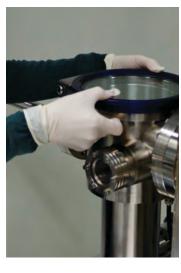


# **Annual Activity Report 2011**

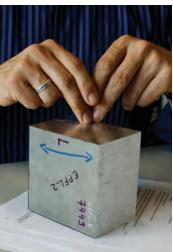




















## **Annual Activity Report 2011**

### **Contents**

Building Enduring Collaboration and Sustainability	2
Education and Outreach Activities	4
CCMX Research Activities	6
An Introduction to Featured CCMX Projects	7
Predicting the Colour of Gold Alloys	8
Investigating the Microstructure of Thick Metal Structures	10
Tuning Nanostructured Ceramics to New Responses	12
Making Dental Implants More Discreet	14
Preventing Infection Thanks to Antimicrobial Surfaces	16
First "Live" View of Cement's Microstructure	18
Turning a Neutron Beam Line into a Unique Instrument	20
2011 Peer Reviewed Publications	22
Projects Funded in 2011	23
CCMX Team 2011	24

## **Building Enduring Collaboration and Sustainabil**



PHOTO ALAIN HERZOG

CCMX Chair Karen Scrivener and Managing Director Nathalie Jongen. The ETH Board formed CCMX in 2006, giving the centre a mandate to build stronger links between the needs of industry and academic research.

Six years later, CCMX has attracted 23 companies supporting 14 projects with funding of more than three million Swiss francs. This investment funds work in fields as diverse as antimicrobial surfaces, *in-situ* mechanical testing and the optical properties of gold, addressing projects that might otherwise fall into the funding gap between research in basic science, typically supported by the Swiss National Science Foundation, and product-oriented development, often funded by the Commission for Technology and Innovation.

Mission accomplished?

"We have been very successful in promoting this interface between industry and academia, but it's never going to be truly finished," said Karen Scrivener, CCMX chair. "There's always more to do."

And CCMX, led by Scrivener and Managing Director Nathalie Jongen, is ready to do what it takes. The focus for the next four years will be on promoting materials science and engineering in Switzerland over the long term. The centre will continue to strengthen the industrial-academic ties that it has worked to build, particularly in pre-competitive research, and forge ahead with successful CCMX-led training and networking activities. But it will also address more fundamental problems — the lack of professors and research activity in fields that are important to industry that is in turn critical to the local economy. The idea now is to build a self-sustaining materials science platform in Switzerland.

"What we hope is that this catalyst will be strong enough for the reaction to continue," Jongen said.

CCMX will support the hiring of four new tenure-track assistant professors in the next two to three years, with two going to EPFL and two to ETH Zurich. The priority areas for

## ity for Materials Science in Switzerland

One significant hurdle is the nature of Materials Science itself — the discipline is highly diverse, with researchers and companies involved in areas as disparate as med-tech and machinery, watches and airplane spars. But even within such diversity, there is a unifying theme — materials are a means to an end rather than ends in themselves.

EPFL are Surfaces and coatings, Electrochemistry and Meso-scale modelling, while those identified at ETH Zurich are Magnetic materials, Soft materials, Multiscale materials modelling, and Multi-level self-assembly.

CCMX is also introducing "Materials Challenges," or research platforms focused on the following fields, identified by partners as "high interest": Advanced metallic materials, Biocompatible surfaces, Functional coatings, Structural coatings, Meso-scale mechanics and fracture of metals. CCMX will keep working with industrial partners to identify a sixth Materials Challenge. Other fields may be added over the next three years.

While the work of the past five years has laid the groundwork, it has also highlighted complexities intrinsically involved in the task at hand.

One significant hurdle is the nature of Materials Science itself. As ongoing projects show, the discipline is highly diverse, with researchers and companies involved in areas as disparate as med-tech and machinery, watches and airplane spars. This makes it difficult to identify first-class projects that could focus a coordinated research effort, and it also requires flexibility and adaptation from CCMX.

"These companies don't have the same priorities," Jongen said. "If you talk with a med-tech company or with a machine or watch company, it's a different approach. I think they're all very keen on developments close to the market, with some, like the med-techs, it's difficult to bring them back to precompetitive research."

But even within such diversity, there is a unifying theme — materials are a means to an end rather than ends in themselves.

"You can make a breakthrough in the materials aspect, but it might still be four or five years before that comes out in a product," Scrivener said. "You have to be a little bit patient and put it into perspective."

Patience and perspective have also played a key role in a critical element of CCMX's success — getting different groups of people to speak the same language and communicate effectively. People working in industry sometimes find it difficult to identify what are the issues in scientific terms, while academic researchers sometimes need assurance that the sort of work they are being asked to undertake is high-level research that they will be able to publish. These different concerns and difficulties are one facet of why the Centre's training, education and outreach activities are so important.

CCMX offers continuing education that includes summer schools, advanced courses and workshops, and also arranges technology aperitifs that give people a chance to learn more about and then discuss various topics: "Solidification of Metal Alloys," "Materials for Energy Applications" and "Optical Effects at Surfaces" have met all with enthusiastic responses from participants. These events introduce companies to the possibilities of research within the ETH Domain, and also sensitise researchers to the ongoing needs of industry. The ultimate goal is for these productive relationships and research collaborations to continue on their own, without CCMX acting as an intermediary.

"The tandem approach we are developing in this five year funding period is meant as long-term strategic reinforcement," Scrivener said. "It is really designed to be something that can carry its own momentum and be a lasting legacy for materials science in Switzerland."

### 2011 Data

Use of funding in 2011 (KCHF)	
Funding of projects	4'444
Education activities, conferences	79
Industrial liaison	145
Management & administration	422
Total	5′090

#### Metrics for 2011

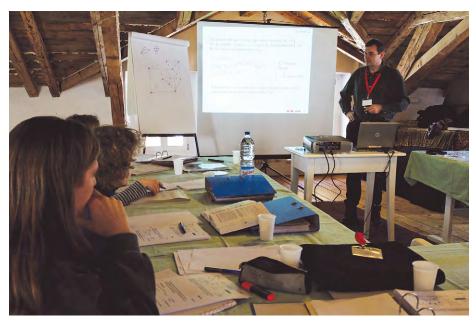
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17

### **Education and Outreach Activities**

Education and outreach activities are one of CCMX's fundamental priorities. The centre organises a variety of advanced training events. The Summer and Winter Schools, offered in alternating years, are designed primarily for doctoral students. Advanced courses and workshops are oriented more towards engineers and scientists from industry who are interested in continuing education and hands-on training, featuring lab visits and the opportunity to transform theory into practise. CCMX has built its training programme by collaborating with motivated lecturers from within its network of academic and industrial subject matter experts. The offering is broad in terms of topics taught, from materials in electronics to materials for the life sciences, and varied in terms of format, from ex-cathedra courses to hands-on workshops. These training events are an occasion to learn, and have also proven to be valuable networking opportunities.

The 2011 CCMX Winter School, "Thin Films: Fundamentals, Structure and Properties", and the Interdisciplinary Training for Young Scientists jointly organised with the NRP62 Smart Materials, "Growth and dissolution of solids on the molecular, cellular and atomic scale" attracted primarily PhD students and were intended as opportunities to create informal connections amont the ETH Domain and other institutions. The two-day advanced course, "Atomic Force Microscopy", emphasising hands-on sessions, gave 21 participants deeper insights into the utility and value of AFM techniques. The contribution of technical experts from the three leading companies Bruker, Nano-Scan and Nanosurf, were essential components contributing to the course's overall success. Based on participant feedback, the most valuable aspect of the course was the blend of theory and practice.

CCMX "Technology Aperitifs" have dual goals of knowledge transfer and networking, where participants meet and discuss materials science in all its diversity. These end-of-afternoon/early-evening get-togethers feature scientific presentations on hot topics to inspire participants' interest in future applications. These presentations are followed by an informal aperitif where people can interact and potentially create contacts for future partnerships.



CCMX Winter School lecture session on thin films, Les Diablerets.



Instrument demo during CCMX Advanced Couse, AFM Theory and Practice, Empa Dübendorf.

"On the whole, the Winter School was an extraordinary opportunity to establish a network among participants and speakers, and to discover the many opportunities that CCMX offers young researchers." Participant in the 2011 CCMX Winter School, Les Diablerets

"When the time came to leave, we packed our blue ring binders full of useful lecture notes, and headed back to the lab with new contacts and a wider perspective of the possibilities for our own research." Participant in the joint NRP62-CCMX Smart Materials Interdisciplinary Training for Young Scientists, Lugano

"I really enjoyed this course. It was a very good overview of AFM and its possible applications, and the hands-on segments offered the chance to compare different instruments and techniques."

Participant in the 2011 CCMX Advanced Course, AFM Theory and Practice, Empa Dübendorf

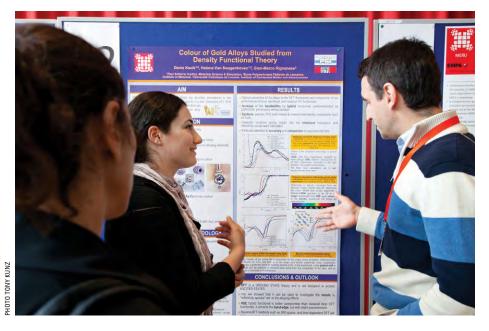
### **Education and Outreach Activities**



Small group discussions at the Interdisciplinary Training for Young Scientists, Lugano.



2011 CCMX Annual Meeting, Bern.



Poster session at the 2011 CCMX Annual Meeting, Bern.

CCMX has organised five annual meetings since 2007. These well-attended meetings address active CCMX members and participants from academia and industry as well as those who do not yet know the Centre.

The programme typically features presentations from researchers and industrial partners involved in current CCMX projects. The poster session offers an opportunity to meet one-on-one with the research teams and to have contact with other materials science experts from the ETH Domain institutions and industry.

### CCMX training activities in 2011

16–21 January CCMX Winter School, Thin Films: Fundamentals, Structure and Properties

25–28 May Joint NRP62–CCMX Interdisciplinary training for young scientists

Growth and dissolution of solids on the molecular, cellular and atomic scale

6–8 June CCMX Advanced Course, Powder Characterisation: From Nanometers to

Millimeters and from Theory to Practice

10–11 November CCMX Advanced Course, Atomic Force Microscopy (AFM): Theory and Practice

### CCMX outreach and networking activities in 2011

2 February Workshop "Materials Challenges for 2020"

5 April CCMX Annual Meeting

3 May Technology Aperitif "Solidification of metallic alloys"

### Research Activities

CCMX creates unique opportunities for interdisciplinary teams from the four ETH Domain institutions and CSEM to reach the critical mass needed for carrying out targeted, pre-competitive research that is relevant to Swiss industry.

Because the application of materials science is very diverse, CCMX has operated at thematic and sectorial levels to facilitate more successful interactions between academia and industry. The Public Private Partnership (PPP) approach, focusing on pre-competitive research, has brought researchers from EPFL, ETH Zurich, Empa, PSI and CSEM together to work in innovative collaborations. To date, CCMX has brought more than 30 companies into close working relationships with ETH Domain research groups.

In addition to large PPP projects under the three Education and Research Units (ERUs), a last Call for Proposals was launched for smaller projects related to nano- and microscale materials, which resulted in four new projects. Funds for the current period have been awarded to 65 projects, with at least one third of the overall funding having come from companies and/or research institutions in the case of the Analytical Platform.

CCMX continues to offer a dynamic programme of education and outreach activities. Seven courses, workshops and events held in 2011 involved more than 300 participants overall. These events have been a particularly effective way of engaging smaller companies, informing them about the latest research developments.



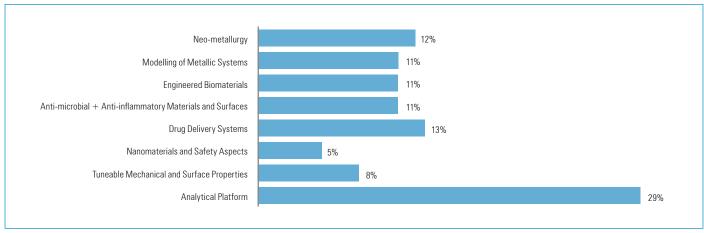
Poster session at the 2011 CCMX Annual Meeting, Bern.

Researchers involved in CCMX value the opportunity for their PhD students to work in an interdisciplinary context and to interact with other ETH Domain researchers. In this way, CCMX contributes to the development of well-trained and team-oriented researchers who are attentive to industry demands. Since 2006, CCMX has established over 100 interactions among a broad range of research teams, who work and meet regularly to discuss project advancement.

The new funding period 2012–2016 sees an evolution in CCMX strategy. At the heart of this strategy is a tandem approach to supporting new faculty and to establishing targeted research platforms, by means of "Materials Challenges". These Challenges will tackle underlying scientific questions

critical to the future of Swiss industry and will strengthen the existing collaborative research among ETH Domain institutions and industry. The CCMX Analytical Platform will continue co-funding efforts in materials characterisation at the nano- and microscale.

As Materials Challenges evolve, the Centre's educational programme will respond by offering relevant topics and pragmatic teaching methods—from excathedra courses to hands-on workshops. CCMX Technology Aperitifs will carry on, inspiring participants' interest for future applications, catalysing contacts for future partnerships and providing occasions for informal networking.



2011 research expenditures by research area [%]

## An Introduction to Featured CCMX Projects

Ever since becoming CCMX Managing Director, Nathalie Jongen has wanted to visit running projects. As planning began for the 2011 Annual Activity Report, it seemed that such visits would be an excellent way to provide material. To make the interviews more effective, and to give Nathalie the opportunity to take photos, journalist Carey Sargent was engaged to accompany her on the visits.

From November 2011 through January 2012, Nathalie and Carey met with seven project leaders and their co-applicants, along with the PhD students, post-docs and researchers involved, in laboratories located all across Switzerland. During these visits, the researchers shared their projects' success stories and challenges, revealing great enthusiasm and spirit. Their generous cooperation in this year's report is much appreciated.

When asked what value CCMX has brought to their projects, researchers emphasised that many of their projects would not be supported by the other funding sources available in Switzerland. We hope the stories on the following pages will appeal to a wide audience, revealing some intriguing aspects of the unique multi-disciplinary, multi-partner projects that are co-funded by industry and CCMX

### 33 companies involved in 2011

ABB Turbo Systems

Alcan

Alstom

Asulab

Attolight

**BASF** 

**Bobst** 

**DECTRIS** 

HeiQ Materials

IG DHS

**ION-TOF Technologies** 

KonMed

**Kugler Bimetal** 

Lyncée Tec

Matter Aerosol

NanoScan

Nanosys

**Novartis Pharma** 

Novelis

OC Oerlikon Balzers

Plansee Powertech

**RMS** Foundation

Rolex

**Rolic Technologies** 

**SCANCO Medical** 

Sika

Straumann

swissnuclear

**Synthes** 

TSI

Varinor

Wessling

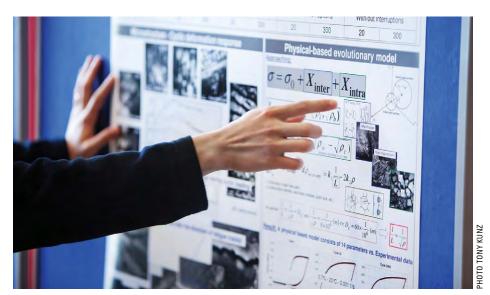
Zeiss

## 2 federal offices involved in 2011

Federal Office for Public Health
Federal Office for the Environment

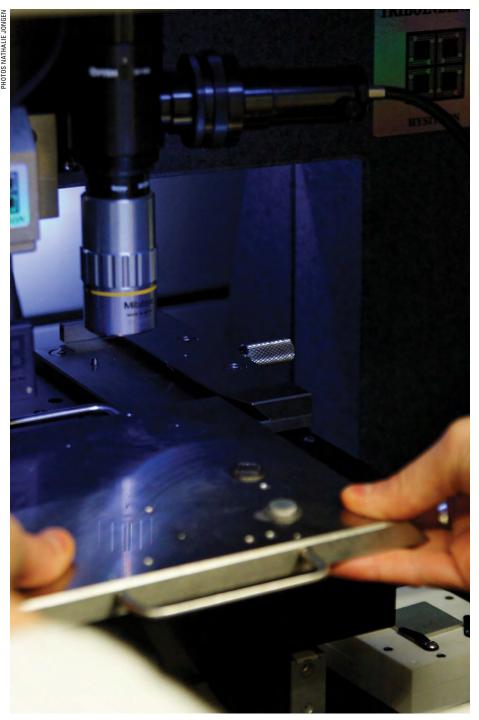
**Bold** signifies these companies joined CCMX in 2011

In 2011, CCMX ran 28 projects, involving 33 industrial partners and two federal offices; including over 150 scientists and engineers from more than 66 laboratories at eight different institutions. Eight new companies joined CCMX projects approved in 2011, together with four companies that will continue their commitment by participating in new projects.



Poster session at the 2011 CCMX Annual Meeting, Bern.

## Theoretical Models, Lab Experiments Designed



A. Furrer inserts a sample into the nanoindentation instrument to measure its hardness.

Metallurgists have long mixed elements such as silver and copper into gold to make alloys that are more durable and available in a wider range of colours than the deep yellows of the pure metal.

And while "recipes" for making white and red gold are well established, customer demand for novel shades of purple, blue and even black have led to costly processes of trial and error for the companies that sell the metal to jewellers and watchmakers. These suppliers want to offer new colours, but also avoid expensive experimentation with the precious metal. They would like to know exactly what the alloy will look like from the outset

PhD students Angela Furrer at ETH Zurich and Deniz Kecik at the Paul Scherrer Institute are developing models to help them figure this out. Guided by advisors Ralph Spolenak and Helena Van Swygenhoven, the two PhD students are evaluating both experimental approaches and simulation models for characterising the optical properties of gold and gold-based alloys. And while the project may have seemed straightforward at first, Spolenak said, it has turned out to be a "huge challenge." Van Swygenhoven agrees that the project has taken more of a focus on basic science.

"At the end of the thesis we will not be able to say 'add this element and you will have black gold' or whatever," said Van Swygenhoven, head of the Materials Science and Simulation group at PSI. "That was maybe industry's hope in the beginning. I don't think they're unhappy about it though — they know that this work is about the future."

It all goes back to band theory, which can be used to explain colour. A material's hue is related to the character of the range of excitations between the valence band, which describes occupied electron energy states, and the conduction band, which involves states that are unoccupied when the system is not excited.

Some materials, such as colourless diamond, have a band gap — the difference in energy between the top of the conduction band and bottom of the valence band — that is larger than the whole energy range of visible light. This means that even violet

## to Predict the Colour of Gold Alloys [MERU]



What we aim to do is see how accurate and close-to-experimental results we can get from these different approaches.

light, which has the highest energy of the visible wavelength spectrum, is insufficient to make an electron overcome the band gap. No light is absorbed and the material appears colourless. For materials with a band gap smaller than the lowest energy of visible light, all visible light interacts with electrons and is absorbed, making the material opaque.

In the case of gold, the absence of a bandgap leads to a complete absorption of light. However, as the electrons in metals are highly mobile, their motion leads to a re-emission of photons. The variation of the electronic band structure results in a more effective reflection of red-yellow wavelengths than the green-blue to violet shades, causing a strong yellow colour to appear.

The team started off by using *ab-initio* calculations — formula that rely on first principles of quantum mechanics rather than empirical data — based on density functional theory (DFT) to determine gold's electronic and optical properties. They found however that DFT, the basic idea of which is to replace a complicated mathematic object with something simpler was insufficient for what they wanted to do.

"Even if physicists and chemists have spent decades on developing algorithms to calculate the electronic state, they have mostly focused on the states that are occupied, not those that are unoccupied," said Spolenak, professor at the Department of Materials at ETH Zurich, "However, to model colour you need the transition between the two states and you need to know them with the same kind of accuracy." Kecik has since been working on calculations based on Green's function (GW method), a method that can be used to solve certain complicated differential equations. This, in turn, should allow the team to understand the interaction between electrons in excited and unexcited states. Though the GW method has some limitations — notably. that it requires a lot of CPU time and is limited to very small systems — it is being continually improved. In addition to DFT and GW methods, the team is also using a hybrid functional method.

The initial focus was on modelling the properties of pure gold, but the team has also begun examining the optical properties of intermetallic phases known to produce appealing colours such as those based on gold-indium, gold-gallium and gold-aluminium combinations. "What we aim to do is see how accurate and close-to-experimental results we can get from these different approaches," Kecik said.

Some of those experimental results are coming from ETH Zurich labs, where Furrer produces alloys with these appealing

colours. Working with thin films of about 500 nanometres' thickness gives Furrer some freedom to experiment with alloys that would be too expensive to manipulate in larger quantities. She nonetheless faces an industrial constraint — the alloys need to have enough gold in them to be classified as 18-karat.

A recent experiment produced a gold-platinum aluminide with a pretty peach colour that just meets these purity requirements. Furrer created it by depositing a thin film of aluminium, then a top-mixture of gold and platinum and finally performing a heat treatment to allow for the formation of intermetallic phases. The team is now determining what they have produced.

This is a lengthy process involving some very high-tech equipment: they first cut thin lamellas using a focused ion beam, look at them in a transmission electron microscope, take diffraction images, and then make sense of the data. They will look at the distribution of elements to find out whether there is segregation or rather a single phase consisting of gold, platinum and aluminium — Furrer hopes the latter will be the case.

## Investigating Microstructure May Yield Gains in

Companies could reduce material consumption by working closer to the limit, but doing this requires much more reliability.







Various aluminium samples supplied by the industrial partners for the project.

The Airbus 380 can carry as many as 853 passengers on two decks that offer 50% more floor surface than any other big passenger plane. It is almost 25 metres high and has a wingspan of about 80 metres.

It may nonetheless one day seem average among new models from airplane makers who "want to go big," says Jean-Marie Drezet, a scientist at the Computational Materials Laboratory at EPFL. "A plane can be 200 metres wide, there's no problem, it will fly . . . . provided we have more powerful engines, but also stronger materials."

One way of making metal stronger is to encourage the formation of precipitates, or fine particles that can prevent the movement of dislocations in the lattice of metal atoms. If the dislocations cannot move, the metal will not deform. It is stronger. The problem is that creating these precipitates causes stresses in the metal that are currently hard to characterise or predict, particularly in thick components. Companies manufacture certain products such as airplane parts without incorporating the complex microstructure into their models. Knowing that these models are not as accurate as they should be, manufacturers

use more metal to build parts with significant safety factors. This wastes metal and makes parts heavier than they need to be.

"We always take higher-than-necessary security factors because we are too afraid of not being able to estimate the actual stresses," Drezet said. "We take something that's a metre thick, when maybe half as thick would be enough. Companies could reduce material consumption by working closer to the limit, but doing this requires much more reliability."

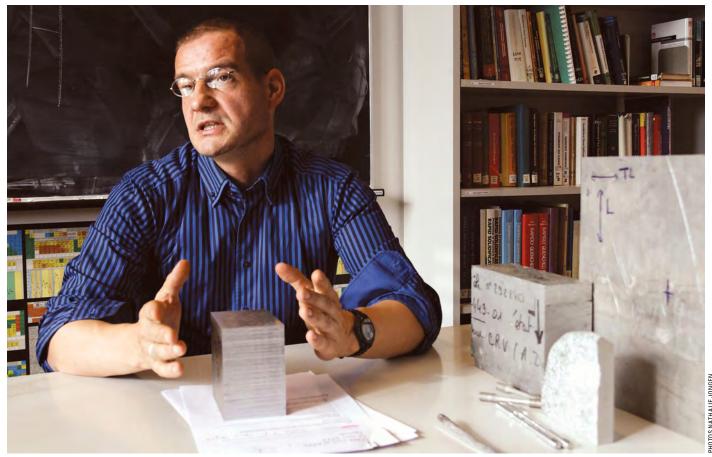
This is where the CCMX project team comes in. Drezet and his PhD student Nicolas Chobaut, together with Helena Van Swygenhoven, head of the Materials Science and Simulation group at the Paul Scherrer Institute, her post-doc Julia Repper and PhD student Patrick Schloth, help industrial partners Constellium and ABB-Turbosystems figure out the best way of describing and modelling the influences of this precipitation on the mechanical properties of thick aluminium components. The companies have asked the team to begin by looking at parts used in spars, the main structural elements of airplane wings, and at forgings for turbochargers.

"We're very happy to have these applications because we feel useful, and that's important," Drezet said. "Frankly, our industrial partners know what they want and they want it now."

Heat treatment of metals involves heating the alloy to the solutionising temperature and then cooling it very fast in water. The follow-up aging treatment consists of reheating the alloy to a much lower temperature, typically 150-200°C, for a given time. There is a compromise between very efficient quenching and resulting residual stresses: if a process quenches a metal component very quickly, it will also produce more of the residual stresses that induce distortion or other complications during the machining of the parts. If the quenching process proceeds too slowly, there is not enough precipitation and the material is not as strong.

Additionally, the cooling rate during quenching is very high on the surface, but much slower in the centre in thick components. This means the efficiency of the whole treatment is much lower and that the final material has a gradient of precipitation and mechanical properties.

## Thick Metal Structures [MERU]



J-M Drezet explains the challenges arising when quenching thick aluminium parts.

"The problem is very complex," Drezet said.
"To fully couple the phenomena — the interaction between the microstructure state and the stress build-up — the inferred mechanical behaviour has to be very well known."

Drezet and Chobaut are using a Gleeble machine to characterise the properties of the thick parts by simulating fast and slow quenching and then mechanically working the sample while various parameters, including strains and stresses, are measured and recorded. The team then incorporates these properties into a finite element model of quenching and compares the results to stresses measured on "as quenched" samples using neutron diffraction — because precipitation is not yet taken into account, residual stresses are underestimated by about 30%, the same margin of error encountered in industry.

The samples are passed on to PSI, where they are characterised by a number of methods so that the team can explore the nature of the precipitate materials and their distribution as a function of thickness of the plate. They will use this information to adjust the data entered into a model for predicting the formation of precipitates as a function of temperature and time.

The group has already measured large forgings from ABB on an instrument known as POLDI (Pulse Overlap time-of-flight Diffractometer), which uses neutron scattering to determine atomic structure. They found that the material was inhomogeneous, which means that it is difficult to set up reliable models to predict residual stresses. The team plans to measure the material again to better understand what they saw.

The group has also started in-situ mechanical testing on samples from Constellium and has developed plans to optimise the process. These tests aim to help the scientists understand the influence of the different microstructures, which result from factors such as different quenching paths, on the mechanical behaviour and thus, on the build-up of residual stresses. They will also consider a new experimental set-up for better investigating the quenching process, using tools including high-resolution transmission electron microscopy and small angle X-ray scattering to build the full picture of the microstructure. Information from the EPFL and PSI legs of the project will then be merged to form a more accurate model.

According to Drezet, "In three years from now, hopefully less than that, we hope to be able to predict these stresses accurately."

## Tuning the Mechanical, Wear and Wetting Respo



S. Rowthu and E. Balic present a specimen and sample holder in order to evaluate response to wear.

The word "Biomimicry" originates from its Greek components — bios, meaning life and mimesis, meaning to imitate. It refers to the scientific practice of observing nature to discover ways to solve engineering problems. Birds inspired da Vinci's flying machines, and termite mounds have led to innovation in climate control.

The team, composed of Empa's Laboratory for Advanced Materials Processing and EPFL's Laboratory of Applied Mechanics and Reliability Analysis, aims at surpassing traditional approaches. Nature has produced very hard materials like sapphire and diamond. It has also created plants like the lotus flower, with waxy, water-repellent leaves. Bios has not, to our knowledge, produced a material that combines the two properties.

"We want to have a hard, very resistant material that mimics the surface wetting properties of lotus plants," said Patrik Hoffmann, head of the Empa lab. "Our work is based on principles and learning from

nature, but we're going beyond, because we have not seen in nature, as far as our knowledge takes us, porous nanoparticulate alumina with control of surface chemistry." Hoffmann and his team at Empa, which includes scientist Edin Balic and PhD student Sriharitha Rowthu, complemented by colleagues John Botsis, head of the Laboratory of Applied Mechanics and Reliability Analysis and his post-doc Marco Lai at EPFL, are collaborating to investigate the mechanical, wear and wetting properties of nanostructured alumina material filled with liquid.

Such a material may eventually have a variety of uses because nanostructuring can be exploited to tune mechanical, chemical, optical and electrical properties. Nanosized liquid-filled pores present in the material could lead to an improved response by the surface to wetting and wear. The first step towards designing materials with specific characteristics is a better understanding of how various parameters influence their properties. That is what inspired this project.

The team starts with particles of commercial alumina with a diameter of about 150 nanometres. These particles are then shaped into a body, with a process called slip casting, in order to generate porous green bodies. These bodies encompass about 60% of the fully dense alumina material. The subsequent sintering, or heating, process increases this density, while assuring that the open porous network is maintained. Hoffmann, Balic and Rowthu are focusing on synthesis and characterisation of the nanoporous material. They also evaluate the effect that filling the pores with liquids of different surface energies has on the mechanical, wear and wetting response. "This is a true multidisciplinary effort," Balic said.

EPFL's Botsis and Lai are focused on developing simulations of the fracture and deformation mechanics of the porous ceramic materials. They will look at the size of the pores and grains and their distribution and, based on that, will construct models for

### nse of Nanostructured Ceramics [SPERU]



S. Rowthu places a sample in the instrument used for assessing the response to wear.

the fracture characterisation of such materials. They will also examine the samples with some of "Empa's and EPFL's very advanced tools," including focused ion beam tomography, digital holographic microscopy and *in-situ* mechanical testing with the help of an electron microscope, Botsis says.

"To understand fracture of such materials you have to consider the effects of different spatial scales," Botsis said. "This means looking at not only the overall specimen size, but also at microstructural features like pores, crystal phases, dislocations, voids, dust particles and heterogeneities. These can be very important in the overall fracture behaviour."

Hoffmann and Balic say an exciting and novel aspect of this effort would be to manipulate the filling of pores with liquids of different wetting behaviour as a response to applied pressure and surface interactions. The material can be viewed as a sort of

sponge, where applying pressure results in a switching of surface chemistry.

While Hoffmann first thought of this approach while listening to managers at Bobst AG — the world's biggest packaging-machinery maker and a partner in the project — complain about workers destroying the surfaces of metal machines with screwdrivers to remove oil-based ink, he said there could also be more day-to-day applications.

Frying pans are a good example. The coatings on today's pans are easily damaged by dishwasher detergents and by metal utensils. Sponges made for cleaning these surfaces have a dual function — while they do not scratch the surface, they are designed to deposit a thin layer of Teflon onto the pan, rendering the surface less wettable. Hoffmann's team is working on a material that will not need this kind of repair because it will always replenish itself.

Our work is based on principles and learning from nature, but we're going beyond, because we have not seen in nature, as far as our knowledge takes us, porous nanoparticulate alumina with control of surface chemistry.

## Multidisciplinary Team Aims to Develop More Di



I. Sailer selects a dental implant from a collection of various examples.





Typical testing machines that subject dental implants to the same chewing conditions experienced by natural teeth.

We're trying to combine
two materials that are
completely different,
mechanically and physically,
to a compound that exhibits
all the advantages that we
would like to have clinically
and aesthetically.

Dental implants have come a long way since the Maya used sharpened seashells to replace missing teeth. Today's devices, generally based on titanium screws implanted into the jawbone, are strong, resistant to corrosion and usually last for decades.

They do have a drawback though: their dark grey colour can sometimes be seen when patients smile. Companies that make these replacement teeth are trying to address the flaw because it is a drawback that customers are increasingly unwilling to accept. "Patients are becoming more and more demanding today," said Irena Sailer, a prosthodontist and associate professor at the University of Zurich's Clinic for Fixed and Removable Prosthodontics and Dental Material Sciences. "They want beautiful smiles and perfect looks."

A team of materials scientists, chemists and biologists working at ETH Zurich, the University of Zurich, Empa and Institute Straumann AG, the world's biggest manufacturer of dental implants, are hoping to provide more natural-looking restorations with a coloured ceramic coating that can be placed over the titanium implants. The final product, which will have to go through years of rigorous testing before it is allowed on the market,

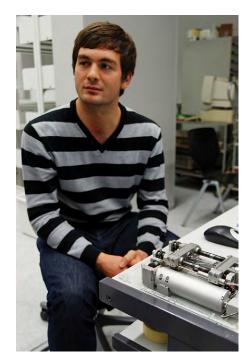
should be as aesthetically pleasing as it is mechanically sound. And while the concept of coating metal with a ceramic may seem simple, the execution is complex. The material has to have the right colour to camouflage the metal, withstand the harsh, wide-ranging conditions present in the mouth and integrate properly into the bone.

"It's a challenging project," Sailer said.
"We're trying to combine two materials
that are completely different, mechanically
and physically, to a compound that exhibits
all the advantages that we would like to
have clinically and aesthetically."

The experimental coatings are first developed in the lab of Prof. Ralph Spolenak, principal investigator of the CCMX project, at the Department of Material Science at ETH Zurich, where the group already faces a significant hurdle: ceramics usually get their colour from internal defects that scatter light and so need to have a certain thickness to be white rather than transparent. The problem is that the material also becomes mechanically weaker as the thickness increases.

"If you just take a ceramic and make it thick enough that it gives you the colour, the mechanical integrity will be a problem,"

## screet Dental Implants [MatLife]



D. Muff performs tensile testing with a stage that can also be used in-situ with various microscopes.

Spolenak said. "That's why, even if it sounds very simple, many approaches in that regard have failed."

The fundamental challenge is therefore to figure out how to colour a ceramic film that is still thin enough to have the desired mechanical stability; it should not crack, delaminate or degrade. Spolenak, who works on the project with PhD students Daniel Muff and Christina Pecnik, thinks the answer may lie in interference, the colour phenomenon that can be seen when a drop of oil is added to water. The team has successfully developed coatings of three different colours — "whitish, yellowish and pinkish" — based on this and deposited different thicknesses onto flat titanium disks that have been either polished or blasted with sand or glass. Coatings that pass an initial colour test go through more rigorous assessment. Muff examines mechanical properties of the coatings through tensile and bending tests. Meanwhile, Pecnik runs tests with some assistance from Sailer and Mutlu Özcan, head of the materials science unit at the University of Zurich's Centre of Dental Medicine. Their main goal is to assess the colour more closely, and also see how the coating may perform in the mouth, an extreme environment that subjects materials



S. Lischer observes cells in culture flasks using a light microsope.

to wide variations in temperatures, acidities, extreme loads and constant moisture. Spolenak says his group has solved the issue of mechanical properties and found a good colour, but still needs to increase the luminosity of the coating to keep the implant from being seen through the gum. A joint brainstorming session generated four ideas for how to do this, and the group has already tried the first. The resulting coating is good from an aesthetic point of view, but still has to undergo mechanical and corrosion testing.

"If it's not good, we have to go to option number two, three or four," he said. "We may still try other ways than the one we have chosen now because there may be some biocompatibility drawbacks."

If there are such drawbacks, Katharina Maniura, co-head of the Laboratory for Materials-Biology Interaction at Empa St. Gallen, and scientist Stefanie Lischer will be the first to know. They are responsible for looking at whether a given coating will affect the implant's ability to anchor into the bone and gum. Biocompatibility is critical because it determines both healing rates at the beginning of the procedure and can affect the lifespan of the implant.

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The Empa team studies this so-called osseointegration by measuring the ability of pre-osteoblasts to form into mature bone cells while in contact with the coating and the degree of mineralization of cells on the material. They compare results of the new coatings - measured through gene and protein expression in the case of cell maturation and with dyes in the case of mineralization — with those measured from Straumann's gold standard implant material, SLActive. In a second step, the team will try a new technique of assessing soft tissue integration through in vitro tests that combine keratinocytes and fibroblasts, two different types of cells present in gums. When the teams are satisfied with the colour of the ceramic as well as its properties, they will try to coat a real implant rather than the flat disks they have been using in the initial trials. The prototype will then be passed on to Pecnik at the dental clinic and to Straumann for more testing.

"Even though we know how well some implants like the one on the market from Straumann work, there's still room for improvement," Maniura said.

## Antimicrobial Surfaces for Prosthetics May Spar



J. Nowakowska showing blood agar plates used for growing two major pathogens associated with implant-related infections.

Less than eight percent of orthopaedic implant surgeries result in device-related infections. Irene Wells was one of the few.

An invasion of bacteria on her prosthetic knee led to two surgeries and setbacks including a fractured leg, a long course of debilitating antibiotics, a blood transfusion and clots. "I have never been in so much pain," said Wells, who lives in Essex, U.K. "It was a year from hell and I would not want to go through that again."

Doctors are concerned that the number of people going through such experiences will rise as aging populations result in more of the procedures. Researchers from EPFL, ETH Zurich and the University of Basel have joined together to reverse the trend by developing antimicrobial implant coatings that prevent bacteria from clumping together to form the films that make infection hard to combat.

Mirren Charnley, who worked in Marcus Textor's group at ETH Zurich, says there are two main strategies for preventing implant-related infections. Coatings can either be biopassive and simply prevent bacteria from attaching to the implant, or bioactive, incorporating antimicrobial substances to kill bacteria that come close or in contact. Both approaches have advantages and disadvantages — that is why the group has decided to focus on a dual-function coating that incorporates both components.

The teams are focusing on two such surfaces. One features a covalent link that keeps the antimicrobial tethered to the material it coats, while the other incorporates the antimicrobial through a cleavable bond, which they hope can be triggered by signals from the bacteria themselves. The first solution would result in a slow, constant release of the antibiotic from the implant, the other would release the drug only in the presence of the bacteria.

Harm-Anton Klok, professor of Materials Science at EPFL, says his team, which also includes PhD student Sorin Ibanescu and

## e Patients from Debilitating Infections [MatLife]



Crystal violet staining of the biofilm formed by a pathogen reveals the anti-adhesive properties of a new polymer coating the specimen surface.

post-doc Li Chen, is trying to answer a few basic questions: what the polymer should be, which antimicrobial should be used, how much of the drug can be linked to the coating and how it can be released.

"If we have the right answer, then in principle it should not matter onto which substrate we want to apply the polymer," he said.

Klok's group has run some initial experiments on a few materials and is now aiming to get a coherent set of data from layers of different thicknesses and with different amounts of antimicrobial to assess bacteria-killing properties. Charnley and her colleague Canet Acikgöz at ETH Zurich have run initial tests on the covalent attachment of the established antibiotic vancomycin onto a biopassive surface and say they are seeing an effect with this dual-functional approach.

A team at the Infection Biology laboratory at the University of Basel's Department of Biomedicine is responsible for another aspect of the project — investigating the bioactivity and mode of action of the compound serrulatane EN4. The compound, which is extracted from the Eremophila neglecta plant found in Australia, is used topically in the country's aboriginal culture.



C. Acikgöz selects a specimen to be characterised by X-ray photoelectron spectroscopy.

While researchers had shown that it is active against Gram-positive bacteria such as staphylococci, which are responsible for half of all implant-related infections, it was not clear how the substance worked. Antibiotics generally combat infection by acting on a single target, including either the bacteria's DNA, protein synthesis, RNA, the cell membrane or the cell wall. The team in Basel used radioactive monomers to show that EN4 acts on all of these targets, which suggests an antiseptic, non-specific mechanism of action.

While these results are promising, the team, which includes Régine Landmann, one of the original principal investigators on the project, her successor Nina Khanna and PhD student Justyna Nowakowska, has also run into challenges — EN4 is also toxic to human cells and, in a surprising twist, the team found that it wasn't able to kill bacteria *in vivo*.

"In vitro it worked very well, meaning very fast, it was very efficient, so we were really very surprised," Nowakowska said. "Even after reapplication of EN4 three times, after 24 hours we didn't see any effect on either planktonic or adherent bacteria."

Researchers from EPFL,
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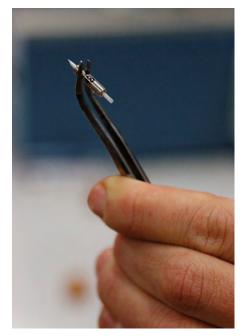
Nowakowska has since been able to show that this is because EN4 is strongly inhibited by albumin, a protein found in blood. Despite these challenges, the team thinks that EN4 might still form the basis for a new sort of antimicrobial substance.

Once the teams at EPFL and ETH Zurich run preliminary screening tests on their materials — experiments that Nowakowska helped set up on trips to Lausanne and Zurich — they will send their best samples off to their Basel colleagues for more detailed microbiological investigation into how well the surfaces kill bacteria.

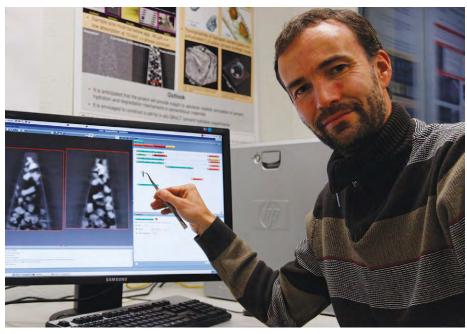
"For us chemists, it's easy to make the compounds," Klok said. "We can make many more than we're able to scan, so we've developed a two-step process, with the first, quick evaluation done here at EPFL, followed by a more indepth screening in Basel."

The Basel team has run tests on the dualfunctional surfaces developed at ETH Zurich and is ready to evaluate additional promising samples as they are developed.

## Novel Nanotomography Technique Allows First



Capillary used for measuring the hydration of tricalcium silicate particles using synchrotron phase-contrast nanotomography.



P. Trtik holding a capillary in front of the images from the first round of measurements by synchrotron phase-contrast nanotomography.

While the process of making cement is considered very efficient, rising energy prices and increased environmental awareness have spurred more fundamental research into the material's properties.

The process for producing Portland cement, the most widely used variety in the world, was first patented in 1824. The material nonetheless remains in some respects more poorly characterised than high-tech ceramics and alloys that were created just decades ago.

"Cement is very complex," said Pietro
Lura, head of the Concrete and Construction
Chemistry Laboratory at Empa in Dübendorf.
"A material scientist or chemist wouldn't
deal with it if not forced to. It's not your
ideal material and doesn't have any
particular beauty about it."

Portland cement is a "low-tech" product made from locally available limestones and clays and is the principal ingredient of concrete, stucco, mortar and many grouts. It is one of the most commonly used building materials and its widespread application — 3.3 billion tons were used in 2010 — also means that about 3% of global primary energy consumption and 5% to 8% of all carbon-dioxide emissions are linked to its production. While the process of making cement is considered very efficient, rising energy prices and increased environmental awareness have spurred more fundamental research into the material's properties.

"The idea is that if you know cement better, you're also able to use it better and in a more efficient way," Lura said.

Portland cement is made from clinker — small lumps of ground limestone and clay that have been heated to between 1,400 and 1,500°C — that is then ground and mixed with some gypsum. The substance sets after a few hours when mixed with water and then hardens over a period of weeks. The aim of this project, which involves researchers from Empa Dübendorf, Paul Scherrer Institute and EPFL, is to better understand the parameters that control cement hydration, including dissolution of cement particles, growth processes and the morphology of early hydration products, called hydrates.

EPFL's Cédric Patapy, a post-doc in the Laboratory of Construction Materials and Amélie Bazzoni, a PhD student with Karen Scrivener, head of the lab, are responsible for producing the sample used in this project. It is a laborious process. Because the tubes used in the testing phase are so small, the team needed to produce a sample consisting of very fine particles of tricalcium silicate — the most abundant phase in cement — with a median size of about five microns and a very narrow size distribution.

## "Live" View of Cement's Microstructure [NMMC]



A. Bazzoni places a cement sample into the X-ray diffractometer to assess its crystal structure.

Getting to this point involved synthesizing the initial material, grinding it, characterising it in terms of crystal structure and then using repeated rounds of sedimentation. It took at least five to six months to perfect the sample, Bazzoni said.

The team was then ready to move on to the next phase of the project: a novel kind of imaging. Earlier studies on hydration have been hindered by the fact that scientists needed to actively stop the process to get a snapshot in time. Drying the samples to halt hydration and sample preparation steps modified the microstructure, making it difficult to say what was an effect of hydration and what resulted from stopping the process itself. This also meant analysing a huge number of samples to get representative results.

Synchrotron phase-contrast nanotomography, developed over the last five years at PSI in Villigen, allows the team to make in-situ measurements that observe hydration over time without this destruction, and at the appropriate scale.

"Our instrument is very special because we can offer this resolution in time and that's unique," said Rajmund Mokso, a scientist

in the X-ray tomography group at PSI. "The other element is that the high sensitivity of our contrast mechanism allows for the distinction of individual particles. They're usually very difficult to see."

The instrument goes beyond classical tomography — a method of imaging by cross-sections — by making use of the fact that a material's density affects how quickly light waves can pass through it. The new technique involves manipulating the phase of the light waves, in this case X-rays, before they enter the sample and then measuring the difference in phase after they pass through. This allows the team to see the microstructure in the small sample sizes needed to get the required resolution of about 100 nanometres or better.

Though the machine has since been used by a few groups studying topics ranging from crystal formation within droplets to the musculature of micro-insects, Pavel Trtik, a scientist who works with Lura at Empa, was the first person external to PSI to use it, Mokso said.

In the experiment, Trtik filled a capillary just a few hundreds of nanometres across at its widest point with some of the sample

provided by the EPFL lab. Managing to prepare samples featuring realistic water/ solid ratios in sizes to fit the nanotomographic field of view, he placed it on the instrument to get a first look at the particles in the dry state to establish a reference point. After starting the hydration process with an injection of water, the sample was rotated and a snapshot taken at every angle through 180 degrees, a process lasting about 20 minutes. The team then reconstructed the images to form 3D views of the sample at certain periods of time after hydration.

Cement hydration with

this resolution, in 3D live

at this level hasn't been

seen, ever...

The team is now analysing the images to observe how particles of tricalcium silicate erode and dissolve, and to see where the hydration products that build cement's microstructure actually form. They may need to repeat the experiment, but have nevertheless already broken new ground.

"Hydration with this resolution, in 3D live at this level hasn't been seen, ever, this is already something," Lura said. "However, I would not do things just because we're the first, but because they're useful and bring us forward."

### Mechanical Innovations to Turn PSI Neutron Be

This interdisciplinary project involves a large team including departments at PSI (NUM and NES), Empa (HTIG) and EPFL (CRPP).



Adjusting a traction instrument on the sample stage at the POLDI neutron beam line at PSI.

The project involves most Swiss academic partners performing research in modern steels and is supported by eight Swiss companies that are already using or plan to use the facilities.

Metallurgical engineers do their best to simulate how alloys and composites deform during under certain conditions. Tensile, torsion and/or compressive loading may be applied, often at elevated temperatures.

Increasingly, they need to see what happens at the micro-level too. This is because demand for lighter, higher performance materials is resulting in alloys with complicated nanostructures — these crystal phases, dislocations and voids influence behaviour in ways not fully understood.

In-situ mechanical testing under X-ray or neutron beam diffraction is an excellent way of examining this scale. It gives details of microstructural changes under mechanical load, shows load transfer in multiphase materials and generates data for predictive models. While increased beam intensity, improved detector efficiency and better focusing techniques mean X-ray and neutron facilities increasingly draw materials scientists to look at the micro scale, there are limits to mechanical equipment on the beam line — rigs can exert tension, but only along one axis.

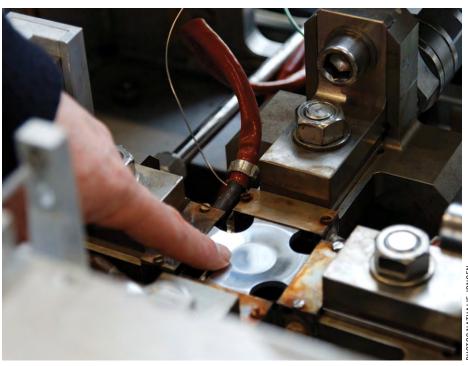
The team behind this project wants to develop equipment that can execute in-situ mechanical tests of these modern industrial alloys in more realistic conditions. They are designing a modular test rig that includes a number of elements — a bi-axial rig will perform tension, compression and fatigue tests and uniaxial tests when the second axis is removed. The uniaxial set-up is itself outfitted to perform tension, compression, torsion and fatigue tests. The whole rig can fit on POLDI (Pulse Overlap time-of-flight Diffractometer), which uses neutron scattering to determine microstructure, and part of it can be used in a furnace. Researchers will be able to recreate realistic conditions by deforming the sample in three ways, sometimes at high temperatures, and observing what happens.

"You can imagine that when you make a car, for example, the deformation of the material doesn't always correspond to just tension," said Helena Van Swygenhoven, head of the Materials Science and Simulation group at PSI. "It's clear that if you can do multi-axial deformation, which is more realistic in some

## am Line into a Unique Instrument [NMMC]



J. Repper, V. Davydov and S. Van Petegem show the dimensions of the modular test rig to be installed at the POLOL neutron beam line.



Metal sample under testing in the Bi-axial Tensioner at the Laboratory for Nuclear Materials at PSI.

applications, this is unique. There are no beam lines equipped with such modular systems."

The team faced two big constraints right from the start, said Werner Wagner, head of the Spallation Neutron Source Division at PSI. The equipment, which needs to be capable of producing 100 kN of force, also has to fit inside a relatively small room. The equipment must allow the neutron beam to come in one pathway and exit in two different directions, without any obstruction. Normally, these types of machines are about 3.5 metres high and have thick frames that would interfere with the beam. The machine also has to be easily moved through a hole in the roof of the bunker. It cannot take two weeks to put the rig away.

"It's really very challenging to design such a machine that fits into this instrument," Wagner said. "We have had a lot of discussions to try to find a way to get a company to build a machine that fits our requirements." The team met with a handful of manufacturers to discuss possible solutions, but encountered various problems. "Off-the-shelf" machines were too tall, weighed about six tons and had thick, surrounding frames that would obstruct the beam line. Larger companies were not enthusiastic about working on a custom solution, while smaller ones may have lacked the know-how. The team eventually found a company in Germany, and came up with an innovative design featuring two frames in a modular design.

The team is now negotiating with the company and hopes to reach agreement in the next couple of months. Taking manufacturing and testing time as well as the beam line's annual shut down into account means the rig may be ready to use a year from now.

The project involves most Swiss academic partners performing research in modern steels and is supported by eight Swiss companies that are already using or plan

to use the facilities. The team expects a number of requests — the rig is unique and should appeal to a wide variety of industrial and academic users.

Some of the earliest tests will be from PSI groups doing deformation behaviour studies of advanced materials used in high temperature areas of nuclear reactors. The types of testing will eventually expand to include fundamental research into all kinds of novel metallic microstructures. The rig is likely to be useful not only for investigating high temperature alloys, but also for research at lower operating temperatures in other alloys such as those used in construction, transport or energy applications.

"Such a machine allows for the very complex strain paths that are usually applied to components in the production process and during service, for example," PSI postdoc Julia Repper said. "It is really needed."

### 2011 Peer Reviewed Publications

#### MatLife

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- U. Müller, C.V. Falub, G. Thorwarth, C. Voisard, and R. Hauert, Diamond-like carbon coatings on a CoCrMo implant alloy: A detailed XPS analysis of the chemical states at the interface, *Acta Materialia*, 59 (2011) 1150-1161.
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- M.S. Pham, C. Solenthaler, K.G.F. Janssens, S.R. Holdsworth, Dislocation structure evolution and its effects on cyclic deformation response of AISI316L stainless steel, *Mat. Sci. Eng.*, A528 (2011) 3261-3269.
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## Projects Funded in 2011

Project Title	Principal Investigator (PI)	PI's Institution	Other Institutions	ERU/Analytical Platform
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	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 modelling of optical properties of gold alloys	Ralph Spolenak	ETH Zurich	PSI	MERU
Study of complex interfacial properties with nanoscale resolution optical microscopy*	Christian Depeursinge	EPFL	CSEM	NMMC
Microtome4SIMS: Chemical tomography of biological material with 100 nm resolution	Beat Keller	Empa	Unispital Basel	NMMC
Adaptation of the Nano-XAS instrument to a dedicated beamline at the SLS*	Christoph Quitmann	PSI	Empa	NMMC
MaCH2: The Analytical Equipment Database	Susan Meuwly	Empa		NMMC
Colored ceramic surfaces for metallic dental implants and prosthetic appliances	Ralph Spolenak	ETH Zurich	Empa, UZH	MatLife
Structural evolution and rheological properties in gel carrier	Christopher Plummer	EPFL	ETH Zurich	MatLife
Low wear articulating implants employing DLC coatings on CoCrMo and TiAlNb with predictable, long-lasting coating adhesion lifetime	Roland Hauert	Empa	ETH Zurich	MatLife
Fibroblast Growth Factor 2 delivery for tissue repair: From Natural Concepts to Engineered Systems	Viola Vogel	ETH Zurich	PSI	MatLife
Liquid repellent wear resistant coatings (LIRE-WERE-CO)	Patrik Hoffmann	Empa	EPFL	SPERU
Serrulatane-based antimicrobial surface platforms	Harm-Anton Klok	EPFL	ETH Zurich, Unispital Basel	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
Synchrotron phase-contrast nanotomography of fresh and hardened cementitious materials	Pietro Lura	Empa	EPFL, PSI	NMMC
Development of computational tools for molecular modelling and X-ray spectroscopy, with application to the understanding and design of molecular alignment technology in commercial LCDs	Daniele Passerone	Empa	PSI, UZH	NMMC
Equipment for in-situ mechanical testing of nanostructured alloys under service-type loading	Helena van Swygenhoven	PSI	EPFL, Empa	NMMC
Measurements and modelling of residual stresses in aluminium components in relation to their microstructure	Jean-Marie Drezet	EPFL	PSI	MERU
Arc erosion processes in contact materials: modelling and model experiments	Ralph Spolenak	ETH Zurich	Empa	MERU
Protective coatings with managed thermal conductivity for machining difficult-to-cut materials	Valery Shklover	ETH Zurich	Empa	SPERU
Quantitative Magnetic Force Microscopy platform (qMFM)	Miguel Marioni	Empa	ETH Zurich	NMMC
OMNY (tOMography, Nano, crYo stage)	Mirko Holler	PSI	CSEM	NMMC
Development of a He-Ion Beam Induced Charge scanning system (He-IBIC)	Urs Sennhauser	Empa	ETH Zurich	NMMC
Ultrasensitive sensing transducer based on Fano interferences n plasmonic metamaterials—FANOSENSE	Olivier Martin	EPFL	CSEM	NMMC
Gantry-based X-ray Phase Contrast Scanner for MicroCT Applications	Marco Stampanoni	PSI	ETH Zurich, CSEM	NMMC
Study of biological processes in cell membranes at nanoscale by Near Field Digital Holographic Microscopy	Pierre Magistretti	EPFL	CSEM	NMMC
NanoAir—Online coupling of a scanning mobility particle sizer SMPS to an inductively coupled plasma mass spectrometer CP-MS for size fractionated, elemental analysis of nanoparticles in aerosols	Andrea Ulrich	Empa	EPFL, PSI, FHNW	NMMC
Failure and defect analysis of fibre composite materials by means of X-ray interferometry	Rolf Kaufmann	CSEM		NMMC

\* Finished in 2011 CCMX Annual Activity Report 2011 23



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Materials Challenges working session, Bern.

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Texts: Carey Sargent, Nathalie Jongen, Sue Niewiarowski Photos: Nathalie Jongen, Tony Kunz, Alain Herzog, PSI

Design & Layout: Sue Niewiarowski

Printing: Repro EPFL













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