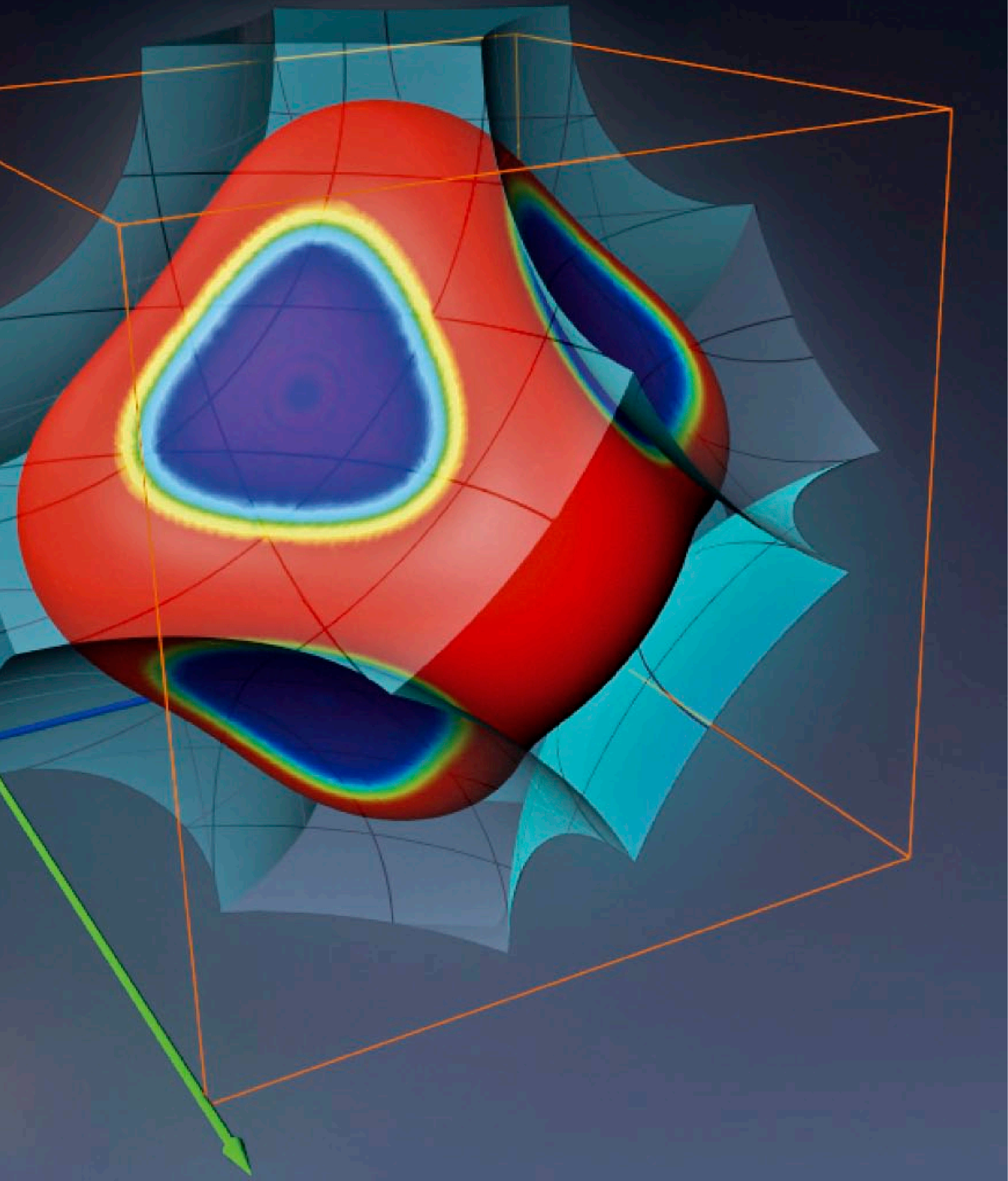
CCMX
Competence Centre for
Materials Science and Technology

ANNUAL ACTIVITY REPORT 2010



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Annual Activity Report 2010

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Message from the Chair

With the close of 2010, CCMX moves into the final year of the 2008–2011 funding period. Our Public-Private Partnership approach has been a great success. All the funds for the current period have been awarded to high quality pre-competitive projects, with at least one third of the funding having come from companies and/or institutions. With this scheme CCMX has brought more than 20 companies into close working relations with research groups in the ETH Domain. This report highlights the achievements of these ongoing projects.

As in previous years, the research projects have been complemented by a dynamic programme of education and outreach, with 9 courses, events and technology aperitifs held in 2010. These events have attracted a high participation from the industry and are a particularly effective way of engaging smaller companies, keeping them up to date with the latest research developments.

The ETH board has already announced that during the 2012–2016 funding period the competence centres will be expected to find a progressively increasing proportion of their funding from sources other than the ETH board. In response to this, CCMX proposes to launch «Materials Challenges 2020», a series of platforms for pre-competitive research to be co-funded with industry. Their aim will be to tackle the underlying scientific questions critical to the future of Swiss industry. Through questionnaires and workshops involving representatives of industry and the academic institutions making up CCMX, we have already actively engaged participants in developing this programme.

Sincerely,
Professor Karen Scrivener
Chair CCMX

ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

ETH Eidgenössische Technische Hochschule Zürich
Swiss Federal Institute of Technology Zurich

EMPA Materials Science & Technology

PAUL SCHERRER INSTITUT

csem

How to be Involved With CCMX

Photos by Tony Kunz

Knowledge transfer and networking opportunities are an important aspect of CCMX events.

Education and Training

CCMX organises different sorts of advanced training events. The summer and winter schools are aimed primarily at doctoral students. Courses and workshops are oriented more towards engineers and scientists from industry who are interested in continuing education and hands-on workshops, featuring lab visits and the opportunity to transform theory into practice. Please see page 5 for a summary of the CCMX courses in 2010.

Outreach Activities

Knowledge transfer and networking opportunities are an important aspect of CCMX events in which participants meet and discuss materials science in all its different aspects. These networking events are in the form of "Technology Aperitifs", end-of- afternoon/ early-evening get togethers that feature scientific presentations on a particular topic followed by an informal aperitif where people can interact. Please see pages 6–7 to find out what happened in 2010.

Research

CCMX funds research projects in several thematic areas. Please see page 27 for a list of all the projects funded in 2010. These research projects are considered "pre-competitive", which is a middle ground between fundamental basic research conducted mainly in universities, and the ETH Domain and proprietary research, performed in corporate laboratories. Additionally, the CCMX analytical platform funds projects aimed at the development of new analytical tools, methods, and instrumentation for characterisation on the scale below 100 nm.

Learning With CCMX

2010 was an active year for education and training, with more than 160 participants in five courses and workshops, bringing together PhD students, engineers, and scientists from industry and academic institutions in a variety of settings.

CCMX Travelling Lab Workshop

This week long workshop took place from 8–12 November 2010, and covered topics relevant to materials science for biomaterials and diagnostic research and applications. Participants travelled to multiple industrial and clinical locations, learning about the perspectives and needs relevant to each setting. This interdisciplinary experience provided exposure to academic, industrial, and clinical sites, and allowed students to actively participate in case studies and problem solving at each site. The programme also included cultural events and evening activities, creating opportunities for meeting and networking with other researchers.

The week began in Zurich with visits to the University Hospital Balgrist and to the Centre for Dental Medicine – Clinic for Fixed and Removable Prothodontics and Dental Material Science. Tours of the facilities and presentations from medical experts and researchers offered insights into particular kinds of injuries and medical interventions, and highlighted the value of interdisciplinary cooperation between clinicians and researchers. At Medicoat in Mägenwil, the site visit featured construction and buildup of coating systems for the manufacture of hip joint replacements. The next stop was Neuchâtel and CSEM, where a fascinating presentation was given about manipulating cells with AFM (Atomic Force Microscopy). Again, emphasising the value of collaboration among researchers from different scientific backgrounds. At the RMS Foundation in Bettlach, collaboration was again the theme, in this case having to do with cooperation between clinics, research institutes, companies in order to improve medical devices and techniques.

A highlight of the day was a hands-on workshop with the head of the orthopedic surgery clinic in Fribourg, where participants manipulated plastic bones using authentic instruments for plate/screw fixation, giving students first-hand experience with some of the problems faced by surgeons. The visit to the Inselspital Bern featured biodegradable materials used in cardiovascular medicine. Afterwards, the Cardiovascular Engineering Group at the Artificial Organ Center presented ongoing research in the field of analytical and surgery tools. Participants broke into small groups and attended actual heart examinations and surgeries in the clinic. The final site visit included a tour of the Straumann production facility in Villeret BE, known for its excellence in implant and restorative dentistry. The week ended on a high note, with a delicious Thai meal organised by Straumann.

«I really took advantage of the course, not only for the technical knowledge but also to make new contacts for possible future collaborations».
EPFL participant in the CCMX Summer School

Photos by Stefanie Lischer, Empa

"The workshop gave a good overview of experimental techniques available to characterise materials at the nanoscale".
Participant in a CCMX Workshop

2010 Programme of CCMX Courses

Date	Course	Title
3 June	CCMX Workshop	Nanoscale analytics of materials for industrial application
6-8 July	CCMX Continuing Education Course	Powder Characterisation: From nanometers to millimeters and from theory to practice
25-27 August	CCMX Summer School	Advanced Characterisation Techniques in Materials
29 September – 1 October	CCMX Continuing Education Course	Advanced X-ray diffraction methods for coatings: Strain, defects and deformation analysis of thin films
5-12 November	CCMX Continuing Education Workshop	Travelling Lab Workshop: Materials for Life Sciences

"After several years working in the industry it was good having a refresh. When you are studying it is not always easy to understand exactly what use you will be able to make of the theory you are learning. This course will allow me to better understand the results of the materials characterisation that we subcontract to outside companies".
Industry participant in the CCMX Summer School

Outreach and Networking Activities

The 'Technology Aperitifs' are a series of events held throughout the year at which CCMX provides information on hot topics related to materials science, encourages creativity for future applications, and allows contacts for partnerships in a pleasant networking environment.

When Lars Lundquist, an expert on environmental sustainability at Nestlé, comments on how the company uses Life Cycle Assessment (LCA) as a strategic tool to meet its environmental sustainability commitment, he can be sure of finding an attentive audience! At Nestlé, sustainability means adopting practices that help the company to meet the needs of the present without compromising the world's ability to satisfy the demands of the future. LCA is a strategic tool, and is integrated as a systematic decision support tool in the innovation and renovation process in the fast-moving consumer goods sector, as well as food packaging eco-design using PIQET (Packaging Impact Quick Evaluation Tool). The designated objective of LCA is to facilitate communication throughout the supply chain and reduce complexity, cost, and confusion.

Getting to know the state-of-the-art

Life Cycle Assessment was the focus of a Technology Aperitif event organised by CCMX at EPFL in March 2010. The partner was Quantis, an EPFL spin-off specialising in the quantification of environmental impacts of products, activities, or companies using LCA methodology. The aim of this event was to give participants an idea of how LCA helps to build a strategy towards the environment, to introduce environmental considerations into product conceptions, and managing and communicating strategic risks.

With its Technology Aperitif events, CCMX helps to create contacts for future partnerships and provides an informal networking environment, tailored to the needs of industry. The meetings, which always take place in late afternoon and are related to current trends, consist of six to eight short presentations given by representatives of academia or dynamic companies. Participants can discuss materials-related aspects with the speakers and initiate cooperation to solve specific problems of key importance to industry.

A good example of a highly topical event was the Technology Aperitif *Materials for Energy Applications*, held at Alstom Ltd (Switzerland), Baden, in June 2010, organised by CCMX and the Alstom Power Technology Center. Specialists from Alstom, and scientists from EPFL, ETH Zurich, Empa, and the Paul Scherrer Institute, gave participants insights into the latest developments in materials for energy applications, from cover corrosion of power plants and low thermal conductivity coating to solar cells and batteries. At PSI, materials research focuses on structural materials for high-temperature applications and on functional materials. The studies include zeolite and supported metal catalysts, electro-catalysts with reduced platinum content, and proton conducting membranes for polymer electrolyte fuel cells with enhanced stability and lifetime. The PV-Lab at EPFL – Neuchâtel conducts research in the field of process and device optimisation for silicon-based photovoltaic applications. The emphasis is on amorphous and micro-crystalline thin-film silicon layers deposited by a plasma process on glass and plastic substrates, hetero-junctions, transparent conducting oxides, and encapsulation technology.

Latest research results 'on a silver platter'

The Technology Aperitif "Mechanical Properties of Materials", held at Empa Akademie, Dübendorf in October 2010, proved particularly popular. This event was prompted by recent advances in measuring techniques. It is now possible to measure mechanical properties using small or micro specimens *in situ*, allowing crystallographic and microstructural changes arising from directly applied loading to be monitored simultaneously. Such techniques also provide an opportunity to determine the mechanical properties of extremely fine-grained materials and materials fabricated in small-section product forms. On this occasion, Empa offered insights into *in situ* experiments studying plasticity and fracture of small structures. ETH Zurich addressed the scaling in the mechanical properties of materials and evaluated what happens at the nanometre length scale. As for PSI, its researchers showed the investigations possible with the *in situ* beamlines, such as POLDI for TOF-neutron diffraction, MS for X-ray powder diffraction, and MicroXAS, the white beam Laue. Another core theme for PSI scientists was the environmentally-assisted

Technology Aperitifs – Time to Meet the Experts

Photos by Nathalie Jongen, CCMX

2010 CCMX Outreach and Networking Actvities

Date	Title
23 March 2010	**Technology Aperitif " Life Cycle Assessment of Materials"**
28 April 2010	**CCMX Annual Meeting**
8 June 2010	**Technology Aperitif "Materials for Energy Applications"**
20 October 2010	**Technology Aperitif "Mechanical Properties of Materials"**

cracking of LWR structural materials. A joint presentation by ETH Zurich and Empa dealt with service-like thermo-mechanical fatigue experiments for the validation of lifetime assessment procedures.

CCMX in a mirror

A desire to learn about the latest developments in materials research prompted Paul J. Würsch of *Franke Industrie AG* to attend the Technology Aperitif. This company supplies hot gas path parts, components, and subassemblies in stainless steel, high-grade alloys, and heat-resistant materials. "We are an SME with about 50 people and serve as an extended work bench for our clients", says the sales manager for Franke. "So we have to know all about new materials, how to handle them and the necessary manufacturing techniques. In this respect, the Technology Aperitif was a real eye-opener!" It is important for Würsch to meet experts who can identify future trends at an early stage. He particularly appreciated the time for networking. "The organisation and the setting make it easier to establish contacts with scientists and other companies, hear interesting presentations, and learn new dynamics."

The idea of approaching people from research institutions and industry was what encouraged Jiri Nohava of the internationally active *CSM Instruments*, to attend. This company has been a leader in the development of instruments for surface mechanical properties characterisation, in both the research and industrial fields, for over 30 years. Their expertise ranges from nano-indentation and scratch testing to tribology pin-on-disk, coating thickness characterisation with Calotest, and 3D imaging and topography. "We wanted to make the most of this opportunity to meet people from different research institutions and private industry to discuss their needs and offer our services."

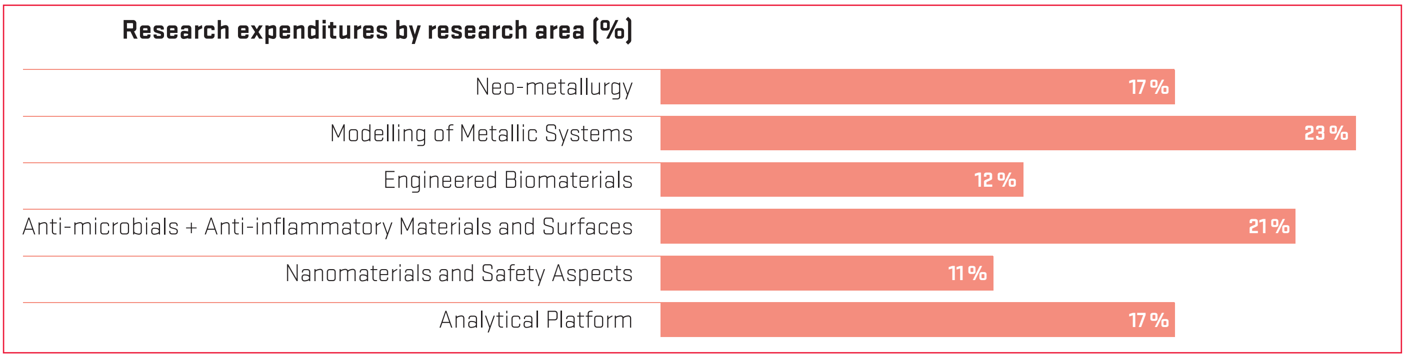
Dirk Weid's field of interest is simulations for materials development to create customised material properties. In August 2009, he attended the CCMX Summer School organised at EPFL on the topic of *Modelling in Materials Sciences*. After meeting Prof. Michel Rappaz, Head of the Computational Materials Laboratory, who told him that a Technology Aperitif was being organised on the *Mechanical Properties of Materials*, Weid decided to attend. "The talks are given at a high level from a scientific point of view and give a perfect insight into the state-of-the-art in materials research", says this mechanical engineer from the Research and Development Department of George Fischer AG. He attached particular importance to the possibility of meeting experts in materials technology. "I met an interesting contact and was able to pass his business card to the appropriate person in our company." Weid also appreciated that enough time had been scheduled between the talks. "It makes sense for speakers to keep to allotted times and offer participants an opportunity to ask questions."

Access to up-to-date information was the impetus for Qi Zhang. "I was looking to obtain information on the latest developments, share experiences with old friends, and get to know new people", says the Health & Environmental Sustainability Project Manager of Nestec SA. "The content of the talks was most interesting. It doesn't matter whether they are more general or go into detail — any information is welcome!"

Technology Aperitifs offer the benefit of meeting experts in a selected field — within the local academic networks. These specialists can contribute to solving specific materials-related problems of key importance to industry, whether through a CCMX project or through another channel. So, although the expectations of the attendees differed, everyone agreed that they enjoyed the event and would certainly attend another Technology Aperitif.

The Research We Focus On

Photo by Nathalie Jongen, CCMX



Fostering Public-Private Partnerships
The Research Ticket Programme is designed to foster Public-Private Partnerships in materials science in order to ensure the longevity of the interactions between industry and CCMX institutions. Instead of engaging in research on a project-by-project basis as it has been in the past, industrial partners collaborate with academic groups in a consortium focusing on a selected "Thematic Research Area". The creation of multilateral partnerships for medium term pre-competitive research is mutually beneficial and is a good approach to bridging the funding gap between the fundamental research supported by SNSF and the applied, "close to market" projects supported by CTI.

What is pre-competitive research?
Pre-competitive research is a middle ground of focused, cutting-edge research that lies between fundamental, basic research conducted mainly in universities/ ETH Domain and proprietary research performed in corporate laboratories.

Members from the industry identify priority research topics with CCMX
The Research Ticket Programme allows flexibility for companies of different sizes to participate in the research activities of the Centre. Companies take a lead in identifying the priority research topics and may influence the choice of research projects to be carried out within a thematic research area. Not only is research funding more than doubled, since funding comes from both CCMX and two or more industrial partners, but companies also share the risk of funding long-term strategic research. Four new projects involving nine new entities started in 2010 and another series of projects is currently being launched.

Keeping materials characterisation in the loop
CCMX's Analytical Platform brings together expertise in the development of analytical methods and broad analytical resources from different institutions within the ETH Domain, industrial partners and other universities. On one hand, the analytical

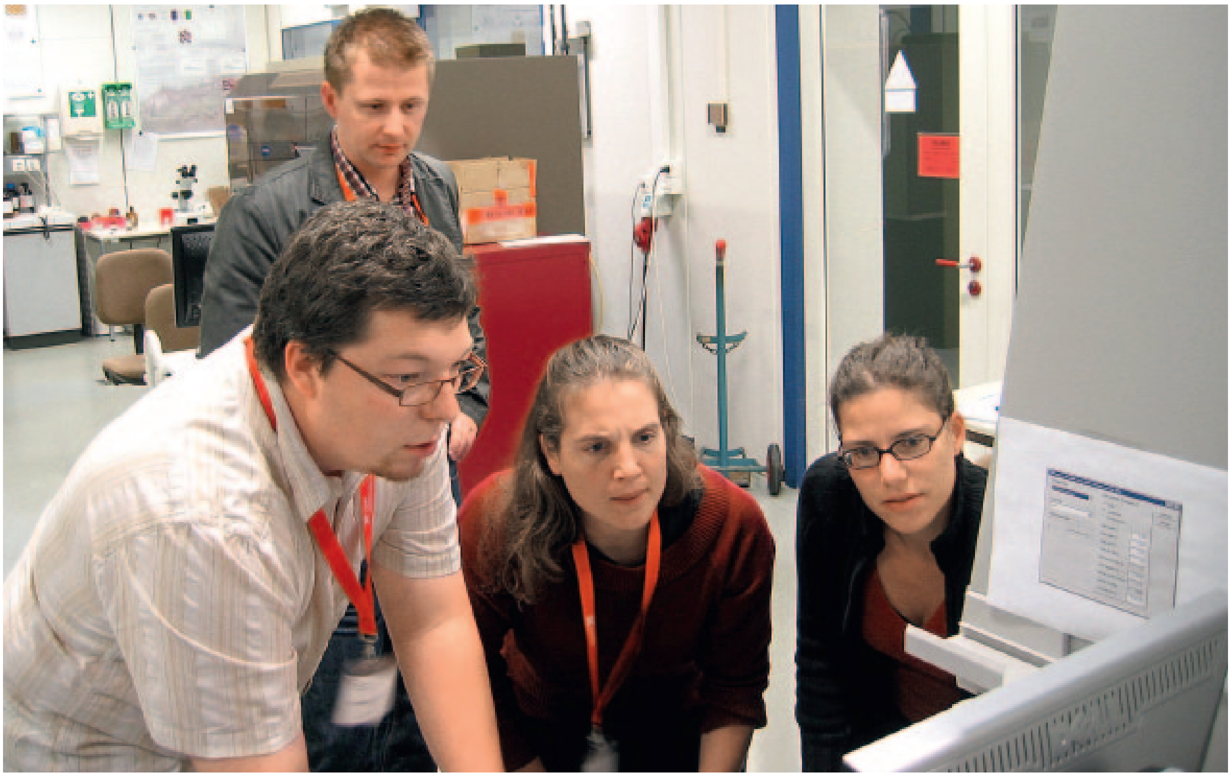


Photo by Antonia Neels, CSEM

Photo by Nathalie Jongen, CCMX

platform funds projects aimed at the development of new analytical tools, methods, or instrumentation for the analysis of physical, biological, or chemical properties on the scale below 100nm. On the other hand, the projects funded concern the exploitation of existing analytical techniques for nanoscale analysis in new fields of application. Co-funding from industrial partners and/or other institutions is a key aspect of these projects.

Companies involved in 2010 in projects (22) *Alphabetical order*

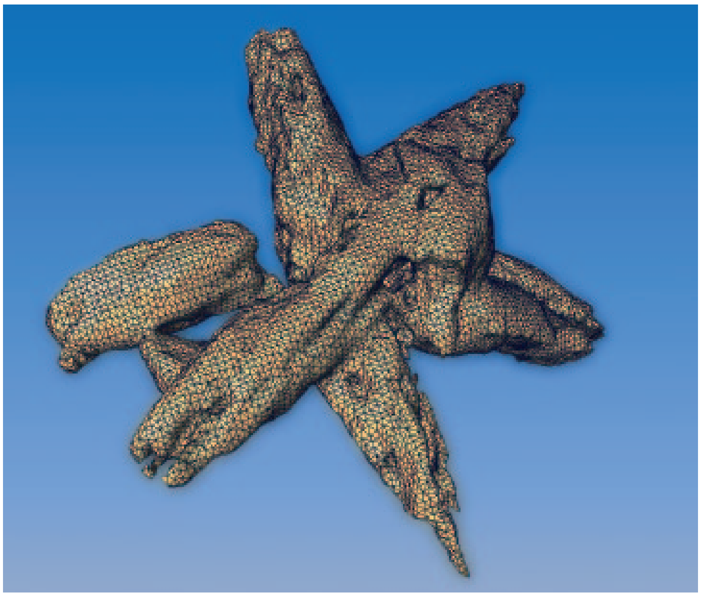
- ABB Turbo Systems
- Alstom
- Alcan
- AO Foundation
- Asulab
- Attolight
- BASF
- IG DHS
- ION-TOF Technologies
- Kugler Bimetal
- Lovalite
- Lyncee Tec
- Novelis
- OPEA
- RMS Foundation
- Rolex
- Sika
- Straumann
- Swissnuclear
- VV SA Varinor
- Wessling
- YXLON International X-Ray GmbH.

Other entities involved in 2010 (2)

- Federal Office for Public Health
- Federal Office for the Environment

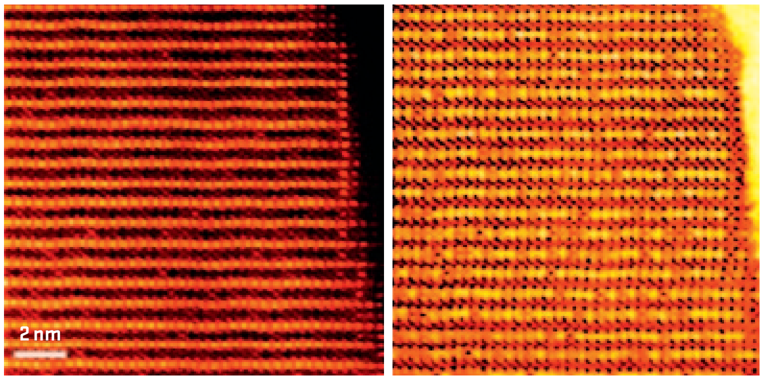
Valuable Insight into Internal Struct

What are the crucial features of composition and structure of a material allowing a particular preparation, a study of properties, an exciting new development and directed reproduction? Characterisation is the key to seeing into materials. That is why CCMX created MaCH², an analytical equipment database, tailored to the requirements of Swiss industry.



Left: Tomographic reconstruction of an iron-oxide nanostar; approximate frame size of 150 nm. Sample courtesy of Dr. A. Braun and D.K. Bora, Empa.

Bottom: Atomic resolution scanning transmission electron micrographs of an edge of a Li-conducting $NdLiTiO_3$ crystal; while the dark field micrograph (left) provides an image of the heavy cations containing information about the reorganisation of the Nd at the very surface, the bright field micrograph (right) reveals the location of oxygen vacancies as faint but distinct bright speckles. Scale bar is 2 nm. Courtesy of Empa.



When PhD student Ghim Wei Ho at Cambridge University applied a methane gas flow over silicon carbide wires she obtained – depending on the temperature and pressure of the growth process – beautiful 'nanoflowers', which amazed the scientific world. But these 'nanobouquets' are not just for show, these nanoscale structures have potential as water-repellent coatings and could provide the base material for new solar cells.

The starting point for such discoveries is accessing state-of-the-art analytical equipment, which is where the MaCH² project comes into play. Within MaCH² (**Ma**terials **CH**aracterisation in Switzerland (**CH**)), research institutions can collaborate by offering their instruments, laboratory infrastructure, and special expertise to help solve tricky characterisation problems. To demonstrate the value of the MaCH² database, it is interesting to look at the four primary centres for electron microscopy within the CCMX network: the Electron Microscopy Centre at Empa in Dübendorf, the Interdisciplinary Centre for Electron Microscopy at EPFL in Lausanne, the Department for Microscopy & Nanoscopy at CSEM in Neuchâtel, and the Electron Microscopy ETH in Zurich. MaCH² makes it easy to find these centers and to compare their fields of expertise.

Empa: Correlation of structure and function

At the *Electron Microscopy Centre (EMC) at Empa*, the team conducts cutting-edge research in development and application of new electron microscopy techniques, carries out research tasks on-demand, and provides consulting services in the area of electron microscopy in materials science. Their expertise encompasses a wide range of topics, including the analysis of functional nanoparticle coatings with exceptional behaviour based upon the nanosized flowers example in cooperation with Basel University.

Together with ETH Zurich and European research groups, Empa researchers are investigating the molecular structure of new types of polymers, and exploring atomic-level mechanisms for the exceptionally high lithium conductivity in complex perovskite-type ion conductors suitable for batteries. They are also applying electron tomography to the analysis of nanoparticles. "This method is particularly appropriate to measure the exact 3D shape of nanosized objects," comments Dr. Rolf Erni, Head of EMC.

"A tilt-series of images is recorded as the object is being tilted inside the microscope. Series of 2D projections are processed to mathematically retrieve the 3D shape of the object." The Empa team recently succeeded in enhancing this 3D technique with experts from ETH Zurich and European Universities to enable atomic resolution imaging, a breakthrough that has been pursued by various groups around the globe. Empa is also active with new carbon-based nanomaterials; they keep a close eye on the complex interfaces of novel carbon nanotubes integrated into electronic devices and they investigate novel routes for the synthesis of graphene. This hexagonal array of carbon atoms with the thickness of one atom is one of the most attractive research topics of condensed matter physics. "Electron microscopy has its price, requiring space and careful sample preparation", Erni admits. "But it is the appropriate method to depict fine structural correlations, to resolve the

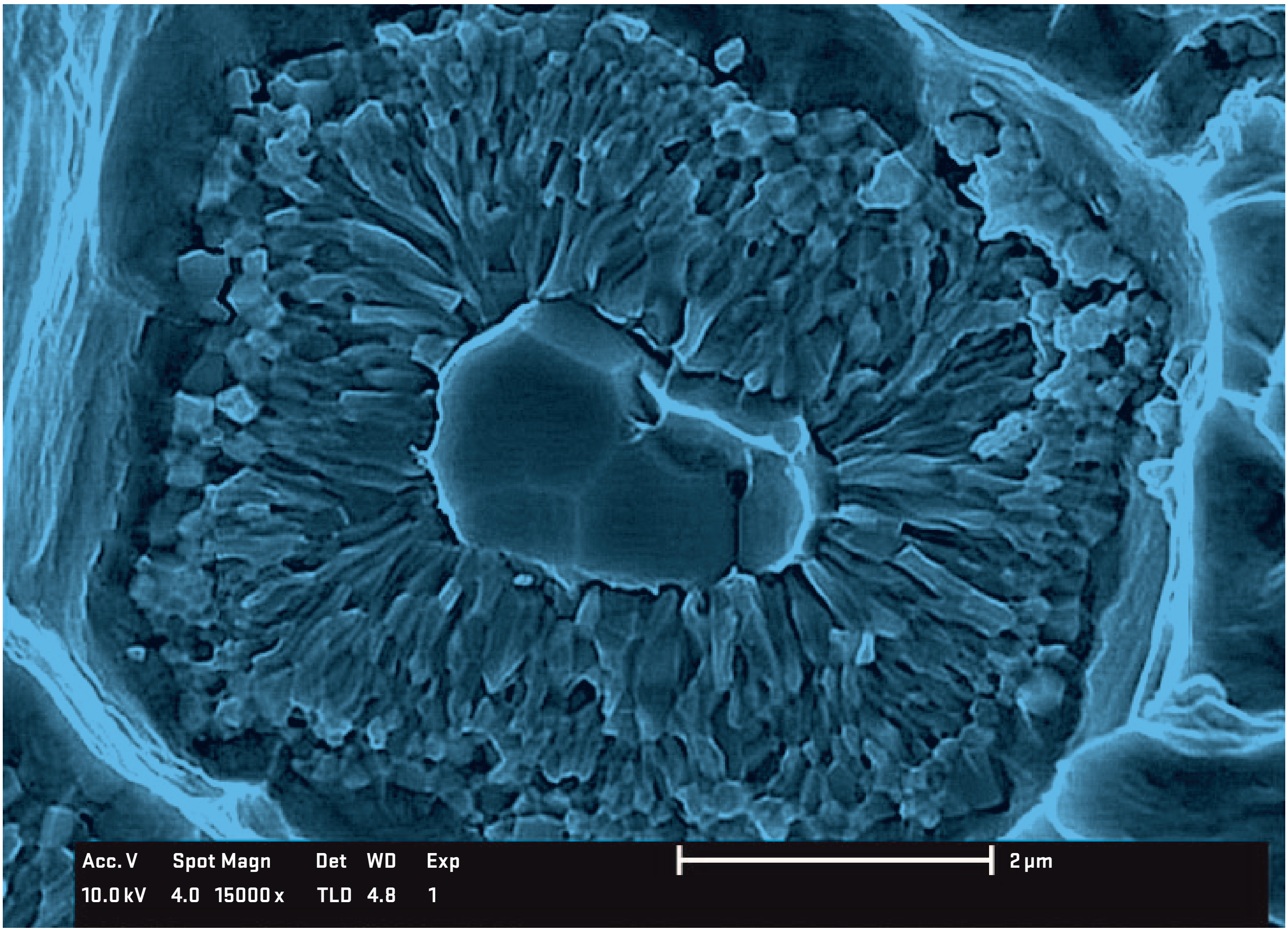


Fig 1a | Secondary electron image (topography contrast). Courtesy of CIME, EPFL.

atomic skeleton of solids and to localise individual molecules in a context, by correlating structure and function. The wealth of information obtained from advanced electron microscopy methods is definitely worth the effort and is often key in solving materials science problems."

Leap into the world of micro- and nanoscopy

Giving academia and industrial partners access to state-of-the-art analytical techniques is the mission of the *Interdisciplinary Centre for Electron Microscopy (CIME)* at EPFL. Professor Cécile Hébert's experienced team offers a wide range of techniques such as scanning electron microscopy, transmission electron microscopy and, surface analysis by XPS and auger electron spectroscopy. The centre aims at developing cutting-edge characterisation techniques applied to challenging problems. The team covers, for example, computational electron microscopy or 3D imaging and chemical analysis in a focused ion beam.

The sample surface is scanned with a focused beam of electrons in the scanning electron microscope (SEM) and signals are collected as a function of probe position to form an image. Very rich information such as the topography (Fig 1a) of the specimen, its chemical composition or its crystallinity can be retrieved from the signals obtained (e.g. channeling contrast Fig 1b). The dual beam SEM/focused ion beam (FIB) instrument, which combines a focused Ga-ion source and a complete SEM, can in particular be used to remove material layer by layer, allowing 3D imaging of the sample (Fig 1c).

Standard imaging using transmission electron microscopy (TEM) provides information about crystallinity, grain size, mass density, defects structure as well as on micro- and nanostructure. Electron diffraction provides local information about the crystal structure. High-resolution microscopy resolves the position of the atomic columns across the thin sample, which is particularly interesting at interfaces, grain boundaries or near defects. Energy dispersive X-ray (EDX) analysis enables mapping of chemical composition but with much higher spatial resolution than in the SEM (Fig 2).

The CIME team is responsive to industry needs and collaborates on numerous projects. For instance, the researchers are studying the SLID process (solid-liquid interdiffusion) in a project dealing with bonding methods aiming to achieve hermetic seals. In cooperation with industry partners, they intend to improve the design and production of very small medical devices used mainly in brain vessel procedures.

Valuable Insight into Internal Structure and Properties

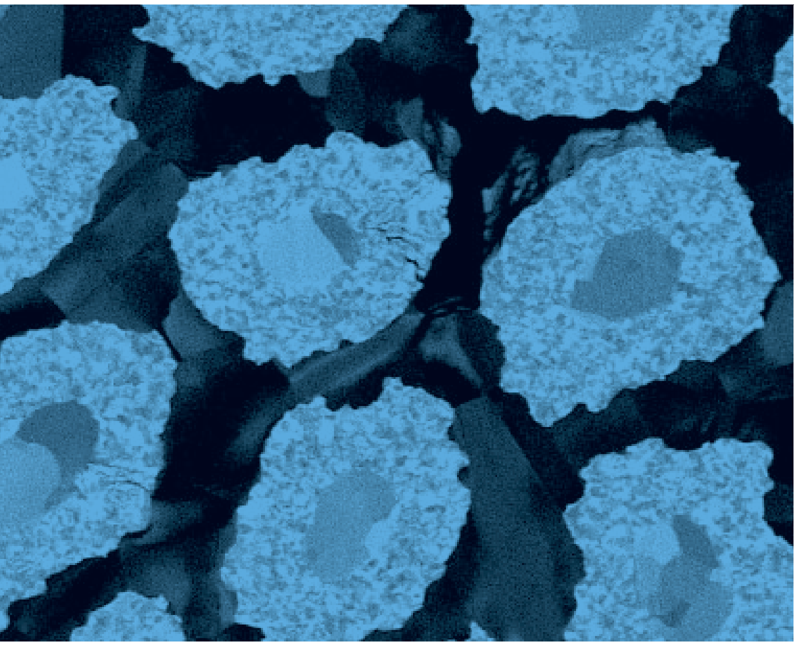


Fig 1b | Channeling and mass density contrast from backscattered electrons.

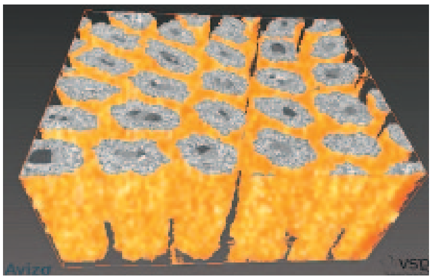


Fig 1c | volume reconstruction.

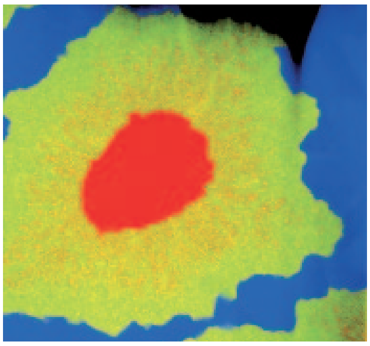
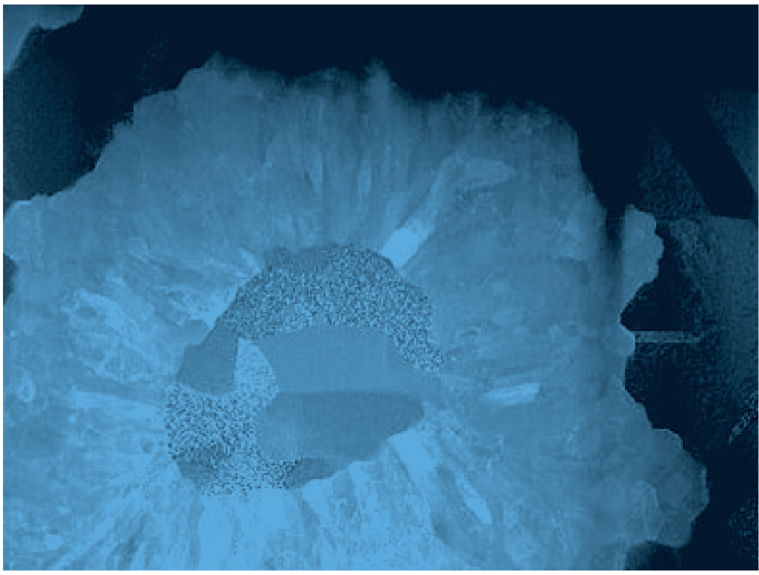


Fig 2 | Result of EDX element mapping; the concentration of three different elements.



STEM image with mixed diffraction (orientation of small grains) and z-contrast (density of different materials).

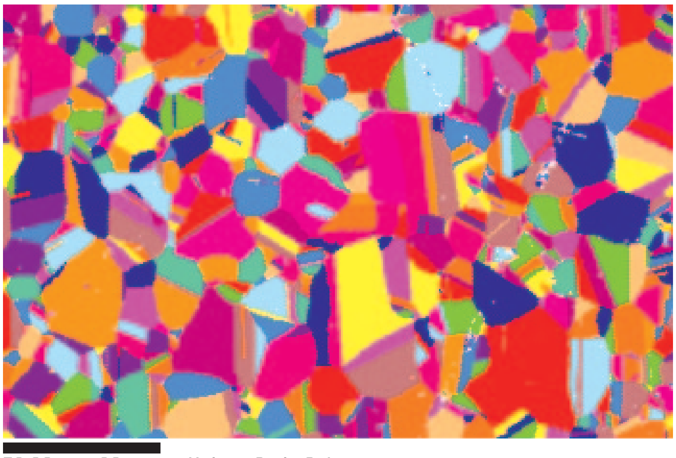
All images on this page courtesy of CIME, EPFL.

Microsystems technology lined up as a target

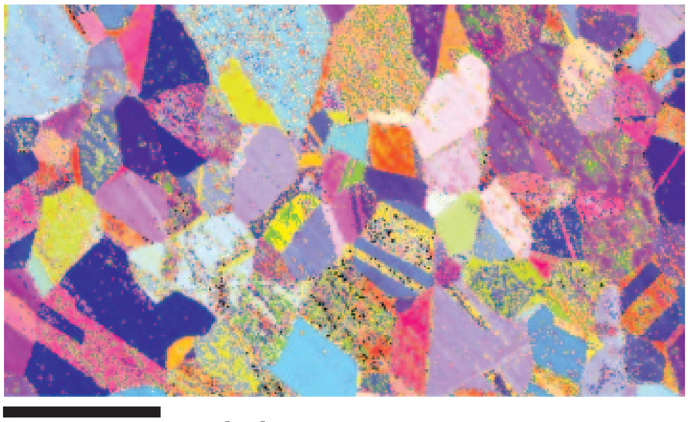
Dr. Massoud Dadras heads the SMN, the Department of Microscopy and Nanoscopy of the *Centre Suisse d'Electronique et de Microtechnique SA (CSEM)*, which offers a range of highly specialised microscopes for advanced investigation. Although his original expertise lies in metallurgy, his current research activities focus on micro-structural studies and the relationship of microstructure to properties, especially to improve thin layers properties. Because of its specific optical design, the available confocal microscope is capable of obtaining optical cross-sections of transparent or reflective samples, enabling the analysis of polymeric, biological, textile, and fibre specimens. CSEM also owns an environ-mental scanning electron microscope ESEM FEG, one of the most powerful analytical tools currently available to scientists, that carries out high-resolution secondary electron imaging at pressures as high as 10 torr and sample temperatures as high as 1000°C. This means that wet, oily, dirty, outgassing and non-conductive samples can be examined in their original condition, without sample alteration or preparation. The microscope manages specimens at very high spatial resolution, in some cases as high as 2 nanometres.

The transmission electron microscope TEM CM200 is the perfect tool for high resolution imaging and spectroscopy. This instrument operates at up to 200 kV and achieves a resolution of 0.14 nm with ultrathin specimens. Absorption contrast is typically useful for non-crystalline samples such as polymers and life science specimens while diffraction contrast of crystalline samples spots defects, dis-location, grains and grain boundaries. Valuable information about the chemical composition of the sample is obtained thanks to the energy dispersive X-ray spectro-meter (EDS) installed on the microscope. Dr. Dadras turns to the Atomic Force Microscope (AFM) if a surface topography and the roughness of a sample have to be determined. The AFM was quite helpful in studying the influence of crystallographic orientation of oxidation of zircaloy. The rate of oxidation of Zr depends on the material's composition. "It follows a cubic rate law in early stages, transforming after what is known as a transition period to a linear oxidation rate", explains Dadras, who is collaborating with Dr. Sousan Abolhassani of PSI. "Certain aspects of the oxidation are however not clear."

The scientist analysed the relation between the crystallographic orientation and the growth of the oxide layer in an early phase of oxidation of Zr using the AFM. The electron backscatter diffraction (EBSD) pattern and the corresponding AFM image of the region concerned revealed a height difference between the grains present after heat treatment. "These height differences prior to oxidation could be related to the prepa-ration and electro-polishing conditions", concludes the researcher. "Our observation



56.00 µm = 80 steps Unique Grain Color



54.00 µm = 90 steps IPF [001]

FCCC recrystallized grain presenting different crystallography orientations. All images on this page courtesy of CSEM.

52.00 µm = 80 steps IPF [001]

confirms that in the polycrystalline Zr, the grains near 0001 crystal orientation oxidise more rapidly than the grains having an orientation near 0-111."

EMEZ ETH Zurich: From biological systems to solid materials

Electron Microscopy ETH Zurich (EMEZ) is a centralised facility for user-focused interdisciplinary research and service activity that employs electron microscopes and micro-analytical tools to explore structure-function relationships in biological systems, as well as structure-property relationships of solid materials. Both man-made and natural materials, including biological specimens, are characterised at the micro- and nanoscale. The available expertise comprises sample preparation, observation using electron and ion beams, as well as data analysis and interpretation. The scale ranges from atoms to micro-domain for any materials of interest. The four major application fields are materials science and engineering, life science, physics/chemistry and earth science. "Our centre features 54 user groups from ten different ETH Zurich departments and partners from academia and industry", explains Dr. Roger Wepf, EMEZ director. "Thanks to this diversity we can push electron microscopy applications to the frontiers of science by transferring techniques from one research field to another."

Electron microscopic techniques offer a large variety of modes for structural investigations for a wide range of materials comprising ceramics, metals, semiconductors, organic materials, and building materials. The extraordinary strength lies in the broad range of accessible scales up to very high spatial resolution (from millimetres to the sub-nanometre and atomic range) of a single selected region of interest, and in the ability to investigate the same specimen area by different techniques, providing complementary information. The combination of morphological, chemical, and crystallographic data from the same sample area offers very comprehensive knowledge about the material with high spatial resolution. Highly sophisticated – and sometimes time consuming – preparation techniques are available for the investigation of specimens preserved as close as possible to the native state.

A main EMEZ focus is to establish interfaces to different imaging modalities, such as the novel nano-tomography method established in 2010 at PSI, different light microscopy techniques at the LMC/ETH Zurich, and a new approach to atomic spatial resolved mass spectroscopy with the recently acquired atom probe system (LEAP) from Cameca.

For complete contact details and further information about these four centres for electron microscopy, and for complete access to the instruments and expertise available in the MaCH² database, see: http://mach2.ccmx.ch.

Tracking Down Metallurgical Phenon

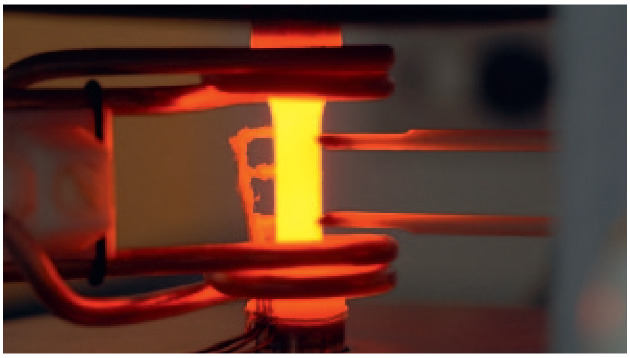
CCMX supports research in metallurgy in order to improve the modelling of metallic systems and to develop new alloys and processes. This application-driven research is conducted via collaborations between universities and research institutes in the ETH Domain. Industry reaps a two-fold benefit from this cooperation: companies not only acquire new knowledge in the form of novel products, but they also have access to materials science engineers who have followed a state-of-the-art educational programme.

PhD students Stéphane Pierret and Michael Weisser prepare a tensile test on the neutron diffractometer POLDI at PSI.
Photo courtesy of PSI.

From an industrial perspective, metallurgy remains a very timely and important subject. "Even though metallurgy can be thought of as a mature field, there are still many interesting problems that need to be solved, such as laminating aluminium sheets, reducing the thickness of steel or aluminium cans, manufacturing single crystal turbine blades and combining dissimilar materials", comments Michel Rappaz, EPFL professor and Director of the CCMX Metallurgy Education and Research Unit (MERU). CCMX builds bridges between academic researchers in the ETH Domain and companies, so that the most recent research results in key areas for industry are readily accessible and to ensure a continued pool of well-trained skilled professionals. Two projects involving industrial partners and ETH Domain institutes are currently being carried out in MERU. The first of these, neo-metallurgy, focuses on the development of new alloys, processes, and analysis techniques. Novel alloys are very sought after, particularly those that are well adapted to the increasingly exacting demands of the energy production, petrochemical, and aeronautical sectors. However, developing these new alloys and putting them to use in extreme conditions requires a thorough understanding and mastery of the phenomena governing their thermo-mechanical behaviour. Empa, ETH Zurich and the Paul Scherrer Institute (PSI) have joined forces to study the interaction between microstructure and mechanical behaviour of technologically important refractory materials.

Steel for steam turbines

At Empa, doctoral students Thomas Mayer and Son Pham-Minh are studying a bainitic steam turbine rotor steel that softens during low cycle fatigue (LCF) loading due to dynamic recovery processes, and an austenitic steel for steam pipes in nuclear power plants that initially hardens and then softens under LCF conditions. The young engineers' task is to characterise the high temperature fatigue properties of these low-alloy steels and study their dislocation and subgrain structures at different temperatures and under varying degrees of deformation and fatigue. The researchers would like to establish a basis for building deformation models that would be capable of describing the extent to which a material's mechanical properties are dependent on past deformation. They have carried out many short-term fatigue tests at varying degrees of deformation and temperature in Empa's Corrosion and Materials Integrity Laboratory, which has a large range of samples that can be used for short-term testing. These tests can last from a few hours to weeks, during which extremely strict standards must be adhered to. At ETH Zurich and Empa, Mayer and Pham-Minh then characterised the complex microstructure of samples that underwent fatigue testing using microscopy techniques



This set-up is ideal for thermo-mechanical fatigue tests involving relatively rapid temperature changes. For example, it is possible to reproduce the conditions present in turbine rotors. Photo courtesy of Empa.

(transmission electron microscopy, electron backscattered diffraction, and scanning electron microscopy). Colleagues from the Institute of Nuclear Physics at the Czech Republic Academy of Sciences (UJF), specialists in this field, measured the density of dislocations on the test samples. They argue that in order to develop better plasticity models, the physical phenomena taking place in the material during high-temperature fatigue resistance tests must be quantified. Current models generally describe the material's behaviour in a purely phenomenological manner. But the extrapolation of fatigue behaviour observed in simple tests to more complex conditions using real elements has only extremely limited relevance. The models developed by the Empa researchers will improve this situation in the sense that their tests, based on physical factors, are not limited to describing the behaviour of the materials, but also provide physics-based interpretations of the behaviour.

Looking into the heart of alloys

As part of a collaborative project between the Paul Scherrer Institute (PSI), Empa and industrial partners, PhD students Michael Weisser and Stéphane Pierret are studying the mechanisms that define how the microstructure of modern alloys affects their deformation behaviour. By doing mechanical testing while simultaneously making neutron diffraction and X-ray diffraction measurements, the students are trying to show the importance of *in situ* techniques for understanding the dynamic evolution of a material's microstructure when it is subjected to external forces. Their research focuses on two kinds of alloys. The first is a nickel-based single-crystal superalloy used for turbine blades. This bi-phase material's sub-micrometer grain structure is known to become less fine when subjected to pressure under high temperatures, thus changing its performance characteristics. The second kind of alloy is creep-resistant steel, which is studied for its deformation mechanisms and the formation of internal strain. "Up to now, these kinds of materials were studied using traditional methods", notes Weisser, who studied engineering at the University of Erlangen-Nürnberg in Germany. "But thanks to modern analysis methods using synchrotron radiation generated by the Swiss Light Source (SLS) at the Paul Scherrer Institute, we can observe the behaviour of components in multiphase steel under various forcing situations." Pierret adds, "Having this detailed understanding is particularly useful in situations in which materials are subjected to extreme conditions, for example in nickel-based superalloys used in turbines or steel used in reactors." The Empa tests are done under the supervision of Dr. Stuart Holdsworth (Mechanics for Modelling and Simulation). The PSI research (Materials Science and Simulation) is directed by Adjunct Professor Helena Van Swygenhoven, who also teaches at EPFL.

Defect Formation in the Spotlight

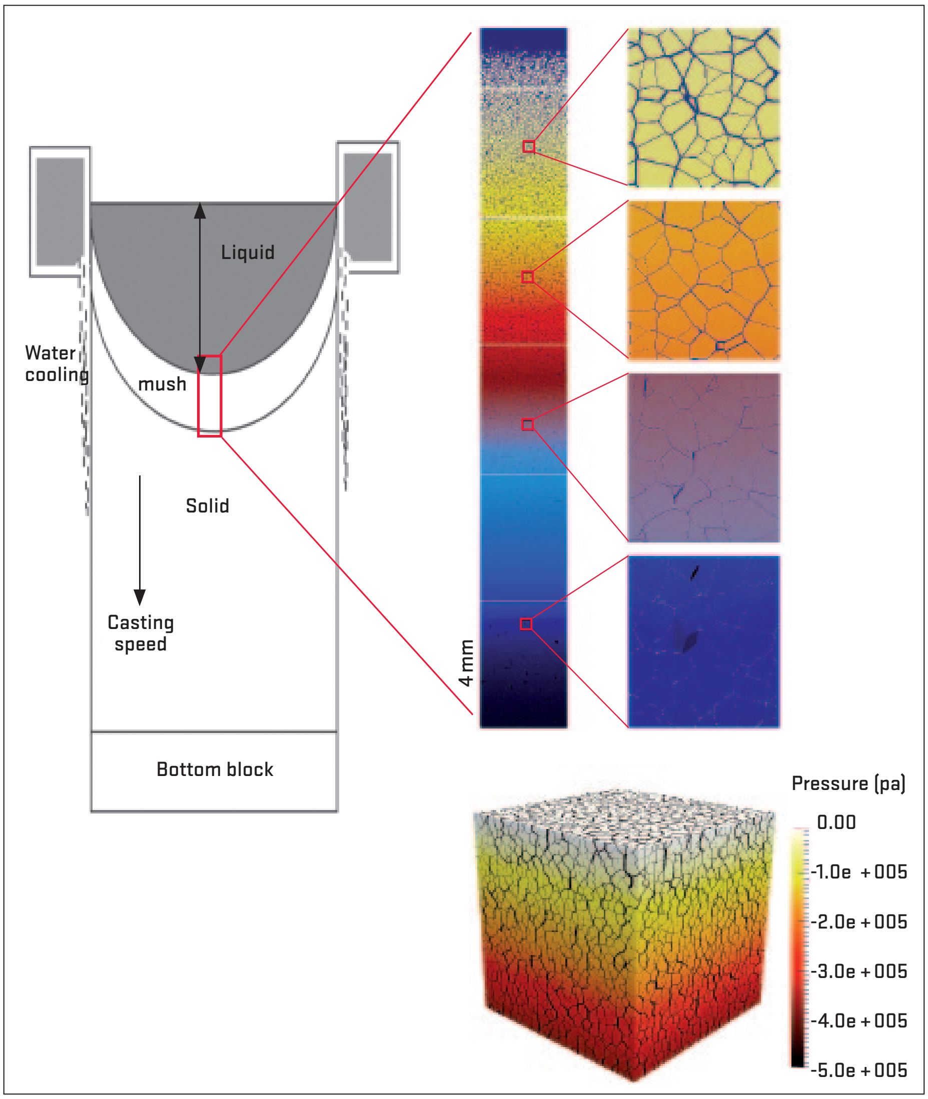
Scientists from EPFL and the Paul Scherrer Institute (PSI) are combining modelling techniques and X-ray tomographic microscopy to penetrate the secrets of defect formation in solidification processes. Their innovative approach is the key to a deeper understanding of materials structure and properties.

Prof. Michel Rappaz with PhD students Hossein Meidani and Meisam Sistaninia. Photo by Nathalie Jongen, CCMX.

Microporosity and hot tearing are two major defects in solidification processes. This is why the team at EPFL's Computational Materials Laboratory (LSMX), headed by Prof. Michel Rappaz, are seeking a better understanding of such defect formation through a combined modelling-experimental approach.

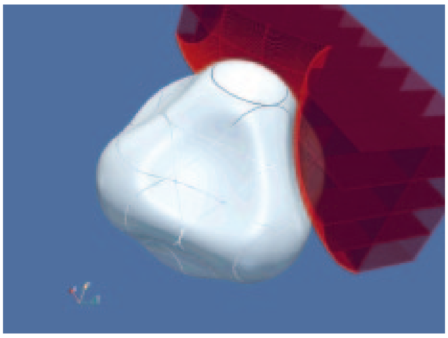
In the first part of this project, the researchers are developing a multiphase-field model incorporating three phases — solid, liquid, and gas — in order to directly describe the morphology of micropores constrained to grow in a solid network. First developed and tested in 2D for simple solid geometry, this model accounts for the curvature of the pores as well as for gas diffusion. The aim was to make a direct comparison with analytical pore pinching models available in the literature. The model was then extended to 3D. To overcome the high computational cost, a new parallelized computer code was developed to distribute the calculations over a user-specified number of processors. The results, obtained in the context of a PhD thesis, show that the phase field model is capable of describing the pore morphology in both 2D and 3D. With the help of the 3D model, "pinching effects" — increase in pore curvature due to limited volume left by a solid — can be investigated in a more realistic way. Moreover, the 3D code will generate results enabling direct comparisons to be made with X-ray tomographic microscopy data obtained at PSI, within the group of Prof. Marco Stampanoni, head of the X-ray Tomography Group at the Swiss Light Source (SLS). Such data provides both the inputs for the actual solid geometry for pore calculations and curvature estimations for validating the calculations. "Based on this experiment-modelling approach, we are trying to derive simplified analytical solutions for pore pinching effects that can be used in a macroscale model of pore formation", concludes Prof. Rappaz.

In the second part of the project, the scientists have chosen a granular dynamic approach to model hot tearing formation in solidifying alloys, a defect that shares similarities with shrinkage-induced microporosity — such as a lack of liquid feeding — but in this case induced by tensile stresses in the coherent solid network. In order to account for the



Right: 2D Hot Tear Simulations

Below: A pore grown between spherical solid grains arranged in a cubic network. While two of the eight solid grains (red) are at gs = 0.7, the pore has penetrated in areas between the solid grains.
Images courtesy of EPFL.



localisation of stresses and liquid feeding at high solid fraction, a Representative Volume Element (RVE) of the solidifying alloy, typically including over a thousand grains, is considered. The 3D model then consists of a solidification module, a mechanical module — a 3D semi-solid deformation model based on a combined Discrete/Finite Element approach — and a fluid flow module. Using a Voroinoï tessellation of randomly distributed nucleation centres, the solidification model generates a set of interconnected triangular elements, representing the intergranular liquid channel elements located between partially coherent grains approximated by a set of polyhedrons. The deformation of the solid is then calculated using the Abaqus(R) software, thus giving access to the openings (closures) of grain boundaries. Combined with solidification shrinkage, this then enables the induced intergranular fluid flow to be calculated using specifically developed software. "We are at the final step of iteratively coupling the fluid flow model and the semi-solid deformation model", says Rappaz. "Hence, we will then use the granular hydro-mechanical coupling to predict and better understand the formation of localized hot tears." The hot tearing model has been applied to the DC casting of round billets in order to see whether experimentally observed defects can be reproduced — meaning, the formation of three curved branches with associated microcracks at the centre of the billet when the casting speed is increased.

In addition to *ex situ* X-ray tomography characterisation performed at PSI, a new set-up for the *in situ* observation of microstructure and defect formation is being built on the SLS TOMCAT beamline. Such direct observations are critical for a better understanding of solidification phenomena. The scientists involved in this project have devised a moderate- to high-temperature furnace made of two diode lasers (980 nm wavelength), each with 150 W of power, symmetrically installed on each side of the X-ray beam. Recently received at PSI, this two-laser system furnace will allow the scientists working in this CCMX project to observe in 3D not only the formation of defects, but also the formation of dendrites and other microstructures, key factors in understanding and improving solidification processes.

Nanoparticles – Put to the Acid Test

In the CCMX project VIGO, the group headed by Professor Harald Krug at Empa, is developing a platform for evaluating the possible biological effects of nanoparticles. EPFL synthesises and characterises various types of nanoparticles, and this work shows that comprehensive particle characterisation is a prerequisite for understanding the behaviour of particles in physiological media and their influence on test methods.

Photos by Elsbeth Heinzelmann

Thanks to nanotechnology, fantastic products are now within reach, whether for innovative materials that consume fewer resources, for environmentally friendly procedures, or for optimised diagnostics and medical therapies.

Knowledge about nanoparticles a 'must'

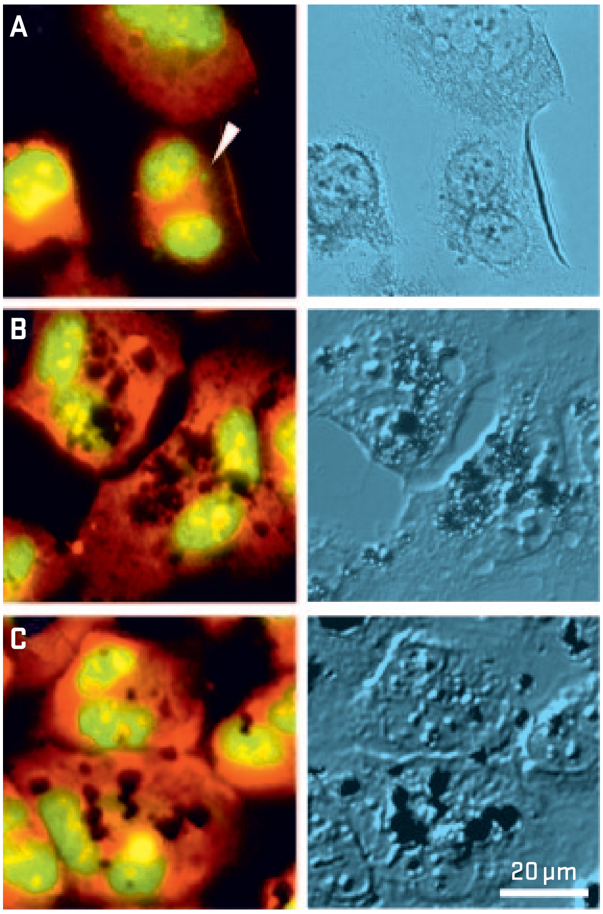
Although in nanotechnology — maybe much more so than in any past technology — scientists began to discuss potential risks at a very early stage, we still know precious little about the possible impact of synthetic nanomaterials on the health and safety of humans and their environment. Standard guidelines on health and safety at work and waste disposal are essential in an industrial environment. But for the research community, it is quite challenging to make safety evaluations when production volumes are not tailored to the nanoscale, and, neither toxicological data nor appropriate methods are available for defining risk thresholds or exposure limits. Prof. Harald Krug and his team at Empa St. Gallen, intend to bridge this gap. In the VIGO project they are developing an *in vitro* evaluation system that will explore the four key aspects of cytotoxicity: Viability, Inflammation, Genotoxicity and Oxidative stress. The aim is to determine, describe, and compare the potential biological effects of nanoparticles with at least two highly standardised, validated, reproducible, and reliable test methods for each of the four endpoints. The platform should allow measurements with a high degree of accuracy, sensitivity, and specificity, and to characterise different nanomaterials in terms of their surface chemistry, particle size distribution, shape, and crystallinity.

Measuring viability

In a first step, the scientists turned their attention to suitable *viability* assays. To this end, they implemented comprehensive controls capable of assessing nanomaterial interference with processed and unprocessed MTS assay reagents. "This allowed us to pre-test every nanomaterial of interest in terms of undesirable effects independent of cellular reactions", explains Dr Cordula Hirsch, a postdoc biologist at Empa. "Together with the optimised plate layout and pipetting scheme, this viability assay now serves as an adequate and fast screening tool for measuring dose-response-curves for any non-interfering nanomaterial."

Inflammation – natural reaction to infection

Regarding the topic *inflammation*, the Empa researchers are cooperating with colleagues from all over Europe in the NANOMMUNE project. Funded by the European Commission, this partnership is committed to a comprehensive assessment of engineered nanoparticles, with a particular focus on effects on the immune system. A plate layout and an experimental set-up have been created to address nanomaterial interference at several levels of an enzyme-linked immunosorbent assay (ELISA). As indicated by preliminary data, this approach makes it possible to detect potential quenching effects of coloured particles, signal reduction due to agglomeration or aggregation



Genotoxicity was analyzed using the Comet Assay in the lung epithelial cell line A549. This assay detects changes in the genome as e.g. chromosomal aberrations or whole chromosome loss. These changes becomes obvious in a small micronucleus (arrow head in Fig A). The dye Acridine Orange is used to stain the cytoplasm red and nuclei green. (A) Control sample of untreated cells. (B) A549 cells were treated for 24 hours with 11 g/cm² multi-walled carbon nanotubes (MWCNT) or 25 g/cm² carbon black (CB) (C), respectively. Due to the clearly visible black precipitates on top or in the cells a reliable evaluation of the test results is not possible. This particular assay should not be used for nanoparticles that form this kind of agglomerates.
Image courtesy of Empa.

events with proteins as well as signal-enhancing effects due to particle-intrinsic activities. "This kind of pre-testing is a prerequisite for reliable, comparable, and robust test methods", concludes Dr. Hirsch.

Genotoxicity – integrity at risk

Genotoxicity refers to the harmful effects of specific substances on the genetic information contained in organisms. Although many different factors can affect DNA, RNA and other genetic materials, the aspect of genotoxicity is only concerned with substances that actually cause harm to the genetic information. In order to detect DNA-strand breaks and alkali labile sites, a prerequisite for evaluating a genotoxicity test, the scientists chose, on the one hand, the alkaline single cell gel electrophoresis or comet assay and, on the other hand, the cytokinesis-block micronucleus cytome assay. "This comprehensive system, which is OECD certified for standard chemicals, is able to measure chromosome breakage and/or whole chromosome loss as well as cytostatic and cytotoxic effects", comments Hirsch. A third assay to be established detects the most common DNA lesion resulting from reactive oxidative species: 8-oxoguanine. The scientists analyse the modified DNA residue immunochemically and quantify the amount of damage by flow cytometry.

Oxidative stress = oxidative damage

Finally, they had to deal with *oxidative stress*. This results from an imbalance between the production of reactive oxygen and the ability of a biological system to detoxify the reactive intermediates and repair the resulting damage. In a first step, the researchers used the dichlorofluorescein (DCF) assay, a simple assay that detects an increase in reactive oxygen species (ROS). The presence of ROS transforms a non-fluorescent substrate into its fluorescent form DCF. "The measurement can easily be done in 96-well plates, allowing for a high-throughput in the future", Hirsch explains. "In preliminary experiments we were able to implement an internal positive control and check for nanoparticle interference. As a result, we succeeded in defining an efficient set-up for detecting possible quenching effects."

Characterisation – the key to understanding particle behaviour

Prof. Heinrich Hofmann at EPFL's Powder Technology Laboratory (LTP) is responsible for synthesising and characterising nanoparticles with different methods. "His work gives us valuable insights and confirms that only through comprehensive particle characterisation will we be able to understand the behaviour of nanoparticles in physiological media and their influence on the test methods", states Harald Krug, who has been studying the opportunities and risks associated with nanotechnology for 20 years.

Efficient and reliable tools for determining, describing and comparing the biological effects on nanoparticles could have a considerable impact on animal testing. Currently, such animal testing seems to be unavoidable, as we need to be sure that chemical products do not have deleterious effects on humans and their environment. This is a consequence of REACH (Registration, Evaluation, Authorisation and restriction of Chemical substances), the European Community Regulation on chemicals and their safe use, that has been in force since 1 June 2007.

Just how important the research within the VIGO project is for the economy is shown by the fact that the project is being supported by companies like Wessling GmbH in Germany, one of the leading analytical and testing companies in the field of quality, safety, environment and health, and by the Swiss retailers' association (IG DHS). "They realise that it is only once they have everything under control in the domain of nanomaterials that they will be able to achieve success in the global markets in a sustained manner", concludes Prof. Krug. The VIGO project has three more years to produce 'airtight' assays that are made-to-measure for industrial use.

3D Tissue Imaging for 'Nanomedicine'

The German company ION-TOF GmbH and Basel University Hospital are working with Empa researchers in Dübendorf to develop a label-free sectioning apparatus for 3D tissue imaging. Based on a time-of-flight secondary ion mass spectrometer (TOF-SIMS), the Microtome4SIMS realized within a CCMX project is particularly suitable for analyzing biological samples with a view to creating nanoscale objects for medical use.

Iñaki Maiztegui, Technical Specialist at Empa (left) and Dr Mirko Milas, specialised in in-situ *manipulation and characterisation of nanomaterial, inserting a sample into the microtome device.*

Photo by Elsbeth Heinzelmann

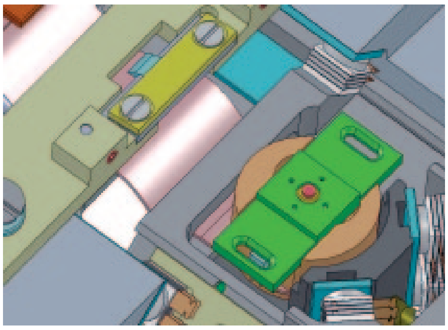
Synthetic nanoscale objects with complex functionality such as 'nanocontainers' have the potential for use in novel medical applications including cancer therapies because they can selectively target the diseased organs. There can be no doubt that they enhance effectiveness and reduce side effects, but it is still not clear how these objects interact with a healthy body.

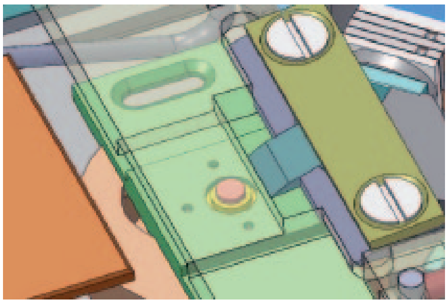
Collaboration brings about innovation

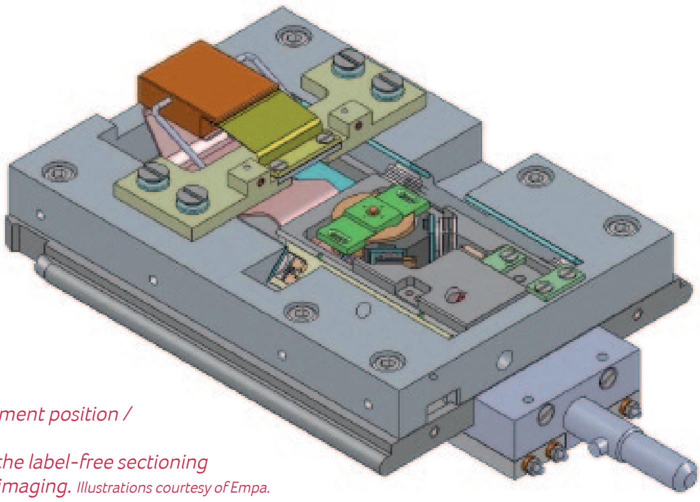
In order to gain insight into the processes and interactions taking place between the synthetic organelles with their active agents and the surrounding tissue, researchers at Empa had the idea of developing an *in-situ*, low-temperature, precision-segmenting stage for the analysis of biological material. The resulting microtome had to be integrated in their time-of-flight secondary ion mass spectrometer (TOF-SIMS). This very sensitive surface analytical technique provides detailed elemental and molecular information about the surface of the sample and gives a full 3D analysis. After bombardment of a surface with energetic projectiles, the emitted secondary species originating from the topmost layers of the material are collected and analysed with high mass resolution. The parallel detection scheme not only provides spectral information with a high degree of accuracy, but also allows the instrument to be operated in a microprobe mode to obtain chemically resolved images of the surface. TOF-SIMS is applicable for various materials, from semiconductors, polymers and paint to coatings, glass, metals and ceramics, to biomaterials, pharmaceuticals and organic tissues. The following researchers are cooperating on the CCMX project under the aegis of Dr. Beat Keller, Empa Advanced Materials and Surfaces: PD Dr. Patrick Hunziker, chief physician at Basel University Hospital and a specialist in copolymer nanocontainers for active drug delivery, and the German company ION-TOF GmbH, known for its innovative ion beam technology for surface analysis.

Pioneering work a 'must'

The development was quite a challenge. First, a handling and transport procedure for samples had to be realised and a method devised to transfer the sample inside the TOF-SIMS chamber without losing structural integrity of the material. Next, the geometric and size restriction of the piezo-motor assembly and the movement range of the piezo-motors were defined. Finally, an electronic concept was designed for the piezo-motor control and the temperature measurement. For an effective surface analysis, the biological material, with maximum 5 mm diameter and 200 µm thickness, must be in a frozen state. After freezing, the samples are transported and stored inside a liquid nitrogen container. For insertion into the TOF-SIMS, they are fixed on a sample holder for safe and simple handling in vacuum. While Empa was responsible for the design and implementation, Basel University Hospital prepared the biological samples and ION-TOF GmbH took care of the operational integration and electronics engineering. ETH Zurich helped with the high pressure freezing and the sample handling, while Diatome AG assisted with the selection of the diamond blades and sample cutting.







Cuting position / Measurment position / Microtome device
Computer simulation of the label-free sectioning apparatus for 3D tissues imaging. Illustrations courtesy of Empa.

"One of the trickiest problems in sample handling was to make sure that the frozen biological samples reach the inside of the TOF-SIMS without thawing or condensation", comments Dr. Mirko Milas, specialised in *in-situ* manipulation and characterisation of nano-materials. "We succeeded by slightly modifying the existing TOF-SIMS loading chamber — load-lock — and by adding a secondary loading chamber connected with load-lock through the vacuum valve". The atmosphere in the secondary loading chamber is exchanged with nitrogen and vacuum-pumped to the level of approximately 1 mbar. Using a wobble stick, the sample is transferred to the pre-cooled microtome stage waiting inside the load-lock. The microtome with the sample is moved inside the TOF-SIMS main chamber and connected to the cooling pad. This ensures that the sample remains frozen and free of contamination or condensation.

Delicate manipulation

The distance between the sample surface and the extraction lens of the TOF chamber is only 1.5 mm. There are several components in the immediate vicinity, such as the primary ion source guns or the electron flood gun. A special design prevents them from coming into contact with parts of the sample holder or the sample itself, avoiding any hardware damage. The researchers also ensured that sample cooling was not interrupted during manipulation and that the sample tissue structure is preserved. Of the two independently driven microtome stages, one determines the cutting thickness (z-axis) and the other performs the cutting (x-axis). The sample is moved relative to the fixed diamond blade using stick-slip shear piezo-motors. "We chose these motors as they guarantee very precise movement over macroscopic distances", explains Iñaki Maiztegui, Technical Specialist at Empa. The cutting procedure is relatively simple and straightforward. The researchers start with the sample in the measuring position and move the sample along the x-axis behind the diamond blade into the cutting position. Thanks to the z-axis, they are able to lift the sample for the desired cutting thickness and then move it back to the measuring position. The sample is cut and the freshly exposed surface is ready for imaging. "Once the TOF-SIMS is aligned and focused on the surface, each new cut is made in exactly the same focal plane, making it possible to automate the data acquisition", adds Iñaki Maiztegui.

Before fixing the diamond blade on the microtome, the researchers attached the blade to a metallic shaft and polished it. "We chose this configuration to produce a variety of possible diamond shapes and to facilitate the handling of the diamond itself", explains Dr. Milas, who formerly worked at the Center of Functional Nanomaterials at the Brookhaven National Laboratory. "We also realized a testing stage and succeeded in performing a cut on the frozen sample and obtaining reliable and controllable movement of the piezo-motors."

The prototype entered production at the end of 2010 and has already resulted in a further collaboration between Empa and Basel University Hospital in the "Smart Materials" National Research Programme NRP62.

New Ground for Materials Science

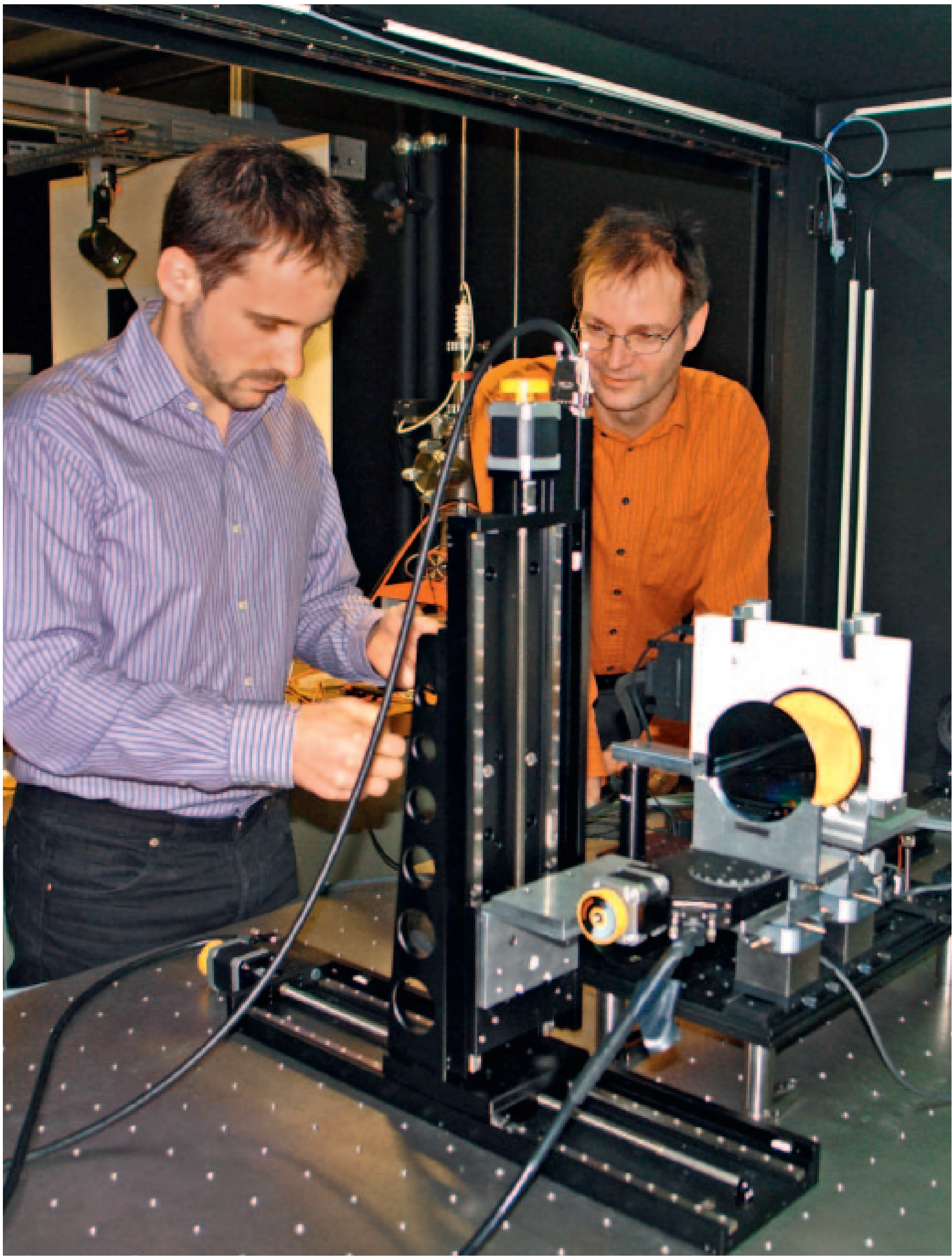


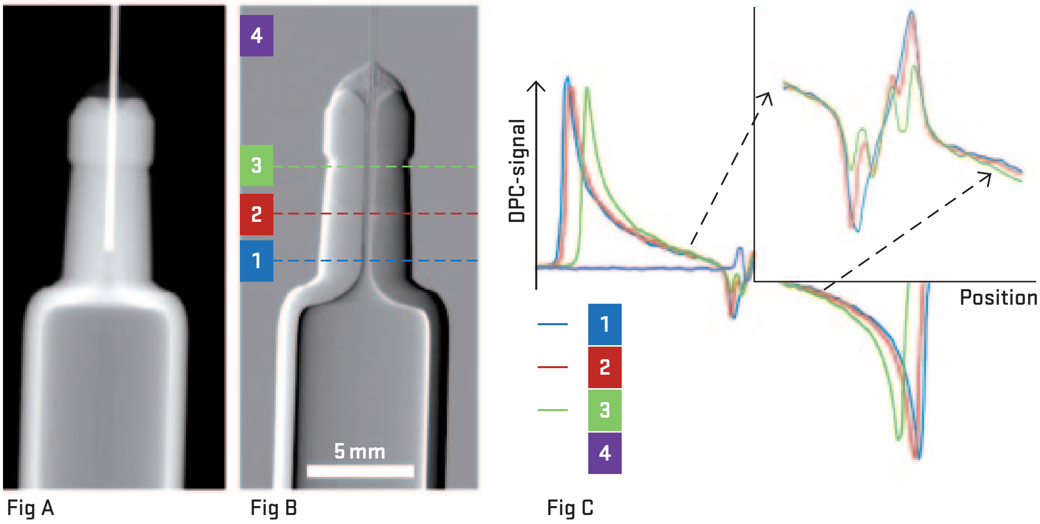
Photo by Elsbeth Heinzelmann

Experts in optoelectronic systems at CSEM are extending the capacity of x-rays by developing a system capable of reducing the exposure to radiation and producing much better contrast with enhanced image quality. Their innovation opens up new possibilities in materials science and medical technology.

Ever since 1895, when Roentgen discovered that x-rays can pass through human tissue and visualise bones and metals, radiology has been the diagnostic technique par excellence for materials science, life sciences and medical applications. For years it was based on the absorption of x-rays, making it possible to explore the inside of objects, though hampered by limited contrast when used on weakly absorbing specimens. This shortcoming has a negative impact on examinations like screening for breast cancer or cardiovascular diseases. Scientists at the Paul Scherrer Institute (PSI) wanted to remedy this situation. With the help of advanced x-ray sources based on synchrotron radiation at the Swiss Light Source (SLS), they produced monochromatic x-rays with a high level of spatial coherence. As a result they were able to access radiology based on refraction and other phase-related phenomena. They succeeded in extending radiology down to the microscopic scale, with a spatial resolution better than 1 micron and real-time monitoring below 1 millisecond, and obtained an excellent image quality. Nevertheless, such synchrotron-based technologies are too costly to be used in mainstream industrial applications and need to be translated to conventional x-ray systems. In fact, researchers have demonstrated that phase contrast technology is also possible when using conventional x-ray tubes.

At the front line of research

At CSEM, Dr. Claus Urban, Section Head Optoelectronic Systems, took this matter in hand. He set up a team of Empa and CSEM scientists and in the summer of 2008, assisted by Ph.D. student Vincent Revol, he initiated a project supported by CCMX for constructing and building a new x-ray instrument for material characterisation suitable for use in industrial quality control. It is based on differential phase contrast imaging. This means that — in contrast with classical x-ray imaging — the phase shift of x-ray radiation transmitted through



A) and (B) show the absorption radiograph and the differential phase contrast image of a syringe, respectively. The syringe is composed of a metal needle glued into a glass body. The coverage of glue along the needle is of interest for the determination of the resistance of the needle to pull-out forces. This task is typically challenging for absorption-based imaging but becomes achievable thanks to differential phase contrast imaging. The latter method is indeed more sensitive to light elements, like polymers. The profiles of the differential phase contrast signal in (C) represent vertical positions through the syringe only (1), syringe and needle without (2) and with (3) glue and through the needle only (4) and illustrate the previous statement.

Figures courtesy of CSEM.

an object, rather than its absorption, is probed by means of an interferometer. This allows for the characterisation of materials with low atomic weight, like polymers or ceramics with x-ray imaging. Since phase contrast imaging is highly sensitive to interfaces, the image contrast is improved for heterogeneous or composite materials like fibre-reinforced polymers or multilayer systems.

To achieve their goals, the scientists chose x-ray design energies up to 50 keV, corresponding to a tube voltage of approx. 80 kV. These x-ray tube voltages required special interferometer gratings for a wide field of view, and were developed by their CSEM colleagues in Neuchâtel, who are experts in this domain. A stable fabrication process was established and the gratings were supplemented by appropriate holders and alignment procedures. Their innovation was to create *bent* gratings. Since x-rays are produced in a point-like source, the objects are generally illuminated with a cone beam rather than a parallel one. Consequently, the angle of incidence at the periphery of the field of view can be quite high in a compact apparatus. Since the gratings are optimised for normal incidence, the image contrast decreases towards the periphery in the direction perpendicular to the grating lines. The CSEM specialists managed to avoid this decrease by using cylindrically bent gratings.

Reaching the finish

"One of the highlights was the successful implementation of the dual-energy imaging with one set of gratings", comments Vincent Revol, who is writing his thesis on this challenging development. "This allows us to carry out measurements with two different x-ray energies in the same geometry." In order to obtain a system suitable for industrial use, the researchers had to develop stable and user-friendly software for laboratory automation, data acquisition, and image reconstruction. It also proved necessary to optimise the algorithms for tomographic phase image reconstructions and to perfect the acquisition of sample images.

The laboratory version, completed during 2010, is a real success. "Our measurements on weakly absorbing materials and on composite materials show that additional information can be obtained from phase contrast measurements compared to classical x-ray imaging", concludes Dr. Claus Urban. "We can reduce the radiation exposure and obtain a much higher contrast with optimal image quality". He envisages a wide range of applications, both for materials technology with polymers and ceramics, and for medical technology. X-ray phase contrast imaging could be particularly helpful in mammography, where mass screening remains controversial due to the still unknown impacts of radiation. The new method is also valuable in cases where, previously, the tissue has prevented precise interpretation of mammograms.

With respect to materials technology, the results achieved are amazing, as demonstrated by a metal needle glued into a glass syringe, which is a fine example of a component consisting of a combination of strongly and weakly absorbing materials. The glue has to extend into the syringe sufficiently to hold the needle, but it should not flow into the fluid-containing section of the syringe (see image). The differential phase contrast image gives a clear indication of how far the glue extends into the syringe body. "These details cannot be visualised with classical absorption imaging, since the strong absorption of the steel needle completely conceals the weak signal from the glue", states Dr. Urban. Pioneering investigations have been undertaken with Fibre Composite Materials. "For the first time it has proved possible to show delamination processes in a clear and unequivocal way". Considering the growing demand for these materials, primarily in the transport sector, and the need for reliable quality checks, the new development faces a bright future.

2010 Peer Reviewed Publications

SPERU

A. Tricoli, S.E. Pratsinis, **Dispersed nanoelectrode devices**, *Nature Nanotechnology*, 5 (2009) 54-60.

A. Evans, A. Bieberle-Hütter, H. Galinski, J.L.M. Rupp, T. Ryll, S. Barbara, R. Tölke, L.J. Gauckler, **Micro-solid oxide fuel cells: status, challenges, and chances**, *Chemical Monthly*, 140 (2009) 975-983.

N.I. Karageorgakis, A. Heel, A. Bieberle-Hütter, J.L.M. Rupp, T. Graule, T., L.J. Gauckler, L.J., **Flame spray deposition of $La_{0.6}Sr_{0.4}CoO_{3-\partial}$ thin films: Microstructural characterization, electrochemical performance and degradation**, *Journal of Power Sources*, 195 (2011) 8152-8161.

S. Heiroth, T. Lippert, A. Wokaun, M. Döbeli, J. L. M. Rupp, B. Scherrer, L. J. Gauckler, **Yttria-stabilized zirconia thin films by pulsed laser deposition: Microstructural and compositional control**, *J. Eur. Ceram. Soc.*, 30 (2010) 489-495.

S. Heiroth, J. Koch, T. Lippert, A. Wokaun, D. Günther, F. Garrelie, M. Guillermin, **Laser ablation characteristics of yttria-doped zirconia in the nanosecond and femtosecond regimes**, *J. Appl. Phys.*, 107 (2010) 014908.

J.L.M. Rupp, B. Scherrer and L.J. Gauckler, **Engineering disorder in precipitation-based nano-scaled metal oxide thin films**, *Phys. Chem. Chem. Phys.*, 12 (2010) 11114-11124.

J.L.M. Rupp, B. Scherrer, N. Schäuble and L.J. Gauckler, **Time-temperature-transformation (TTT) diagrams for crystallization of metal oxide thin films**, *Adv. Funct. Mater.*, 20 (2010) 2807-2817.

J.L.M. Rupp, U.P. Muecke, P.C. Nalam and L.J. Gauckler, **Wet-etching of precipitation-based thin film microstructures for micro-solid oxide fuel cells**, *Journal of Power Sources*, 195 (2010) 2669-2676.

T. Ryll, S. Ellenbroek, A. Brunner, A. Bieberle-Hutter, J.L.M Rupp, L.J. Gauckler, **Crystallization and electrical properties of amorphous bismuth ruthenate thin films deposited by spray pyrolysis**, *Phys. Chem. Chem. Phys.*, 12 (2010) 13933-13942.

N.I. Karageorgakis, A. Heel, J.L.M. Rupp, M.H. Aguirre, T. Graule, L.J. Gauckler, **Properties of flame sprayed $Ce_{0.8}Gd_{0.2}O_{1.9-\partial}$ electrolyte thin films**, *Adv. Funct. Mater.*, 21 (2011) 532-539.

F. Krauss Juillerat, U. T. Gonzenbach, A. R. Studart, and L. J. Gauckler, **Self-setting particle-stabilized foams with hierarchical pore structures**, *Materials Letters*, 64 (2010) 1468-1470.

J.C.H. Wong, E. Tervoort, S. Busato, U. T. Gonzenbach, A. R. Studart, P. Ermanni, and L. J. Gauckler, **Designing macroporous polymers from particle-stabilized foams**, *Journal of Materials Chemistry*, 20 (2010) 5628-5640.

F. Krauss Juillerat, U. T. Gonzenbach, P. Elser, A. R. Studart, and L. J. Gauckler, **Microstructural control of self-setting particle-stabilized ceramic foams**, *Journal of the American Ceramic Society*, 94, (2011) 184-190.

B. Gallinet, A. M. Kern, and O. J. F. Martin, **Accurate and versatile modeling of electromagnetic scattering on periodic nanostructures with a surface integral approach**, *Journal of the Optical Society of America A: Optics and Image Science, and Vision*, 27 (2010) 2261-2271.

B. Gallinet, and O. J. F. Martin, **Scattering on plasmonic nanostructures arrays modeled with a surface integral formulation**, *Photonics and Nanostructures - Fundamentals and Applications*, 8 (2010) 278-284.

W. Zhang, B. Gallinet, and O. J. F. Martin, **Symmetry and selection rules for localized surface plasmon resonances in nanostructures**, *Physical Review B - Condensed Matter and Materials Physics*, 81 (2010). DOI: 10.1103/PhysRevB.81.233407.

T. Ryll, J.L.M Rupp, A. Bieberle-Hutter, A. Galinski, L.J. Gauckler, **Characterization of thin films for solid oxide fuel cells facilitated by micropatterning**, *Scripta Materialia*, in press (2011).

N.J. Simrick, J.A. Kilner, A. Atkinson, J.L.M. Rupp, T.M. Ryll, A. Bieberle-Hütter, H. Galinski, L.J. Gauckler, **Micro-fabrication of patterned LSCF thin-film cathodes with gold current collectors**, *Solid State Ionics*, in press (2011).

MatLife

P. Gunde, A. Furrer, A.C. Hänzi, P. Schmutz, P.J. Uggowitzer, **The influence of heat treatment and plastic deformation on the bio-degradation of a Mg–Y–RE alloy**, *Journal of Biomedical Materials Research A*, 92A (2010) 409-418.

M. Rottmar, M. Håkanson, M. Smith, K. Maniura-Weber, **Stem cell plasticity, osteogenic differentiation and the third dimension**, *J Mater Sci Mater Med.*, 21 (2010) 999–1004.

O.V. Semenov, S. Koestenbauer, M. Riegel, N. Zech, R. Zimmermann, A.H. Zisch, A. Malek, Multipotent mesenchymal stem cells from human placenta: critical parameters for isolation and maintenance of stemness after isolation, *Am J Obstet Gynecol.*, 202 (2010) 193. e1-193.e13.

A.C. Hänzi, I. Gerber, M. Schinhammer, J.F. Löffler, P.J. Uggowitzer, On the in vitro and in vivo degradation performance and biological response of new biodegradable Mg–Y–Zn alloys, *Acta Biomaterialia*, 6 (2010) 1824-1833.

M. Ochsner, M. Textor, V. Vogel, M.L. Smith, Dimensionality controls cytoskeleton assembly and metabolism of fibroblast cells in response to rigidity and shape, *PLoS One*, (2010). DOI 10.1371/journal. pone.0009445.

A. J. van der Vlies, C. P. O'Neil, U. Hasegawa, N. Hammond, J. A. Hubbell, Synthesis of pyridyldisulfide-functionalized nanoparticles for conjugating thiol-containing small molecules, peptides and proteins, *Bioconjugate Chemistry*, 21 (2010) 653-662.

S. Bürgel, O. Guillaume-Gentil, L. Zheng, J. Vörös and M. Bally, Zirconium ion mediated formation of liposome multilayers, *Langmuir*, 26 (2010) 10995-11002.

O. Guillaume-Gentil, N. Graf, F. Boulmedais, P. Schaaf, J. Vörös, and T. Zambelli, Global and local view on the electrochemically induced degradation of polyelectrolyte multilayers: from dissolution to delamination, *Soft Matter*, 6 (2010) 4246-4254.

L. Isa, K. Kumar, M. Müller, J. Grolig, M. Textor, E. Reimhult, Particle lithography from colloidal self-assembly at liquid-liquid interfaces, *AVS Nano*, 4 (2010), 5665-5670.

G. Weder, O. Guillaume-Gentil, N. Matthey, F. Montagne, H. Heinzelmann, J. Vörös, M. Liley, The quantification of single cell adhesion on functionalized surfaces for cell sheet engineering, *Biomaterials*, 31 (2010) 6436-6443.

O. Guillaume-Gentil, D. Abbruzzese, E. Thomasson, J. Vörös, T. Zambelli, Chemically tunable electrochemical dissolution of noncontinuous polyelectrolyte assemblies: An *in situ* study using ecAFM, *ACS App. Mater. Interfaces*, 2 (2010) 3525-3531.

O. Guillaume-Gentil, M. Gabi, M. Zenobi-Wong, J. Vörös, Electrochemically switchable platform for the micro-patterning and release of heterotypic cell sheets, *Biomedical Microdevices*, 13 (2010) 221-230.

A. Sala, M. Ehrbar, D. Trentin, R.G. Schoenmakers, J. Vörös, F.E. Weber, Enzyme mediated site-specific surface modification, *Langmuir*, 26 (2010) 11127-11134.

MERU

M. Rappaz, F. Kohler, J. Valloton, A.B. Phillion, M. Stampanoni, Connectivity of phases and growth mechanisms in peritectic alloys solidified at low speed: an x-ray tomography study of Cu-Sn, *Met. Mater. Trans.*, A 41 (2010) 563-567.

J.-M. Drezet, M. Sistaninia and M. Rappaz, Modeling of hot tearing: two-phase models, coalescence and mesoscale granular models, *Matériaux & Techniques*, 98 (2010) 261–267.

M. Sistaninia, A.B. Phillion, J.-M. Drezet, M. Rappaz, Simulation of semi-solid material behavior using discrete finite element method, *Met. Mater. Trans.*, 42A (2011) 239-48.

H. Meidani, A. Jacot, Phase-field simulation of micropores constrained by the dendritic network during solidification, *Acta Mater*, in press (2011).

MS Pham, C Solenthaler, KGF Janssens, SR Holdsworth, Dislocation structure evolution and its effects on cyclic deformation response of AISI316L stainless steel, *Mat. Sci. Eng. A*, in press (2011).

MMNS

E.P. Dupont, E. Labonne, C. Vandevyver, U. Lehmann, E. Charbon, M. Gijs, Monolithic silicon chip for immunofluorescence detection on single magnetic beads, *Anal. Chem.*, 82 (2010) 49–52.

E.P. Dupont, R. Luisier, M. Gijs, NOA 63 as a UV-curable material for fabrication of microfluidic channels with native hydrophilicity, *Microelectronic Engineering*, 87 (2010) 1253-1255.

B. Städler, M. Limacher, N. Li, J. Vörös, Photobleaching induced damage of biomolecules: streptavidin as 'bio'-photoresist, *Surface Science*, 604 (2010) 898-905.

2010 Peer Reviewed Publications

R. MacKenzie, C. Fraschina, T. Sannomiya, J. Vörös, **Controlled *in situ* nanoscale enhancement of gold nanowire arrays with plasmonics**, *Nanotechnology*, 22 (2011) 055203.

NMMC

P. Corfdir, P. Lefebvre, J. Ristic, J.-D. Ganière, B. Deveaud-Plédran, **Electron localization by a donor in the vicinity of a basal stacking fault in GaN**, *Physical Review B*, 80 (2009) 153309.

P. Corfdir, J. Ristic, P. Lefebvre, T. Zhu, N. Grandjean, B. Deveaud-Plédran, **Low-temperature time-resolved cathodoluminescence study of exciton dynamics involving basal stacking faults in a-plane GaN**, *Applied Physics Letters*, 94 (2009) 201115.

P. Corfdir, P. Lefebvre, J. Levrat, A. Dussaigne, J.-D. Ganière, D. Martin, J. Ristic, T. Zhu, N. Grandjean, B. Deveaud-Plédran, **Exciton localization on basal stacking faults in a-plane epitaxial lateral over-grown GaN grown by hydride vapor phase epitaxy**, *Journal of Applied Physics*, 105 (2009) 043102.

P. Corfdir, P. Lefebvre, J. Levrat, J. Ristic, P. Valvin, E. Calleja, A. Trampert, J.-D. Ganière, B. Deveaud-Plédran, **Time-resolved spectroscopy on GaN nanocolumns grown by plasma assisted molecular beam epitaxy on Si substrates**, *Journal of Applied Physics*, 105 (2009) 013113.

T. Colomb, S. Krivec, H. Hutter, A. Akatay, N. Pavillon, F. Montfort, E. Cuche, J. Kühn, C. Depeursinge, Y. Emery, **Digital holographic reflectometry**, *Opt. Express*, 18 (2010) 3719-3731.

T. Sannomiya, T. Balmer, C. Hafner, M. Heuberger, J. Vörös, **Optical sensing and determination of complex reflection coefficients of plasmonic structures using transmission interferometric plasmonic sensor**, *Rev. Sci. Instr.*, 81 (2010) 053102

C. Roth, D. Passerone, L. Merz, M. Parschau, K.-H. Ernst, **Two-dimensional self-assembly of malic acid on Cu(110)**, *J. Phys. Chem. C*, 115 (2011) 1240-1247.

P. Corfdir, P. Lefebvre, L. Balet, S. Sonderegger, A. Dussaigne, T. Zhu, D. Martin, J.-D. Ganière, N. Grandjean, B. Deveaud-Plédran, **Exciton recombination dynamics in a-plane (Al,Ga)N/GaN quantum wells probed by picosecond photo and cathodoluminescence**, *Journal of Applied Physics*, 107 (2010) 043524.

C. Roth, K.-H. Ernst, **Surface explosion decomposition chemistry of malic acid on Cu(110)**, *submitted to Top. Catal.*

C. Roth, M. Parschau, K.-H. Ernst, **Chiral reconstruction of a metal surface by adsorption of racemic malic acid**, *submitted to Chem Phys Chem.*

2010 Data

Use of funding in 2010 (KCHF)	
Funding of projects	4'307
Education activities, conferences	149
Industrial liaison	141
Management & administration	403
Total	**5'000**

Metrics for 2010

17	Running projects
16	Professors involved in projects
3	Senior scientists (FTE)
4	Post docs paid by CCMX (FTE)
0.3	Post docs not paid by CCMX (FTE)
9	PhD students paid by CCMX (FTE)
1	PhD students not paid by CCMX (FTE)
34	Publications (peer reviewed)
4	Patents

Projects Funded in 2010

Title of project	Principal Investigator (PI)	PI's Institution	Other Institutions	ERU/ Platform
Studying single cells in engineered 3D microenvironments	**Viola Vogel**	ETH Zurich	ETH Zurich, Empa	MatLife
Colored ceramic surfaces for metallic dental implants and prosthetic appliances	**Ralph Spolenak**	ETH Zurich	EPFL, Empa, UNIZ	MatLife
V.I.G.O. — A new evaluation tool for determination, description, and comparison of the biological effects of nanoparticles / nanomaterials	**Harald Krug**	Empa	EPFL	MatLife
Serrulatane-based antimicrobial surface platforms	**Marcus Textor**	ETH Zurich	EPFL, Unispital Basel	MatLife
Evolution of microstructure and mechanical response due to cyclic deformation at elevated temperatures	**Stuart Holdsworth**	Empa	ETH Zurich, PSI	MERU
In-situ mechanical testing	**Helena Van Swygenhoven**	PSI	Empa(2)	MERU
Modelling of defect formation in solidification processes using granular dynamics and phase field approaches	**Michel Rappaz / Alain Jacot**	EPFL	Empa, PSI	MERU
Combinatorial study and modeling of optical properties of gold alloys	**Ralph Spolenak**	ETH Zurich	PSI	MERU
Label free imaging of molecular adsorption on *in-situ* surface-functional patterns	**Nicholas Spencer**	ETH Zurich	Empa	NMMC
Nanoscale resolution optical microscopy for material imaging and spectroscopy	**Christian Depeursinge**	EPFL	EPFL, CSEM	NMMC
Vibrational spectroscopy of nanostructured surface systems	**Davide Ferri**	Empa	ETH Zurich, Empa	NMMC
Time-resolved cathodoluminescence (TRCL)	**Jean-Daniel Ganière**	EPFL	ETH Zurich	NMMC
Microtome4SIMS: Chemical tomography of biological material with 100 nm resolution	**Beat Keller**	Empa	Unispital Basel	NMMC
Adaptation of the NanoXAS instrument to a dedicated beamline at the SLS	**Chistoph Quitmann**	PSI	Empa	NMMC
Development of a X-ray phase contrast instrument for the characterisation of materials with low atomic mass	**Claus Urban**	CSEM	Empa	NMMC
Synchrotron phase-contrast nanotomography of fresh and hardened cementitious materials	**Pietro Lura**	Empa	EPFL, PSI	NMMC
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A pore grown between spherical solid grains arranged in a cubic network.