# Performance Report 2009/2010/2011

Institute for Electron Microscopy and Nanoanalysis Graz University of Technology

Graz Centre for Electron Microscopy ACR Austrian Cooperative Research







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#### Life in the Institute

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#### Statement from the Rector of Graz University of Technology

I have great pleasure in presenting this report from the Institute for Electron Microscopy and Nanoanalysis of Graz University of Technology. As you will see from the following pages, successful operation of this facility has both a national as well as an international impact.

The installation of the first electron microscope in Styria more than 60 years ago followed a decision taken by the university board in 1949 and was a key element of the former Technische Hochschule Graz. From the very beginning, electron microscopy became an invaluable tool both for university research and industrial collaborations. In these early days, the electron microscopy group headed by Dr. Fritz Grasenick paved the way to the Institute for Electron Microscopy and Nanoanalysis (FELMI), which is now a member of the Faculty of Technical Mathematics and Technical Physics. Today, the Institute significantly contributes to several fields of expertise at Graz University of Technology, especially to "Advanced Materials Science" and is now also a member institute of the joint NAWI faculty of Graz University of Technology and the University of Graz.

Over the past decade, methods for developing new materials and examining their detailed nature have become more subtle, sensitive and precise. Nowadays, electron microscopes can identify the location of individual atoms in materials and focused ion beams can create structures with dimensions of less than 10 nanometers. These powerful capabilities are essential in the era of nanosciences and nanotechnology. However, these achievements come at a price: today's sophisticated microscopes have become so expensive and complex that individual researchers cannot own or adequately operate or maintain them. It is therefore clear that such resources must be concentrated and managed in special institutions (mid-size multi-user facilities). In the case of the Institute this works very well via the close collaboration with the Graz Centre for Electron Microscopy thus creating the basis for cuttingedge instrumentation such as the Austrian Scanning Transmission Electron Microscope.

I hope that the Institute will continue to work in this cooperative way and I also wish great success for the years to come.



Univ.-Prof. Dipl.-Ing. Dr.techn. Dr. h.c. Harald Kainz

#### Statement from the Heads of the Institute

For centuries scientists have used microscopes to explore the previously unseen world of small dimensions. Nowadays, in the nanotechnology era, the science of microscopy essentially contributes to the new discoveries taking place in many scientific and technological areas. In particular, electron microscopy has encountered ground-breaking improvements during the last ten years, having a direct impact on materials and life sciences and technology. As a leading Austrian research institution, the FEL-MI-ZFE actively participates in this endeavour. In this way, we support researchers from university institutes and Austrian companies with our expertise in advanced microscopy in all fields of science and technology.

With the goal to strengthen our unique position, we have identified two aspects to be keys for success: • Bringing together the best people: The success is visible, when looking at our multistranded collaboration network in science and industry, collaborative research projects and the organization of workshops and big conferences such as the Microscopy Conference 2009.

• Providing leading edge instrumentation: With the Austrian Scanning Transmission Electron Microscope (ASTEM), advanced experimental research has made a big leap forward.

This can be justified by the extraordinary manifold activities ranging from the organisation of conferences, to the acquisition of new instrumentation, institute renovation and enhanced teaching duties during this time.

It goes without saying that all these activities represented a big burden for the lab team and we would like to thank them for their support, enthusiasm and dedication.



Ferdinand Hofer



Gerald Kothleitner



Peter Pölt

#### Statement from the Presidents of the Association

We are pleased to present the sixth performance report of the Institute, which also marks the 60th anniversary of electron microscopy in Styria. Several years after the installation of an electron microscope at the former Technische Hochschule Graz, five leading Austrian industrial companies and the Government of Styria established the Association "Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung" in 1959. This was an important step, because electron microscopy immediately proved to be a key tool for the development of natural sciences and technology in Styria. In particular, close collaboration with industry was one of the main success factors forin the years to come.follow. Since then we have seen the Association grow continuously and today it has some 33 members from industry and research sorganizations.

The Graz Centre for Electron Microscopy (ZFE) of the Association has developed into one of the leading research institutions in the field of advanced microscopy in Europe. The ZFE employs a staff of 25 researchers dedicated to the development of microscopy in materials and biological sciences and to its application in around 120 industrial collabora-



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

tions a year – mostly with small and medium -sized companies.

The Association's close cooperation with Graz University of Technology led to the establishment of the Austrian Centre for Electron Microscopy and Nanoanalysis, which has the critical mass for developing leading-edge instrumentation such as the Austrian Scanning Transmission Electron Microscope (ASTEM), which was unveiled in 2011. It was of course no mean feat for the Association to build up this world-class instrument in Graz, but finally we succeeded twith the help of funding provided by the Austrian Research Promotion Agency (FFG), the Styrian Business Promotion Agency (SFG) and the Styrian Government. Now the ASTEM serves as a national resource for electron microscopy at atomic resolution thus supporting the research needs of both industrial companies and university institutes. We would like to take this opportunity to extend our thanks to our members, our industrial customers, our partners at Graz University of Technology and Austrian Cooperative Research (ACR), funding institutions at the regional and federal level and the members of the Advisory Board.



KR Dipl.-Ing. Ulrich Santner Vice-President of the Association

## 60 Years of Electron Microscopy in Graz

1949	The senate of Graz University of Technology (formerly known as "Technische Hochschule Graz") made a trend-setting and far-reaching decision: Following the suggestion of Professor Bernhard Baule, an industrial donation intended as an acknowledgment for organizing the first International Congress on Powder Metallurgy was used to provide the basis for the acquisition of an electron microscope.
1950	A committee consisting of Professors Gustav Jantsch, Gustav F. Hüttig, K.W.F. Kohlrausch and Peter Klaudy agreed to establish an electron microscopy group in the physics department.
1951	Dr. Fritz Grasenick was employed to operate the new microscope and lead the group and later the Institute until 1981.
1951	The first electron microscope was installed, the "Übermikroskop UEM100" engineered by Sie- mens & Halske, Berlin. Opening ceremony with Ernst Ruska (Nobel Prize winner in 1986) Berlin, Bodo v. Borries, Düsseldorf, Werner Glaser, Vienna and Otto Wolf, Berlin.
1959	The association "Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung" was founded under the guidance of Josef Krainer sen., the Governor of Styria. The non-profit research institute "Zentrum für Elektronenmikroskopie Graz" was established to support industry and universities in the field of advanced electron microscopy.
1959	The ZFE becomes a member of the Austrian Cooperative Research (ACR).
1960	The "Forschungszentrum der Hochschulen in der Steiermark" was established at the former Tech- nische Hochschule Graz (Graz University of Technology).
1960- 1970	Development of new specimen preparation methods for electron microscopy, sputtering, evapo- ration, low temperature methods.
1963	Installation of the second transmission electron microscope at the institute.
1965	The institute moves from the main building of the TH Graz to the new building at Steyrergasse 17.
1966- 1974	New electron microscopes were introduced, e.g. scanning electron microscopes (Cambridge, Leitz), transmission electron microscopes (Philips) and emission electron microscopes (Trüb- Täuber and Jena).
1965- 1985	New developments in specimen preparation and materials characterisation, e.g. cryo-preparation methods for SEM, X-ray fluorescence analysis in the SEM and X-ray microscopy in the SEM
1970- 1995	Construction of specimen preparation units with the company Anton Paar, Graz, e.g. cryo-transfer unit, neutral particle sputtering unit, freeze fracturing, plasma cleaning, ion-milling,
1978	Restructuring into a central university facility for electron microscopy named "Forschungsinstitut für Elektronenmikroskopie und Feinstrukturforschung / FELMI" at Graz University of Technology.
1982	Dr. Herwig Horn becomes Head of the Institute until 1988.
1983	Analytical transmission electron microscope (Philips EM420), equipped with a serial electron energy-loss spectrometer (Gatan).
1984- 1995	Developments in the field of analytical electron microscopy (quantification of electron energy- loss spectra).
1989	Installation of the second analytical transmission electron microscope (Philips CM20) with a Noran HPGe X-ray detector.
1989	Habilitation of DI Dr. Ferdinand Hofer.
1989	Ao. UnivProf. Dr. Wolfgang Geymayer becomes the Head of the Institute until 1999.

1990	The research group for polymer microscopy and ultramicrotomy is formed (group leader Elisabeth Ingolic).
1993	A new research group for Raman and infrared microspectrometry is established (group leader Peter Wilhelm).
1993	Introduction of one of the first energy-filtering spectrometers in Europe (Gatan).
1995	Analytical high resolution scanning electron microscope with FEG (Gemini, ZEISS-LEO).
1995- 2010	Contributions to energy-filtering TEM for nanoanalysis of materials, steels, semiconductor devices and biological tissue.
1996	Research in the field of biomass ash aerosols by means of scanning electron microscopy and automatic particle analysis.
1997	The first Raman microscope (Renishaw) arrives in the Institute.
2000	Ao.UnivProf. Dr. Ferdinand Hofer becomes the Head of the Institute.
2000- 2003	The high resolution TEM (FEI Tecnai F20) equipped with a monochromator, a high resolution energy filter (Gatan) and an X-ray spectrometer is introduced.
2002	The first environmental scanning electron micr.oscope in Austria is installed (FEI Quanta 600F).
2002	A new research group for advanced scanning electron microscopy is established (group leader Peter Pölt).
2003	The dual-beam focused ion beam microscope (FEI Nanolab Nova 200F) opens new chances for nanotechnology research in Graz.
2004	New research focus on in-situ electron microscopy and preparation of nanostructures
2004	The "Institut für Elektronenmikroskopie und Feinstrukturforschung / FELMI" is incorporated into the Faculty for Technical Mathematics and Technical Physics.
2004	Habilitation of DI Dr. Werner Grogger and DI Dr. Gerald Kothleitner
2006- 2008	Introduction of a new high resolution scanning electron microscope (Zeiss Ultra 55) with EBSD, EDX and WDX detectors.
2007	Habilitation of DI Dr. Peter Pölt.
2009	Organization of the Microscopy Conference 2009 in Graz with 988 participants and with a trade exhibition with 42 companies.
2010	A new research group for FIB and AFM methods is formed (group leader Harald Plank).
2010	Installation of a confocal Raman microscope.
2011	Installation of ASTEM (Austrian Scanning Transmission Electron Microscope), Cs-probe correc- ted Titan 60-300 (FEI Company Eindhoven) with Quantum energy filter (Gatan) and ChemiSTEM detector (FEI).



The first symposium about electron microscopy in Graz on 29 - 30 June 1951 with prominent speakers and the second electron microscope in Austria "UEM 100".



The Austrian President Dr. Rudolf Kirchschläger and the Governor of Styria Dr. Josef Krainer jun. visit the institute in 1980 (Dr. Herwig Horn, Dr. Josef Krainer jun., DDr. Wilfried Schönauer, Dr. Fritz Grasenick, Dr. Wolfgang Geymayer, from left).

1951-1981 Hofrat DI Dr. Fritz Grasenick



Institute Directors from 1951-2012

1982-1988 Hofrat Dr. Herwig Horn (left) 1989-1999 Ao.Univ.-Prof. DI Dr. Wolfgang Geymayer (right)



2000-Ao.Univ.-Prof. DI Dr. Ferdinand Hofer

#### Presidents of the Verein zur Förderung der Elektronenmikroskopie



1959-1971 Governor of Styria Josef Krainer sen.



1971-1986 Governor of Styria Dr. Friedrich Niederl



1986-1996 Dr. Friedrich Pfohl



1996-Prof. Dipl.-Ing. Dr.-Ing.h.c. Helmut List



Foundation of the Verein zur Förderung der Elektronenmikroskopie on 24 June 1959.

# The Institute

### The Institute

#### The Institute at a Glance

The institute concentrates on interdisciplinary research and teaching in physics, materials science, micro- and nanotechnology and biological sciences.

Since the beginning of electron microscopy in Graz, the institute has been built on two main columns:

- the Institute for Electron Microscopy and Nanoanalysis (FELMI) at the Faculty of Technical Mathematics and Technical Physics of Graz University of Technology.
- the Graz Centre for Electron Microscopy (ZFE), a member of Austrian Cooperative Research (ACR) and held by the association "Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung".

Although the two organizations have their own legal status and budget, both institutes work in close alliance to ensure efficient use of personnel and instrumental resources. The Institute is located in the city of Graz, on the campus of the Graz University of Technology. It is organized in research groups focused on specific aspects of microscopy or important material classes.



The Institute in the building Steyrergasse 17 is located on the 2<sup>nd</sup> and 3<sup>rd</sup> floor.



Organigram of FELMI-ZFE on 31 Dec 2011

#### **Research Focus**

A unique feature of the Institute is its broad field of activities ranging from fundamental to applied research and contractual projects with Austrian and European companies. Major areas of research include the micro- and nanoanalysis of materials, electronic devices and biomaterials.

The Institute brings together

- well-accepted laboratory methods,
- cutting edge instrumentation, and
- advanced research skills.

The primary aim is to study fundamental scientific problems and to transfer the knowledge about advanced microscopy methods into practical collaborations with other university institutes and industry (with a clear focus on small and medium-sized enterprises). However, the spectacular developments of the Institute's research groups also rely on an increasing number of high level research projects supported by European and Austrian funding organisations.

During the past few years we have focused on the following research topics, which are described in detail in the research chapter of this report:

- In-situ electron microscopy and surface analysis
- Nanoanalysis of materials
- Soft-matter characterization
- 3D microscopy
- Nanoprototyping and preparation



Operation areas of the FELMI-ZFE

#### A Unique Position in Austria

The institute is one of the leading microscopy facilities in Europe and has a global profile worldwide. Its unique position is based on the leading-edge instrumentation ranging from advanced light and probe microscopes via specialized scanning electron microscopes to transmission electron microscopy at atomic resolution.

On this basis the Institute is endeavouring to develop new microscopy methods and to improve special preparation methods, especially for the materials and life sciences.

These techniques are used intensively to characterize all kinds of materials, providing efficient answers and solutions to scientific and industrial problems.

#### The Institute in Figures

The development of personnel is increasingly influenced by funded research projects and contractual research for industry.

The Institute's staff has been dedicated to special programmes in recent work to compensate for the decrease in industrial projects in 2009 and is very successful with this strategy.

The number of scientists has increased during this period, a factor which is helping to expand the expertise into new research areas, e.g. cryo-microscopy and atomic resolution microscopy.

Since we are also hosting more master students the space pressure we face remains very tight. The budget development also shows very high expenses for the ASTEM microscope and for the ongoing renovation of the Institute and the Steyrergasse 17 premises.

FELMI-ZFE Staff in	FTE	2009	2010	2011
Professors	TU	3.5	3.0	3.0
Scientists	TU	8.0	7.5	7.5
Scientists	ZFE	5.5	8.0	9.0
Scientific Support	TU	7.5	7.0	6.5
	ZFE	6.5	6.5	6.5
A duala interation	TU			1.0
Administration	ZFE	4.0	3.5	2.5
DhD Ctudanta	TU	4.5	5.0	4.0
PhD Students	ZFE	2.0	1.0	1.5
Apprentices	TU	4.0	3.0	3.0
		45.0	44.5	44.5

Development of FELMI-ZFE staff (in FTE)



Financial situation of the FELMI (2008-2011)

#### **Quality Assurance**





Financial situation of the ZFE Graz (2008-2011)

The institute works under an advanced quality management system according to the rules of EN ISO 9001. The aim is to maintain the outstandingly quality of our work and analysis results and to continously improve our organization & management structures.

Following the successful audits performed by TÜV Austria, the Institute was awarded the EN ISO 9001 certificate, which is valid until May 2012. The certificate covers "Research and teaching in the field of microstructure research and materials characterization by electron microscopy, micro- and nanoanalysis and the development of analysis and preparation methods".

#### Collaborations

The Institute has collaborated with around 39 university institutes and more than 120 companies on average each year – mainly from Austria, but increasingly also from other European countries. It should be mentioned that two main directions have been followed during recent years:

Firstly, collaborations within the ACR group which are developing well, and secondly, the incorporation into important European research networks such as the STREP project "CopPeR", the ENIAC initiative or the forthcoming ESTEEM2 project.

Our activities in technology transfer are also manifested in the 300 visitors from other research groups and companies each year. During the report period 133 master and PhD students from other institutions benefited from the scientific and technical support of the Institute.

#### **Research Expert for Austrian SMEs**

For around sixty years the Institute has supported Austrian companies in the innovation process. We have made a considerable contribution to the competitiveness of the Austrian economy through our numerous projects and short-term activities.

The strengths of the Institute are its research and development competence with close links between the academic world and business, strong and flexible research groups in close contact with SMEs and expert knowledge of the national and international funding landscape.



Scientific collaborations with local universities and research institutes



Scientific network of the institute with ongoing international collaborations

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#### The Institute Staff

#### Head of Institute

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

#### Scientific staff

ALBU Mihaela, Dipl.-Ing. Dr.\* CHERNEV Boril, Mag. Dr.\* FISSLTHALER Evelyn, Dipl.-Ing. Dr.\* GROGGER Werner, Dipl.-Ing. Dr., ao.Univ.-Prof. GSPAN Christian, Dipl.-Ing. Dr.\* HABER Thomas, Dipl.-Ing. Dr.\* INGOLIC Elisabeth, Dr.phil.\* (till March 2012) LETOFSKY-PAPST Ilse, Dipl.-Ing. Dr. MATSKO Nadezda, Dr.\* MITSCHE Stefan, Dipl.-Ing. Dr. PLANK Harald, Dipl.-Ing. Dr. PÖLT Peter, Dipl.-Ing. Dr. Univ.-Doz. RATTENBERGER Johannes, Dipl.-Ing. Dr.\* REICHMANN Angelika, Dipl.-Ing. Dr. \* RIEGLER Katharina, Dipl.-Ing. Dr.\*\* (till Mar. 2011) SEZEN Meltem, Dipl.-Ing. Dr.\* (till Jan. 2011) SCHRÖTTNER Hartmuth, Ing. WAGNER Julian, Dipl.-Ing. Dr.\* WEWERKA Karin, Dipl.-Ing. Dr. WILHELM Peter. Dr.phil. ZANKEL Armin, Dipl.-Ing. Dr. ZECHMANN Harald, Dr. phil.\*\* (till Nov. 2011)

#### PhD students

FLADISCHER Stefanie Dipl.-Ing.\*\* GANNER Thomas\* (Mar. 2011 till Mar. 2012) HAAS Wernfried, Dipl.-Ing.\*\* (till Oct. 2012) MEINGAST Arno, Dipl.-Ing.\*\* REINGRUBER Herbert, Dipl.-Ing.\*\* SCHMIDT Franz, Dipl.-Ing.\*\* SCHMIED Roland, Dipl.-Ing.\* UUSIMÄKI Toni, MSc.\*

#### General staff

BAHR Peter, Microscope operator BIRNSTINGL Gerhard, Mechanic BRANDL Christian, Microscope operator\* BRUNEGGER Margit, Chem. lab assistant\* DIENSTLEDER Martina, Chem. lab assistant \*

FREUND Angela, Cleaner\* GOGER Sabine, Secretariat\* MAYRHOFER Claudia, Ing., Chem. lab assistant\* MERTSCHNIGG Sabrina, Microsc. operator\* MITTERBACHER Sabine, Accounting (since Jun. 2011) PALLER Manuel. Chem. lab assistant\* RAUCH Sebastian, Chem. lab assistant ROßMANN Anita, Chem. lab assistant\* (maternity leave since April 2011) SCHMOLTNER Jan-Wolfgang, Design & image lab apprentice SCHREINER Daniel, Chem. lab apprentice SIMIC Sanja, Microscope operator\* SITTSAM Markus, Mechanic\*\* STREUSSNIG Fatima, Secretariat\* (till Feb. 2011) STÜRZENBECHER Ulrike, Mag., Secretariat\* TÖGLHOFER Julia, Chem. lab apprentice WALLNER Margit, Design & image lab WEER Birgit Mag.\*\*, Secretariat (till Apr. 2011) WINDISCH Gerhard, Design, PC & LAN Admin. (till June 2012) WINKLER Silke, Secretariat (since Nov. 2011) \* ZFE staff, \*\* staff supported by projects

#### **Guest Scientists**

- Dr. Vicky KEAST, School of Mathematical and Physical Sciences, University of Newcastle, Australia
- Prof. Dr. Velimir RADMILOVIC, University of Belgrade, Serbia and National Center for Electron Microscopy, Lawrence Berkeley Laboratory, Berkeley, USA
- Prof. Dr. W. Mark RAINFORTH, Department of Materials Science, University of Sheffield, U.K.
- MSc. Jonathan WINTERSTEIN, School of Engineering, University of Connecticut, Storrs, USA
- Dr. Matthew WEYLAND, Centre for Electron Microscopy, Monash University, Victoria, Australia
- Jenny ANGSERYD, Applied Physics, Chalmers University of Technology, Gothenburg, Sweden

#### Scientific staff



Ferdinand Hofer



Christian Gspan



Stefan Mitsche



Gerald Kothleitner

Thomas Haber

Harald Plank

FELMI-ZFE



Mihaela Albu











Boril Chernev



Ilse Letofsky-Papst







Evelin Fisslthaler



Nadejda Matsko



Johannes Rattenberger Angelika Reichmann





19

Werner Grogger



Meltem Sezen



Katharina Riegler



Hartmuth Schröttner

PhD students

# Julian Wagner











Franz Schmidt



Stefanie Fladischer Thomas Ganner



Wernfried Haas



Arno Meingast







Roland Schmied

#### **General staff**



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Claudia Mayrhofer



Jan-Wolfgang Schmoltner



Julia Töglhofer





Sabrina Mertschnigg



Daniel Schreiner



Margit Wallner





Sabine Mitterbacher



Sanja Simic



Birgit Weer



Martina Dienstleder



Manuel Paller





Gerhard Windisch



Angela Freund



Sebastian Rauch



Fatima Streussnig



Silke Winkler







#### Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung

The industrial associates' organisation was established in 1959 to support the institute and to strengthen the interaction between industrial and academic scientists.

On the one hand the "Verein" supported the institute in terms of improvement of instrumentation thus enabling cutting-edge instrumentation which was always important because of the limited resources of the university. On the other hand it allowed maintaining a high-skilled and well trained permanent staff in the Graz Centre for Electron Microscopy (ZFE).

The program is designed to provide industry with useful results from established and emerging new microscopy techniques and to keep the in-house specialists in industry in touch with the latest developments in the field.

Policies and procedures of the non-profit organisation are established by a steering committee consisting of academic and industrial scientists.

Since 1995 the Verein is headed by Professor Dipl.-Ing. Helmut LIST (AVL Graz) and presently the Verein has 33 members mainly from Austria. Since the general business meeting in 2008 the administrative body for the next six years is given as follows:

#### **Presidential Committee:**

President: Prof. Dipl.-Ing. Dr-Ing.h.c. Helmut LIST 1. Vice president: KR Dipl.-Ing. Ulrich SANTNER 2. Vice president: Mag. Christian KNILL

#### Managing Committee:

Head: KR Dipl.-Ing. Ulrich SANTNER Financial referee: DDr. Wilfried SCHÖNAUER Representative of the Styrian Universities: O.Univ.-Prof. Dipl.-Ing. Dr. Franz STELZER

Head of ZFE:

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

#### Accounting Controller:

1. Controller: Dr. Hermann PUCHER

2. Controller: Mag. Petra SCHACHNER

#### Advisory Board:

Univ.-Prof. DI Dr. Wolfgang BAUER, Graz University of Technology

Univ.-Prof. DI Dr. Helmut CLEMENS, University of Leoben

DI Dr. Markus GAHLEITNER, Borealis AG, Linz

Univ.-Prof. Dr. Georg HOINKES, University of Graz DI Dr. Armin HOLZNER, Semperit Technische Produkte, Wimpassing

Univ.-Prof. Mag. Dr. Wolfgang KERN, University of Leoben

Univ.-Prof. DI Dr. Johannes KHINAST, Graz University of Technology

Ing. Bernd KRAUS, Gatan GmbH

Univ.-Prof. Mag. Dr. Joachim KRENN, Graz University of Technology

Univ.-Doz. Dr. Gerd LEITINGER, Medical University Graz

Dipl.-Ing. Hubert LENGER, Böhler Edelstahl GmbH & Co KG

Univ.-Prof. DI Dr. Emil LIST, Nanotec Center, Weiz DI Dr. Harald OKORN-SCHMIDT, LAM-Research AG, Villach

Univ.-Prof. DI Dr. Wolfgang PRIBYL, Joanneum Research, Graz

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

Univ.-Prof. DI Dr. Klaus REICHMANN, Graz University of Technology

DI Dr. Stefan SCHERER, Alicona Imaging GmbH, Grambach

Univ.-Prof. DI Dr. Christof SOMMITSCH, Graz University of Technology

Univ.-Prof. DI Dr. Walter STEINER, Graz University of Technology

Dr. Birgit STRIMITZER-RIEDLER, Amt der Steiermärkischen Landesregierung, Graz

Univ.-Prof. Dr.phil. Günther ZELLNIG, University of Graz

#### Members of the "Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung"

- Honorary Members: Univ.-Prof. DI Dr.techn. Wolfgang GEYMAYER
- Company Members:







Development of company members of the Association

#### Austrian Cooperative Research (ACR)

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

The strengths of ACR-members:

- Applied research, development and innovation
- Technology and knowledge transfer
- Project management for R&D projects
- Efficient funding advice
- High-grade testing and measuring accredited at EU level
- Certification and audits
- Training courses, special seminars, company events

Currently, ACR has 17 full members. In 2011, ACR had a total of 503 full-time equivalent employees and produced a turnover of 52.9 million EUR of which 77% was produced with SMEs.

The ZFE Graz cooperates with the following ACR institutes:

Austrian Foundry Institute (ÖGI), Leoben

Austrian Research Institute for Chemistry and Technology (OFI), Vienna

Research Institute of the Austrian Cement Associ ation (VÖZFI), Vienna

Austrian Institute of Material Science and Welding Technology (SZA), Vienna

Holzforschung Austria (HFA), Vienna

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



ACR - Austrian Cooperative Researc...



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#### Photo Release -- FEI Extends ChemiSTEM Technology to Enable Atomic-Level Spectroscopy

Elemental Mapping at the Atomic-Level is Now Possible Across the Periodic Table Using X-Ray Spectroscopy Techniques

HILSBORO, Ore., May 3, 2011 (GLOBE NEWSWRE) – EE Company (Nasdaq:FEIC), a leading instrumentation company providing systems for research and industry, today announced that it is extending its OhemiSTEM<sup>™</sup> Technology to enable, fo the first time, atomic-level energy dispersive X-ray (EDN) spectroscopy across the periodic table. The combination of increased current in an atomic-sized probe by Cs-correction and the increase in X-ray detection sensitivity and beam ournent of the ChemiSTEM Technology allows results to be obtained within minutes.

A photo accompanying this release is available at http://www.globenewswire.com/newsroom/prs/?pkgid=9611

6

Wirtschaftspanorama

Steirisches

The powerful containation of the groundbreaking ChernSTEM Technology and an aberration corrector offers unique capabilities for material science; aid Professor Ferdinand Hörder of Graz University of Technology, valutia, "One of the most important applications for the new technology will be element-specific marging at atomic resolution. We will apply the technology study interfaces in semiconductors, solar cell materials, LEDs and cerami materials with previously unhown detection semicitivity and accuracy.





W STEIERMARK W Eine der größten mitteleuropäischen Investitionen in Wissenschaft und Forschung an TU Graz:



Grazer TU täglich die mikroskopischen Sphären der Nanowelten. Mit einer weltweit einzigartigen "Super-Lupe" können sie ab Juni in noch weitere Dimensionen vordringen. Das hilft nicht nur dem Forschungs standort Steiermark, sondern auch unserer Wirt schaft: Neue Medikamente können entwickelt, Auto teile und Computerchips perfektioniert werden. Wirt-

So können Materialien rer Ebene in völlig neuer im Nanobereich mehr als eine Million (!) mal größer den; sogar mittels Fernbe-dargestellt und auf atoma-dienung. Ein besonders



ACR bündelt Kompetenz in der Materialcharakterisierung

AUSTRIAN COOPERATIVE RESEARCH KOOPERATION MIT KOMPETENZ

Mit der Plattform Materialcharakterisierung liefert

na sam numora manaraanaaranenserung tietert das ACR-Netzwerk Unternehmen ganzheitliche Lösungen für materialispezifische Fragestellungen – von Nanoanalytik, makroskopischer Werkstoffprüfung, über zerstörungsfreie, dreidimensionale Gefügecharakterisierung bis hin zu chemischer Analytik.

#### Die Presse.com > Wissenschaft

Politik Wirtschaft MeinGeld Panorama Kultur Tech Sport Leben Bildung Wissenschaft Gesund × zurück

#### Wie man einzelne Atome sehen kann

In Graz wurde feierlich ein neues Hochleistungsmikroskop eingeweiht. "Astem" hat stolze vier Millionen Euro gekostet



Das Institut für Elektronenmikroskopie der TU Graz hat ein neues Flaggschiff. Es hört auf den Namen Astem, hat stolze vier Millionen Euro gekostet, und wird Grundlagenforschern sowie Industrie neue Einsichten in die Eigenschaften von Materialien gewähren. Eine Substanz Atom für Atom auflösen zu können, und noch dazu genau bestimmen zu können empfindlicher Röntgende-tektor stellt zusätzlich fest, aus welchen Elementen sich eine Probe zusammensetzt. Finanziert wurde das vier Millionen Euro teure Mess-instrument "ASTEM" auch durch Förderungen des Landes Steiermark. "Mit ASTEM verfügen wir über ein Gerät, das derzeit welt-weit einzigartig ist", ist In-stitutsleiter Ferdinand Ho-fer – er arbeitet mit einem fer – er arbeitet mit einem Team aus 57 Mitarbeitern zusammen – stolz. Der Zuzusammen – stolz. Der Zu-sammenbau am Grazer Zen-trum für Elektronenmikros-kopie wird zwei Wochen in Anspruch nehmen; die ab-schließende Gerätkekonfigu-ration vermutlich noch eini-ge Monate.



lionen Euro

Pharma-, Halbleiter- und Autoindustrie profitieren

Autoindustrie profilieren Vor allem die steirische Wirtschaft (Pharma-, Halb-leiter-, Fahrzeugbereich) verlangt nach dem "Super-Mikroskop": So sollen künf-tig Werkstoffe verbessert, Medikamente entwickelt, Computerchips verkleinert und elektronische Bauteile perfektioniert werden. "Auch so genannte Bioma-terialien können so genau untersucht werden wie nie zuvor", freut sich der

Das neue Mikroskop schafft eine millionenfache Vergrößerung

(Noch-)TU-Rektor Hans Sünkel über die Mega-In-vestition – übrigens eine der größten in wissen-schaftlichen Infrastruktur in Mitteleuropa!

#### Laboratory Facilities

#### Scanning electron microscopes (SEM)

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

**Analytical high resolution SEM:** LEO Gemini DSM 986; 0.1-30 kV, field emission gun, with EDX detector Noran Voyager 3105A, with TSL EBSD detector and cryogenic specimen transfer system (developed at FELMI-ZFE).

**Environmental Scanning Electron Microscope (ESEM):** FEI Quanta 600 equipped with Noran Vantage EDX system, heating stage (up to 1500°C), Peltier cooling-stage, tensile testing stage and 3ViewTM ultramicrotome (Gatan).

**Environmental SEM (ESEM):** FEI Quanta 200; W-cathode with EDAX Saphire EDX-system.

#### Focused ion beam instrument (FIB)

**FEI NovaTM 200 NanoLab:** DualBeamTM FIB/ SEM; equipped with OmniprobeTM manipulator, various gas injection systems (Pt deposition,  $I_2$  enhanced metal etch, XeF<sub>2</sub> insulator enhanced etch, TEOS-oxide deposition), XFlash 4010 X-ray spectrometer (Bruker AXS), cryo specimen holder and direct ion detector (CDEM).

#### Transmission electron microscopes (TEM)

Austrian Scanning Transmission Electron Microscope (ASTEM): FEI Titan 60-300 cubed; quipped with a Cs probe corrector, a high brightness gun (X-FEG), a Wien filter monochromator, with ultrafast STEM, an x-ray detector (ChemiSTEM), a high resolution energy-filter (GIF Quantum, Gatan), a Lorentz lens and remote control.

**Analytical high resolution TEM:** FEI TECNAI F20; 80-200 kV, field emission gun, supertwin objective lens, STEM (0.2 nm probe) with HAADF detector, with Wien filter monochromator, with EDX Si(Li) light element detector (EDAX) and high resolution Gatan imaging filter (HR-GIF, Gatan) with 2kx2k CCD camera (Gatan), magnetic field compensation system.

**Analytical TEM:** FEI TECNAI 20, 200 kV,  $LaB_{6}$ cathode, supertwin objective lens, STEM with bright and dark field detectors, with EDX Si(Li) light element detector (EDAX), Lorentz lens and 1kx1k CCD camera.

**Analytical TEM:** Philips CM20; 200 kV, LaB6-cathode, twin lens, STEM with SE detector and Gatan BF/DF detector, EDX detector (HPGe, Noran) and Gatan Imaging Filter (including a 1kx1k CCD camera).

**Analytical TEM:** FEI Tecnai 12; 80-120 kV, LaB6cathode, twin lens, and EDX Si(Li) detector with ultrathin window (EDAX), 1kx1k CCD camera, lowdose CCD camera (Gatan).

**TEM specimen holders:** Philips double tilt holder, Gatan cryo-transfer and cooling holder, Gatan double tilt cooling holder for analytical work, low background holders, rotation and tomography holders.



The Austrian Scanning Transmission Electron Microscope (ASTEM)

#### Atomic force microscopes (AFM)

Atomic force microscope: VEECO Dimension 3100; equipped with EFM and KPFM modes in a glove-box "Unilab MBraun 20"

Atomic force microscope: SNOTRA SPM; with cryo ultramicrotome, cryo SPM head CERTUS cryo and drive digital controller EG-3000

#### Light microscopes

**FT-IR microscope Bruker:** Bruker Equinox 55 spectrometer with Hyperion 3000 microscope, ATR objective (Ge crystal), grazing angle objective, MI-Racle single reflection horizontal ATR unit (diamond and Ge crystal), Sadtler "KnowltAll" spectral libraries and search software.

**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 and hot-cold stage.

**Raman microscope:** HORIBA LabRAM HR 800 confocal Raman microscope (pinhole), spectrograph with 800 mm focal length spectral resolution: 0,35 cm-1 (with 633 nm excitation and 1800 gr/mm grating); red laser: 633 nm, 17 mW; blue laser: 473 nm, 20 mW; BX41 microscope (10X, 50X, 100X objectives)

Motorised XYZ stage;1024x256 pixel multichannel; CCD detector (spectral range: 200-1050 nm); Lab-SPEC software and module SWIFTTM for fast Raman imaging; DuoScan<sup>™</sup> imaging system.

**Light microscope:** Zeiss Axioplan for transmitted and reflected light with bright field, dark field, polarization, interference contrast (DIC), phase contrast and a Polaroid DMC camera.

Advanced light microscope: Alicona Infinite Focus microscope with automated xy- and Z-lifting table and CMOS-camera.

#### Preparation equipment

#### **Microtomy laboratory:**

Universal Zentrifuge (Hettich) Knifemaker LKB7800 (LKB) Taper apparatus EM TRIM (Leica) Ultramicrotome OMU3 (Reichert-Leica) Ultramicrotome Ultracut UCT with EM FCS for low temperature sectioning (Leica) Microtome Supercut 2050 for light microscopy Ultramicrotome Ultracut E (Reichert-Leica) Sawing microtome 1600 (Leitz)

#### Sample preparation laboratory > cutting, polishing, embedding:

Diamond wire saw (Well)

Diamond blade saw ISOMET 1000 (Buehler) Polisher MiniMet (Buehler) Ultrasonic disc cutter (Gatan) Dimple grinder (Gatan) Manual Tripod polisher (Southbay Technology, Allied High Tech) Semiautomatic Wedge Polishing System Multi-Prep (Allied High Tech) Mounting Press SimpliMet 1000 (Buehler) Vibratory Polisher VibroMet 2 (Buehler) Automatic Polisher Tegramin 25 (Struers) Vacuum-Mounting-System CitoVac (Struers) Polisher LaboPol (Struers)



Manual Tripod polisher

Sample preparation laboratory > thin films:

Evaporation and sputtering apparatus PSC (ZFE) Sputtering apparatus GEA (ZFE) Thin film preparation system EPA 101 Spin Coater (Laurell)

Sample preparation laboratory > ion milling:

lon milling and polishing system PIPS with digital zoom camera (Gatan)

Low angle ion milling apparatus (ZFE), equipped with low energy ion guns (Technoorg Linda) Precision ion beam cross-sectioning system Ilion with digital zoom camera and cold stage controller (Gatan)

Sample preparation laboratory > misc:

Electrolytic thinning device TenuPol 5 (Struers) Plasma cleaner Model 1020 (Fischione) Cryo-preparation system EPA 101 with quadrupole mass spectrometer QMG311 (ZFE) Critical point dryer CePeDe8 (ZFE)

#### Computer system

Central server based on HP DL30 units with storage area network (4 TByte) consisting of a mail server, a file server, a backup server and a 5 TByte tape library

1 GBit network with 4 Cisco switches 85 desktop computers and 4 network printers Canon color laser printer CL30 and several high quality printers



The new server room with the new uninterruptible power supply system.

# Research

### Research

#### **Microscopy Gives New Insight**

Advanced microscopy is making an essential contribution to understanding the structure, property and function of all kinds of solids, materials and biological samples (alloys, steels, metals, ceramics, composites, minerals, polymers, nanoparticles, clusters and biological tissue). The institute's main research activities are therefore devoted to developing new microscopic characterization methods and their practical application in the "real-world".

Additionally, several methodical developments were partially driven by our industry and academic partners resulting from their increasing demands for advanced materials characterization.

New research funds were allocated by the FFG, the FWF, the FP7 of the EU, the Austrian Nanoinitiative and the Provincial Government of Styria (pp. 38 - 65). In the following pages we take a glimpse at the Institute's research projects. Each article highlights a different aspect of materials microscopy.



3D reconstruction of a polymer membrane recorded in the FEI ESEM Qunata 600 by serial sectioning and BSE imaging.

#### Multiscale and 3D microscopy

Generally, we are familiar with many material-related phenomena whose essential role often extends over many scales in space (and also time). Living matter or advanced materials and devices, products and processes are influenced by underlying phenomena, which span a broad and hierarchically organized sequence of length and time scales. Consequently, we have focused our activities in three-dimensional (3D) structure elucidation using microscopic methods and in-situ microscopy for time resolved studies.

Recent advances in microscopy and software techniques enable a new way to look at materials, allowing us to visualise nano- and microstructures in three-dimensional space. This allows us to directly see the 3D relationships between different phases, as well being able to determine the size of nanoparticles or pores.

Starting already in 2004, the Institute pioneered several new 3D microscopy techniques. Four main directions are presently followed:

#### 3D elemental mapping with EDX-spectrometry in the FIB microscope

The group of Hartmuth Schröttner developed the method of 3D elemental mapping by combining a focused ion-beam instrument (FIB) with energy-dispersive X-ray spectrometry (EDXS). The FIB-EDXS method is especially powerful for inorganic materials, such as for studying secondary phases in steels, alloys, casting materials, hard metals, ceramics and composites (Stotter et al, Int. J. Mat. Res., 2008).

#### 3D imaging by serial sectioning and imaging in the ESEM

The group of Peter Pölt developed serial section ultramicrotomy for materials science applications. The internal structure of specimens can be elucidated by serial sectioning with an ultramicrotome directly installed in the environmental SEM (ESEM) and simultaneous image acquisition with backscattered electrons (Gatan 3VIEW<sup>™</sup> system). This method permits high resolution tomography of soft materials, polymers and biological samples (Zankel, J. Microscopy, 2009). An important breakthrough was achieved with the study of polymer membranes, which is the first example for the application of serial sectioning in ESEM to the quantitative description of microfiltration membranes (Reingruber et al., J. Membrane Sci., 2011).

Recently, Armin Zankel and Hartmuth Schröttner were the first to adapt this new method for 3D elemental mapping in materials, e.g. the distribution of precipitates in aluminium alloys (Zankel et al., Imaging & Microscopy, 2011).



Elemental distributation maps of precipitates in an aluminium alloy recorded in the FEI ESEM Quanta 600 by combining the serial sectioning method and X-ray spectrometry (SSD detector provided by Oxford Instruments).

#### 3D imaging by means of an AFM

Nadejda Matsko has developed the first cryogenic atomic force microscope (AFM) in the world, which is directly mounted in the cryogenic chamber of an ultramicrotome. This new combination allows scanning immediately after sectioning, so that structural changes can be avoided as the whole structure will be stabilised by cold. Direct observation of the block surface structure of the sample by cryo-AFM will provide information about the native structure of the bulk polymer which is not chemically or mechanically modified during sample preparation or observation.

This new method has been developed by Nadejda Matsko in cooperation with Nanoscan Technologies, Moscow, Russia (Efimov, Matsko et al., Soft Matter, 2012).

#### Electron tomography in the TEM

Electron tomography in the TEM is a means by which the 3D structure can be reconstructed from a series of images or projections taken at regular tilt intervals. In the TEM these projections allow the 3D structure to be determined at a resolution of about 1 nanometre with a field of view of hundreds of nanometres making it ideal for the characterisation of nanoscale devices.

Electron tomography was introduced in the Institute by the group of Gerald Kothleitner especially for applications in semiconductor research within the project CopPer and for the study of porous materials (Uusimäki et al., in preparation).



Tomographic reconstruction of iron oxide nanocrystals (red) in a porous material. The reconstruction was made from a tilt series of TEM images obtained at 200kV on the FEI Tecnai F20 over a range of 112°.

#### In-situ microscopy in the ESEM

Due to its broad applicability the environmental SEM is one of the key instruments in the Institute. The ESEM allows the study of practical any specimen under gaseous conditions, unlike conventional SEMs, which operate in high vacuum. Any specimen, wet or dry, insulating or conducting in situ and close to its natural state can be investigated. Consequently, research of the Pölt and Schröttner groups focus on the improvement of the ESEM methodology and to explore new application fields:

#### 1. Methodical improvements of the ESEM

Current instrumental limitations of the ESEM have been identified by the Schröttner group in collaboration with G. Danilatos. Therefore recent research activities concentrate on finding new ways to overcome efficiency problems of commercial ESEMs and to further enhance the applicability of the ESEM in soft materials research (Rattenberger, Scanning, 2009 and Danilatos et al., J. Microsc., 2011).

#### 2. Advanced in-situ experiments

The ESEM was not only used for materials analysis, however also as a kind of micro-reactor. A variety of processes can be controlled, while simultaneously documenting the changes of the specimen with high magnification and great depth of focus on video. in-situ experiments in the ESEM are especially important to study the fracture behaviour of both polymers and textile fibres and the corrosion of steel surfaces at high temperatures (Reichmann, 2008).



The orientation contrast image recorded with backscattered electrons in a SEM reveals the domain structure of a NaNbO<sub>3</sub> ceramics (Reichmann, J. Eur. Ceram. Soc., 2011).

#### Nanoanalysis of materials

The ability of modern TEMs to produce images at near atomic resolution has led to a tremendous progress in materials science. If the TEM is combined with analytical techniques such as electron energyloss spectroscopy (EELS) and energy-filtering TEM or the new SDD detectors, it is now routinely possible to record elemental distribution maps at nanometre resolution.

#### EELS and EFTEM

are particularly useful for studying precipitation or segregation at grain boundaries in materials such as steels, alloys and ceramics. Particular progress was achieved on the quantitative analysis of complex nitrides in steels (Kothleitner group). The studies have been performed in collaboration with the Institute for Materials Science and Welding, TU Graz and Chalmers University of Technology in Sweden (Mitsche et al., Mat. Science & Engineering, 2011 and Angseryd et al., Micron, 2011).

#### X-ray spectrometry in the TEM

Due to the increasing need for quantitative data recorded with the new SDD detectors, the group of Werner Grogger improved the  $\zeta$ -factor method which provides two important advantages: (i) use of pure-element standard samples, rather than multielement standards and (ii) a built-in X-ray absorption correction with simultaneous mass thickness determination. The built-in absorption correction facilitates light-element analysis (Fladischer et al., MCM 2011).



 $\zeta$ -factors for EDX quantification in the TEM, comparison of measured values with different interpolation models.

#### Nanoplasmonics

The TEM is an ideal tool for mapping physical properties of individual nanostructures at the nanometer level. Here, the Institute works on the investigation of surface plasmons of noble metal nanoparticles and nanostructures by means of advanced EFTEM and EELS spectrum imaging techniques. Due to the excellent energy resolution of the monochromated TEM/STEM system (FEI Tecnai F20), which is in the range between 0.15 and 0.20 eV, it is now possible to image the local surface plasmons on Au and Ag nanoparticles and structures. For example, we have been the first to demonstrate that energyfiltered TEM can be efficiently used to image these surface plasmons of chemically prepared Au nanorods (Schaffer et al., Phys. Rev., 2009 and Schaffer et al., Ultramicroscopy, 2010).

Recently, these studies have been extended to artificial nanostructures which have been prepared by electron beam lithography. In this respect, we could firstly identify dark plasmonic breathing modes in silver nanodisks (Schmidt et al., Nano Letters, 2012).



Radially propagating surface plasmons in a silver nanodisc revealed by EELS spectroscopy and spectrum imaging and the simulation by U. Hohenester, Institute of Physics, University of Graz.

#### Microscopy at atomic resolution

The past years have seen many new developments in all forms of microscopy. The most spectacular progress, however, was achieved in transmission electron microscopy, mostly due to the development of aberration correctors for both TEM and STEM imaging. For example, TEMs, which have been equipped with correctors to eliminate the spherical aberrations, now provide an information limit in the range of 70 picometers, thus enabling the imaging of atoms.

In order to follow this dramatic progress the Institute had to introduce a new Cs corrected scanning TEM system. The essential benefit of this new microscope is that a high electron current can be packed into an atom-sized electron probe, which allows signals with weaker cross-sections (EELS & EDXS), to be collected with atomic resolution and sensitivity. As a consequence, we are focusing our research now on the quantitative analysis of atomic sized phenomena in materials such as nanoparticles, interfaces and defects in semiconductors.



STEM-HAADF image of the atomic structure of zinc oxide doped with indium oxide, viewed in [110] direction, recorded with the ASTEM at 300 kV (by Dr. Mimo Radmilovic).

#### Soft matter characterisation

"Soft matter", i.e. polymers, hybrid coatings, biomedical materials and nanocomposites form an important research area nowadays with many new discoveries, developments and industrial applications. Our main research goal in this area is to understand the organisation or (self-) assembly of molecules and nanostructures to control the properties and performance of materials and devices. The challenge in soft matter microscopy is thus not only to image molecular states of the materials but also to analyse their 3D structural arrangement, since both are essential in soft matter science.

#### Electron microscopy of soft materials

Over the years, the institute has developed unique knowledge in studying the morphology of technical polymers, the phase distribution in polymers, distribution of fillers, secondary phases and interfaces in polymers and metal-polymer composites. These investigations are mostly performed by means of transmission electron microscopy (TEM) of specimens, which have been prepared via ultramicrotomy. Increasingly, we also employ scanning electron microscopy (SEM) for the study of polymers and composite materials, in most cases combined with in-situ investigations in the ESEM (Pölt et al., Polymer, 2010).

#### Microscopy of biological tissue

A broad range of biological tissue and medical materials were investigated in collaboration with institutes of the Medical University of Graz, the University of Graz and the University of Ljubljana: Firstly, we worked on the electron microscopical investigation of skin tissue of the Ötzi and Peruvian mummies (Pabst et al., J. Archaelog. Sci., 2009), secondly on special spherite structures in tissue of insects and spiders (Lipovsek et al., Microsc. Res. Techn., 2011) and thirdly silicone was identified for the first time as the key element for the early stages of biocalcification (Matsko et al., J. Struct. Biology, 2011). Additionally, we could establish a close collaboration with the Institute of Plant Sciences of the University of Graz, where we worked on electron microscopy of plant tissue (Zechmann et al., and Zankel et al., Protoplasma, 2011).

The Institute is also collaborating with the Institute for Biotechnology and Biochemical Engineering (BIOTE) of the TU Graz in the field of visualization enzymatic cellulose degradation. While the BIOTE provides the entire enzyme systems as well as biochemical investigations, emphasis of the FELMI is put on real time visualization on the nanoscale via atomic force microscopy (AFM) in liquid environments.



SEM-image of the nanostructure of a spherulite of i-polypropylene after surface etching.



TEM image of tattoed skin area of the Tyrolean Ice Man Ötzi revealing ash particles.
#### Chemical imaging

One key element of the institute's soft matter studies is the long enduring tradition in combining electron microscopy and vibration spectroscopy: Electron microscopy of soft matter is embedded in a sophisticated characterisation environment providing light microscopy with vibration spectroscopy (infrared and Raman) which enables the chemical characterisation of samples or domains as small as 10 µm (infrared) or 1 µm (Raman). The group of Peter Wilhelm applies these methods to analyse polymers (identification, defects, impurities, stress and density), rubbers, paper, inorganic and biological materials. Results are obtained by spectral interpretation (band allocation to functional groups, comparison with reference spectra) or imaging (2D mapping of functional groups, e.g. Chernev et al., Appl. Spectrosc., 2011).



Raman spectrum of a pharmaceutical substance (Ibuprofen). ecitation laser 633 nm; acquisition time 1 s.

#### Cryo-microscopy for soft matter research

Cryo-methods in TEM have been proven to be powerful tools in the characterisation of soft matter nanostructures. By rapid cooling (cryo-fixation) molecular movements are stabilised and the structure is preserved and observed in its natural state. The successful introduction of cryo-TEM was followed by detailed studies of polymeric, pharmaceutical and biological emulsions (Klang, Matsko et al, Micron, 2011).

Additionally, we could establish a new research focus on the microscopical characterization of pharmaceutical materials via the collaboration with the Research Centre for Pharmaceutical Engineering (RCPE) and the Department of Pharmaceutical Technology of the University of Vienna.

#### Soft matter processing

During the last decade focused ion beam (FIB) processing has become a unique tool for the site specific fabrication of ultrathin lamellas suitable for TEM. Due to its versatile functionalities, this method has also attracted increasing attention as a rapid prototyping tool for surface modification and 3D surface structuring with spatial resolutions down to the nanometer range. However, when combined with soft matter, such as polymers or biological materials, morphological instabilities and severe chemical damage are often observed results.

The group of Harald Plank developed a new patterning strategy which allows for increasing morphological stability and decreasing chemical degradation (maintained functionality), Plank et al., ACS Nano, 2012).



Direct comparison of a FIB processed polymer sample, structured via regular (left) and alternative (right) patterning strategies (SEM micrographs with a horizontal image width of  $6 \mu m$ )

# **Funded Research Projects**

#### Austrian Scanning Transmission Electron Microscope – A National Resource

Project leader: Ferdinand Hofer, Werner Grogger, Gerald Kothleitner

Since the first electron microscope was constructed in 1932 by Ernst Ruska and Max Knoll, we have seen a continuous improvement in spatial resolution. First atomic resolution images were claimed in the early 1970s. Since the introduction of aberration corrected electron optics in the past decade these instruments have now been commercially available for about seven years and have been installed in various research institutions throughout Europe.

The project to establish such a leading-edge transmission electron microscope (TEM) in Austria was originally launched by Graz University of Technology, but was later taken over by the ZFE (Fig. 2).

The first project stage was granted by the Austrian Research Promotion Agency (FFG) under the COIN programme in early 2009. After a careful evaluation of several systems, it was decided to install a Titan3 60-300 with a Cs probe corrector (FEI Company, Eindhoven, The Netherlands). This basic instrument was later extended with the help of additional funding from the Styrian Provincial Government and the Styrian Business Promotion Agency (SFG).

Graz University of Technology provided considerable support in the technical preparation of the microscope room (construction, air-conditioning, etc.). The instrument was delivered in two stages with final installation in early summer 2011 (Fig. 1).

The core of this project comprises the build-up of a new scanning TEM (STEM), the only one of its kind in Austria and one of just a few world-wide. The microscope integrates an aberration correcting lens assembly for the illumination system (Cs probe corrector), thus enabling the acquisition of STEM images with million-fold magnification and unsurpassed spatial resolution. In combination with unique analytical accessories, the system will allow for the investigation of materials and electronic devices at an atomic level (Figs. 3-4).

The ASTEM microscope has the following features:

 The ASTEM can be operated from 60 to 300 kV with a special high-brightness electron gun (X-FEG).

- The microscope is equipped with a Cs probe corrector for high resolution STEM imaging (correction of spherical aberrations).
- A new x-ray spectrometer consisting of a four quadrant silicon drift detector (ChemiSTEM) for fast elemental mapping.
- A special energy filter (Quantum filter, Gatan Inc, USA) for high energy resolution EELS spectroscopy and EFTEM mapping.
- Lorentz lens for imaging of magnetic materials
- Tomographic acquisition capabilities.

The ASTEM provides a wide range of operating conditions and can be used for the study of all kinds of materials. Gaining insight down to the atomic level is indispensable for the development of novel materials. This microscope will thus help to answer some of the fundamental questions associated with modern materials research. The microscope is thus expected to become a valuable tool for research and development for the Institute's industrial and university partners. The ASTEM project also aims to provide remote access to the microscope, which means that researchers from collaboration partners will be able to remotely control and use the ASTEM.

The microscope will allow the following scientific challenges to be addressed:

- Direct atomic resolution imaging of materials and devices
- Elemental analysis at atomic resolution (Fig.4)
- Local electronic structure and bonding in inter faces and nanostructures
- Nanotomography of functional materials
- Nanometrology of semiconductor devices (inorganic and organic).

#### Funding organisations:

Austrian Research Promotion Agency (FFG), Vienna, Project No. 821074, COIN-Aufbau

Verein zur Förderung der Elektronenmikroskopie und Feinstrukturforschung, Graz

Styrian Provincial Government, Graz Styrian Business Promotion Agency (SFG), Graz Chamber of Commerce of Styria, Industry Section Federal Ministry of Economy, Family and Youth, Vienna



Fig. 1. Installation of the ASTEM microscope at the building Steyrergasse 17 premises.



Fig.2. Development of the ASTEM project



Fig.3. Improvement of FEG-STEM resolution in HAADF-imaging using a silicon crystal viewed in [110] direction; a) Tecnai F20 without Cs correction, b) Titan3 60-300 with Cs-probe corrector.



Fig.4. a) STEM-HAADF image of GaN showing the Ga atom columns in [110] direction; b) atomically resolved X-ray map of Ga recorded with ChemiSTEM technology (FEI Company) including a four quadrant SSD detector and a high-brightness X-FEG electron source.

# **Copper Interconnects for Advanced Performance and Reliability**

#### Project leader: Gerald Kothleitner

The EU project CopPeR launched in 2008 was aimed at finding new ways of non-aqueous directon-barrier copper plating to support further device scaling in sub-32 nm technologies. The CopPeR project (the acronym 'CopPeR' stands for 'Copper Interconnects for Advanced Performance and Reliability') was run at FELMI together with a consortium of seven leading European companies and academic institutions in the area of plating technologies. This vertically integrated consortium spanned the entire range from basic research to the design and marketing of products, including the production, evaluation and impacts on the International Technology Roadmap for Semiconductors (ITRS).

The project aimed to develop a novel copper deposition process based on the use of non-aqueous solvents in order to overcome the limitations of current interconnect formation processes enabling device scaling beyond the 32 nm technology node. This non-aqueous process opens novel routes to implement direct-on-barrier plating, focusing on tantalum and ruthenium as diffusion barriers. The process developed and implemented within the CopPeR project significantly improves the quality of Cu metallization due to the fact that the conductivity limiting seed-Cu will be eliminated and thinner barrier films can be applied, e.g. by ALD (atomic layer deposition) so that more volume is available in trenches for high quality, low resistivity Cu.

CopPeR consisted of three phases: In the first phase, electrolyte ingredients and wafer materials were selected, their basic physical properties investigated and a deposition cell designed through modelling and simulation, and new analytical techniques were evaluated to enable adequate analysis of the deposited films. The second phase focused on the development of the copper deposition process based on the findings from phase one with the additional support of micro-modelling, and the process was scaled and integrated into a 300 mm proof-of-concept. In the third and final phase, the process aimed for a complete interconnect scheme, optimized according to the industrial chip manufacturer's needs. FELMI's task consisted of both supporting characterization and, more importantly, developing new metrology methods for key parameters such as physical and chemical properties like feature dimensions/sizes, chemical composition in bulk material and at interfaces, structure, pore shapes, surface topography, roughness, adhesion etc.

Within the project new routes have been shown to further investigate and explore features and phenomena at the nanometer scale and in 3D (nanotomography). Questions that could not be solved before due to the projection character of the TEM, e.g. through-the-sample surface/interface defects, can now be tackled by reconstructing a tilt series (projection series) using various algorithms. Structured materials and deposited elements could be analysed in terms of volumetric concentrations and roughness.

#### Funding organization:

CopPeR, Project No. 216474, FP7 ICT STREP (European Commission)

#### Project period:

1 Jan. 2008 - 31 Dec. 2010

Selected publications:

L. D'Urzo, S. Schaltin, A. Shkurankov, H. Plank, G. Kothleitner, K. Binnemans, J. Fransaer, "Direct on barrier copper electroplating on ruthenium from the ionic liquid 1-ethyl-3methylimidazolium dicyanamide", J. Electrochem. Soc. 158 (2011) 647-653.

S. Schaltin, N.R. Brooks, L. Stappers, L. D'Urzo, H. Plank, G. Kothleitner, C. Gspan, K. Binnemans, J. Fansaer, "Electrodeposition from a liquid cationic cuprous organic complex for seed layer deposition", J. Electrochem. Soc. 158 (2011) D647-D650.

#### Team:

Gerald Kothleitner, Harald Plank, Christian Gspan, Toni Uusimäki, Mihaela Albu, Martina Dienstleder

#### Collaborations:

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LAM Research, Villach, Austria

Katholieke Universiteit Leuven, Belgium



ELSYCA N.V., Wijgmaal, Belgium Vrije Universiteit Brussel, Brussels, Belgium

# Infineon Technologies AG, Munich, Germany



Fig.1 Cartoon figure of a new deposition cell and FIB cross-section of a Cu interconnect.



Fig.2 Surface rendered visualization of the incoherent bright (IBF-STEM) reconstruction of a copper interconnect; the background shows an image of the central plane of the reconstruction; Ta layer in green, oxygen voids in red.

# **Microscopical Analysis of Composites for Automotive Applications**

#### Project leader: Hartmuth Schröttner

The use of composite materials and multi-material compounds was a significant milestone of achievement in the automotive industry. Such materials found their way into the production of sports cars and aerospace components, and in the 21st century, have become more or less the norm in automotive manufacturing. The advantage of composite materials over single layered materials is the fact that they are much stronger and lighter. As a result, composite materials help to save energy and resources as well as to provide cost effective production.

In 2009, Austrian Cooperative Research (ACR) launched the competence platform "Multi-Material Compounds". The platform combines the know-how, competences and measurement capabilities of four ACR institutes (ÖGI, OFI, SZA and ZFE) in order to support small and medium-sized enterprises in the automotive sector with additional research capabilities.

The main research focus of the project was to find optimal ways of joining different lightweight materials and composites, to characterize their distinctive properties and to examine their suitability for automotive applications. In order to control their functionality, electron microscopy methods were used for characterizing the morphology of these materials, their interfaces, defects and fracture surfaces over an extended period of time. Consequently, ZFE concentrated on improving sample preparation of new compounds for electron microscopy analysis and on developing advanced microscopy methods for defect and interface characterization of automotive materials (Fig.1).

For the first time, it was possible to combine a serial block face SEM with X-ray spectrometry (EDXS) for recording 3D elemental maps of materials. The serial block face SEM uses an ultramicrotome integrated in an environmental scanning electron microscope (ESEM), which enables automated slicing and imaging, delivering a 3D reconstruction of a specimen's volume.

The 3D structural information is now complemented by chemical information using EDX spectrometry

(Fig. 2). This technique has the potential to provide new insights into sliceable composite materials with complex chemical structures at the sub-micrometer scale.

Funding organization:

Federal Ministry of Economy, Family and Youth, Vienna; Project No. 98.175.

Project period:

1 Jul. 2009 – 30 Jun. 2012

Selected publications:

H. Schröttner, "Grundlagen der Rasterelektronenmikroskopie und Mikrobereichsanalytik", Gießerei-Praxis 10 (2010) 307-320.

A. Zankel, H. Reingruber, H. Schröttner, "3D elemental mapping in the ESEM", Imaging & Microscopy 2 (2011) 35-37.

Team:

Hartmuth Schröttner, Julian Wagner, Johannes Rattenberger, Sabrina Mertschnigg

Collaborations:

Austrian Foundry Research Institute (ÖGI) Leoben

- Austrian Research Institute for Chemistry and Technology (OFI), Vienna
- Austrian Institute of Material Science and Welding Technology (SZA), Vienna

Alicona Imaging GmbH, Grambach



Fig.1. Mono-stone chipping test of an anti-corrosion coated sheet steel; a) SEM image of the surface after the test, b) 3D model of the surface after the test, light microscopy image recorded by an infinite focus microscope (IFM by ALICONA, Grambach).



Fig.2. Worldwide first 3D EDS elemental map of the most important intermetallic phases and precipitates in an aluminum-copper alloy (Al2024) based on a dataset of 200 slices with a thickness of 100 nm cut by an in-situ ultramicrotome in an ESEM.

# **Electronic Circuits for Hot-embossed Organic Devices (NILecho)**

#### Project leader: Werner Grogger

As the field of organic electronics is developing from purely laboratory based research to standard production procedures, fabrication methods allowing high throughput at low costs are gaining more and more attention. Nano-imprint lithography (NIL) appears to be a suitable tool for mass production of organic devices. The main focus of the NILecho project is the development and improvement of NIL fabrication techniques with respect to organic electronic devices and circuits.

The common NIL process requires an anisotropic etching step under vacuum conditions, which is both costly and time consuming. This step was eliminated by developing special resist materials that can be imprinted residue-free, thus allowing a complete production line on air [1]. An ideal method to investigate the results of individual fabrication steps is to analyse cross sections of the imprinted areas. The cross section lamellas are cut using a focused ion beam instrument (FIB) thinning only the region of interest for transmission electron microscopy (TEM) and nanoanalysis. This preserves the major part of the lamella as static support and thus prevents bending of the soft materials.

A main topic of the project is to optimize the fabrication process in order to improve device performance. To achieve higher switching frequencies the devices need to become smaller and smaller. Since aspect ratios have to be preserved to avoid downgrading effects (e.g. short channel effects), however, downscaling enhances other detrimental effects (e.g. parasitic capacitance). The overlap of source/ drain electrodes and the gate electrode must be as small as possible to minimize these effects. A minimum overlap was achieved by developing a method where the gate electrode acts as a shadow mask for source and drain (referred to as self-aligned process) [2]. In particular, the NIL fabrication process is applied to structure a sub-micron gate electrode which is subsequently used as a shadow mask for the channel between source and gate electrode as sketched in Fig. 1. Depending on the substrate materials and their thicknesses, electrode overlaps down to several nanometres were achieved (Fig.2 and 3). The specimens were again analysed using analytical transmission electron microscopy, i.e. EELS and EDXS spectrometry.

The introduced methods were used to produce organic field effect transistors and even electronic circuits with reasonable performance. The project also involves the preparation of stamps using a FIB, which allows structuring of channel lengths as small as 100 nm. This is a promising approach for circuits with higher frequencies.

Based on the substantial progress made, efforts are now being concentrated on implementing the developed techniques into a roll-to-roll (r2r) fabrication line using different stamp materials. A NIL r2r fabrication line for organic electronic devices would enable a substantial increase in throughput at reduced costs.

Funding organization:

Austrian Research Promotion Agency (FFG), Vienna, Project No. 830269.

Project period:

1 Apr. 2008 – 31 Mar. 2014.

Selected publications:

[1] C. Auner; U. Palfinger, H. Gold, J. Kraxner, A. Haase, T. Haber, M. Sezen, W. Grogger, G. Jakopic, J. Krenn, G. Leising, B. Stadlober, Organic Electronics 10 (2009) 1466-1472.

[2] U. Palfinger, C. Auner, H. Gold, A. Haase, J. Kraxner, T. Haber, M. Sezen, W. Grogger, G. Jakopic, J.R. Krenn, B. Stadlober, "Fabrication of n- and p-Type Organic Thin Film Transistors with Minimized Gate Overlaps by Self-Aligned Nanoimprinting", Adv. Mater. 22 (2010) 5115-5119.

#### Team:

Thomas Haber, Meltem Sezen, Werner Grogger

Collaboration and Coordinator:

Institute for Surface Technologies and Photonics, Joanneum Research GmbH, Weiz



Fig.1 Scheme of the self-aligned process; completed device



Fig.2 TEM bright-field image and EFTEM elemental maps of the channel border showing the electrode overlaps for different substrates.

Fig.3 TEM bright field image at higher magnification.



# Soft Matter Processing via Focused Ion Beams

#### Project leader: Harald Plank

Focused ion beam (FIB) processing has attracted enormous attention over the last few decades, not only due to its unique capabilities as a site-specific lamella preparation tool for transmission electron microscopy, but also as a method for 3D surface structuring from the micro- to the nano-scale. Because of its flexibility and its rapid implementation character, it is of particular importance for prototyping applications in science and technology.

Despite the undoubted advantages of FIB processing, there are also problems such as spatially confined ion implantation, surface amorphization and partial high thermal stress. While the former two are intrinsic properties and therefore invariable, local heating effects can considerably alter or chemically damage sensitive samples such as nano-scale devices, thin films, or materials with a low melting point such as polymers and biological material. A possible counterstrategy is the application of low temperature stages (down to liquid nitrogen temperatures) which, however, are not always available and can be complicated to use. Hence, alternative process strategies need to be developed which reduce the overall thermal stress for the samples without necessarily using low temperature stages.

The mechanism of local heating can be divided into 1) intrinsic events due to ion impact and entailed temperature rise; and 2) patterning related influences which reveal significant potential for improvements. By varying the process parameter such as the distance of two consecutive points (point pitch; PoP) during patterning and the ion beam pulse duration for single points (dwell time; DT) it is found that classical serpentine- or raster-like patterning strategies can induce additional heating. Finite element (FE) simulations suggest a temperature rise of more than one order of magnitude above the intrinsic temperature increase during one single ion beam pulse depending on the PoP used.

Figure 1 shows FE based temperature rises for different point pitches ranging from 10 nm (standard) to 500 nm revealing the massive effect of technically induced heating and its impact on polymer materials (scanning electron micrographs on the right). Based on these findings, an alternative patterning sequence has been introduced, which is able to eliminate this technically induced heating while not affecting process times and sample surface roughness.

Atomic force microscopy, scanning electron microscopy, transmission electron microscopy, simultaneous thermal analyses, and Raman spectroscopy have revealed that the chemical damage is strongly reduced by the alternative patterning sequences. As can be seen in Figure 2, the novel processing method using integrated Raman signals causes much less damage (green versus red bars) and can be compared to the results of classic FIB processing with low ion beam currents (blue bars) or at cryogenic sample temperatures of -150 °C (left versus right block).

The successful reduction of local thermal stress towards the intrinsic and unavoidable limit – related to single pulse effects – might open up new possibilities for focused ion beam processing of soft matter.

Funding organization:

Austrian Research Promotion Agency (FFG), Vienna, Project No. 830186

Project period:

1 Jan. 2010 - 31 Mar. 2013

Selected publications:

- H. Plank, D.A. Smith, T. Haber, P.D. Rack, F. Hofer, "Fundamental proximity effects in focused electron beam induced deposition", ACS Nano 6 (2012) 286-294.
- H. Plank, G. Kothleitner, F. Hofer, S.G. Michelitsch, C. Gspan, A. Hohenau, J. Krenn, "Optimzation of postgrowth electron-beam curing for focused electron-beam-induced Pt deposits", J. Vac. Sci. Techn. A29 (2011) 051801, 1-7.

#### Team:

Harald Plank, Roland Schmied, Boril Chernev, Claudia Mayrhofer

Collaborations:

Institute for Chemistry a. Technology of Materials, University of Technology, Graz



Fig.1. Finite element based simulations for the temperature rise of polymer samples (polypropylene) during FIB processing for different point distances of consecutive patterning points (point pitch). The temperature rise for typical point pitch distances of about 10 nm (black) is 20 times higher than for 500 nm distances (grey), which involve only intrinsic heating due to ion-sample interactions. The impact on polymer samples can be seen in the scanning electron microscopy images on the right.



Fig.2. Raman intensities of polypropylene (integrated between 2700-1 nm and 3000-1 nm according to the characteristic CH2 and CH3 stretching vibrations bands) structured with ion energies and beam currents of 30 kV and 500 pA, respectively. The summary compares the results for standard (red) and alternative (green) patterning strategies at room temperature (left block) and -150 °C conditions (right block) for different dwell times (see X-axis). The direct comparison clearly reveals clearly that chemical damage via alternative patterning strategies is strongly reduced, much more independent on process parameters and can even compensate for cryogenic low temperature stages.

# Nanoanalytical Characterization of Organic Sensor Devices

#### Project leader: Werner Grogger

The aim of the project cluster "Integrated Organic Sensor and Optoelectronic Technologies" (ISO-TEC) is to fabricate opto-electronic components for light guiding and light coupling and to develop integrated organic sensors for multi-analyte detection. The focus is to realize an integrated organic optical sensor platform suitable for the parallel detection of multiple parameters such as oxygen, carbon dioxide, pH, temperature and ammonia. This cuttingedge sensor technology approach combines optical sensor layers, realized by optical waveguides, with organic light emitting devices (OLEDs) and organic photodiodes (OPDs) as light source and detection unit, respectively. Work area 4 of ISOTEC ("Nanoanalytical Characterization of Organic Sensor Devices") concentrates on nanoanalytical methods such as transmission electron microscopy (TEM), scanning electron microscopy (SEM), atomic force microscopy (AFM) and X-ray scattering techniques, which are applied for device, process and system characterization regarding morphology, nanostructure and chemical composition.

In organic devices the topographies and roughnesses of the interfaces as well as the thicknesses of the individual layers decisively influence the device properties. These parameters of the multilayer systems were therefore analysed using TEM, SEM, AFM and X-ray scattering techniques. Figure 1 shows the setup of an OPD consisting of stacked layers of organic p- and n-type materials sandwiched between two metal electrodes and a TEM bright-field image of the cross-section of this OPD. Topographies, roughness and layer thickness obtained with the above mentioned techniques are in very good agreement.

Furthermore, the deposition of the top cathode has an essential influence on device performance. The penetration of Ag into different organic materials is a crucial factor and was thus systematically investigated for various deposition methods such as physical vapour deposition, ion-beam sputtering and electron-beam deposition using TEM and X-ray scattering techniques.

In addition, materials used for two photon absorp-

tion (TPA) technology were investigated by TEM to elucidate the chemical structure and changes of the chemical bonding within the structure.

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In addition, materials used for two photon absorption (TPA) technology were investigated by TEM to elucidate the chemical structure and changes of the chemical bonding within the structure.

#### Funding organization:

Austrian Research Promotion Agency (FFG), Vienna, Project No. 819718

Project period:

1 Feb. 2005 - 30 Apr. 2012

Selected publications:

S. Fladischer, A. Neuhold, S. Mitsche, E. Kraker, A. Haase, B. Lamprecht, R. Resel, W. Grogger, "Nanoanalytical characterization of an organic photodiode by electron microscopy and atomic force microscopy", Proceedings Int. Conf. on Organic Electronics (2010) pp.235-236.

A. Neuhold, S. Fladischer, S. Mitsche, E. Kraker, B. Lamprecht, A. Haase, W. Grogger, R. Resel, "Structure and Morphology of an Organic/Inorganic Multilayer Stack: An X-ray Reflectivity Study", Journal of Applied Physics 110 (2011) 114911.

#### Team:

Werner Grogger, Stefanie Fladischer, Thomas Haber, Claudia Mayrhofer

#### Collaborations:

Institute of Solid State Physics, Graz University of Technology

NANOTEC Center Weiz GmbH, Weiz

- Institute for Surface Technologies and Photonics, Joanneum Research GmbH, Weiz
- Institute of Materials Science and Technology, Vienna University of Technology



Fig.1 a. Schematic of the organic photodiode with nominal layer thickness values and b. TEM bright-field image of the cross-section of a real device.

# NanoProbe – Analytics on Nanosized Objects

#### Project leader: Gerald Kothleitner

NanoProbe is a cooperation project between FEL-MI-ZFE and ZONA of the Johannes-Kepler University of Linz and is embedded in the NSI cluster (Austrian Nanoinitiative). It contains two work packages that deal with high resolution TEM and element specific mapping of two materials systems of great importance for the NSI, namely magnetic phases in a crystalline host (here: GaN), and magnetic core-shell nano crystals. Nanoanalytical techniques such as electron microscopy and spectroscopy and surface-sensitive optical measurements, which are complementary in several aspects, are brought together for studying these magnetic nanostructures.

The main work was performed on the analysis of Fe- and Mn-doped GaN layers grown by metalorganic vapour phase epitaxy (MOVPE) on GaN/csapphire at a substrate temperature of 850°C. First experiments were done on a probe-corrected TITAN microscope from FEI with 300 kV at the FZ Jülich in order to bridge the time for the ramp-up of the ASTEM in Graz. Mn-doped GaN layers were investigated in high resolution STEM mode with a 0.1 nm probe combined with EELS investigations, carried out in the "stripe-EELS" configuration. A typical STEM image of this sample is shown in Figure 1 with a significant variation of the Mn-doping perpendicular to the growth direction.

A highly critical task of this work package was to optimize specimen preparation based on FIB crosssections and low-energy ion milling, which is an essential prerequisite for high resolution STEM imaging (to avoid any contamination and amorphous layers).

The operational start of the ASTEM (FEI TITAN 60-300) in Graz enabled more detailed investigations. First experiments focused on the acquisition of atomic resolution HR-STEM of undoped GaN (Fig. 2a). This Z-contrast image clearly reveals the Ga columns in the lattice. Another new possibility is the acquisition of annular bright-field images (AFB) in the STEM mode, which uses the outer angles of the bright-field disc recorded in the annular dark-field detector. These images have a higher sensitivity to light elements (such as N and O) and combine large signal rates with low exposure times, at lower doses than in HAADF or EELS and EDX spectroscopy. Fig. 2 shows the simultaneously recorded STEM HAADF and ABF images in combination with the crystal structure of GaN in [110] orientation. A zoom into the image reveals the nitrogen atoms (pointing alternatively upwards and downwards).

The ASTEM microscope incorporates the ChemiSTEM technology (X-ray spectroscopy with a four quadrant SSD detector and a high brightness electron source from FEI) which opens the route to atomic resolution analysis of dopants in the GaN lattice. The final part of the project therefore concentrated on the direct imaging of Mn and Fe in doped GaN layers.

Funding organisation:

Austrian Research Promotion Agency (FFG), Vienna, Project No. 819703

Project period:

1 Mar. 2009 – 29 Feb. 2012

Selected publications:

A. Bonanni, M. Sawicki, T. Devillers, A. Meingast, G. Kothleitner et al., "Ga1-xMnxN – Experimental Probing of Exchange Interactions between Localized Spins in a Dilute Magnetic Insulator", Phys. Rev. B84 (2011), 035206 – 1 – 035206-11.

C. Gusenbauer, T. Ashraf, J. Stangl, T. Plach, A. Meingast, G. Kothleitner, R. Koch, "Interdiffusion in Heusler film epitaxy on GaAs (001)", Phys. Rev. B 84 (2011) 0353319-1 – 035319-8.

#### Team:

Arno Meingast, Gerald Kothleitner, Sebastian Rauch

#### Collaboration:

- ZONA, Johannes-Kepler University Linz Institute of Semiconductor and Solid State Physics, Johannes Kepler University Linz
- Ernst-Ruska-Centre for Microscopy and Spectroscopy, Research Center Jülich, Germany



Fig.1. a) STEM image of a GaN cross-section with GaN doped with Mn in [110] orientation; b) STEM-HAADF image showing the Ga columns overlaid with measured signal of the Mn  $L_{2,3}$  ionization edge along each atomic line (stripe EELS).



Fig.2. a) HR-STEM-HAADF image of GaN in [110] orientation; b) corresponding ABF image; c) zoom into the ABF image of b; d) crystal structure of GaN in [110] orientation; e.) contrast inverted ABF image of b.

# **Microscopical Characterization of Nanocomposite Solar Cells**

#### Project leader: Ferdinand Hofer

The Institute cooperates with the Christian Doppler Laboratory for Nanocomposite Solar Cells, which is directed by Gregor Trimmel (Institute for Chemistry and Technology of Materials, Graz University of Technology). The Laboratory has set its focus on the development and characterization of new nanocomposite materials with controlled morphology for photovoltaic applications. Together with the industrial partner ISOVOLTAIC new materials and processes for low-cost, polymer based photovoltaics are explored. The research programme is devoted to developing strategies and materials for nanocomposites with enhanced photovoltaic activity.

Besides classic inorganic photovoltaic devices that are currently on the market, mixed inorganic/organic cells are being intensively investigated in many research labs. The advantages of these materials are, among others, easy and non-expensive processing, low cost and the possibility to vary the composition and structure over a broad range.

In this project, we concentrate on nanocomposites consisting of semiconducting nanoparticles in a conjugated polymer matrix, where the inorganic nanoparticles are usually responsible for electron transport, while the conjugated polymer acts as a hole transporting material.

In order to optimize the efficiency and the design of the solar cells, it is important to control the morphology, local chemistry and the interface structure of the devices. FELMI-ZFE therefore concentrates on the characterization of the processed devices by means of scanning electron microscopy, focusedion beam microscopy (FIB) and cross-section transmission electron microscopy (TEM) (Fig. 1).

Another important aspect of these studies lies in the development and characterization of the inorganic nanoparticles. Chalcopyrite-type semiconductors like  $Cu(In,Ga)S_2$  or  $Cu(In,Ga)Se_2$  have very beneficial properties for photovoltaic application, but the Ga and In used for the active layer are very rare and expensive elements.

To overcome these limitations, alternative materials are also being explored by means of high resolution STEM, such as  $Cu_2ZnSnS_4$ , which is among the most promising materials as it consists of relatively cheap elements (Fig.2).

Funding organization:

Christian Doppler Research Society, Vienna, Project No. 821074

Project period:

1 Jul. 2008 - 31 Oct. 2012

Selected publications:

A. Pein, M. Baghbanzadeh, T. Rath, W. Haas, E. Maier, H. Amenitsch, F. Hofer, O. Kappe, G. Trimmel, "Investigation of the Formation of CuInS<sub>2</sub> Nanoparticles by the Oleylamine Route: Comparison of Microwave-Assisted and Conventional Syntheses", Inorganic Chemistry 50 (2011) 193-200. W. Haas, T. Rath, A. Pein, J. Rattenberger, G. Trimmel, F. Hofer, "The Stoichiometry of Single Nanoparticles of Copper Zinc Tin Selenide",

Chem. Commun. 47 (2011) 2050-2052.

#### Team:

Wernfried Haas, Roland Schmied, Armin Zankel, Martina Dienstleder, Ferdinand Hofer

#### Collaborations:

Institute for Chemistry a. Technology of Materials, Graz University of Technology ISOVOLTAIC AG, Lebring

NANOTEC Center Weiz GmbH, Weiz



Fig.1. TEM cross-section of nanocomposite solar cell, prepared in the focused ion beam microscope (FIB).



Fig.2. Electron microscopy reveals the chemical inhomogeneity of  $Cu_2ZnSnSe_4$  nanoparticles, measured in the FEI Tecnai F20 (STEM-HAADF image) and confirmed by EDX spectra for selected copper-(A), tin-(B) and zinc-rich (C) nanoparticles.

# ESiP 2 (Efficient Silicon Multi-Chip System-in-Package Integration)

Project leader: Ferdinand Hofer, Werner Grogger

Miniaturization and enhanced functionality of integrated devices open new markets and improve quality of life by mobile communication, safety and health supporting devices. As a result higher system integration in nanoelectronic technologies like multi-chip packaging, through silicon via technologies or package stacking approaches are growing in importance.

The ESiP project cluster investigates latest silicon based integration technologies such as through silicon via (TSV) and wafer level packaging. This includes integration of silicon chips into a systemin-package (SiP) considering also the interface to the board. The main roadblocks for these new system integration technologies are reliability, test, and cost.

FELMI-ZFE contributes via the development of failure analysis methods on SiP level, which is required for a better assessment of reliability. Our focus lies on improvements in specimen preparation like large area, X-section ion milling, focused ion beam preparation and wedge polishing. Analytical transmission electron microscopy and EELS and EDXS spectrum imaging are used to investigate defects and interfaces of TSV technology at high spatial resolution.

Typical defect types were observed during the development of open TSVs, occasionally close to the wafer edge. The defects had the appearance of lines running parallel to the scallops of the TSVs (Fig.1). They are relatively difficult to characterize as they are located 180  $\mu$ m to 250  $\mu$ m below the device surface. In order to prepare TEM lamellas from a depth of more than 180  $\mu$ m, the TSV had to be prepared by special lateral grinding to allow FIB preparation from the side.

Figure 2 shows that Ti and TiN layers are deposited on the positive slope of the scallops. The film tails out towards the reverse deepening and abruptly ends on the scallop crest, i.e. it is interrupted in the negative slope of the scallop. The de-lamination of the Ti layer starts from the positive slope in the buckling parts and bends outwards towards the centre of the TSV. Figure 2 shows a gas sensitive  $SnO_2$  layer (from AIT), where wedge polishing was successfully used to minimize the specimen induced amorphization. This was essential as the investigation focused on the differentiation between amorphous and crystalline regions.

The project consortium consists of 41 partners with complementary skills from nine European countries (Austria, Belgium, Finland, France, Germany, Italy, The Netherlands, Norway, United Kingdom).

Funding organizations:

ENIAC and Austrian Research Promotion Agency (FFG), Vienna, Project No. 824957

#### Project period:

1 May 2010 - 30 Apr. 2013

Selected publications:

J. Kraft, F. Schrank, J. Teva, J. Siegert, G. Koppitsch, C. Cassidy, E. Wachmann, F. Altmann, S. Brand, C. Schmidt, M. Petzold, "3D Sensor Application with Open Through Silicon Via Technology", Electronic Components and Technology Conference (2011) 560-566.

#### Collaborations:

Austriamicrosystems AG, Unterpremstätten Austrian Institute of Technology, Vienna Infineon Technologies, Regensburg, Germany and other partners from Belgium, France, Italy, The Netherlands, Norway, U.K. and Finland.



Fig.1. FIB cross-section of TSV sidewall, a) TEM image of a scallop with partial Ti/TiN coverage. b) elemental distribution map of the TSV sidewall investigated by means of energy-filtering TEM (EFTEM). The RGB-image shows two interface layers.



Fig.2. HR-TEM image of gas sensitive SnO<sub>2</sub> layer. Use of a suitable specimen preparation technique (wedge-polishing) minimized the preparation induced amorphization so that amorphous and crystalline regions can be clearly visualized.

# **Confocal Raman Microscopy for Materials Characterization with High Resolution**

#### Project leader: Peter Wilhelm

This project aimed at installing a new confocal Raman microscope at the Institute to complement the existing methods for the characterization of new materials. Three-dimensional chemical (molecular) analyses ("chemical imaging") with the highest lateral resolution currently available will support the development of new materials (polymers, composites and soft materials) with fine structured details.

Since Raman microscopy connects light microscopy with vibrational spectroscopy, it is possible to analyse materials in two, or even three dimensions, with lateral resolutions of a few hundred nanometers and measuring times below 1 second per point spectrum. Depth resolution may be obtained by microtoming the sample or by confocal techniques. Dispersive Raman microscopes use excitation lasers in the visible, UV or NIR range with very low intensities to avoid damage to sensitive samples (biological materials, soft polymers).

Raman microscopes from three manufacturers were investigated, looking for best lateral and confocal resolution, as well as fast and precise imaging. Finally, the LabRam HR 800 system from Horiba-Jobin-Yvon was selected and installed in June 2010. This microscope has two excitation lasers (red: 633 nm, 17 mW, and blue: 473 nm, 20 mW). The spectrograph has a focal length of 800 mm, providing a spectral resolution down to 0.35 cm-1 (with 633 nm excitation and 1800 gr/mm grating), and a 1024x256 pixel multichannel CCD detector (spectral range: 200-1050 nm).

Fast imaging is accomplished by SWIFTTM (software module) and DuoScan<sup>™</sup> (hardware option for fast and uniform scanning over small and large sample areas with variable pixel size).

The main application fields of the new microscope are new functional materials (polymers, organic light emitting diodes, organic solar cells), semiconductors (silicon wafers, ceramics), pharmaceutical and biological samples (tablets, emulsions, bones, teeth, implants) with very thin layers or small domains to be analysed.

The new Raman microscope will be mainly used

within the core area "Advanced Materials Science" of the Graz University of Technology.

Funding organization:

European Funds for Regional Development (EFRE) Styrian Provincial Government, Graz Graz University of Technology

Project period:

1 Jan. 2010 - 30 Jun. 2011

Selected publications:

G. Eder, B.S. Chernev; "Spectroscopic characterization of the oligomeric surface structures on polyamide materials formed during accelerated aging", Applied Spectroscopy 65 (2011) 1133-1143.
M. Schiller, B. Pelzl, J. Woschitz, S. Schwarz, A. Jahnke, P. Wilhelm, B. Chernev, "Environmental factors on weathering of plastic windows", GAK – Gummi, Fasern und Kunststoffe 62 (2009) 428-438.

#### Team:

Peter Wilhelm, Boril Chernev

Collaborations:

- Institute for Chemistry a. Technology of Materials, Graz University of Technology
- Institute of Process and Particle Engineering, Graz University of Technology
- Austrian Research Institute for Chemistry and Technology (OFI), Vienna



Fig.1: LabRAM HR 800 confocal Raman microscope at FELMI, Graz University of Technology



Fig.2: Raman image of a pharmaceutical tablet (3 components imaged in red, blue and green). Left image: whole tablet (scanned area: 12 mm x 12 mm; 160,000 spectra collected, step width 30  $\mu$ m; measuring time: 3.5 hours); right image: area within the white square (500  $\mu$ m x 500  $\mu$ m; 10,000 spectra, step width 5  $\mu$ m; measuring time 20 minutes).

# Microanalysis with High Detection Sensitivity

#### Project leader: Stefan Mitsche

Microanalysis with high detection sensitivity, i.e. elemental analysis in the ppm range, is an important issue in materials characterization. The Institute's high resolution scanning electron microscope (Zeiss Ultra 55) was therefore equipped with a LambdaSpec wavelength dispersive X-ray spectrometer (WDXS) from EDAX. Fig. 1a shows the setup of the microscope and Fig. 1b the outstanding performance of the new WDX system in comparison to the energy dispersive X-ray system (EDXS).

The project was carried out to gain practical experience in microanalysis by WDXS. The first part of the project focused on the development of methodical principles and the second part on the application of WDXS to materials science problems, which are briefly summarized in the following.

We were able to show that proper specimen preparation is essential to obtain reliable quantitative results for trace elements. This influence was demonstrated on a multilayer device consisting of ceramics with thin noble metal layers in between, where the diffusion of the noble metal in the ceramic was measured. Whereas the noble metal concentration in specimens prepared by mechanical grinding and polishing followed by Argon etching was determined in the range of 1000 ppm, the values for specimens prepared with the focused ion beam (FIB) were around 100 ppm (see Fig. 2).

Since the microscope is additionally equipped with a STEM detector, it was also possible to explore the potential of a combined STEM and WDXS analysis. Using very thin specimens (<100 nm) prepared in the FIB and an improved specimen holder we were able to combine STEM with both WDXS and EDXS. The simultaneous use of WDXS and EDXS is essential to reduce analysis time and, as a consequence, the effect of specimen drift.

This setup allowed us to achieve a lateral analytical resolution in the 10 nm range, which is an improvement of at least one order of magnitude over conventional scanning electron microscopy. Additionally, we developed a procedure to obtain reliable results for the quantitative analysis of carbon down to the 100 ppm range.

A general problem for the quantification of carbon in the SEM is the electron beam induced deposition of carbonaceous material at the sample surface. This problem was solved by building a decontamination device, which enabled the measurement of carbon contents in the 100 ppm range. This was proven by use of carbon standards with carbon concentrations between 0.03 % C and 1.06 % C.

Funding organization:

Project period:

1 Jul. 2008 – 31 Dec. 2010

Team:

Stefan Mitsche, Meltem Sezen, Wolfgang Czapek, Peter Pölt, Angelika Reichmann

#### Collaborations:

EPCOS OHG, Deutschlandsberg, Austria

Austrian Research Promotion Agency (FFG), Vienna, Project No. 818816 and 825165



Fig.1. a) HR-SEM Zeiss Ultra 55 equipped with a) EDX-spectrometer, b) WDX-spectrometer, c) CCD-camera for EBSD, d) STEM-detector; b) comparison of a WDX-spectrum (blue) und an EDX-spectrum (red), x-axis: energy in eV, y-axis: count of X-rays.



Fig.2. a) FIB cut of the ceramic with measurement points marked; b) diffusion profile of the noble metal, x-axis: measuring point, y-axis: weight% noble metal.

# **3D Microscopy of Polymers and Biomaterials**

#### Project leader: Nadejda B. Matsko

The study of the ultrastructure and properties of soft and hydrated materials (plastics, engineering resins, polymers, nanoliquids, biological liquids and materials) is a rapidly developing field. Additionally, the commercial importance of understanding the relationships between the manufacturing process, the structure produced, and the resulting physiological properties of soft and hydrated materials for medical and pharmaceutical applications cannot be overestimated.

The real three dimensional phase morphology of soft/hydrated materials can only be reconstructed with nanometer resolution using tilt-series electron tomography under cryo conditions. The main drawbacks of this method are cryo-section shape (compression, chattering, crevasses) and thickness (few hundred nanometers maximum), poor electron microscopy contrast and high proneness to electron beam induced damage.

Atomic force microscopy (AFM) is a surface characterization technique that, in contrast to TEM, can be considered non-destructive when analysing soft matter. Information about the location, architecture and mechanical properties of macromolecules or polymer chains can be obtained directly from the surface of the unstained block face, free from sectioning artifacts - even at low temperatures.

We have successfully developed a unique cryo atomic force microscope system (SNOTRA) based on a specially designed cryo-AFM directly mounted in the cryogenic chamber of an ultramicrotome.

This combination of instruments allows scanning a sample immediately after sectioning at ambient conditions and for the first time also under cryo conditions. Any configurational, constitutional and conformational change of macromolecules such as ageing or recrystallization during preparation can be avoided as the whole structure is stabilized by cold.

Consequently, this instrument can be applied to serial section 3D tomography of a wide range of soft polymer and biological materials. Sections of good quality can also be collected and used for correlative electron microscopy analyses. In addition, SNOTRA provides the possibility to investigate samples in a wide range of temperatures (from -120°C to 50°C), and can be used for the investigation of thermotropic dynamic processes in soft materials in vivo.

Funding organisation:

Austrian Research Promotion Agency (FFG), Vienna

Project period:

1 Nov. 2008 - 31 Jan. 2011

Selected publications:

A.E. Efimov, H. Gnaegi, I. Haynl, V. Sevastyanov, R. Schaller, W. Grogger, F. Hofer and N. Matsko, "Analysis of native structures of soft materials by cryo scanning probe tomography", Soft Matter (in press).

N. Matsko, N. Žnidaršič, I. Letofsky-Papst, W. Grogger, J. Štrus, F. Hofer, "Silicon: the key element in early stages of biocalcification", J. Struct. Biol. (2011) 174 (1), 180-186.

#### Team:

Nadejda B. Matsko, Werner Grogger, Ferdinand Hofer, Helmut Gnaegi, Anton E. Efimov

#### Collaborations:

Nanoscan Technologies, Moscow, Russia Department of Pharmaceutical Technology and Biopharmaceutics, University of Vienna

Semperit Technische Produkte, Wimpassing



Fig.1. AFM and TEM images and 3-D AFM image of the polyamide 6/ acrylonitrile-butadiene-styrene copolymer captured at room temperature; a) AFM topographical image of the cross-section of the PA6/ABS system, obtained at room temperature with SNOTRA; b) TEM image of an ultrathin section of the same sample, c) 3-D structure of PA6/ABS obtained with SNOTRA (13.5×13.0×1.5  $\mu$ m, 15 sections, section thickness 100 nm); scale bars in a and b are 500 nm, the topographical variation in each section is 250 nm.



Fig.2. AFM and TEM images and 3-D AFM image of polyamide 6/styrene-acrylonitrile copolymer obtained at room temperature and under cryo conditions. Topographical AFM images of PA6/SAN system (30/70w/w), a) cryo sectioned and immediately scanned at -80°C, and b) cryo fractured and immediately scanned at -120°C using SNOTRA; c) topographical AFM image of block face; d) TEM image of ultrathin section of the same copolymer sample, sectioned and examined at room temperature by AFM and TEM, e) three dimensional structure of PA6/SAN obtained with SNOTRA; scale bars in e) 7.9×6.2×0.75 µm, 6 sections, section thickness 125 nm; scale bars in a, b, c, and d are 2000 nm.

# Mapping Surface Plasmons of Designed Metal Nanostructures with an Electron Beam

#### Project leader: Ferdinand Hofer

Metallic nanoparticles and their extraordinary optical properties have become of great interest in the past decade. Their ability to transform light into areas much smaller than its wavelength and to strongly increase the local electric field intensities lead to an immense motivation for scientific research. A multitude of various technological applications are already in development.

The reason for these exciting properties is that electromagnetic illumination of metallic nanoparticles leads to collective resonant excitations of their conduction electrons. These so-called surface plasmon resonances (SPRs) can be triggered either by light or electron irradiation (Fig.1).

One important aspect of these SPRs is their local distribution on the nanoparticles, which can only be investigated via microscopic imaging methods. Until recently, the scanning near field optical microscope (SNOM) has been the standard method for SPR imaging despite its lateral resolution, which is limited to some tens of nanometers.

The situation changed with the advent of instrumental improvements in transmission electron microscopy, such as electron monochromators, energy filters and spectrometers with high energy resolution. The superior lateral resolution of the TEM, which is only limited by delocalization of the inelastically scattered electrons, opened the rapidly evolving field of real-space imaging of nanoplasmonic resonances. It has already become clear that both STEM-EELS and EFTEM can be used to study SPRs on nanoparticles.

At the beginning of these studies all work was performed using the STEM-EELS method. We were able to show for the first time, however, that surface plasmon resonances can also be studied using EF-TEM spectrum imaging. Key to this success was the availability of the monochromated TEM Tecnai F20, the parallel development of advanced EELS spectrum imaging techniques (PhD thesis B. Schaffer) and the preparation of "exotic" gold nanoparticles (PhD thesis M. Rogers). This project was launched in cooperation with the University of Graz in order to achieve a better understanding of the fundamental behaviour of surface plasmons on metal nanoparticles. The aims of the project are to study surface plasmons on artificially designed nanoparticle arrays of different shape and various configurations prepared by electron beam lithography (EBL) on very thin substrates. Both energy-filtering TEM and STEM-EELS are used to study pairs of electro-dynamically coupled particles (bowtie and dipole antennas) and nanorings (see Fig.2).

#### Funding organization:

Austrian Science Fund (FWF), Vienna, Project No. P21800.

#### Project period:

1 Oct. 2010 – 31 Mar. 2014

Selected publications:

B. Schaffer, U. Hohenester, A. Trügler, F. Hofer, "High-resolution surface plasmon imaging of gold nanoparticles by energy-filtered TEM", Phys. Rev. B 79 (2009) 041401.

B. Schaffer, W. Grogger, G. Kothleitner, F. Hofer, "Comparison of EFTEM and STEM EELS plasmon imaging of gold nanoparticles", Ultramicroscopy 110 (2010) 1087-1093.

#### Team:

Franz Schmidt, Ferdinand Hofer (TU Graz) and Harald Ditlbacher, Ulrich Hohenester, Joachim Krenn (Institute of Physics, University of Graz)

#### Collaborations:

Institute of Physics, University of Graz



Fig.1. shows one basic principle in plasmonics: Light is transformed in a 2-dimensional wave of collectively oscillating electrons on the surface of a metal, so called surface plasmons. This new "state" of light shows exceptional properties like a reduction of the lateral distribution far below the diffraction limit of light and a strong enhancement of the electric field. (from Harry A. Atwater: "The promise of plasmonics", Scientific American

(from Harry A. Atwater: "The promise of plasmonics", Scientific American 2007)



Fig.2. demonstrates lateral field distributions of 3 different plasmon modes (a - c) on a trimer configuration of 3 gold nanotriangles. Each mode is shown for 3 different distances between the triangles. The colour scale is proportional to the electric field distribution of the corresponding mode.

# **Cooperation Platform for Materials Characterization**

Project leader: Julian Wagner

In 2009, four Austrian Cooperative Research (ACR) institutes established the platform for materials characterization. This platform combines the unique expertise of the institutes in analysing different materials and devices. The idea was to develop a one-stop shop for the Austrian economy focused especially on the needs of small and medium-sized enterprises.

The knowledge of varying and changing properties of different materials is an indispensable component for the manufacturing process as well as for quality control. In order to support our customers with comprehensive approaches solving individual materials and production problems, we started cross-linking the unique analysis methods and facilities used at the ACR institutes.

The spectrum ranges from nanoanalysis to macroscopic materials testing, from non-destructive 3D microstructure characterization to wet chemical decomposition and measurement of physicochemical properties. The materials focus of the cooperation lies on minerals, building materials, concrete, coatings, composites, metals and electronic materials. The cooperation activities and intense scientific exchange of the institutes provide the basis for tailored customer service.

Funding organisation:

Federal Ministry of Economy, Family and Youth, Vienna.

Project period:

1 Oct. 2009 - 30 Jun. 2010

Team:

Julian Wagner, Hartmuth Schröttner, Ferdinand Hofer

Collaborations:

- Salzburg Research and Testing Institute for the Construction Industry (bvfs), www.bvfs.at
- Austrian Foundry Research Institute (ÖGI) Leoben, www.ogi.at
- Research Institute of the Association of the Austrian Cement Industry (VÖZFI), www.zement.at



#### ENTWICKLUNGSSCHWERPUNKTE

Die Insthute des Kooperationsfeldes Materialdhanatterisierung verfügen Über eine einzigznigtige Expertise auf dem Seitor der Materialshanatterisierung verschiedenster Werkstoffe, die der österreichischen Wirtschaft, insbesondere Kleinen und mittleren Behrieben, zur Verfügung gestellt wird.

Die Kenntnis der unterschliedlichen Materialeigenschaften ist heute ein unverschlatzene Belandteil. Daher wurde durch die Vernetzung der einzelnen in den KCR-Instituten genutzten Methoden ein ganzheitlichter Ansatz für Problemlösungen in der modernen Materialforschung geschaffen.

Dis vorhandene Spektrum reicht von der Nancanalytik bis zu makroskopischer Werkstoffprüfung, von der zerstforungsfreien, dreidimensionalen effolgednarktensierung bis zu nasschemischen Aufschlussverfahren. Durch die langlristige Kooperation der beteiligten Institute und ihren intersiven Austausch untereinander wird eine optimale Betersung der Kunden sichergestellt.

#### BETEILIGTE ACR INSTITUTE



# MATERIALCHARAKTERISIERUNG

EINE ACR-KOMPETENZ-PLATTFORM bvfs Bautechnische Versuchs- und Forschungsanstalt Salzburg VÖZFI Forschungsinstitut der Vereinigung der österreichischen Zemeentindustrie GGI Osterreichischen Zeißerei-Institut

ZEE Zentrum für Elektronenmikroskom



Fig.1 Information folder of the "Platform for Material Characterization"

# **Academic Education**

# **Academic Education**

The FELMI-ZFE offers modern and flexible courses for Bachelor, Master and PhD courses. The training of students provided by active involvement in research plays a crucial role. The following courses are presented in technical physics, biotechnology, chemical engineering and materials science at Graz University of Technology.

## Winter Semester

Courses for Bachelor Students

645.905 Basics of Applied Analytical Chemistry VT, 3 LU (Pölt)

511.121 Advanced Laboratory Exercises, 5 LU (Reingruber, Zankel)

Courses for Master and PhD Students

519.001 Project Laboratory Electron Microscopy and Fine Structure Research, 2 PR (Grogger, Hofer, Kothleitner, Pölt)

519.003 Scanning Electron Microscopy, 2 VO (Grogger)

519.005 Colloqium Micro- and Nanoanalysis, 2 SE (Grogger, Hofer, Kothleitner)

519.007 Electron Microscopy in Materials Science, 2 VO (Kothleitner)

519.009 Microscopy and Structuring of Materials Surfaces, 2 VU (Pölt, Plank)

519.011 Scientific Methods, 2 SE (Hofer)

519.015 Special Aspects of Analytical Electron Microscopy I, 2 PV (Hofer)

519.017 New Methods in Electron Microscopy I, 2PV (Kothleitner)

519.019 Special Aspects of Transmission Electron Microscopy I, 2 PV (Grogger)

519.023 Privatissimum Microanalysis I, 2 PV (Pölt) 519.025 Advanced Physics Seminar for Doctoral Students, 1 SE (Pölt)

519.300 Materials Characterization II, 1.33 VO (Hofer)

519.301 Microscopy of Polymers, 2 VO (Pölt, Wilhelm)

519.310 Project Laboratory Metals and Ceramics, 8 LU (Grogger, Hofer, Pölt)

519.311 Project Laboratory Semiconductor Processing and Nanotechnology, 8 LU (Grogger, Hofer, Pölt)

511.202 Physics (ET), 1 UE (FissIthaler, Haber, Letofsky-Papst, Plank, Zankel)

513.301 Materials Characterization III, 1.33 (Plank)

513.010 Experimental Laboratory Exercises, 3 LU (Grogger. Kothleitner, Plank, Pölt, Zankel)

513.011 Experimental Methods 1, 3 LU (Grogger, Kothleitner, Plank, Pölt, Zankel)

513.014 Experimental Methods 2, 3 LU (Grogger, Kothleitner, Plank, Pölt, Zankel)

MOL.915 Environmental and Food Biotechnology Laboratory, 5 LU (Kothleitner)

MOL.991 Structure Characterization by High Resolution Electron Microscopy (Biosciences), 2 VO (Hofer)

PHY.001 Introduction to Nanoanalytical Methods, 2 VO (Grogger)

PHY.031 Laboratory Course Nanoanalysis, 4 LU (Grogger, Hofer, Letofsky-Papst, Mitsche)

439.222 Survey on Methods for IC Evaluation, 1 VO (Mitsche)

446.017 Electron Microscopy Imaging, 1 VO (Hofer) 446.009 Imaging Laboratory, 2 LU (Mitsche, Wilhelm)



Development of the teaching reference number (LKZ) of the FELMI.

# **Summer Semester**

Courses for Bachelor Students

511.121 Advanced Laboratory Exercises, 5 LU (Reingruber, Zankel)

Courses for Master and PhD Students

519.004 Transmission Electron Microscopy, 2 VO (Grogger)

519.006 Colloqium Micro- and Nanoanalysis, 2 SE (Grogger, Hofer, Kothleitner)

519.008 Materials Characterization with Electron Microscopy, 2 LU (Kothleitner)

519.014 Structure Characterization by High Resolution Electron Microscopy (Physical Sciences), 2 VO (Hofer)

519.016 Special Aspects of Analytical Electron Microscopy II, 2 PV (Hofer)

519.018 New Methods in Electron Microscopy II, 2PV (Kothleitner)

519.020 Special Aspects of Transmission Electron Microscopy II, 2 PV (Grogger)

519.024 Privatissimum Microanalysis II, 2 PV (Pölt)

519.026 Nanotomography, 1 VO (Weyland)

519.028 Advanced Physics Seminar for Doctoral Students, 1 SE (Pölt)

519.030 Characterizing Materials across the Length Scales, 1 VO (Rainforth)

519.310 Project Laboratory Metals and Ceramics, 8 LU (Grogger, Hofer, Pölt)

519.311 Project Laboratory Semiconductor Processing and Nanotechnology, 8 LU (Grogger, Hofer, Pölt)

513.010 Experimental Laboratory Exercises, 3 LU (Grogger. Kothleitner, Plank, Pölt, Zankel)

513.011 Experimental Methods 1, 3 LU (Grogger, Kothleitner, Plank, Pölt, Zankel)

633.902 Practical Class in Basic Chemistry (VT), 4 LU (Mitsche, Wewerka)

CHE.578 Solid State Spectroscopy, 2 VO (Kothleitner)



Stefanie Fladischer gives a demo for students.



Hartmuth Schröttner explains the ESEM to participants of the LLL course.

# LLL Courses



# Problem Solving with Scanning Electron Microscopy and X-ray Microanalysis

(S. Mitsche, P. Pölt, A. Reichmann, H. Schröttner)

The course benefits scientists, engineers and technicians by helping them to solve their analytical problems. It is a concentrated three day hands-on laboratory workshop taking participants step-bystep through the use of advanced scanning electron microscopy. The course will familiarise them with the latest equipment and will cover the fundamental principles and methods critical to obtaining meaningful images, spectra and elemental maps.

The methods are applicable to fields ranging from materials research (steels, ceramics, semiconductors, polymers, etc.) to biological research. An important aspect of the course is the practical use of the microscope. Several advanced microscopes are available and the participants are invited to bring their own samples and are given the opportunity to analyse them themselves with the help of the advisor.

#### **GIF-School in Cooperation with Gatan, USA**

(T. Haber, G. Kothleitner, W. Grogger)

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

Participants are familiarized with the latest EELS & EFTEM equipment and will teach the fundamental principles and methods critical to obtaining meaningful EELS spectra and energy-filtered images or elemental maps. The techniques are applicable to fields ranging from biological to materials research. The participants benefit from tightly coupled lectures and discussions with some of the top experts in the field and will gain hands-on experience in various techniques.

# **Presentations and Lab Tours**

Presentations and tours of the Institute including lectures and demonstrations have also been organised for groups of physics and chemistry teachers and for students from TU Graz, schools and local universities. Around 250 pupils, teachers and students from other institutions visited the Institute during the period 2009-2011.

#### - Tours 2009

4 Dec. 2009 for the Rector of Tomsk Polytechnic University, Russia (N. Matsko)

3 Dec. 2009 for participants of the URANIA course "Electron Microscopy" (W. Grogger)

29 Oct. 2009 for students of the Chemie-Ingenieurschule in Graz (G. Kothleitner)

1 Oct. 2009 for Tokyo University of Agriculture and Technology, Japan (P. Pölt)

27 Feb. 2009 for students of the Chemie-Ingenieurschule in Graz

#### Tours 2010

13 Jan. 2010 for VDI Austria (A. Zankel)

25 Feb. 2010 for pupils from the "Kirchengasse Gymnasium" in Graz

5 Mar. 2010 for Institute of Molecular Biotechnology (N. Matsko)

28 Mar. 2010 for visitors from Infineon (G. Kothleitner, H. Plank)

26 Apr. 2010 for Austrian Academy of Sciences (W. Grogger, N. Matsko, W. Haas)

30 Jun. 2010 for employees of the Harbin Institute of Technology, China (M. Albu)

19 Aug. 2010 for participants of the ICPS-Comfernce in Graz (S. Fladischer)

18 Aug. 2010 for visitors from the Keio University in Japan and for Orbita Film GmbH (A. Zankel)

10 Oct. 2010 for Chalmers University of Technology, Sweden (M. Albu)

3 Nov. 2010 for AUDI AG (H. Schröttner, J. Wagner) 9 Nov. 2010 for physics students and TU Graz employees 23 Nov. 2010 for National Academy of Science of Ukraine (F. Hofer)

9 Nov. 2010 for the Chemie-Ingenieurschule in Graz (G. Kothleitner)

6 Dec. 2010 for a delegation from the "Industriellenvereinigung" in Graz

## Tours 2011

31 Mar. 2011 for visitors from the Swiss Federal Institute of Technology in Lausanne (P. Pölt, A. Reichmann)

1 Apr. 2011 for students of the Chemie Ingenieurschule in Graz (K. Wewerka, M. Brunegger)

4 Feb. 2011 for "Fachhochschule Kärnten" (P. Pölt, P. Reichmann)

14 Jun. 2011 for visitors from India and the IWS institute (Ing. Schröttner)

8 Aug. 2011 for visitors from "Philipps Universität Marburg" (H. Reingruber, A. Zankel)

12 Dec. 2011 for participants of the URANIA course "Electron microscopy" (W. Grogger, C. Mayrhofer)



Claudia Mayrhofer explains the operation of the TEM to the participants of the Urania course.

# Speakers at the Institute

#### Speakers 2009

13 Jan. 2009, Werner MADER

Institut für Anorganische Materialforschung, Universität Bonn, Deutschland, "Neues über Domänenbildung und Nanostrukturen mit Zinkoxid"

#### 16 Jan. 2009, Thomas WEINBERGER

Institut für Werkstoffkunde, Schweißtechnik und Spanlose Formgebungsverfahren, TU Graz, "Friction Stir Spot Welding"

#### 30 Jan. 2009, Erminald BERTEL

Institut für Physikalische Chemie, Universität Innsbruck, Austria, "Nanostrukturen: Ein Schlüssel zur Entwicklung neuer Materialien"

# 27 Feb. 2009, Peter GNAUCK

Carl Zeiss NTS GmbH, Oberkochen, Deutschland, "Neue TEM-Entwicklungen & Helium-Ionenmikroskopie"

#### 16 Mar. 2009, Nick SCHRYVERS

EMAT, University of Antwerp, Belgium, "Recent TEM and FIB/SEM studies on advanced shape memory alloys"

#### 20 Mar. 2009, Friedrich AUMAYR

Institut für Allgemeine Physik, TU Wien, Austria, "Langsame hochgeladene Ionen – ein neues Werkzeug für die Nanotechnologie?"

# 31 Mar. 2009, Hartmuth FUESS

Technische Universität Darmstadt, Deutschland) "Piezokeramiken, In-situ Transmissionselektronenmikroskopie und Röntgenbeugung"

29 May 2009, Francisca MENDEZ-MARTIN Institut für Werkstoffkunde und Schweißtechnik, TU Graz, "Experimental investigations on nitrides stability in a 12CrWMoVNb steel"

#### 5 Jun. 2009, Dieter FISCHER

Leibniz-Institut für Polymerforschung Dresden, Deutschland, "Neue Methoden zum Nachweis von Partikelgröße und Dispersion von Nanofüllstoffen in polymeren Nanocompositen"

# 30 Sep. 2009, Raymond EGERTON

National Institute for Nanotechnology, University of Alberta, Canada, "Basic questions involved in electron-induced sputtering"

19 Oct. 2009, Tatiana PEROVA

Department of Electronic Engineering, Trinity College Dublin, Ireland, "1D photonic crystals"

#### 23 Oct. 2009, Bernd O. KOLBESEN

Institut für Anorganische u. Analytische Chemie, Universität Frankfurt am Main, Deutschland, "Nitridund Oxynitridschichten von Hartmetallen und thermische Kurzzeit-Prozesse: eine harte Nuß?"

#### 6 Nov. 2009, Stavros NICOLOPOULOS

NanoMEGAS SPRL Brussels, Belgium, "Precession electron diffraction, 3D electron diffraction tomography and EBSD-TEM"

#### 21 Nov. 2009, Dangshen SU

Department of Inorganic Chemistry, Fritz-Haber-Institut of the Max-Planck-Society, Berlin, "Structure and cytotoxicity of Diesel engine soot particulates"

#### Speakers 2010

26 Jan. 2010, Mark RAINFORTH

Department of Engineering Materials, University of Sheffield, U.K., "EBSD Characterization of the Microstructure of Materials"

29 Jan. 2010, Barry CARTER

Department of Chemical, Materials & Biomolecular Engineering, University of Connecticut, Storrs, USA, "Interfaces in Ceramics: Challenges for Microscopy"

#### 23 Mar. 2010, Mathieu KOCIAK

Laboratoire de Physiques des Solids, Universite of Paris-Sud, Orsay, France, "Nanoplasmonics and nanophotonics with electrons"

24 Mar. 2010, Alberta BONANNI,

Institut für Halbleiter- und Festkörperphysik, Universität Linz, Austria, "High-Tc magnetic semiconductors"

#### 15 Apr. 2010, Mario STRASSER

Oxford Instruments GmbH, Wiesbaden, Germany, "Großflächige Einzel-Siliziumdriftdetektoren für die EDS Analytik am REM und TEM"

30 Jun. 2010, Masashi WATANABE

Lehigh University Betlehem, Pennsylvania, USA, "Atomic resolution chemical analysis in aberrationcorrected STEM"

#### 1 Oct. 2010, Gerasimos DANILATOS

ESEM Research Laboratory, North Bondi, Australia, "Implementations of ESEM and its overdue promise"

20 Oct. 2010, Merle HURRLE

BMW AG, München, Germany, "Ferromagnetische Pulverwerkstoffe"

#### 22 Oct. 2010, Andreas SCHÖNBERGER

BMW AG, München, Germany, "Prognose des Steinschlags und der induzierten Korrosion am Fahrzeug"

3 Dec. 2010, Jürgen STAMPFL

Institut für Werkstoffwissenschaft und Werkstofftechnologie, TU Wien, Austria, "Materialentwicklung für die lithographie-basierte generative Fertigung"



Dr. Gerasimov Danilatos presenting his talk in the Institute.

#### Speakers 2011

28 Jan. 2011, Christian TEICHERT

University of Leoben, "Nanostructure characterization by atomic-force microscopy and related techniques"

#### 29 Mar. 2011, Frank ALTMANN

Frauenhofer Institut für Werkstoffmechanik, Halle, "Effiziente FIB-Zielpräparationsverfahren f. die höchstauflösende Transmissionselektronenmikroskopie"

7 Apr. 2011, Jannik MEYER, University of Vienna, "The physics of nanocarbons explored by atomic resolution TEM"

6 May 2011, Gunther FIGNER (STIRZONE and Graz University of Technology), "Friction Stir Spot Welding"

20 Mai 2011, Peter RÖDIGER

Vienna University of Technology, "Hydrocarbonace-
ous contamination in SEMs: Evaluation and removal"

8 Jun. 2011, Andreas KENDA, Gudrun BRUCKNER CTR Carinthian Tech Research AG, Villach, "Photonische Mikrosysteme für die Analytik und Wireless SAW Sensoren"

10 Jun. 2011 Katrin REINKE

University of Halle-Wittenberg, Halle, "Investigation of the mechanical properties of polymeric films"

22 Jun. 2011, Christian COLLIEX

University of Paris-Sud, Orsay, France, "Using the fine electron probe of a (corrected) STEM to explore the nanoworld"

22 Jun. 2011, Ulrich DAHMEN

National Center for Electron Microscopy, Lawrence Berkeley Laboratory, USA, "Electron microscopy's answer to the Feynman challenge"

22 Jun. 2011, Christoph MITTERBAUER FEI Company Eindhoven, Nederlands) "Titan G2: Taking the Sub-Ängström era to new limits in material science"

11 Jul. 2011, Bernhard SCHAFFER University of Glasgow and SuperSTEM, Daresbury U.K., "SuperSTEM laboratory, atomic resolution EELS mapping in an aberration corrected STEM at 100 kV"

14 Oct. 2011, Zaoli ZHANG

Erich-Schmied-Institute for Material Science, Leoben, "Advanced characterization of interface structure by Cs-corrected HR-TEM"

7 Nov. 2011, Andreas SCHÖNBERGER BMW AG München, "Puls Phasen Thermographie von Steinschlagbeschädigungen und Korrosion"

11 Nov. 2011, Helmut GNAEGI Diatome, Switzerland, "Das Schneiden von spröden und weichen industriellen Proben"

25 Nov. 2011, Günter RESCH Institute for Molecular Biotechnology GmbH, Vienna, "Cryo-electron tomography"

6 Dec. 2011, Velimir RADMILOVIC University of Belgrade, Serbia, "Electron Microscopy and Spectroscopy of  $L1_2$  complex nanostructures"



Prof. Velimir Radmilovic and Werner Grogger at the AS-TEM.



Boril Chernev and Peter Wilhelm in the Institute seminar.

## Master & Doctoral Theses at the Institute

# Finished master theses

DOHR Judith (2011), Enzymatic degradation of superflat cellulose surfaces (in cooperation with University of Graz).

FLADISCHER Stefanie (2009), New methods of quantitative X-ray analysis in a transmission electron microscope.

HAYNL Ines (2010), Analysis of soft materials using a new cryo atomic force microscope combined with an ultramicrotome.

JANTSCHER Klemens (2011), Crystal structure determination in the scanning electron microscope: fundamental and experimental problems.

KREMSHOFER Eva (2011), TEM investigation of the structural and chemical order of lead-free piezoelectric alkali bismuth titanates.

MICHELITSCH Stefan (2011), Electrical in-situ measurements during electron beam induced platinum deposition.

NEUMAYER Sabine (2011), Wedge Polishing as sample preparation method for transmission electron microscopy.

SCHMIED Roland (2011), Fundamental proximity effects for electron beam induced deposition processes.



Students work with Armin Zankel at the ESEM Q600.

#### Master theses in progress

GANNER Thomas, Liquid cell investigation of enzymatic cellulose degradation.

#### Finished PhD theses

GSPAN Christian, Dipl.-Ing. (2011), Investigation of the superstructure and the domains in the perows-kite  $La_{0.4}Sr_{0.6}CoO_{2.71}$  by transmission electron microscopy.

RATTENBERGER Johannes, Dipl.-Ing. (2010), Improved understanding of scattering processes and their meaning for quantitative image interpretation in scanning electron microscopy.

SEZEN Meltem (2009), Nanostructuring and modification of conjugated based optoelectronic device structures by focused ion beam.

ZANKEL Armin, Dipl.-Ing. (2009), In-situ investigations of polymeric materials in the environmental scanning electron microscope.

#### Doctoral theses in progress

FLADISCHER Stefanie, Dipl.-Ing., Application of new EDXS quantification schemes in TEM on organic semiconducting devices.

HAAS Wernfried, Dipl.-Ing,, Morphology control of high-performance polymer solar cells.

MEINGAST Arno, Dipl.-Ing., STEM investigations of GaN-based semiconductors.

REINGRUBER Herbert, Dipl.-Ing., In-situ experiments with an environmental scanning electron microscope.

SCHMIDT Franz, Dipl.-Ing., Mapping surface plasmons of designed metal nanostructures with an electron beam (in cooperation with University of Graz).

SCHMIED Roland, Dipl.-Ing., Focused Ion Beam Processing of Soft Matter.

UUSIMÄKI, Toni, Mag., Nanotomography in the Transmission Electron Microscope.

# Master and Doctoral Students from other Institutes

Graz University of Technology

# Faculty of Technical Mathematics and Technical Physics

Institute of Solid State Physics Tatjana DJURIC, PhD thesis Piet REUTER. PhD thesis Lisbeth KAPPEL, PhD thesis Wolfgang FISCHER, PhD thesis Nora MAYRHOFER, Master thesis Christian GRUBER. Master thesis Wolfgang FISCHER, PhD thesis Markus POSTL, Master thesis Andreas HIRZER. Master thesis Barbara LENDL, Master thesis Alfred NEUHOLD, PhD thesis Marco MARCHL, PhD thesis Kerstin SCHMOLTNER, PhD thesis Alexander BLÜMEL, PhD thesis Roman TRATTNIG, PhD thesis Christoph AUNER, PhD thesis

- Institute of Experimental Physics Andreas SANBACH, Master thesis Alexander VOLK, PhD thesis
- Institute of Materials Physics Andreas KAUTSCH, Master thesis Mario ARAR, Master thesis Thomas TRAUSZNIG, PhD thesis Bernd OBERDORFER, PhD thesis Eva-Maria STEYSKAL, PhD thesis

### Faculty of Chemistry, Engineering and Biotechnology

Institute for Chemistry a. Technology of Materials Bernadette ALLNOCH, Master Thesis Cornelia BAYER, PhD thesis Ute DASCHIEL, PhD thesis Michael EDER, PhD thesis Achim FISCHEREDER, PhD thesis Christopher FRADLER, PhD thesis Verena KALTENHAUSER, PhD thesis Kathleen KIRSTEIN, PhD thesis Harald KREN, PhD thesis Eugen MAIER, PhD thesis Angelika PATETER, Master thesis Marta PAWLAK, PhD thesis

Andreas PEIN, PhD thesis Alejandro SANTIS, Master thesis Alexander SCHENK. Master thesis Elisabeth STRUNZ, Master thesis Elisabeth ZIEGLER, PhD thesis Institute of Analytical Chemistry and Food Chemistry Günter MISTLBERGER, PhD thesis Elisabeth SCHEUCHER, Master thesis Stefan KOREN, Master thesis Institute of Inorganic Chemistry Kathrin SCHITTELKOPF, PhD thesis Dominik GENSER, PhD thesis Johanna FLOCK, Master thesis Judith BINDER, Master thesis Stefan PADLESAK, Master thesis Institute of Environmental Biotechnology Stefan Weiß, PhD thesis Konstantin SCHNEIDER, PhD thesis Institute of Paper, Pulp and Fibre Technology Johannes KRITZINGER. PhD thesis Harald SCHÄFFER, PhD thesis Institute of Chemical Engineering and Environmental Technology Munazza Mohsin, PhD thesis Markus PERTHALER, PhD thesis Gerd RABENSTEIN, PhD thesis Astrid STADLOBER, Master thesis Nikolaus SCHWAIGER. PhD thesis Eva WALLNÖFER, PhD thesis Institute of Process and Particle Engineering Eva-Maria LITTRINGER, PhD thesis Sabrina SCHNEPFLEITNER, PhD thesis Jakob REDLINGER-POHN, Master thesis Georg LICHTENEGGER, Master thesis Sarah ZELLNITZ, Master thesis Markus MAIER, Master thesis Daniela STROHMEIER. Master thesis

## Faculty of Mechanical Engineering and Economic Sciences

Institute of Materials Science and Welding Francisca MENDEZ-MARTIN, PhD thesis Thomas WEINBERGER, PhD thesis Saeid SABERI, PhD thesis Asmir KUDUZOVIC, PhD thesis Friedrich KRUMPHALS, PhD thesis Alexander TIMOSHENKOV, PhD thesis Mizanur Rachman, PhD thesis Christian SCHLACHER, PhD thesis Rene RADIS, PhD thesis Suleiman SCHWERWAN, Master thesis F. WIRNSBERBER, Master thesis Bernhard THOMAS, Master thesis Christian PFEIFFER, Master thesis Johannes WINKELHOFER, Master thesis Bernhard BERGER, Master thesis

- Institute of Tools and Forming Robert VOLLMER, Master thesis
- Institute of Thermal Engineering Bernhard GATTERNIG, PhD thesis Michael WOHLMUTHER, Master thesis Thomas KIENBERGER, PhD thesis

#### **Faculty of Informatics**

Institute of Computer Vision and Graphics Peter KONTSCHIEDER, Master thesis

### Faculty for Electrical and Information Engineering

Institute of High Voltage Engineering and System Management

Denis IMANOVIC, PhD thesis Jürgen FABIAN, PhD thesis Thomas Berg, PhD thesis

Institute of Genomics and Bioinformatics Michael KARBIENER, PhD thesis

#### Faculty of Civil Engineering

Institute of Hydraulic Engineering and Water Resources Management

Wolfgang DOBLER, PhD thesis

Institute of Applied Geosciences Andrea NIEDERMAYR, PhD thesis

Bioenergy 2020+ Stefan MARTINI, PhD thesis

#### University of Graz

Institute of Pharmaceutical Sciences Sabrina WEBER, PhD thesis Christina PETSCHACHER, PhD thesis Institute of Zoology Tobias PFINGSTL, PhD thesis Anna TRUCK, Master thesis Institute of Chemistry Doris BREITWIESER, PhD thesis Bernhard GUTMANN, PhD thesis Martin DULLE, PhD thesis

Institute of Physics Nadeem KASHIF, PhD thesis Andreas TRÜGLER, PhD thesis Judith DOHR, Master thesis

#### Joanneum Research

Andreas HIRZER, PhD thesis Thomas ROTHLÄNDER, PhD thesis Johanna KRAXNER, Master Thesis Andreas PETRITS, Master Thesis Julien MAGNIEN, Master Thesis Philipp HÜTTER, Master Thesis

#### Medical University of Graz

Division of Preventive and Operative Dentistry Daniela PRAUSE, PhD thesis

Institute of Cell Biology, Histology a. Embryology Stefanie KRASSNIG, Master thesis

Institute of Pathophysiology and Immunology Victor AGUIRIANO-MOSER, Master thesis

#### University of Leoben

Department of General, Analytical and Physical Chemistry

Robert SONNLEITNER, PhD thesis Manuela PROHASKA, PhD thesis Johannes ZAUNER, PhD thesis Clemens VICHYTIL, PhD thesis

Chair of Chemistry of Polymeric Materials Nina MUHR, PhD thesis Mathias EDLER, PhD thesis

Chair of Materials Science a. Testing of Polymers Michael SEEMANN, Master thesis

Chair of Functional Materials a. Materials Systems

Christian TRITREMMEL, PhD thesis Viktoria EDLYMAYR, PhD thesis Markus POHLER, PhD thesis Marlene MÜHLBACHER, PhD thesis Thomas WEIRATHER, PhD thesis

Vienna University of Technology

Institute of Materials Science and Technology Klaus CICHA, PhD thesis Aleksandr OVSIANIKOV, PhD thesis

#### University of Vienna

Department of Pharmaceutical Technology and Biopharmaceutics Viktoria KLANG, PhD thesis Julia SCHWARZ, PhD thesis

### - Johannes-Kepler University of Linz

Institute of Semiconductor a. Solid State Physics Tanweer Ashraf, PhD thesis Christian Gusenbauer, Master thesis

- University of Connecticut, USA
  School of Engineering
  Jonathan WINTERSTEIN, PhD thesis
- University of Marburg, Germany

Institute of Chemistry Tibor MÜLLER, PhD thesis

Munich University of Technology, Germany

Department for Materials and Manufacture Merle HURRLE, PhD thesis

#### University of Erlangen, Germany

Department for Materials Science a. Engineering Andreas SCHÖNBERGER, PhD thesis

 Chalmers University of Technology, Sweden

Department of Applied Physics Jenny ANGSERYD, PhD thesis

Tel Aviv University, Israel School of Chemistry Assaf BEN MOSHE, Master thesis



Johnathan Winterstein (Fulbright-Award) with Werner Grogger at the microscope.



Dr. Christian Gspan after his PhD rigorosum, May 2011.

# Events and Awards

# **Events**

### Microscopy Conference 2009 in Graz

Joint Meeting of

9<sup>th</sup> Multinational Conference on Microscopy & Dreiländertagung für Elektronenmikroskopie

The FELMI-ZFE organized the Microscopy Conference 2009 in Graz in cooperation with the "Austrian Society for Electron Microscopy". The conference was held in the Congress Graz from 30 August 30 – 4 September 2009. MC 2009 was joining up the "Multinational Conference on Microscopy" and the "Dreiländertagung für Elektronenmikroskopie" both having established a strong reputation as key events of the European and international microscopy communities.

The program committee set up a scientifically stimulating, future oriented program in the three main areas of instrumentation and methodology, materials science and life sciences. The program comprised plenary lectures, keynote lectures, poster sessions, symposia and workshops (650 scientific presentations). Special emphasis was given on the importance of the poster sessions.

During the whole conference there was a high quality Trade Exhibition with 42 companies in the same building adjacent to the lecture halls displaying modern instruments and state-of-the-art developments in microscopy of the physical and life sciences and nanotechnology. Together with the exhibitors' presentations this was an important highlight of the conference.

The local organization staff of 35 people from the Universities in Graz could welcome 982 registered participants from 38 countries (from USA to Australia) including 211 students.

International Scientific Advisory Board:

Marie Cheynet (Grenoble) Christian Colliex (Paris) Aleksandra Czyrska-Filemonowicz (Kraków) Bruno Humbel (Utrecht) John Hutchison (Oxford) Jaakko Saraste (Bergen) Dominique Schryvers (Antwerp) José María Valpuesta Moralejo (Madrid)



Program Comitee:

Roberto Balboni (Bologna) Marco Cantoni (Lausanne) Miran Čeh (Ljubljana) Fedor Ciampor (Bratislava) Elisabetta Falcieri (Urbino) Srećko Gajović (Zagreb) Jasmina Grbović Novaković (Belgrade) Pavel Hozák (Prague) H. Peter Karnthaler (Vienna) Agnes Kittel (Budapest) Helmut Kohl (Münster) Aleksandra Korać (Belgrade) Zoltan Kristof (Budapest) Amelia Montone (Roma) Margit Pavelka (Vienna) Reinhard Rachel (Regensburg) Rok Romih (Ljubljana) Anđelka Tonejc (Zagreb) Roger Albert Wepf (Zürich)

The local organization team came from the Graz University of Technology, the University of Graz and from the Medical University of Graz:

Ferdinand Hofer (TU Graz, Chairman) Werner Grogger (TU Graz) Gerald Kothleitner (TU Graz) Peter Pölt (TU Graz) Manfred Leisch (TU Graz) Maria-Anna Pabst (MedUni Graz) Günther Zellnig (KFU Graz)



Rector Hans Sünkel opens the MC 2009



#### Joint Meeting of

on the Schlossberg

## Long Night of Research 2009

"What can we see with the electron micrscope?" 7 November 2009

The electron microscope is the big sister of the light microscope and enables the study of smallest structures in natural and artificial objects – even down to atomic dimensions. More than 200 visitors in 5 hourswere able to gain special insights into the exciting world of the micro- and nanocosmos which is not visible to the naked eye.



Herbert Reingruber presenting the ESEM Quanta 600F.

# Vienna TEC 2010

"Multi-material bonds", 12-15 October 2010

Four ACR institutes presented the results of the research project "Multi material bonds" at Austria's most important industry fair in Vienna. There was great interest in the ACR expertise in functional material bonds in the automotive industry.



### Research 2010

Exhibition for Science, Research and Innovation from 11-12 June 2010

The "Research 2010" congress at the Messe Graz brought together university scientists, companies and research organizations. The Institute presented new research results at an information stand booth which was organized in cooperation with ÖGI Leoben and Austrian Cooperative Research.



Deputy Governor of Styria Hermann Schützenhöfer visiting the ACR booth (left Ferdinand Hofer, right Gerhard Schindelbacher, ÖGI Leoben).



The ACR team celebrating the successful presentation.

The ACR stand: Julian Wagner, Gerhard Weiner, Karl Wizany, Bernd Panzirsch, Gerhard Schindelbacher (from left).

# FELMI-ZFE at the "Ball der Technik"

#### 28 January 2011

The institute had an information stand at the Ball der Technik in the Congress Graz. Many guests of the ball were able to gain an impression of the amazing micro- and nanocosmos. The stand was organized by Margit Wallner and Martina Dienstleder.



Rector Prof. Hans Sünkel, Prof. Helmut List and Kathryn List visiting the Institute's information stand (from left, with Ferdinand Hofer).

# Exhibition "Microworlds-Nanoworlds"

#### at the Greith-Haus in St. Ulrich am Greith.

People are increasingly interested in the amazing landscapes and the hidden beauty of the micro- and nanoworld. The institute's microscopists therefore selected some of the most spectacular micrographs from the thousands of images which are recorded every year in the lab for presentation at the exhibition. The exhibition, which was open from 11 February to 13 March 2011. The exhibition was sponsored by companies and the organization was organized by Margit Wallner, Armin Zankel (both Graz University of Technology) and Helena Wallner from the Greith-Haus.





Exhibition of images at the Greith-Haus in St. Ulrich am Greith, Styria

### 1<sup>st</sup> ASEM Workshop 2011 "Advanced Electron Microscopy"

#### 7-8 April 2011

Graz University of Technology

Workshop Organizer: Ferdinand Hofer and Gerald Kothleitner (Graz University of Technology), Margit Pavelka (Medical University Vienna)

The purpose of the workshop was to create an informal platform for those who are interested in electron microscopy and its applications in modern science and technology. More than 50 scientists from all fields of microscopy in biological sciences, materials and physical sciences participated in the workshop held in Graz. In addition to the 20 contributed talks, two guest speakers gave an overview of the state of the art of electron microscopy.

Reinhard RACHEL, "Cell biology of Ignicoccus hospitalis – a unique Crenarchaeon", University of Regensburg, Germany.

Jannik MEYER, "The physics of nano-carbons explored by atomic resolution transmission electron microscopy", University of Vienna, Austria

For further information, please go to www.univie.ac.at/asem



Dr. Reinhard Rachel (Regensburg) giving his presentation.

#### 60 Years Electron Microscopy in Graz

On 22 June 2011 we celebrated the Institute's 60<sup>th</sup> anniversary with a symposium held in the auditorium of Graz University of Technology – almost exactly 60 years after the inauguration of the first electron microscope in Styria on 25 June 1951.

More than 140 participants joined the meeting, which was combined with the inauguration of the "Austrian Scanning Transmission Electron Microscope - ASTEM".

The anniversary was honored by speeches given by the Rector of the Graz University of Technology, Prof. Dr. Hans Sünkel, by the President of the Association, Prof. Helmut List and by the President of Austrian Cooperative Research, Dipl.-Ing. Martin Leitl.

Dr. Birgit Strimitzer from the Styrian Government of and the Dean of the Faculty of Technical Mathematics and Technical Physics, Prof. Dr. Wolfgang Ernst presented additional aspects of electron microscopy in Styria.



Well-known plenary speakers offered exciting insights into the amazing world of electron microscopy:

Dr. Ulrich Dahmen, Director of the National Center for Electron Microscopy (NCEM) at the Lawrence Berkeley Laboratory in Berkeley (USA) presented a talk entitled "Electron microscopy's answer to the Feynman challenge" and Professor Dr. Christian Colliex from the University of Paris-Sud in Orsay (France) spoke about "Using the fine electron probe of a (corrected) STEM to explore the nanoworld".

Dr. Christoph Mitterbauer from FEI Company Eindhoven (The Netherlands) described the great potential of the new microscope Titan G2, which is the basis for the ASTEM.



Rector Prof. Hans Sünkel with the Vice-President of the Association, Dipl.-Ing. Ulrich Santner at the press conference.



The Symposium in the auditorium of the Graz University of Technology



The President of the Association, Prof. Helmut List presenting his talk.



The President of the ACR, Dipl.-Ing. Martin Leitl giving his presentation.



Dr. Ulrich Dahmen from the NCEM in Berkeley presents "Electron microscopy's answer to the Feynman challenge".



Tour of the ASTEM in the basement of Steyrergasse 17.

# The RFTE visits the Institute

On 7 October 2011, Dr. Hannes Androsch, chairman of the Austrian Council for Research and Technology Development (RFTE), made a visit to the most powerful electron microscope in Austria. He met with the Rector of the Graz University of Technology, Professor Harald Kainz in order to discuss the Austrian situation with regard to scientific infrastructure.

The RTFE delegation was accompanied by Dr. Hans Jäger (ACR) and took the opportunity to look down the "Austrian Scanning Transmission Electron Microscope" in the FELMI-ZFE.

Dr. Androsch was impressed with this new national facility for advanced materials characterization and the way it is organized to support researchers from universities and industry.



Dr. Gerald Kothleitner, Dr. Hannes Androsch, Prof. Dr. Harald Kainz, Dr. Ferdinand Hofer and Dr. Hans Jäger taking a look at the ASTEM microscope (from left).

# **Agilent Workshop**

#### "Imaging Beyond Morphology"

17 November 2011, Graz University of Technology

"Recent advances in nanoscale imaging"

In cooperation with Agilent Technologies, Harald Plank organized a one-day workshop held at the Graz University of Technology. Several advanced scanning probe and electron microscopy techniques, technologies and applications were discussed by speakers from the Graz University of Technology and Agilent Technologies.

More than 40 attendees had the opportunity to participate in hands-on sessions using a new AFM and a compact low-voltage FE-SEM system.

#### Speakers:

Dr. Harald PLANK (Graz University of Technology), "Beyond morphology"

Prof. Dr. Bernd NIDETZKY (Graz University of Technology), "Visualization in biotechnology"

Dr. Gerald KADA (Agilent), "Kelvin force microscopy"

Dr. Johannes RATTENBERGER (ZFE Graz), "Lowenergy electron microscopy: option or demand?"

Dr. Natascha NIERMANN (Agilent), "A new, compact FE-SEM"

# FELMI-ZFE Calendar 2010

Based on an internal image competition, the best micrographs are chosen once a year and published in the Institute's biannual calendar. The calendar for 2010 highlights micrographs from the world of crystals.



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# Awards

### The Fritz Grasenick Prize of the Austrian Society for Electron Microscopy (ASEM)

Since 2007 the Fritz Grasenick Prize has been awarded by the Austrian Society for Electron Microscopy for scientific achievements by young researchers in electron microscopy.

The prize is presented by the Association to honour the memory of Dr. Fritz Grasenick\* (1916-2003), the founder of the Graz Centre for Electron Microscopy.

The following scientists have received the Fritz Grasenick Prize:

2007 Dr. Sylvia NÜRNBERGER (Medical University of Vienna)

Dr. Fernando Aldrian LASAGNI (Vienna University of Technology)

Dr. Gerd LEITINGER (Medical University of Graz)

2008 Dr. Bernd ZECHMANN (University of Graz)

- 2010 Dipl.-Ing. Dr. Johannes RATTENBERGER (Graz University of Technology)
- 2011 Dipl.-Ing. Christoph GAMMER (University of Vienna)

Dipl.-Ing. Stefan LÖFFLER (Vienna University of Technology)

\* more information: www.wikipedia.de



Johannes Rattenberger being presented with the Fritz Grasenick Prize 2010 at the University of Vienna

# Friedrich Schmiedl Research Award for Nanotechnology 2009

Evelin FissIthaler was awarded the prize for her PhD thesis entitled "Structuring Methods for Conjugated Polymers and their Applicability regarding Micro- and Nano-Patterning of Polymer Light Emitting Devices" which was written at the Institute of Solid State Physics (Graz University of Technology).

### **Best Poster Award 2009**

Herbert Reingruber won the award for the best poster presentationat the Microscopy Conference 2009 in Graz on 4 September 2009. The poster was entitled "The ESEM and Water – Prospects and Limits".

# ACR Woman Award 2010 for Nadejda Matsko

The aim of the award is to raise awareness and critically comment on the gender situation in ACR institutes. The award for female ACR scientists was presented for the first time in 2010 to the physicist Nadejda Matsko from ZFE Graz for her development of a new 3D cryo-AFM\*. It was presented during the ACR Conference together with the Federal Ministry of Economy, Family and Youth (BMWFJ) on 27 October 2009.





SC Dr. Michael Losch (BMWFJ) and ACR President Dipl.-Ing. Martin Leitl presenting the award to Nadejda Matsko

# Harald Plank wins the Grand Prize at the FEI Image Contest

in the category Technical Merit for the image "Platinum nanorods on silicon", 16 February 2010. The image shows an array of freestanding Pt nanorods on silicon fabricated by electron beam induced deposition from the gas phase (in the DualBeam FIB Nanolab Nova 200F). The individual rods have a base diameter of about 80 nm and a height of 1  $\mu$ m. The wavy appearance was actually an artefact but too nice to be ignored (colorized by Margit Wallner).



# Third place of the Grand Prize

in the 2011, FEI Owner Image Contest for **Angelika Reichmann** for her entry "Lamnacarus ornatus" (forepart of a mite), captured with a ESEM Quanta 600F (colorized by Margit Wallner).



# Grand Prize for "Microcanyon" by Martina Dienstleder

In April 2011, FEI announced that **Martina Dienst-Ieder** of the ZFE Graz had been awarded the Grand Prize in the 2011 FEI Owner Image Contest for her entry "Microcanyon".

The image was selected by FEI experts from more than 180 entries. Overall, the entries were judged on their aesthetic appeal, application and scientific relevance, and overall creativity. Martina's entry shows a micro-crack in steel after bending tests and the resulting image inspired Manual Paller to colorize the micrograph, creating an amazing likeness of a canyon.



Grand Prize for "Microcanyon" image, captured on an FEI Nova Dual Beam FIB



Martina Dienstleder and Manuel Paller

More information on www.fei.com and www.flickr.com

### Poster Prize for Armin Zankel and Herbert Reingruber

for their presentation "Morphology Characterization of Particle Filled Polymers by Tomographic Methods", presented at the ASPM Conference in Leoben, Austria, 8-10 September 2010.

### ZFE wins the Carbonium Prize presented by ACR

Every year ACR presents the Carbonium Prize to those members who, over the previous year, contributed most towards internal networking and communication of the ACR umbrella brand to people outside ACR. For the 2009 reporting year, ZFE Graz won the Carbonium Prize, which was presented by ACR President Dipl.-Ing. Martin Leitl in the Haus der Forschung in Vienna on 29 April 2010.



Carbonium Prize 2009 was presented to the ZFE Graz

# 2<sup>nd</sup> Prize Image Contest Nadejda Matsko

at the International Microscopy Congress IMC 17 in Rio de Janeiro, Brazil, 17- 24 September 2010.



# ACR Cooperation Award 2011 presented to ZFE Graz

The ACR Cooperation Award underlines how successfully and efficiently SMEs can collaboratewith research institutes. In 2011, the award was presented to the ZFE and Bionic Surface Technologies for research into a new high-tech foil for application in aeroplanes, boats and wind power plants. The award was presented by Federal Minister Dr. Reinhold Mitterlehner (BMWFJ) at the ACR Congress in Vienna on 17 October 2011.



ACR President Dipl.-Ing. Martin Leitl, Federal Minister Dr. Reinhold Mitterlehner, the bionic team and Ing. Hartmuth Schröttner from the ZFE (from left).

# Publications

# **Publications**

Although the institute is not only a pure research institute, but also devoted to teaching activities and service research, the number of peer reviewed publications has arrived at a high level and the number of citations is steadily rising.

These are good figures, which have to be increased especially with publications in high ranking journals, such as ACS Nano, Chem.Commun. Angewandte Chemie, Ultramicroscopy, Advanced Materials, Soft Matter etc..



Development of poster presentations and oral presentations (conferences, workshops, etc.) of FELMI-ZFE staff and of non peer-reviewed publications of the institute.



FELMI-ZFE Publications from ISI Web of Knowledge



Gerald Kothleitner is the guest editor for the proceedings

scopy ZFE



Citations of FELMI-ZFE Publikations from ISI Web of Knowledge

Development of of peer-reviewed publications of the FELMI-ZFE and citations of these publications taken from the ISI Web of Knowledge.

of EDGE 2009.

#### **Peer Reviewed Publications 2009**

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#### Lectures 2009

F. Hofer (invited), "High resolution imaging of surface plasmons by transmission electron microscopy", Symposium "Nano and Photonics", Mauterndorf, Austria, 12 Mar. 2009.

F. Hofer, "Elektronenmikroskopie in Material- und Biowissenschaften", Ingenieur- u. Architektenverein, University of Salzburg, Austria, 2 Mar. 2009.

H. Plank, "Controlling structure, chemistry and morphology of free standing platinum nanorods via process parameters during electron beam induced deposition", MRS Spring Meeting 2009, San Francisco, 12 - 20 Apr. 2009.

S. Mitsche, "Gefügecharakterisierung der Superlegierung Allvac 718 PlusTM mittels elektronischer Methoden", 55. Metallkundekolloquium, Lech, Austria, 15 -17 Apr. 2009.

J. Wagner, "Advanced analytical methods for high resolution characterization of thin layers", Seminar "Topics in applied electrochemistry", Wiener Neustadt, Austria, 23 April 2009.

M. Albu, "Steel related Analytics – Possibilities and Limitations at FELMI", Institut für Werkstoffkunde und Schweißtechnik, TU Graz, Austria, 28 Apr. 2009.

J. Wagner (invited), "3D Elemental Mapping in a Dual Beam System", Helmholtz-Zentrum Berlin für Materialien und Energie, Berlin, Germany, 4 May 2009.

F. Hofer (invited), "High resolution imaging of surface plasmons by energy-filtering transmission electron microscopy", EDGE 2009, Banff, Canada, 17 - 22 May 2009.

G. Kothleitner, "Band-gap mapping with a monochromated EF(S)TEM", EDGE 2009, Banff, Canada, 17 - 22 May 2009.

G. Kothleitner (invited), "Accurate chemical shift measurements using a post-column spectrometer with an electrostatic shutter", EDGE 2009, Banff, Canada, 17 - 22 May 2009.

F. Hofer (invited), "Quantitative low-loss spectroscopy", European Workshop on advanced TEM measurement techniques for materials science, St Aygulf, France, 24 -27 May 2009.

M. Albu (invited), "Comprehensive TEM nano-analytics in steels", Institut für Allgemeine Physik, Vienna University of Technology, Austria, 9 Jun. 2009.

G. Kothleitner (invited), "Low-loss EELS measurements for bandgap and plasmon analysis", French Society of Microscopy, Paris, France, 22 - 27 Jun. 2009.

P. Pölt (invited), "A tensile stage in the ESEM", Seminar "Deformation und Bruchverhalten von Kunststoffen", Merseburg, Germany, 23 - 27 Jun. 2009.

M. Dienstleder, "The effect of FIB process parameters on the surface morphology of thin lamellas", Dreiländer-Arbeitskreis FIB, Halle; Germany, 29 Jun. - 1Jul. 2009.

H. Plank, "How to Maximize the Volume Growth for Electron Beam Induced Deposition Processes", Dreiländer-Arbeitskreis FIB, Halle; Germany, 29 - 30 Jun. 2009.

B. Chernev, "Monitoring of the artificial weathering of PVC formulations with FTIR spectroscopy", APST ONE, Johannes-Kepler-Universität Linz, Austria, 8 -10 Jul. 2009.

E. Fisslthaler, "Structuring methods for conjugated polymers and their applicability regarding micro- and nanopatterning of polymer light emitting devices", European Polymer Conference EPF09, Graz, Austria, 12-17 Jul. 2009.

H. Reingruber, "New micro scale methods for the Characterization of fluid and gas transport through membranes", ICAPM 2009, Istanbul, Turkey, 9-10 Aug. 2009.

N. Matsko (invited), "Structural and mechanical characterization of chitin using correlative AFM-TEM microscopy and spectroscopy", University of Ljubljana, Department of Biology, 19-20 Aug. 2009.

N. Matsko, "Structural and mechanical characterization of chitin using correlative AFM-TEM microscopy and spect-roscopy", Microscopy Conference 2009, Graz, 30 Aug. - 4 Sep. 2009.

J. Rattenberger, "Nonlinear material contrast in low voltage backscatter electron images", Microscopy Conference 2009, Graz, 30 Aug. - 4. Sep 2009.

J. Wagner, "Fast automated 3D EDXS Mapping", Microscopy Conference 2009, Graz, Austria, 30 Aug. - 4 Sep. 2009.

H. Reingruber, "The ESEM and water - prospects and limits", Materials Day, TU Graz, 1 Oct. 2009.

T. Uusimäki, "Electron tomography", Materials Day, TU Graz, 1 Oct. 2009.

H. Plank, "Growth characteristics of electron beam induced deposition", Nanogrowth Workshop, University of Leoben, Austria, 2 Oct. 2009.

G. Kothleitner (invited), "High-resolution EELS measurements for the mapping of direct bandgap semiconductors", 216<sup>th</sup> ECS Electrochemical Society Meeting, Vienna, 6-7 Oct. 2009.

M. Albu (invited), "Dual EELS and new frontiers in analyzing ceramic materials", School & Workshop on Electron Microscopy of Ceramic Materials (EMCM), Eskisehir, Turkey, 8-13 Oct. 2009. P. Pölt (invited), "In -situ SEM experiments in the ESEM", School & Workshop on Electron Microscopy of Ceramic Materials (EMCM), Eskisehir, Turkey, 8-13 Oct. 2009.

W. Grogger (invited), "Energy-filtering transmission electron microscopy", Workshop on Quantitative HAADF-STEM Imaging and EELS, Piran, Slovenia, 13-15 Oct. 2009.

W. Grogger (invited), "Advanced nanoanalysis in the transmission electron microscope", Electron Microscopy and Multiscale Modelling – Fall 2009, Zürich, Switzerland, 27-30 Oct. 2009.

H. Plank, "Nano-Biosensors for Early Cancer Diagnostics", Round Table Discussion – South Korea, Joanneum Research, Graz, 28 Oct. 2009.

F. Hofer, "Angewandte Nanoanalytik für die Wirtschaft", 3. Weizer NanoBusinessTalk, Weiz, Austria, 11 Nov. 2009.

G. Kothleitner (invited), "Low-loss EELS Measurements: Instrumentational Aspects and Applications", LESS 2009, Vienna, 12 Nov. 2009.

S. Fladischer, "First SEM, AFM and TEM results of the layers of an organic photodiode", ISOTEC-Veranstaltung, Reinischkogel, Austria, 19 Nov. 2009.

#### Lectures 2010

G. Kothleitner (invited), "Advances in EELS and electron spectroscopic imaging for low energy-losses", University of Ulm, Germany, 11 Jan. 2010.

N. Matsko, "Cryo-Microscopy in Chemistry and Life Science", Institute of Chemistry, University of Graz,15 Jan. 2010.

N. Matsko, "Structural characterization of chitin using correlative AFM-TEM microscopy and spectroscopy", Beyond Self-Assembly Workshop 2010, Bad Gastein, Austria, 23-27 Jan. 2010.

T. Haber, "Etch-free nano-imprint-lithography for organic electronic devices", Winterschool on Organic Electronics 2010, Donnersbach, Austria, 6-12 Mar. 2010.

F. Hofer (invited), "Electron-energy loss spectrometry of plasmonic nanostructures", Festsymposium "Frontiers in EELS", Vienna University of Technology, 10-12 Mar. 2010.

G. Kothleitner (invited), "Advances in EELS electron spectroscopic imaging for low energy-losses", CD Labor für oberflächenoptische Methoden, Johannes-Kepler University Linz, Austria, 15 Mar. 2010.

F. Hofer (invited), "Neue Anwendungen der Elektronenmikroskopie in der Materialforschung", Seminar "Moderne Analytische Chemie", Institut für Chemische Technologie und Analytik, Vienna University of Technology, Austria, 16 Mar. 2010.

W. Grogger, P. Pölt, "Neue mikroskopische Methoden für

die Materialanalytik", Plansee, Reutte, Austria, 13 Apr. 2010.

A. Meingast, "TEM investigations at FELMI", NSI-Workshop, Johannes-Kepler University of Linz, Austria, 28 May 2010.

F. Hofer (invited), "High spatial-resolution analysis by energy-filtering TEM", Ann. Meeting of the Israel Society for Microscopy, Tel Aviv, Israel, 31 May 2010.

F. Hofer (invited), "Monochromated EELS and EFTEM of nanostructured materials", Israel Institute of Technology, TECHNION, Haifa, Israel, 3 Jun. 2010.

M. Albu, "EELS-Spectrum Imaging-extended analysis for composite materials", European Materials Research Society Spring Meeting 2010, Strasbourg, France, 6-11 Jun. 2010.

G. Kothleitner (invited), "Materials science applications with a new electron energy-loss spectrometer", European Materials Research Society Spring Meeting 2010; Strasbourg, France, 6-11 Jun. 2010.

F. Hofer, "Mikroskopische Nanoanalytik", Research 2010, Messe Graz, Austria, 11-12 Jun. 2010.

F. Hofer (invited), "The advanced sensitivity of EFTEM", Jaszowiec International School & Conference on Physics of Semiconductors, Krynica-Zdroj, Poland, 19-24 Jun. 2010.

M. Sezen, "The aspects of FIB-based processing in nanostructuring and nanoanalysis", Paul Scherer Institut, Villigen, Switzerland, 21 Jun. 2010.

T. Haber, "Novel thermally induced polymerization nanoimprint lithography processes for organic electronic devices", International Conference on Organic Electronics, Paris, France, 21-27 Jun. 2010.

M. Albu, "The boron influence on the microstructure of a 9%-Cr martensitic steel", High temperature defect assessment", International HIDA-5 Conference, Guildford, U.K., 22-23 Jun. 2010.



Prof. Barry Carter from the University of Connecticut (USA) presents a lecture in the institute (Jan. 2010).

H. Plank, "The influence of preparation parameters during electron beam induced deposition on chemistry, structure and volume growth rate of Pt deposits", 5<sup>th</sup> FIB-Workshop, Vienna, Austria, 27-29 Jun. 2010.

H. Reingruber, "New micro scale methods for the characterization of fluid and gas transport through membranes", Microscience 2010, London, U.K., 28 Jun. – 2 Jul. 2010.

G. Kothleitner (invited), "Towards more quantitative EELS: possibilities with a novel electron energy-loss spectrometer", Microscience 2010, London, UK, 28 Jun. – 2 Jul. 2010.

H. Plank, "The influence of preparation parameters during electron beam induced deposition on chemistry, structure and volume growth rate of Pt deposits", Focused Electron Beam Induced Processes (FEBIP 2010), Albany, NY, USA, 13-19 Jul. 2010.

G. Kothleitner (invited), "EELS & EFTEM imaging: instrumentation, applications and artifacts", Microscopy & Microanalysis 2010, Portland, Oregon, USA, 1-5 Aug. 2010.

P. Wilhelm (invited), "Raman microprobe imaging of submicron structures and interdisciplinary characterization of beam damage", EUCMOS 2010, Florence, Italy, 29 Aug.– 3 Sep. 2010.

J. Rattenberger, "Kontrastinversion in Niederenergie-RE-Abbildungen", Gemini User Meeting, Bochum, Germany, 30 Aug. – 2 Sep. 2010.

G. Kothleitner (invited), "Analytical Electron Microscopy", IMPRS Summer School "Advanced Microscopy", Garmisch-Partenkirchen; Germany, 1-3 Sep. 2010.

N. Matsko (invited), "Biocalcification. Correlative AFM and EM microscopy and spectroscopy", 52nd Symposium of the Society for Histochemistry, Prague, Czech Republic, 1-4 Sep. 2010.

I. Haynl, "Cryo atomic force microscopy: a new way to investigate elastomeric samples", 60. Jahrestagung der Österreichischen Physikalischen Gesellschaft; Salzburg, Austria, 6-10 Sep. 2010.

W. Haas, "Copper zinc tin sulfide and selenide nanoparticles: chemical analysis on the single particle level", European MRS 2010 Conference, Warsaw, Poland, 12-17 Sep. 2010.

A. Zankel, "In situ characterization of polymers in the ESEM, even enabling new hybrid testing methods", 14<sup>th</sup> International Scientific Conference on Polymeric Materials, Halle, Germany, 15-17 Sep. 2010.

B. Chernev, "Throw a hyperspectral glance through your samples, but approach the results with caution", 18<sup>th</sup> European Symposium on Polymer Spectroscopy (ESOPS), Zadar, Croatia; 19-22 Sep. 2010.

P. Pölt, "In situ characterization of micro filtration membranes in the environmental SEM", International Microscopy Congress IMC 17, Rio de Janeiro, Brazil, 19-24 Sep. 2010. N. Matsko, "Biomineralization: Correlative AFM -TEM microscopy and spectroscopy", International Microscopy Congress IMC 17, Rio de Janeiro, Brazil, 19-24 Sep. 2010.

A. Meingast, "Overview of TEM results in NanoProbe", NSI Meeting, Johannes Kepler University of Linz, Austria, 22 Oct. 2010.

W. Grogger (invited), "Reliable and accurate nanoanalysis with EFTEM", 7<sup>th</sup> Workshop on EELS/EFTEM, Zurich, Switzerland, 27-29 Oct. 2010.

S. Fladischer, "TEM techniques explained by investigations of a thin Ag layer", ISOTEC Seminar 2010, Reinischkogel, 22 Nov. 2010.

C. Gspan (invited), "Convergent-beam electron diffraction and quantitative electron diffraction for structure analysis", Institute of Semiconductor and Solid State Physics, Johannes-Kepler University of Linz, 27 Nov. 2010.

### Lectures 2011

H. Schröttner, F. Hofer, "Materialcharakterisierung mittels elektronenmikroskopischer Methoden", BMW Group, Munich, 24 Jan. 2011.

W. Grogger, "Hochauflösungselektronenmikroskopie von Nanoteilchen", Nanonet Day 2011, Graz, 14 Feb. 2011.

G. Kothleitner, "Nanotomographie", Nanonet Day 2011, Graz, 14 Feb. 2011.

B. Zechmann, "Subcellular protection of antioxidants during abiotic and biotic stress", Group of Fruit Tree Biotechnology, Department of Plant Breeding, CEBAS-CSIC, Murcia, Spain, 25 Feb. 2011.

F. Hofer (invited), "Monochromated low-loss EELS for studying surface plasmons on nanoparticles", Nano and Photonics 2011, Mauterndorf, Austria, 16 Mar. 2011.

S. Fladischer, "Nanoanalytical characterization of an organic photodiode", ASEM Workshop, Graz, 7 Apr. 2011.

W. Haas, "Chemical (in)homogeneity of copper zinc tin selenide nanoparticles and resulting solar absorber layers", ASEM Workshop, Graz, 7 Apr. 2011.

J. Rattenberger, "Beam transfer characteristics of a low vacuum SEM", ASEM Workshop, Graz, 7 Apr. 2011.

A. Reichmann, "Orientation contrast images of different ceramic materials with the SEM", Jozef Stefan Institute, Ljubljana, Slovenia, 15 Apr. 2011.

H. Plank, "A novel strategy to maximize deposition efficiency and electrical conductivity for electron beam induced Pt deposition as a gentle alternative to ion beam assisted deposition", MRS Spring Meeting, San Francisco, USA, 24 Apr. 2011.

M. Albu, "Comprehensive analysis of precipitates in rich Cr steels by EELS spectrum imaging, CETAS 2011, Luxembourg, 17 May 2011.

B. Zechmann, "Subcellular distribution of glutathione associated with pathogen attack in plants", University of Natural Resources and Life Sciences, Vienna, 20 May 2011.

J. Wagner, "3D structural and chemical characterisation of paper using advanced electron microscopy techniques", Österreichische Papierfachtagung, Graz, 8 Jun. 2011.

H. Plank (invited), "Fabrication of functional nanostructures via focused electron beam induced deposition, Nano TR 7. Istanbul, Turkey, 27 Jun. 2011.

A. Zankel, "Hybrid testing methods in the ESEM for the investigation of polymers", 13<sup>th</sup> Workshop "Deformation und Bruchverhalten von Kunststoffen", Merseburg, Germany, 29 Jun. 2011.

H. Schröttner, "Feinstrukturuntersuchungen an Polymerund Verbundwerkstoffen", Semperit TP, Wimpassing, 17 Jun. 2011.

B. Zechmann, "Sulfur-induced resistance is correlated with enhanced glutathione metabolism in Tobacco mosaic virus–infected tobacco plants", 10<sup>th</sup> International Conference Reactive Oxygen a. Nitrogen Species in Plants, Budapest, 5 Jul. 2011.

B. Chernev, "Visualisierung alterungsbedingter Veränderungen an Kunststoffoberflächen mit spektrokopischen Imaging Techniken", 16<sup>th</sup> Workshop Festkörperanalytik, Vienna, 4 Jul. 2011.

H. Reingruber, "New microscopic characterization methods for porous polymeric membranes". ICOM 2011 Int. Conf. on Membrane and Membranes Based Processes, Amsterdam, Netherlands, 23 Jul. 2011.

S. Mitsche (invited), "Influence of temperature and strain rate on the softening processes in Allvac 718Plus", THER-MEC 2011, Quebec, Canada, 1 Aug. 2011.

G. Kothleitner (invited), "Advances in quantitative EELS electron spectroscopy and imaging" Microscopy Conference 2011, Kiel, Germany, 28 Aug. 2011.

H. Schröttner, "Nano-structured, sol-gel derived  $SiO_2$ ,  $TiO_2$  and  $SiO_2$ - $TiO_2$  thin films", Nanostructured Surfaces and Interfaces Workshop, Bad Schallerbach, Austria, 5 Sep. 2011.

M. Albu, "Mapping the bonding of light elements in a composite cutting tool material by DualEELS spectrum imaging", 10<sup>th</sup> Multinational Conference on Microscopy 2011, Urbino, Italy, 4-7 Sep. 2011.

J. Dohr, "Preparation of nano-flat cellulose substrates for dynamic degradation experiments via AFM", 10<sup>th</sup> Multinational Conference on Microscopy 2011, Urbino, Italy, 4-7 Sep. 2011.

S. Fladischer, "Chemical nanoanalysis of organic semiconducting devices using EDXS and EELS", 10<sup>th</sup> Multinational Congress on Microscopy 2011, Urbino, Italy, 4-7 Sep. 2011. N. Matsko (invited), "3D reconstruction and 3D imaging techniques", Satellite Workshop on Tomography. Ancona, Italy, 3 Sep. 2011.

R. Schmied, "Fundamental proximity effects of electron beam induced platinum deposition", 10<sup>th</sup> Multinational Congress on Microscopy 2011, Urbino, Italy, 4-7 Sep. 2011.

N. Matsko (invited), "Integration of an ultramicrotome and specially designed AFM for cryo serial section tomography of soft materials", 10<sup>th</sup> Multinational Congress on Microscopy 2011, Urbino, Italy, 4-7 Sep. 2011.

T. Uusimäki, "Investigation of nickel and Fe<sub>3</sub>O<sub>4</sub> nanoparticles incorporated within mesoporous silicon by electron tomography", 10<sup>th</sup> Multinational Congress on Microscopy 2011, Urbino, Italy, 4-7 Sep. 2011.

H. Plank (invited), "Alternative patterning strategies for reduced thermal stress during focused Ion Beam Processing", 10<sup>th</sup> Multinational Congress on Microscopy 2011, Urbino, Italy, 4-7 Sep. 2011.

P. Pölt (invited), "In situ experiments with soft materials in the ESEM - Development of new test methods", 10<sup>th</sup> Multinational Congress on Microscopy 2011, Urbino, Italy, 4-7 Sep. 2011.

T. Haber, "Contrasting Polymers used in Organic Electronic Device Fabrication by STEM EELS and EFTEM spectrum imaging", 10<sup>th</sup> Multinational Congress on Microscopy 2011, Urbino, Italy, 4-7 Sep. 2011.

A. Zankel, "Serial block face scanning electron microscopy for three dimensional structural and elemental analysis of materials", 10<sup>th</sup> Multinational Congress on Microscopy 2011, Urbino, Italy, 4 Sep. 2011.

A. Meingast, "Analytical (S)TEM investigations of magnetic semiconductors and Heusler alloys", 10<sup>th</sup> Multinational Congress on Microscopy 2011, Urbino, Italy, 4-7 Sep. 2011.

F. Hofer (invited), "Transmission electron microscopy of nanostructured materials", 10<sup>th</sup> Multinational Congress on



FELMI-ZFE delegation at the MCM in Urbino, Italy, 2011.

Microscopy 2011, Urbino, Italy, 4-7 Sep. 2011.

B. Zechmann, "Compartment specific importance of ascorbate and glutathione during abiotic stress", 3<sup>rd</sup> Sulphyton Meeting, University of Padua, Italy, 20 Sep. 2011.

F. Hofer (invited), "Advanced electron microscopy of complex nanostructured materials", SLONANO- 2011, Ljubljana, Slovenia, 27 Oct. 2011.

A. Zankel (invited), "Hybrid testing methods in the ESEM, a contribution to materials science", Seminar IWM Fraunhofer, Halle, Germany, 8 Nov. 2011.

J. Rattenberger, "Low-energy electron microscopy: option or demand?", Agilent Workshop, Graz University of Technology, 17 Nov. 2011.

F. Hofer, "Mit Elektronen sieht man besser", Österreichischer Ingenieur und Architektenverein (ÖIAV), Graz, 12 Dec. 2011.

F. Hofer (invited), "Advanced electron microscopy of complex nanostructures", Hybrid Inorganic/Organic Systems for Opto-Electronics, Humboldt University, Berlin, Germany, 13 Dec. 2011.

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Grogger, W., Hofer, F., Pölt, P., eds., Proceedings Microscopy Conference, Graz, 30 Aug. – 4 Sep. 2009, Microscopy Conference MC2009, vol. 3 Materials Science, Verlag der Technischen Universität Graz (2009).

Kothleitner, G., Leisch, M., eds., Proceedings Microscopy Conference, Graz, 30 Aug. – 4 Sep. 2009, Austrian Society of Electron Microscopy, ed., vol. 1 Instrumentation and Methodology, Verlag der Technischen Universität Graz (2009).

Albu, M., Martin, F. M., Mayr, P., Kothleitner, G. "Comprehensive TEM studies on Cr -rich martensitic steels", Microscopy Conference MC2009, vol. 3, Verlag der Technischen Universität Graz (2009) pp. 215 - 216.

Chernev, B.S., Sezen, M., Wilhelm, P., Brandl, C., "Approaching nanaoscale with Raman and FTIR imaging", European Polymer Congress (EPF09), Graz, Book of Abstracts (2009) p.251.

Chernev, B. S., Wilhelm, P., Brandl, C., Schiller, M., Huisman, H., Pelzl, B., Klaess, P., "Monitoring of the artificial weathering of PVC formulations with FTIR-spectroscopy" Proceedings Advances in Polymer Science and Technology (APST), Linz, Austria (2009) p.72.

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Fischereder, A., Maier, E., Rath, T., Haas, W., Hofer, F., List, E., Trimmel, G., "Preparation and characterization of metal sulphides / polymer nanocomposite solar cells', European Polymer Congress (EPF09), Graz, Book of Abstracts, p. 234.

FissIthaler, E., Sax, S., Blümel, A., Sezen, M., List, E., "Structuring methods for conjugated polymers and their applicability regarding micro- and nano-patterning of polymer light emitting devices", European Polymer Congress (EPF09), Book of Abstracts (2009) p.251.

Granitzer, P., Rumpf, K., Albu, M., Plank, H., Pölt, P., "Three dimensional quasi-regular arrangement of ferromagnetic nanostructures within porous silicon", Proceedings IEEE NANO 2009, IEEE, Piscataway, NJ, (2009) pp. 655 - 658.

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# Life in the Institute

## Life in the Institute

2009



PhD promotion of Armin Zankel and Meltem Sezen



Barbecue party



Excursion to the Erzberg





December party



Kleeblattlauf / FELMI-ZFE team

"Microscopist" working with a TEM from 1965 !!

### 2010



Excursion to Haus im Ennstal



Kleeblattlauf / FELMI-ZFE team



Excursion to Pöllau



Renovation work on the 2<sup>nd</sup> floor.



December party



ZFE delegation at the ACR Enquete in Vienna

## 2011



Excursion to Slovenia



Excursion to Slovenia



Ulrich Santner thanks Gerhard Windisch for 40 years in the Institute



December party