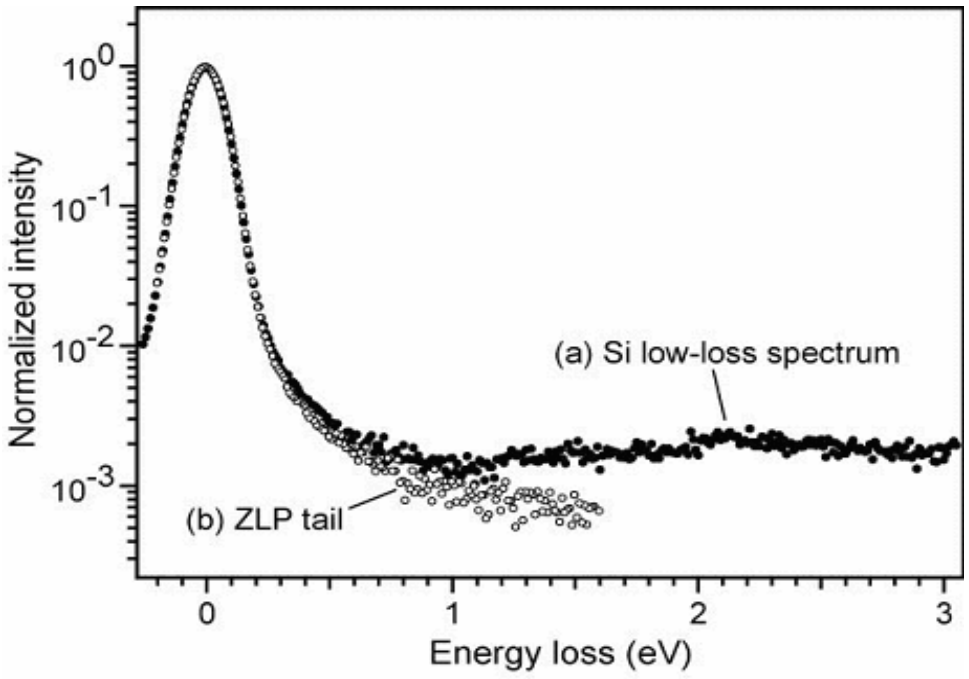

Fig.2a Low-loss spectrum of Si. The 512 EELS spectra obtained with an exposure time of 1.4 ms were summed after drift correction. Open circles (b) are the ZLP estimated from the tail of the negative energy-loss side. The onset is roughly estimated to be 1.1 eV.

Worldwide Database of Free Scripting Resources in Digital Micrograph™

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Gatan Inc's Digital Micrograph™ (DM) software is a worldwide used program for transmission electron microscopy (TEM) imaging and electron energy-loss spectroscopy (EELS). It is optimized for mathematical image processing and also directly communicates with parts of the electron microscope hardware to enable data acquisition within the software. Although a lot of dedicated packages for both data acquisition (e.g. energy filtered image series, image montage for larger fields of view, etc.) and data analysis (e.g. elemental mapping, EELS quantification, particle analysis etc.) exist and can be bought separately, no software can completely fulfill the high and often highly specialized demands of modern research. Especially, developing research fields are in permanent need of customizable software which no commercial product can hope to fully satisfy. Therefore, DM offers an integrated scripting language which allows computer literate users to create their own application tools within the framework of the software, offering optimized routines for image or spectral data processing as well as to some extend control over the microscope hardware for customized acquisition routines.

The DM scripting language is similar to C++ and gives the user access to most of the command and control functionality used by DM itself. The scripting language is relatively easy to use and simple scripts can be written in minutes. The main drawback of the scripting language is the lack of a complete and comprehensive documentation. A somewhat uncompleted beginner's guide can be found at the company's homepage [1] and later versions of the software include a help file with some topics on scripting as well as an incomplete listing of most available commands. To some extent, help can be found within the community of fellow script programmers loosely organized in a worldwide email mailing list [2], but except of these resources the user is left on his own. Especially the lack of comprehensive scripting examples makes understanding of the language a time consuming task.

To improve the situation and encourage more microscopists to take advantage of the scripting capabilities, we created a web-based archive for DM scripts, which is freely accessible via the internet [3]. The database is split into a command example script section and a freeware scripts section. In addition, there is a section with information on type conventions and parameters for certain commands. All scripts are sorted both alphabetically and grouped by an application field. A simple search routine allows to quickly find use-ready scripts for special tasks and each script entry holds contact information of the responsible author as well as a short description of the script. The script source code can be directly copied from the homepage into DM by the clipboard or, alternatively, downloaded as a script file. Whenever a script belongs into a set of scripts working together, the script information is cross-linked for easy navigation. To allow easy uploading of new scripts, a simple online submission form was created.

Since the introduction of the scripting database on 26th November 2002 over 175 scripts were added to the archive and numerous users all over the world have downloaded, used and learnt from the scripts. To further foster an increased awareness and interest in DM scripting, we recently published a paper on this topic [4].

References

[1] Gatan Inc.'s Digital Micrograph™ Scripting Resources: http://www.gatan.com/~software/

[2] Digital Micrograph Scripting Users Group (DMSUG): http://www.public.asu.edu/~perkes/DMSUG.html

[3] Digital Micrograph Script Database: http://www.felmi-zfe.at/dm_scripts/

[4] D. R. G. Mitchell and B. Schaffer, Ultramicroscopy 2005, accepted for publication

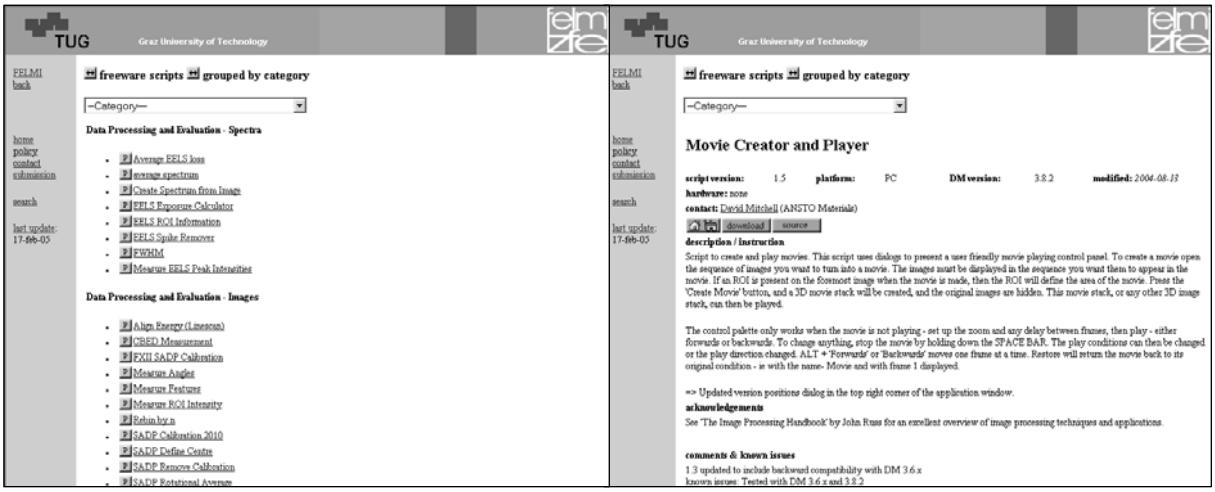

Fig1 The interface of the DM scripting database. Left: Listing of freeware scripts grouped by application field. Right: Database entry of a script.

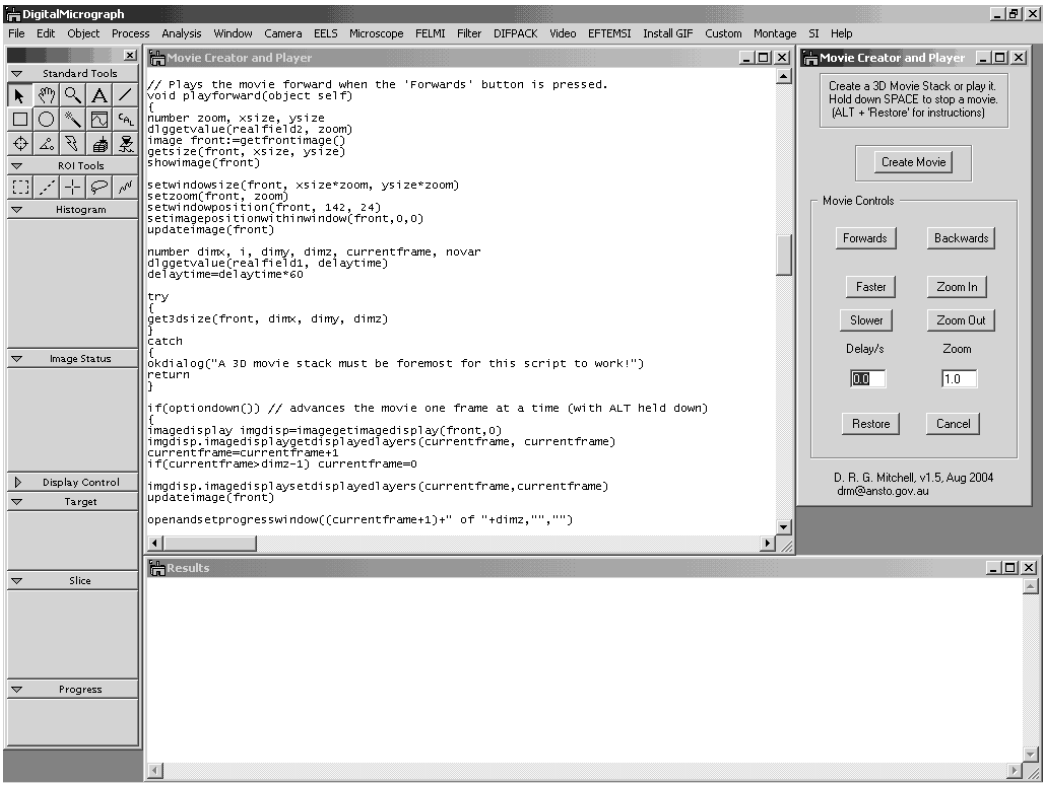

Fig.2 Example script 'Movie Creator and Player' in DM.

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