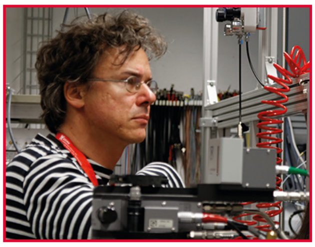
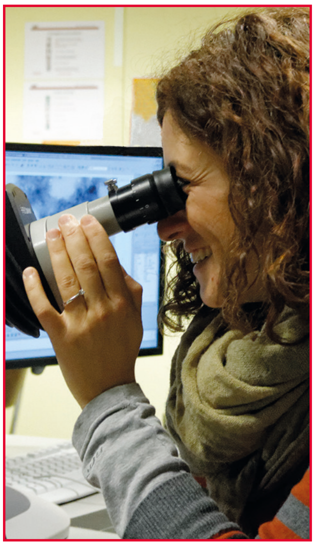
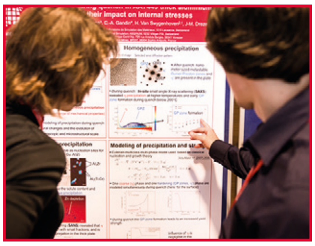
CCMX
Competence Centre for
Materials Science and Technology

Annual Activity Report 2013





Annual Activity Report 2013

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CCMX and Materials Science in Switzerland:

CCMX catalyses cross-disciplinary collaboration and competence

Over the past eight years, CCMX has fostered increased collaboration between Swiss research institutions and industry. The Public Private Partnership (PPP) model has become a proven alternative for funding pre-competitive materials science in a pragmatic and productive way. This unique approach permits multiple companies to cooperate in addressing scientific questions in the same topic area. It also enables a much broader scope of research to be realised with increased efficiency.

The PPP approach has been widely beneficial to ETH Domain and industry partners—numerous offshoot projects have already brought a substantial return on investment with funding from a broad range of Swiss and international sources.

Cross-disciplinary collaboration is a core value at every level of the Centre's activity—from funding to training to networking. Academic and industry partners acknowledge the benefits of their interaction, as it has become critical to innovation. Having established a framework that can address Swiss industry's long-term scientific needs, CCMX now focuses on sustaining connections between industry and academia.

In 2013, the Centre continued implementing its tandem strategy for supporting materials research over the long-term by promoting negotiations for Materials Challenges and by co-funding new professor chairs.

Materials Challenges will foster durable, innovative, and continuing collaboration between ETH Domain researchers and industry partners. Two Challenges are close to finalisation and discussions are ongoing for Challenges in several other topic areas. At the end of 2013, the Centre launched an open Call for Proposals to induce proposals responding to these criteria:

PHOTO TONY KUNZ

PHOTO NATHALIE JONGEN

- Focus on scientific questions critical to industry in areas of durable interest currently lacking in adequately trained personnel
- Promote interactions among CCMX's institutional partners
- Offer training activities for academic and industry partners
- Elaborate a strategy to ensure self-sustainability beyond CCMX's seed funding

The Call has already stimulated institutional and industry interest, with many inquiries from potential applicants.

Three out of four new professor chairs were appointed in 2013. Professors Pietro Gambardella and Fabien Sorin took office, respectively, at ETH Zurich and EPFL in the first quarter. In the last quarter, they were joined by Professor Michele Ceriotti at EPFL. The search for the fourth professor, who will be appointed at ETH Zurich, is well underway.

These scientists were recruited to bring enthusiasm and dynamism into leadership roles in collaborating with industry partners. CCMX looks forward to their participation in networking

Collaborate, Invest and Train!

PHOTOS TONY KUNZ

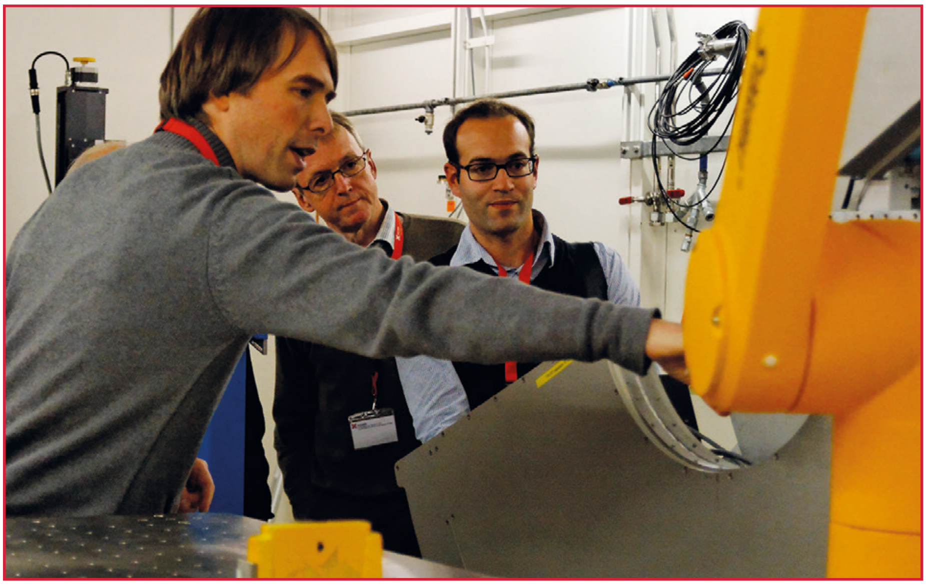


PHOTO NATHALIE JONGEN

CCMX projects are a unique possibility to specifically address Swiss industry's needs via direct collaboration with Swiss research institutes. The change from a ticket-programme model to the Challenges model increases industry's contribution at the same time that it fosters closer and enduring collaborations.

Peter Wick, Empa

and training events. Materials Challenges will continue to develop as the new professors establish their research facilities.

The Analytical Platform also launched a Call for Proposals to fund substantial development or installation of cutting-edge materials characterisation methods and/or analytical tools. Successful proposals to contribute to durable improvements in the ETH Domain's materials science related analytical facilities were evaluated and selected according to four criteria:

- Uniqueness and cutting edge scientific value
- Accessibility
- Sustainability
- Leverage

Four proposals, submitted by EPFL, ETH Zurich, Empa and PSI, were approved in December and will be funded once matching funds have been confirmed.

With the Materials Challenges research initiative, the co-funding of new professors, and a programme of well-attended training and outreach events, CCMX continues to fulfill its mission.

Every aspect of the Centre's activities has advanced materials science in Switzerland through productive and innovative partnerships between academia and industry.

As planning progresses for coming years, CCMX has developed these phrases to guide its focus:

Collaborate to Innovate
Invest for the Future
Train for Life

CCMX Education and Outreach Activities

CCMX creates an enduring network through training and outreach activities

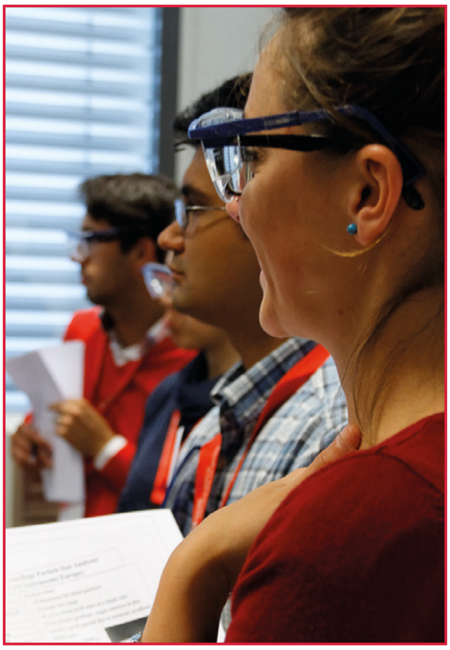
Education is a key element of CCMX's mission. Since 2006, a variety of advanced training events have attracted over 1'100 participants. Courses on recurring themes have established a brand identity—demonstrated by the way students anticipate their occurrence with early registration. Combining state-of-the-art topics with hands-on experience, CCMX courses offer relevant skills and real-world application. Interdisciplinary interactions allow participants to benefit from shared experiences and differing perspectives. Courses are both intensive and comprehensive, enabling participants to put their new skills immediately to use.

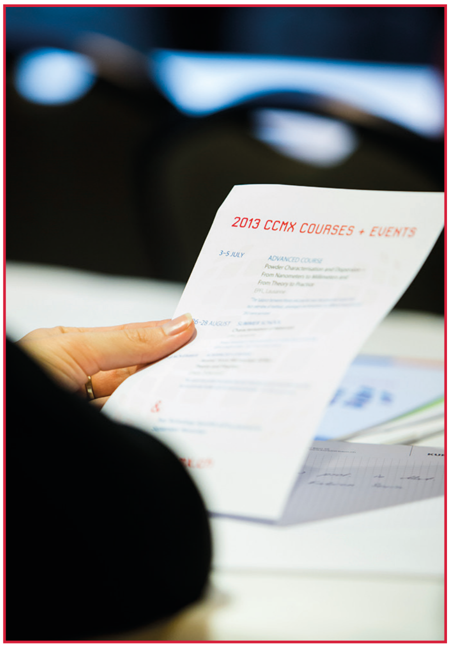
CCMX's educational programme will be aligned with the Materials Challenges as these evolve, contributing to curriculum development and to opportunities for Master and PhD students to intensify their training through practical application.

Events

CCMX Technology Aperitifs provide an informal networking environment that enables contacts for potential partnerships. These events feature concise presentations on current trends, given by academic researchers and associated companies. After the presentations, discussions frequently initiate collaboration.

CCMX has organised seven annual meetings since 2007. These meetings feature presentations from industry and institutional partners, and regularly attract around 100 participants. The always lively and dynamic poster session allows industry participants to engage directly with CCMX project researchers.





PHOTOS ALAIN HERZOG, NATHALIE JONGEN, TONY KUNZ

CCMX Education and Outreach Activities in 2013

13–18	January	Winter School, Nanoparticles: From Fundamentals to Applications in Life Science	Kandersteg
30	April	Annual Meeting	Kursaal, Bern
3–5	July	Advanced Course, Powder Characterisation and Dispersion	EPFL, Lausanne
26–28	August	Summer School, Characterisation of Materials	EPFL, Lausanne
27–28	September	Conference and Working Session: Current Challenges Facing Inorganic Nanoparticles in Medicine and Industry	Bern
30	October	Technology Aperitif, New Trends in Functional Structured Surfaces	Lausanne
7–8	November	Advanced Course, Atomic Force Microscopy: Theory and Practice	Empa, Dübendorf

PHOTOS TONY KUNZ

"It's a very good combination of talks and breaks, which allows enough time and opportunity to interact with people." Participant, CCMX Advanced Course

"It was so interesting to get to know other people who work on similar projects, to discuss the challenges that I have in my experiments right now, and to get some ideas on how to solve them." Participant, CCMX Advanced Course

"It's important for today's PhD students and industry leaders to understand the latest technologies available so they can analyse their materials using the most current methods." Speaker, CCMX Summer School

"People go away from this much more aware of the limitations and assumptions made behind measurements, and consequently will make better use of instruments." Instructor, CCMX Advanced Course

Creating opportunities for networking is an important component of CCMX's educational and outreach activities. Participants continually cite networking as a valuable feature. Designed to bring industry and academic researchers together in both structured and informal ways, the Centre's events introduce companies to research potential within the ETH Domain, while sensitising academic researchers to industry's current needs. To date, CCMX's outreach events have attracted more than 1'900 participants.

Outreach

The monthly e-newsletter, distributed to nearly 1'400 subscribers, is an effective and cost-efficient channel for promoting training and outreach activities, for sharing news and success stories from project researchers, for publishing profiles on PhD students involved in current projects, and for publicising materials science related activities at our partner institutions.

The website is also a means for publishing relevant news items, providing a more in-depth guide to research activities, explains the structure of funding and performing research within the Public Private Partnership (PPP) framework, lists current projects with detailed descriptions available for download, and provides links to project research labs and facilities, and partner institutions.

CCMX Film 2: Train for Life!

What's important about CCMX courses is that people, from Doctoral students to people in industry, have an opportunity to complement the basic knowledge they have with more specialised, state-of-the-art expertise from a variety of domains.

Karen Scrivener, Professor, EPFL

PHOTOS ALAIN HERZOG, NATHALIE JONGEN, TONY KUNZ

In 2012 CCMX produced its first short film, "When Industry Meets Materials Science." While filming interviews with industry partners and project researchers, three themes emerged which have since become guiding themes—Collaborate to Innovate, Train for Life, and Invest for the Future.

Each of these themes represents a core value and corresponding focus. "Collaborate to Innovate" describes CCMX's research and networking activities. "Train for Life" describes the Centre's ongoing educational activities. In 2013, CCMX produced the companion film "Train for Life," in which course participants and instructors talk about the advantages of the Centre's educational offering.

These films are effective tools for introducing CCMX to potential industry partners and course participants, demonstrating the benefits of becoming involved with the Centre's various activities.

Multimedia Complement
See "Train for Life" on your smartphone using this QR code, or go to www.ccmx.ch

Professor Michele Ceriotti, EPFL

Michele Ceriotti, new tenure-track assistant professor and head of the Laboratory of Computational Science and Modelling at EPFL, doesn't like to make definitive decisions.

Faced with the prospect of choosing between physics and chemistry as an undergraduate at the University of Milan-Bicocca, he opted to study the multidisciplinary field of materials science. When deciding on a specialisation as a PhD student at ETH Zurich, he selected computer modelling.

"Simulation allows me to wear multiple hats and change what I'm working on very quickly," Ceriotti said. "It takes lots of effort and resources to build a new laboratory just to investigate an idea. Using a computer as a virtual laboratory only requires changing a few lines of code."

PHOTO ALAIN HERZOG

Simulation allows me to wear multiple hats and change what I'm working on very quickly . . .

Ceriotti researches and develops techniques for computer simulation of materials at the atomic level. That is, he aims to reproduce atomic motion and behaviour at the nanoscale to improve our understanding of mechanisms that account for macroscopic properties, and to then improve these properties through rational design. While computers can now give accurate predictions about material properties such as conductivity and brittleness when fed descriptive parameters, the real goal should be to predict trends and identify critical properties.

"Basically, I try to simulate how atoms behave," he said. "I want to figure out what we can learn about the behavior of materials, and infer from the microstructure information about how to make materials better."

Though the specific techniques vary depending on the application, all simulations need to be accurate, predictive and able to capture both subtle physical effects and complex, large-scale structural features. For now, Ceriotti plans to focus his research on materials such as amorphous silicon that can be used in energy applications, and also on metals. He also wants to develop techniques that contribute to the development of sustainable materials that are easier to recycle and less environmentally damaging to make.

"I think that right now, at the academic level, one is mostly interested in getting materials with exceptional properties and performance," he said. "Typically, we choose a material to work on and think about how to design it, but we don't think about how much of it there is on Earth, how polluting it will be, or how easily we'll be able to recycle it. Steering materials science into a more environmentally aware direction will be a big challenge over the next 10 years."

Ceriotti intends to make it easy for other people to use the methods he develops and is already collaborating with other research groups from Stanford to Trieste. He also wants to use CCMX's connections to work more closely with industry, notably Switzerland's metal companies, who are constantly seeking better alloys and more easily processed materials.

He would also like to see his methods help other scientists in a wide range of fields.

"What has given me the most satisfaction over the last few years was when someone took my methods and used them to accomplish something non-trivial," he said. "That is, when my work can act as a stepping-stone to accomplish something I had not foreseen."

Traditional simulation models treat atomic nuclei as classical particles, describing them with the same equations as billiard balls or planets. This approximation works well for heavier elements, but is problematic for lighter atoms such as hydrogen. Making this assumption about hydrogen nuclei would, for example, suggest that water has a pH of 8.5. During a post-doc at Oxford, Ceriotti developed a technique for simulating nuclei as quantum particles. A researcher at Lawrence Livermore National Laboratory used the method to study hydrogen at very high pressure, an important element of understanding giant planets and astrophysical phenomena.

"It was nice to see someone put my research to use in a field so completely different from mine," he said.

CCMX Materials Challenges

In 2013, CCMX began implementing its tandem approach for supporting materials research over the long term by promoting negotiations for Materials Challenge research initiatives and by co-funding new professor chairs.

Public Private Partnerships (PPP) continue to be the cornerstone of CCMX's funding framework. Materials Challenges are designed to be a means for building on and extending the equity of past collaborations between academic groups and industrial sectors. Each Materials Challenge is to be a single research platform involving one or many academic partners that tackles underlying scientific questions critical to the future of Swiss industry, addressing long-term research needs, while training the researchers who will become a recruitment resource for industry. CCMX matches funds from the private sector for five years, the ultimate goal being to endure sustainability of each initiative beyond 2016.

The open Call for Materials Challenge Proposals launched at the end of 2013 has brought a lot of institutional and industry interest, with many inquiries from potential applicants. This Call will remain open until funds for 2012–2016 have been allocated. For more information, visit www.ccmx.ch/research/call-for-proposals/materialschallenges0/

Significant progress was made in establishing the first two proposals—the Coating Centre and the Nano Screen Materials Challenges. Both proposals are currently completing a rigorous review process by panels of independent international experts. CCMX is committed to assuring parity, transparency, and the highest level of scientific quality in implementing the Materials Challenges.

To move forward with other Challenges, CCMX will organise a series of thematic working sessions to bring together all interested academic and industry parties.

CCMX is confident that it will reach its goal of launching at least six Materials Challenges during this final funding period. This initiative will foster durable, innovative, and continuing collaboration between ETH Domain researchers and industry partners.

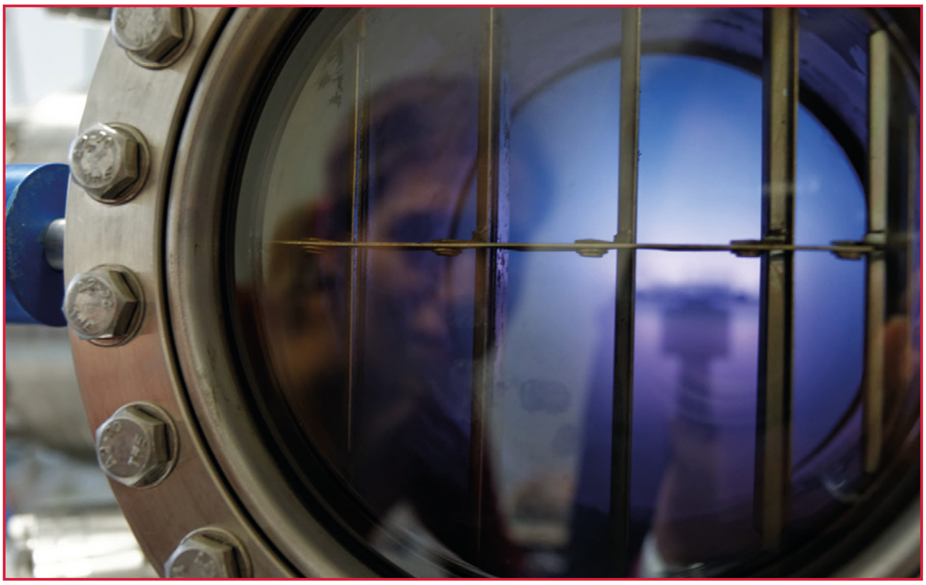


PHOTO NATHALIE JONGEN

CCMX project researcher monitors the progress of an Argon plasma cleaning of a sample, Empa.

52 Companies Involved Since 2006

ABB Turbo Systems (2)
Alstom (2)
AO Foundation Davos
Arrayon Biotechnology SA
Asulab
Attolight Sàrl
Ayanda Biosystems SA
BASF (2)
Biotronik AG
Bobst
Bruker Optics GmbH
Constellium (2)
Crolles R&D
DECTRIS Ltd
HeiQ Materials AG
IG DHS
ION-TOF Technologies (2)
KonMed
Kugler Bimetal
Lyncée Tec (3)
Matter Aerosol
Métalor
Meyer Burger Group
Microsens SA
NanoScan AG
Nanosys GmbH
Nestec Ltd
Novartis (2)
Novelis
OC Oerlikon Balzers
Philips Semiconductors ZH
Plansee Powertech
PreenTec AG
RMS Foundation
Rolex (2)
Rolic Technologies
SCANCO Medical AG
Scientific Visual Sàrl
Sika
Stettler Sapphire AG
Straumann (2)
Sanofi-Pasteur
Surface SolutionS
swissnuclear
Synthes GmbH (2)
TSB GmbH
TSI GmbH
Varinor
Wessling GmbH
Zeiss

2 Federal Offices Involved Since 2006

Federal Office for Public Health
Federal Office for the Environment

3 HES and Universities Involved Since 2006

University Hospital Basel (3)
University of Zurich (3)
FHNW-Fachhochschule Nordwestschweiz (3)

(2) signifies that a company has contributed twice to CCMX projects
(3) signifes that the institution is involved in three CCMX projects

Use of funding in 2013 (kCHF)

Funding of research	3'261
Education activities, conferences, industrial liaison	85
Management & administration	417
Total	3'763

CCMX Research Activity Highlights

PHOTO TONY KUNZ

From October 2013 through January 2014, CCMX Managing Director Nathalie Jongen and journalist Carey Sargent once again went on the road to gather stories and images for this year's annual activity report. They visited four running projects, meeting with project leaders and their various collaborators—co-applicants, PhD students, post-docs, and masters students—working in laboratories located in Dübendorf, Lausanne, Thun, Villigen and Zurich. Sargent has also written some follow-up stories for selected projects that have been previously featured in CCMX annual reports, that bring news about recent achievements.

Over the three years of making these project visits, besides gathering information about ongoing research, what continues to impress Jongen and Sargent are the stories behind, or along-side the research.

They note the way researchers enthuse about their collaboration with fellow scientists and with industry partners, about how the whole sums up greater than its parts. As one post-doc put it, "Good collaboration was the basis for the good results."

When project collaboration is at its best, it succeeds even across long distances. Researchers involved with the same project who work in different parts of Switzerland, use their travel budgets, as well as the telephone, email, and Skype video chats, to keep in touch.

Another valued aspect of collaboration is the contributions made by industry partners, who often take active roles in encouraging regular meetings among project researchers, and in many cases can supply materials that are difficult to procure or particularly specialised.

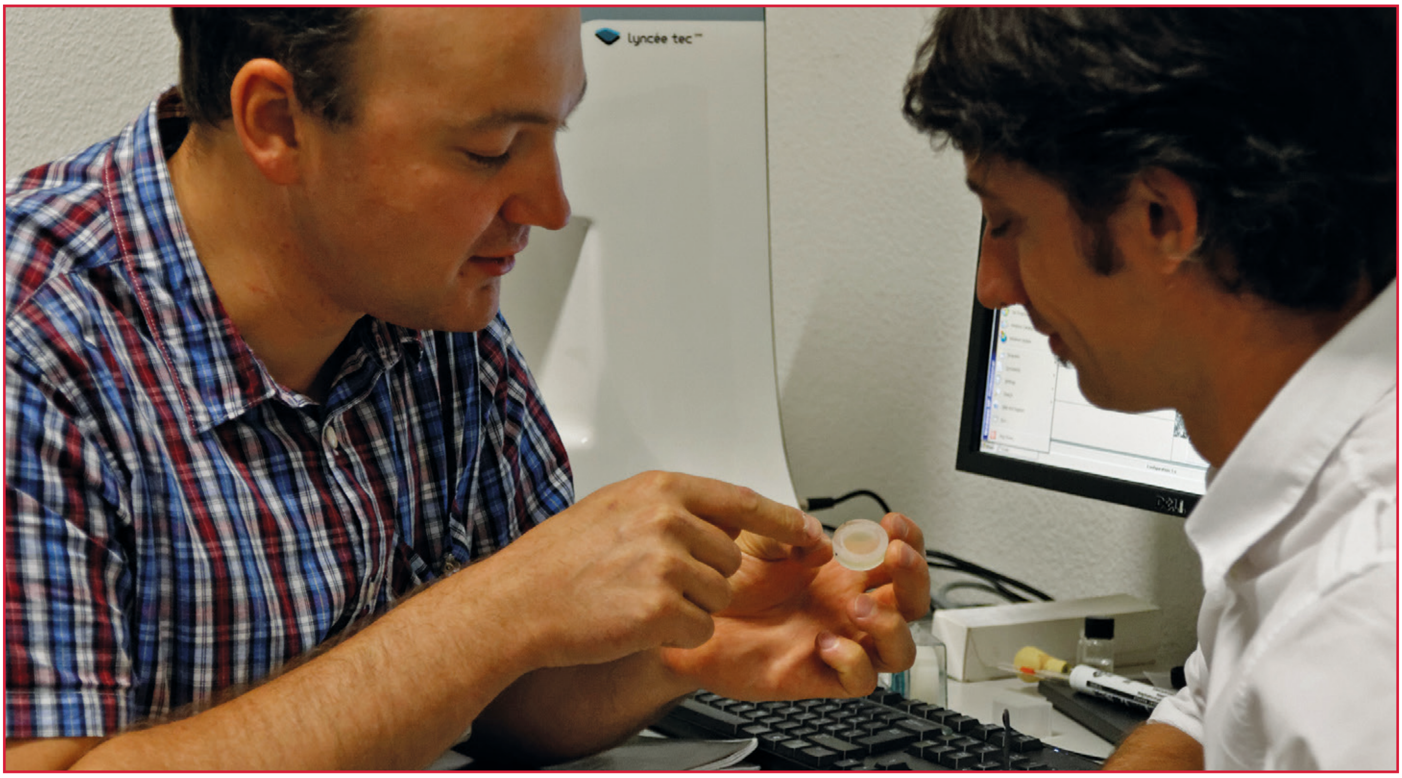
We hope the stories on the following eleven pages will appeal to a wide audience, revealing some intriguing aspects of the unique multi-disciplinary, multi-partner projects that CCMX co-funds with industry.

The researchers' generous cooperation in this effort allowed us to share stories we trust you will find engaging.

The CCMX project allowed us access to experts in the field and start a project that would have not been possible with completely company based resources. CCMX support, matched with governmental funds, allowed tackling the subject on a much larger scale.

Simon Berner, Straumann

Holographic Images Reveal Hidden Flaws



PHOTOS NATHALIE JONGEN

Y. Kuzminykh and S. Equis look at a sapphire piece before using digital holographic optical microscopy to identify the presence of deformities.

The different partners have different needs, and expectations too, and working closely with the companies and continually exchanging ideas keeps things exciting.

Sebastien Equis, Scientist, Empa

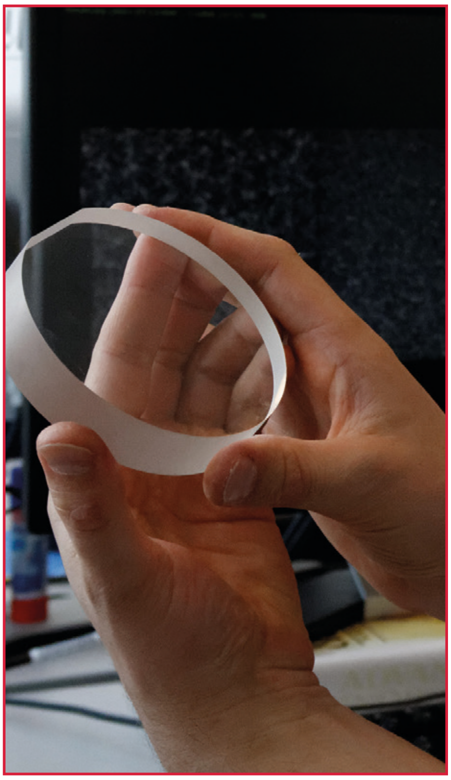
Sapphire piece makers have a problem: they cannot detect whether the material they are using is flawed with deformities until the very end of the manufacturing process. More than a quarter of the parts, used in products including watches and semiconductors, are thrown away at this late stage, wasting both time and money.

Manufacturers do this because the surface of the usually transparent raw material is rendered rough after being cut, and therefore opaque due to light scattering. This means that flaws cannot be seen until the pieces are polished, a step that requires considerable effort for such hard materials. At that point, humans visually inspect each piece. Companies working with diamond face a similar issue. In both cases, detection could be made more efficient and allow companies to reduce their rejection rates. Researchers led by Patrik Hoffmann, head of the Laboratory for Advanced Materials Processing at Empa, hope to help them do this by introducing microscopy-based methods of defect detection.

Hoffmann and colleagues Yury Kuzminykh and Sebastien Equis are doing this with a digital holographic optical microscope, an instrument that generates high resolution, 3D digital images of a sample using the principle of interferometry. The machine records the interferential field formed by the wavefield scattered by an object and a reference wave. Computer processing then allows researchers to obtain both the amplitude and the phase of the field the object disperses. Having this resolution in depth means that different focus levels can be recreated on the computer, and defects in the material can be detected from a single image.

This approach offers a considerable advantage over more standard 2D techniques, which involve scanning the sample through the focused laser spot and then reconstructing the 3D image from the data gathered —a time-consuming process.

"In industrial applications, you cannot scan every lens through the confocal microscope, it would just take too long," Hoffmann said. "It's easier to produce the parts and then let people find the error."



Various sapphire pieces.

Though the new approach offers the advantage of speed, there are still challenges that need to be overcome. One is figuring out how to define a "defect" with this highly precise method.

Though the new approach offers the advantage of speed, there are still challenges that need to be overcome. One is figuring out how to define a "defect" with this highly precise method. A flaw might be detectable by machine, but invisible to the human eye. The researchers need to adapt the machine to such limitations.

Another complication involves the effect of opaque sample surfaces on laser light. If the surface of the sample is too rough, the laser light will be scattered and unable to pass easily through the material. This results in very blurred images, or in no images at all. One approach to solving this problem could be making the wavelengths so long that they elude these small variations in roughness. A disadvantage to this is that it results in a loss of resolution.

"It's a good resolution for the wavelength, but when you see these pictures based on terahertz radiation, they look ugly," Hoffmann said. "We are used to resolutions of half a micrometer or better—these millimetre resolutions are not useful for precision industry."

This leaves the scientists with trying to exploit the difference in the refraction indexes of two materials. Light diffusion is strong when there is a large difference in the refraction indexes—bringing them closer together reduces it. This is why another partner in the project, Scientific Visual Sàrl, is trying to develop an index matching liquid for materials such as sapphire. Some are currently available, though they are highly toxic.

"Everyone knows that they exist, but no one wants to use them," Kuzminykh said.

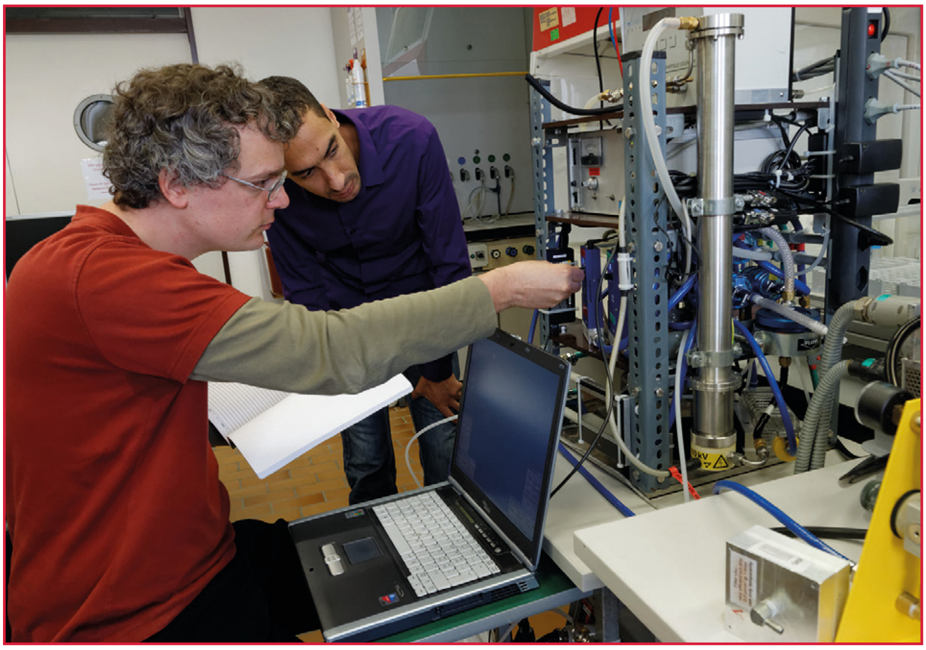
The industrial partner is striving to find a transparent, non-toxic liquid that could be applied to rough sapphire surfaces, allowing the laser light to pass through and reveal flaws. Those close enough to the surface could be polished away; any deep inside would result in discarding the piece. The challenge is finding a transparent liquid that can match high refractive index materials such as diamond.

Because liquids in general have lower refractive indexes than solids, one approach is to add nanoparticles to a liquid with a relatively high index of refraction: the higher the density of nanoparticles, the higher the index of refraction. With higher densities however, the nanoparticles tend to coagulate and precipitate. Empa is using nanodiamonds to clear this hurdle. Nanodiamonds are black though and the liquid should be transparent. Initial efforts to remove the color has led to white and yellowish nanodiamond powders, which is already a significant improvement.

The researchers hope that, once they attain a liquid with a higher index of refraction, they will be able to apply their techniques to diamonds. The same method, though with a different light source, could also be applied to testing silicon wafers.

"We're working on many different things within this project and this wealth of topics makes it very interesting," Equis said. "The different partners have different needs, and expectations too, and working closely with the companies and continually exchanging ideas keeps things exciting."

Innovative Instrument Analyses Airborne Nanop



A. Hess and M. Tarik have to precisely tune the gas flows on the modified SMPS at startup.

PHOTOS NATHALIE JONGEN

Suspended gold nanoparticles used to generate model aerosols for instrument validation.

Such an instrument could be used to evaluate the release of nanomaterials from sprays and polymers or from waste incineration or other combustion processes that may emit nanoparticles.

With companies incorporating nanoparticles into an increasingly wide range of products, it is becoming ever more important to evaluate the possible impact on human exposure, particularly through aerosol spray or incineration.

An essential first step is finding out what is actually released.

Elemental evaluation currently involves collecting airborne nanoparticles on slides, which are sent to a lab where they are digested in acid and then introduced to a type of mass spectrometer. The particles can also be deposited on grids and inspected by electron microscopy. The disadvantages of these approaches are that samples can only be collected over a given period of time and that there is a high risk of contamination. Being able to perform size-classified chemical analysis of airborne particles simultaneously and online would be a better approach.

Researchers at Empa and the Paul Scherrer Institute are working towards this goal by developing an instrument that couples functions performed by a scanning mobility particle sizer (SMPS) with those performed by a plasma mass spectrometer. The modified SMPS enables size fractionation and provides information on size distribution, while the inductively-coupled plasma mass spectrometre (ICP-MS) determines the elemental composition of the airborne nanoparticles.

"This should be an online measurement," said Heinz Vonmont, head of the Analytical Chemistry unit at Empa. "Particles will go in, and at the end you have everything—size, number, and chemistry. We want to find answers about the behaviour, composition and risk to man of nanoparticles in aerosols or materials."

Potential applications abound. Such an instrument could be used to evaluate the release of nanomaterials from sprays and polymers or from waste incineration or other combustion processes that may emit nanoparticles. The researchers would eventually like to have a mobile version of the instrument that could be easily taken into the field

M. Tarik testing the new ICPMS equipment for analysing nanoparticles in process gases from biomass (e.g. natural wood) and biowaste (e.g. waste wood) combustion.

We want to know what the fate of nanoparticles along this pathway is. What we're interested in is materials that become waste and what this means for emission control on the other side.

Christian Ludwig, Professor, PSI and EPFL

to evaluate such particles wherever they are produced. For now, the size of the equipment means it is confined to the lab.

The SMPS works by evaluating the movement of charged particles in an electric field. That is, particles are classified according to electrical mobility, with those of a selected range being guided through an output leading to a counter that determines their concentration. Those outside the range exit through an exhaust flow. The ICP-MS works by ionizing a sample with inductively coupled plasma and then using a mass spectrometer to separate and quantify the ions.

Adrian Hess, the PhD student who has been working on the coupling of the two machines, says doing so has been a challenge. The work involved finding the instruments, building up the whole flow set-up and purchasing all the parts needed for the modifications. The researchers needed, for example, electrically conductive tubing, tube fittings, pumps and flow controllers, and also had to evaluate a number of particle filters—there is little reliable information about filter efficiency in very small particle size ranges for most products on the market.

Hess and colleagues have since coupled the two machines and carried out an initial test to verify calibration. The scientists produced an aerosol with a known concentration by suspending particles in water, then dispersing this suspension into the air with a spray nozzle, producing an aerosol. The water is then removed in a drier, producing a dry test aerosol. This sample was then run through the coupled machine.

The researchers were surprised to find that the SMPS produced a peak in the 20-40 nanometre range, while the ICP-MS signal was strongest at around 100. They recognised that while the SMPS signal is related to the particle number, ICP-MS is linked to the total mass. They therefore calculated an approximated volume-based size distribution and produced a curve that was a much better fit to the data generated by the ICP-MS.

"The correlation between these signals is quite good," Hess said. "Based on that, we decided to continue."

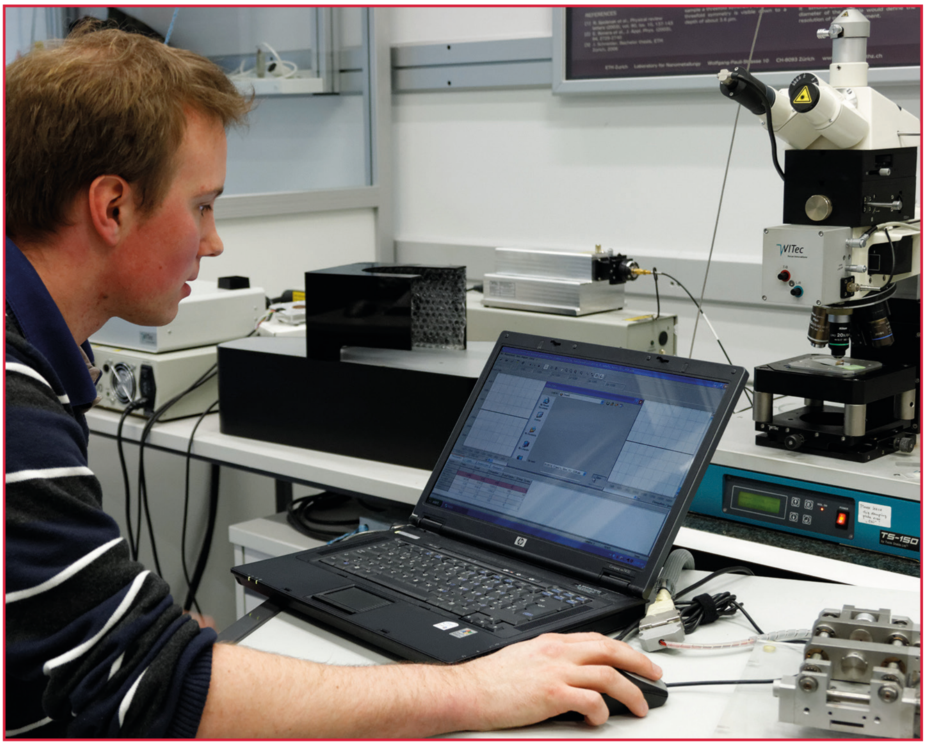
The researchers will now publish a paper about their set-up and show the proof of principle for the coupling and the established method for validating the instrument. Hess then plans to test aerosols containing large and small particles of different chemical compositions. Meanwhile, colleagues at PSI are preparing to investigate thermal incineration processes. PSI post-doc Mohamed Tarik plans to examine commercial nanoparticles mixed with cellulose and wood with ICP-MS in the coming months. The researchers will later compare the results with those generated by SMPS and ICP-MS together.

"In the future we're going to have a lot of materials containing nanoparticles and when they become waste, we may be treating them with incineration," said Christian Ludwig, head of the Chemical Processes and Materials Group at PSI. "We want to know what the fate of nanoparticles along this pathway is. What we're interested in is materials that become waste and what this means for emission control on the other side."

Team Develops Coatings to Extend Lifetime of

We could show that diamond-like carbon coatings are fantastic, it's only adhesion that you need to be worried about. No one investigated the interface to see how stable it is. We're the first ones to do that.

Roland Hauert, Senior Scientist, ETH Zurich



PHOTOS NATHALIE JONGEN

D. Bernoulli performing uniaxial loading with diamond-like carbon coated titanium substrates.

Even today's best hip implants generally fail after 15 to 20 years.

This is because the artificial joint's metal-on-metal friction eventually generates wear particles that can cause inflammation and pain. The implant then has to be replaced using complicated, time-consuming surgery.

Prosthetics manufacturers have experimented with diamond-like carbon (DLC coatings) —already used in a variety of fields to reduce friction and wear—to diminish production of such particles. DLC coatings are appealing because they are hard, have low friction coefficients and are chemically inert. Nonetheless, wear particles between moving parts can lead to locally applied high pressure on the coating and result in damage to both the DLC and the substrate. Initial attempts in knee, hip and toe joints failed after the coatings separated from the underlying structure in unpredictable ways.

Researchers at Empa and ETH Zurich hope to revive the approach by improving surface adhesion and developing ways to predict how long such joints might last.

"Our initial objective was just to understand why implants failed and how you could do better," said Kerstin Thorwarth, a scientist at Empa. "Now we're looking to establish a test setup to predict the lifetime of coated implants."

The team at Empa, led by Roland Hauert, a senior scientist in the Nanoscale Materials Science laboratory, started the project by making calls and emailing people to gather all of the failed DLC-covered joints. Their initial testing showed that the problem was not the DLC, but rather the silicon that was used to promote adhesion at the interface between the prosthetic and the coating. Stress corrosion cracking occurred in the reaction layer, and, mechanical load combined with the permeation of body fluids led to slow-growing cracks, which in turn ultimately caused the DLC coating to detach.

"In people's minds, DLC failed, but this is wrong —it's the interface that failed," Hauert said. "We could show that DLC is fantastic, it's only adhesion that you need to be worried about. No one investigated the interface to see how stable it is. We're the first ones to do that."

What people failed to realise, according to the researchers, is that an entirely new substance is formed at the interface between the coating and the substrate. Solid chemical bonds make for good adhesion, but also produce a few atomic rows of what is, in effect, a new implant material that is being introduced into the body's corrosive environment.

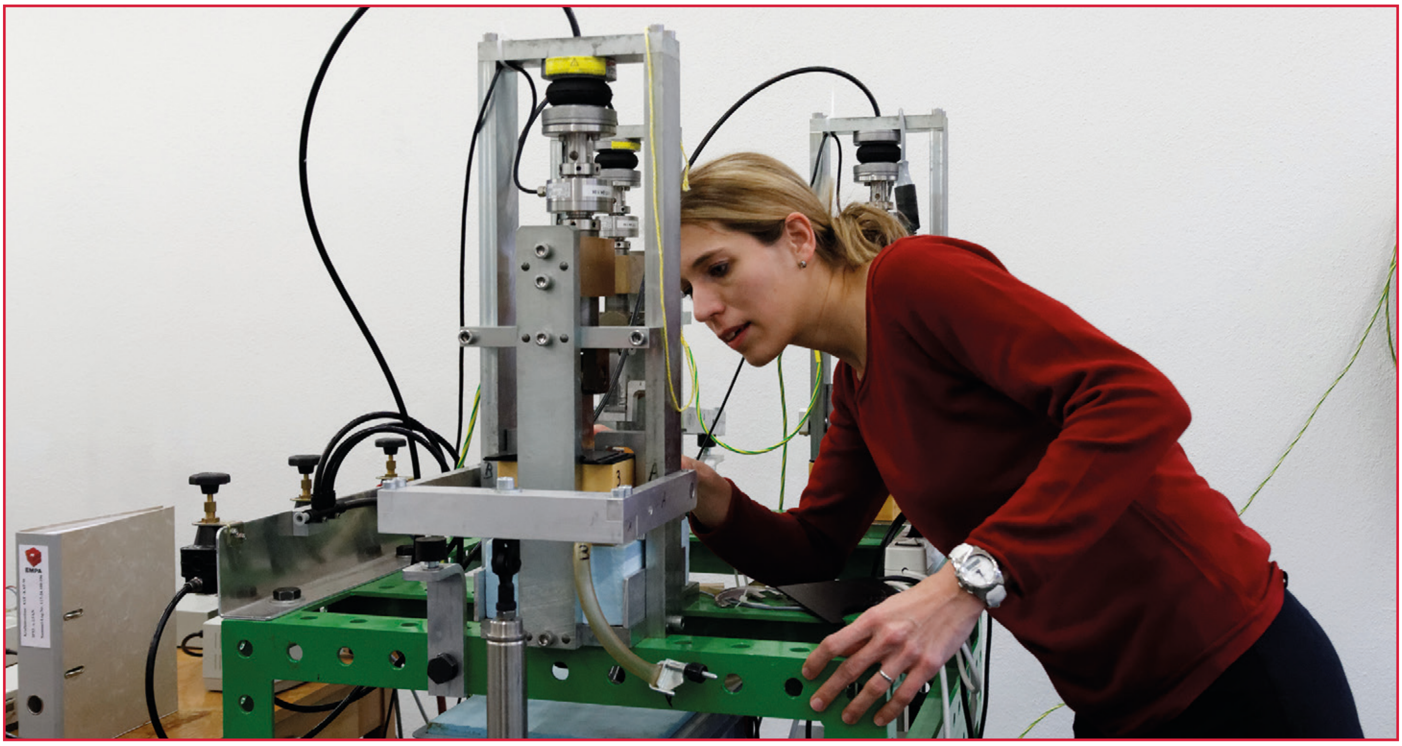
"It's a lot of work to check for all the defects that can destroy the material," Hauert said. "There's no simple solution and that's probably why no one has attempted it. We really want to understand these few atomics rows, what the material is, and what will it do in the future."

Empa researchers started out by developing new, corrosion-resistant, adhesion-promoting interlayer coatings made of tantalum, nitrogen doped tantalum and α-tantalum. The coatings work independently from the substrate and so are suitable for nearly every orthopaedic alloy, including those based on cobalt-chromium and on titanium-aluminium.

The team, which also includes Patrik Schmutz, a group leader at Empa, is continuing with a variety of tests and simulations, including joint simulators to assess the stability of these interfaces on the underlying materials. A particular focus is on estimating crack growth rates under conditions similar to those in the body, as well as the dissolution rate of the reaction layer.

The scientists have been able to ensure a lifetime of more than 100 years of articulation, and corrosion resistance for at least 40 to 50

Implants



K. Thorwarth mounting DLC coated spinal disks to the spine simulator.

years. The group is satisfied with coating performance so far, but intends to develop more fundamental research to analyse probable effects of all corrosion phenomena that will be faced by the new material. The ultimate goal is to calculate the expected operating lifetime of the coated implant in the human body. Current tests still take several years and are only able to predict the effects of up to 50 years of wear. The team is looking for a way to accelerate these tests.

"We need to do a lot more basic research to be able to give a kind of guarantee, although compared to the rest of the world we're pretty far ahead," Hauert said.

Meanwhile, Daniel Bernoulli, a PhD student in Ralph Spolenak's Laboratory for Nanometallurgy at ETH Zurich, is helping Empa researchers improve mechanical stability. Mechanical forces exerted on the implant either in the body or during processing of the joint can lead to crack formation and delaminated areas. Bernoulli is investigating how the coating fails in both cases by simulating contact damage with indentation, and mechanical stress with uniaxial loading. He is also simulating stress distribution with finite element analysis.

The biggest challenge has been finding the ideal length scale for the thickness of all involved materials. Getting it right is a balancing act because there is a trade-off between fracture strength and delamination.

"We found that we can increase the fracture strength if we decrease the film thickness, but then we have a much higher risk for contact damage," he said. "Neither cracks nor contact damage are good for the system, so we have to find something in between."

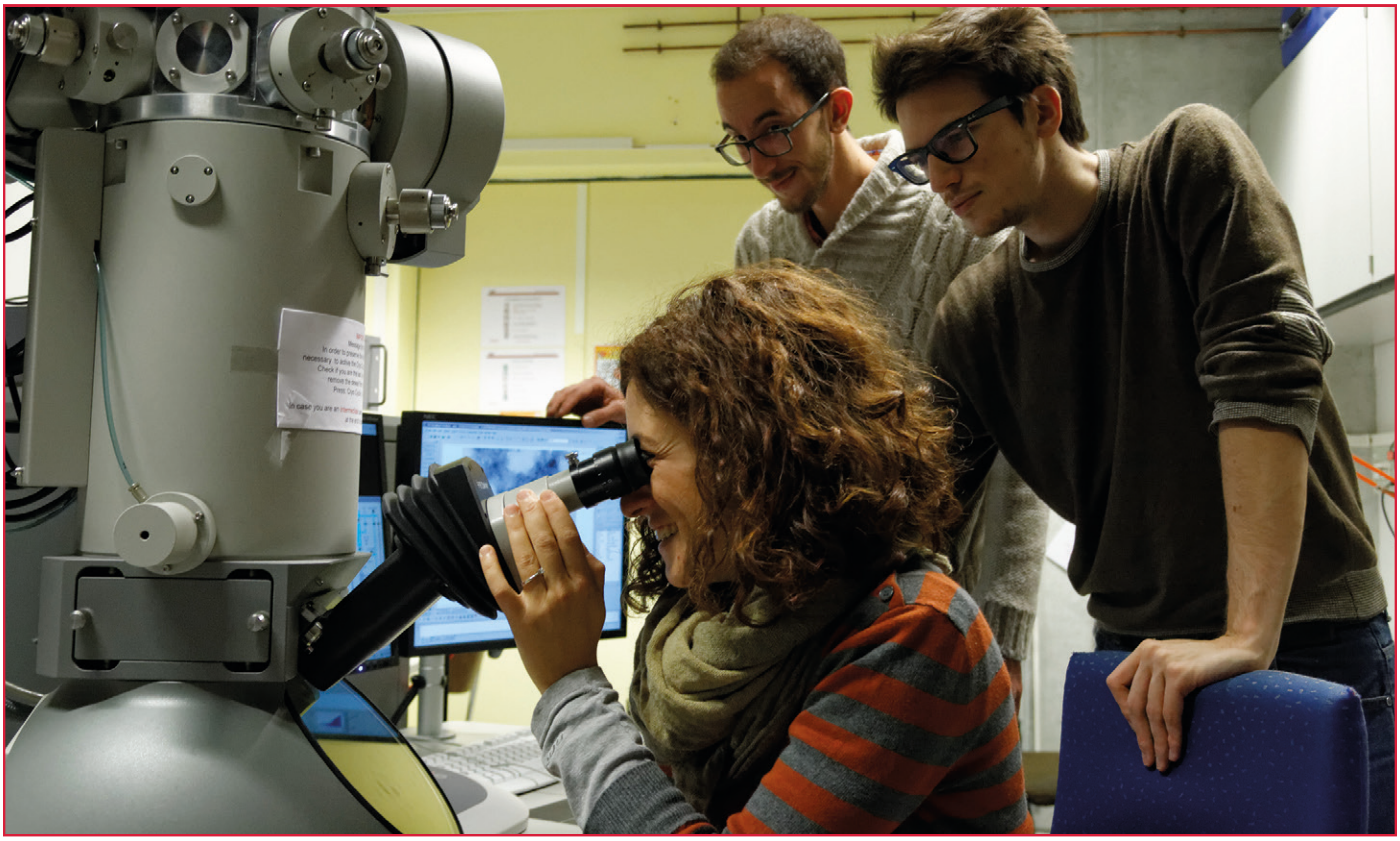
Bernoulli is continuing to work on interlayer characterisation as well as fracture strength and delamination. He is also looking at contact damage problems and with more simulations, using interlayer materials and film thicknesses chosen based on previous experiments. The work has given the researchers insight into the processes that cause damage and how they can optimise the whole system.

The work is even more far-reaching than that. "Everything we've done here can be applied to any two bodies in frictional contact," Spolenak said. "It can be used for any other moving implants, and applied to engines, bearings, and even tooling."

Everything we've done here can be applied to any two bodies in frictional contact. It can be used for any other moving implants, and applied to engines, bearings, and even tooling.

Ralph Spolenak, Professor, ETH Zurich

Dental Gel Set to Get Longer Shelf Life



A. Apicella, M. Marascio and V. Colangelo during a Transmission Electron Microscopy (TEM) session in CIME (EPFL).

Dental enamel is the only tissue in the human body that does not renew itself. This means that every day wear and build-up of plaque eventually decays and erodes it, leading to gum disease and the destruction of what holds teeth in their sockets.

Straumann, the world's biggest maker of dental implants, sells a product that can help tissue regeneration by promoting its growth. Straumann® Emdogain, a combination of enamel matrix derivative (EMD) proteins and propylene glycol alginate (PGA), is directly applied to damaged tissue, where the regenerative action starts. Though the process is promoted by physiological conditions, neither the interactions between EMD and PGA nor the product's mechanism of action are completely understood.

EMD is a mix of about 90% amelogenin and other proteins that self-assemble to form a matrix. Proteins come with a particular set of challenges because they adopt specific shapes that are critical to their functioning. Altering the environment, pH or temperature can affect these configurations and, in turn, have an impact on a given protein's stability and effectiveness. Researchers at EPFL and ETH Zurich are trying to figure out how to make EMD and the EMD/gel complex less susceptible to such changes.

"We'd like to find a way of imposing stability so it can spend more time in storage and be less sensitive to thermal shock," said Christopher Plummer, a senior researcher in EPFL's Lab of Composite and Polymer Technology. "Basically, we want to identify the conditions under which EMD will remain in its initial state for as long as possible."

Researchers at EPFL are focused on gaining a fundamental understanding of how the mix of proteins reacts to changes in variables such as environment and temperature. They are using computer modelling as well as analytical tools such as atomic force microscopy, transmission electron microscopy and infrared spectroscopy in their work.

"The important thing in using these technologies is to be able to compare results from different samples prepared under different pH conditions and at different temperatures," said Alessandra Apicella, a post-doc in EPFL's Laboratory of Composite and Polymer Technology.

These investigations have allowed them to build up a systematic representation of phase behaviour, to see how EMD molecules self-assemble as a function of temperature and pH.

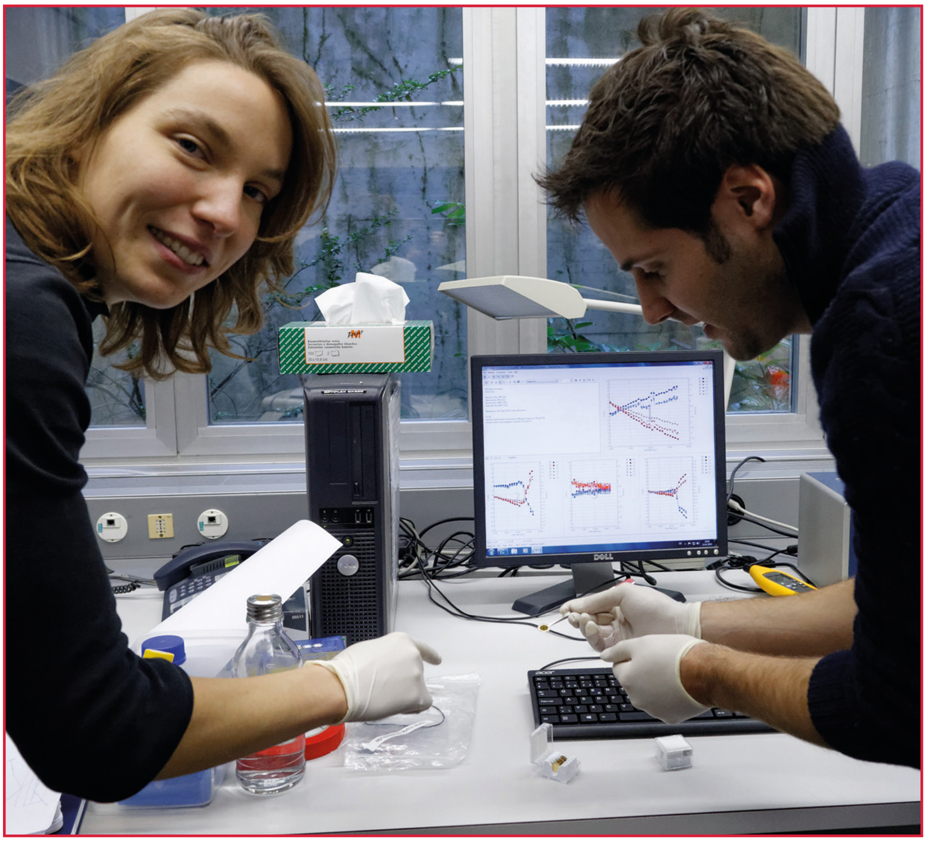
The group's understanding of EMD is being helped along by graduate student Matteo Marascio's theoretical work. Marascio and the team set out to develop a computational approach to modelling the amelogenin, the main constituent protein.

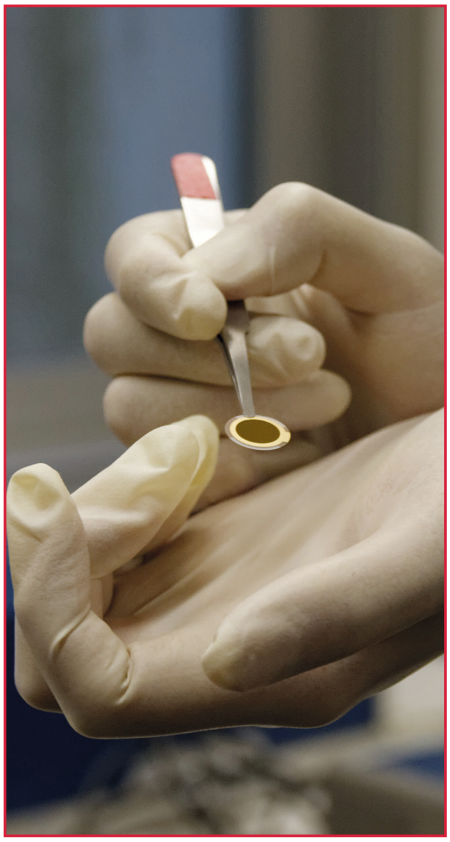
The group already compared its initial model, a fourth of the overall protein length, with experimental data gathered from nuclear magnetic resonance techniques and Fourier Transform Infrared Spectroscopy and found a close fit, validating the approach.

"We started with nothing one year ago and now we have a first part of the model which contains most of the information that we can see by experimental techniques," Marascio said. "What we are doing now is trying to apply the same method to the remainder of the protein."

Altogether, the work has allowed the group to develop practical guidelines for promoting long-term stability of EMD by modifying its environment and using suitable additives.

PHOTOS NATHALIE JONGEN





P. Heunemann and collaboration partner B. Simona at ETH Zurich preparing Quartz Crystal Microbalance.

Meanwhile, colleagues at ETH Zurich have been examining PGA, the polymer carrier used to formulate Straumann® Emdogain's gel matrix. This carrier needs to be able to release the protein when applied to dental tissues, yet remain stable—or show a predictable change in properties such as viscosity, pH and morphology—while in storage.

The current gel is considered a good carrier because it has antimicrobial properties and facilitates the dissolution of the EMD-proteins. The protein is released from the carrier through the neutralisation of the gel's acidity and an increase in temperature during application. It then self-assembles and precipitates on the dental tissue. It is not clear how this process is affected by variables such as temperature, pH or exposure to radiation.

"The good news is that the protein is very stable so the question is really on the side of the polymer," said Peter Fischer, a professor at ETH Zurich's Laboratory of Food Process Engineering. "We're trying to understand what the limiting factors are here."

Fischer and post-doc Peggy Heunemann are looking primarily at the polymer's natural and process induced stability, the polymer interaction with the protein and the deposition of individual and mixed components on tooth model surfaces. To this end, they study the mixing process and do quality control measurements on the polymer and also on the combination with the protein. They also study how the flow properties of the polymer and protein-gel are affected by variable factors that alter Emdogain and thus limit its shelf life.

The scientists have used instruments and techniques such as rheometers, viscosimeters and diffusing wave spectroscopy to gather data about the substance. They have recently started using the stopped flow technique and quartz crystal microbalance to investigate what happens in the mouth itself. The instruments give information about how or whether the protein, the carrier or the mix is absorbed by the tooth. They also help EPFL colleagues with protein stability studies and work together towards improved understanding of the final product.

"In the end, we're developing a good understanding of the polymer and what it's doing in the mix," Heunemann said. "This knowledge can also be used later on for new formulations of these complexes."

In the end, we're developing a good understanding of the polymer and what it's doing in the mix.

Peggy Heunemann, Post-doc, ETH Zurich

CCMX Projects Follow-up

PSI's Imaging Tool Prototype Achieves World-Record Resolution

Researchers at the Paul Scherrer Institute's Swiss Light Source have achieved world-record isotropic 3D resolution of 16 nanometres in X-ray imaging using a prototype of OMNY (tOMography Nano crYo), an instrument that will use ptychographic tomography for reaching high resolution in large samples.

The scientists performed the demonstration on a specially prepared six-micron-thick test object of nanoporous glass. The sample was designed to resist potential deformation from radiation damage, which reduces sharpness and makes it difficult to demonstrate targeted resolution.

"You always have the question whether it's the damaged sample that is frustrating everyone or whether the set-up isn't working," said Mirko Holler, the PSI researcher leading the project. "This special sample allowed us to demonstrate that ptychographic tomography can reach a 3D resolution that is not reached by other X-ray imaging techniques, and that the instrument concepts are working."

The researchers are developing the instrument to fill the resolution gap between electron microscopy and X-ray imaging, allowing for the improved measurement of thick—that is, statistically representative—sample volumes at high resolution.

The instrument achieves this through ptychography, a combination of scanning X-ray microscopy and coherent diffractive imaging. The sample is scanned in 2D with nanometre accuracy perpendicular to the X-ray beam. Rather than only detecting the transmitted X-ray intensity in a scan, the method involves using a 2D detector to measure diffraction patterns at successive, overlapping, regions of the sample. This creates a redundant dataset, allowing the scientists to mathematically reconstruct the object. Applied in 3D via computed tomography, this method is quantitative and provides a 3D dataset of the electron density in the sample. The OMNY prototype is now being used regularly by scientists attracted by its improved resolution and quantitative data. Researchers have come to measure samples of materials including cement, fossils, chalk and catalysts.

While the prototype measures at room temperature and atmospheric pressure, OMNY will feature a cryogenic sample environment in ultra-high vacuum including a cryogenic sample transfer system. This will allow imaging of radiation-sensitive objects such as polymer structures and biomaterials.
The team has completed the CAD design and will start assembling the final version of the instrument in spring 2014.

PHOTO COURTESY PSI

M. Holler, with cSAXS beamline scientists A. Diaz and M. Guizar-Sicairos, presents imaging results using the OMNY test instrument.

We're really getting a lot of interest from user groups all over Europe and beyond

Mirko Holler, Scientist, PSI

Researchers Closing in on Camouflage Layer for Dental Implants

The dark grey colour of dental implants can sometimes be seen through the gum—a serious aesthetic issue for some people.

A team of materials scientists, chemists, biologists and medical doctors working at ETH Zurich, the Universities of Zurich and of Geneva, Empa and Institute Straumann AG, the world's biggest manufacturer of dental implants, have been working to address this flaw with a coloured ceramic coating that can be placed over the titanium implants. The final product needs to be aesthetically pleasing as well as mechanically and chemically sound—to camouflage the metal and withstand the mouth's harsh environment.

Scientists led by Ralph Spolenak, head of the Department of Material Science at ETH Zurich, quickly developed a ceramic thin film with the right mechanical properties and a good colour, but were faced with the challenge of increasing the coating's luminosity to help mask the implant.
Now, two years later, they have figured out two possible ways of doing this.

The first approach involves adding a layer of aluminium or silver between an interference colouring layer and the titanium. The other involves depositing an array of ceramic layers that have very different indices of refraction, a procedure that results in very high reflectivity approaching 100% over a given band in wavelength. The group is now investigating the mechanical properties of these modified films and assessing their performance in the sometimes acidic environment of the mouth.

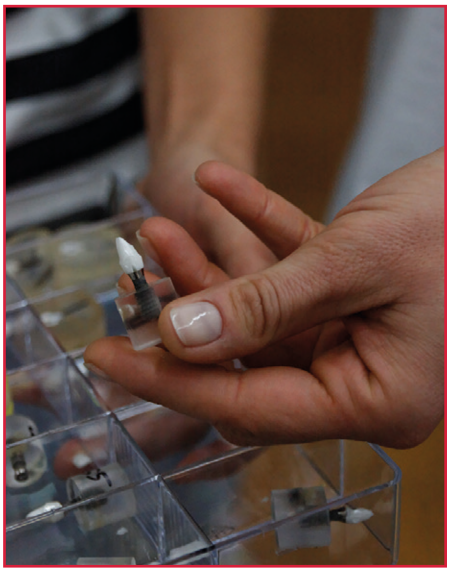


PHOTO NATHALIE JONGEN

Selecting a sample implant from the collection.

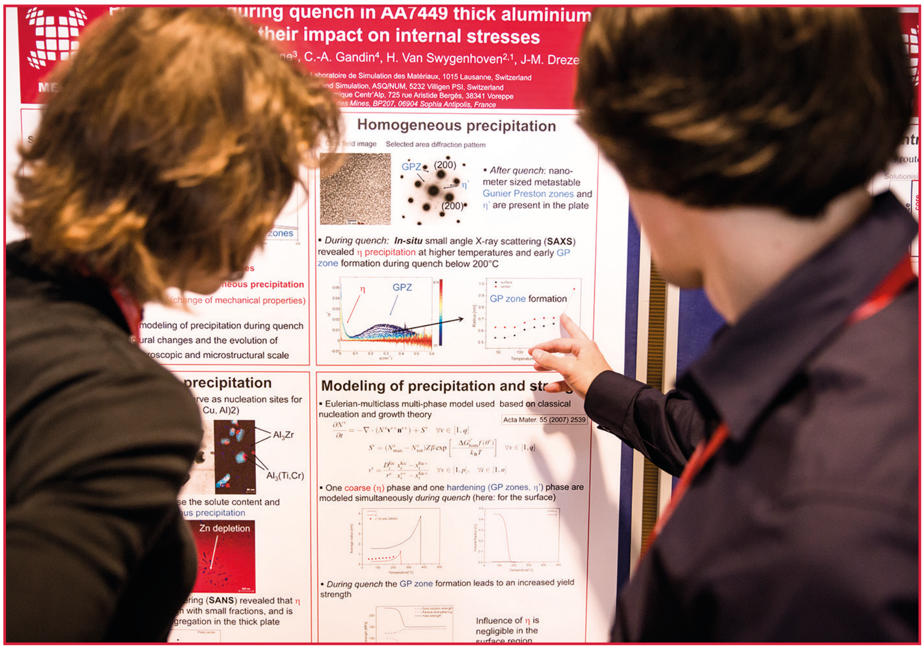


PHOTO TONY KUNZ

Discussing findings during the poster session, 2013 CCMX Annual Meeting, Bern.

We could see directly more or less for the very first time how very small precipitates form at lower temperatures during quench

Patrick Schloth, PhD Student, PSI

Researchers Get First Look at Real-Time Precipitate Formation during Quench

Precipitates can make metals stronger by increasing the yield strength, but these fine particles can also cause higher residual stresses if they form during quenching of large components. Their formation is currently challenging to characterise and predict.

Two PhD students, Nicolas Chobaut and Patrick Schloth, supervised by Jean-Marie Drezet and Helena Van Swygenhoven at EPFL and the Paul Scherrer Institute, respectively, have been helping industrial partners Constellium and ABB-Turbosystems figure out the best way to describe and model precipitation influences on the mechanical properties of thick aluminium components. The undertaking has been more challenging than expected.

Aluminium precipitates evolve, changing size and composition as a function of both temperature and time. This is hard to describe in a precipitation model, and experiments are complicated because precipitate sizes range from half a nanometre to much larger dimensions in a single sample. It is also delicate to quantify the chemical composition of the very small precipitates, which is usually far from the composition at equilibrium.

"This is why it's a bit tricky to try to simulate the precipitates in a physically correct manner," Schloth said. "It's also very difficult to get a good idea about precipitates from the literature. Of course, if this were easy, someone would have done it already."

Precipitation during quench is now generally detected indirectly, through an observed increase in yield strength. No one has really been able to say what actually takes place because the quenching phenomenon is very fast, making direct measurements challenging. The microstructure continues to evolve after quenching, so data gathered at this point is not strictly relevant to the process itself.

Schloth and a team at PSI directly observed the material during the quenching process—using small-angle X-ray scattering (SAXS) on the cSAXS beamline at PSI. The researchers heated small samples with a laser, cooled them at different quench speeds and observed the evolution of the nanostructure during the process.

"Basically, we could see directly more or less for the very first time how very small precipitates form at lower temperatures during quench," Schloth said.

These small precipitates, ranging in radius from about 0.5 to 0.7 nanometres, are of great interest because they increase the yield strength and therefore the residual stresses. The idea now is to take the results gathered from this observation—the volume fraction and average size of the precipitates—and couple it with finite element simulations Chobaut is doing on internal stresses at the macro level . This should result in a multi-scale model for quenching heat treatable aluminium alloys.

Schloth will use the information from the cSAXS experiment, along with data gathered from other experiments to calibrate the model, so it can predict precipitate evolution for different cooling conditions. Chobaut will use the cSAXS experiment results to calibrate a yield strength model. The final result should be a calibrated precipitation model that predicts the microstructure during different quench paths.

The output of this precipitation model will be used to calculate changes in yield strength during quenching. This will ultimately allow Chobaut to predict internal stresses in thick parts using a thermo-mechanical finite element model.

"The outlook is to have a precipitation model that is able to reproduce what we see by the end of the year," Schloth said.

CCMX Projects Follow-up

These coatings provide both high lateral heat dissipation along the tool surface and heat shielding normal to the tool surface.

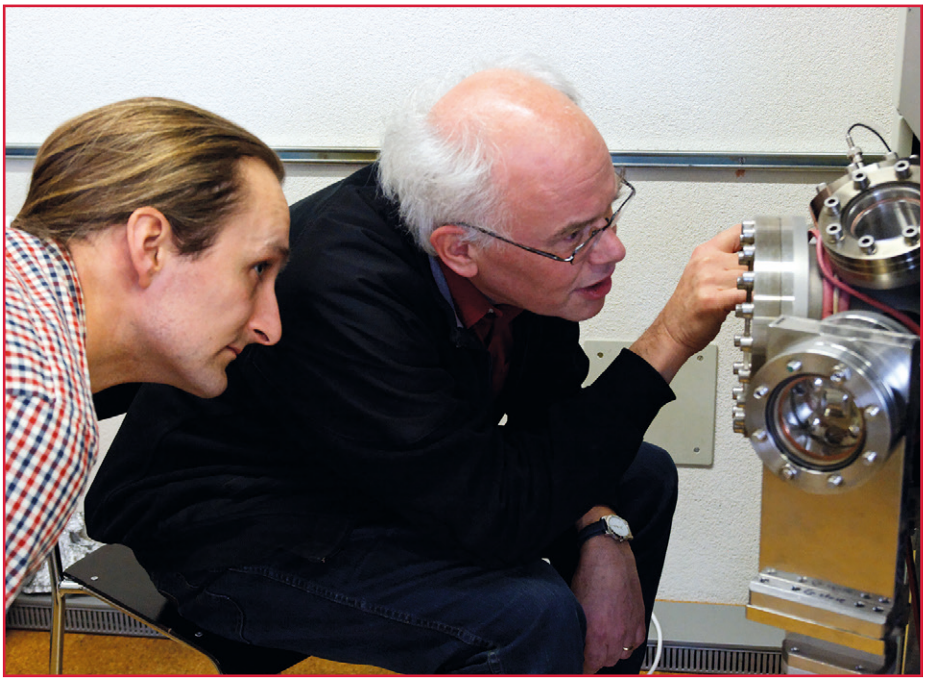


PHOTO NATHALIE JONGEN

M. Böttger and J. Patscheider check samples inside the X-ray photoelectron spectroscopy instrument.

Researchers Tailor Thermal Conductivity to Strengthen Machining Tool Coatings

Titanium and nickel superalloys—modern materials for lightweight or high-temperature applications—are notoriously difficult to machine. The process is associated with high tool wear and slow material removal.

Low thermal conductivity explains a lot of the problem. It necessitates very high process temperatures, and leads to softening of the tool, increased chemical reactivity and local overheating or "hot spots". All of these phenomena contribute significantly to cutting tool wear.

A team of researchers from ETH Zurich and Empa, along with industry partners Oerlikon Balzers Coating AG, are trying to determine the most efficient and least costly way to modify the structure of hard cutting tool coatings to provide high lateral thermal conductivity and therefore increased lifetimes.

The team has so far examined several possible approaches and has found that excellent coatings with high lateral thermal conductivity can be obtained using a method of arc-evaporation. The problem is that the associated rapid erosion of the target material results in a spray of µm-sized metal particles or droplets. These small pieces are then incorporated as flaws into the coating and influence its thermal conductivity. In the end, scientists need to consider not only the intrinsic conductivity of individual layers, but also droplet concentration, shape and the possible influence of interface conduction when engineering thermal conductivity in arc-evaporated multilayer materials.

During the course of the project, post-doc Michael Böttger used Finite Element Method (FEM) simulations to quantitatively characterise the effects of droplet size, material and orientation on the anisotropy, or direction, of thermal conductivity. He found that for spherical droplets at concentrations usually seen in arc-deposited coatings, anisotropy decreases linearly in proportion to droplet concentration and almost independently of their thermal conductivity. For ellipsoidal droplets, anisotropy strongly depends on the material and on droplet orientation.

The work has shown that creating coatings with high lateral thermal conductivity that consist of multiple layers with alternating thermal conductivities is possible even in the presence of such droplets. Controlling droplet formation can even be used as a way to engineer the direction of thermal conductivity in the arc-deposited coatings. The scientists also compared different approaches to inducing thermal conductivity anisotropy.

In the end, the group, which also included teams from University of Illinois (USA) and the École Nationale d'Ingénieurs de Saint-Étienne (France), produced multilayer coatings with alternating layers of high and low thermal conductivity. These coatings provide both high lateral heat dissipation along the tool surface and heat shielding normal to the tool surface.

The two resulting patents will help industrial partner Oerlikon Balzers Coating AG offer new industrial solutions to the worldwide hard coating tool market, underscoring its leadership in the design of hard coatings with engineered thermal conductivity.

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M. Weisser, **Effect of different loading conditions on the accumulation of residual strain in a creep resistant 1%CrMoV steel: A neutron and X-ray diffraction study,** *EPFL Thesis 5722* (2013).

H. Meidani, **Phase-field modeling of micropore formation in a solidifying alloy,** *EPFL Thesis 5653* (2013).

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R. Hauert, K. Thorwarth, G. Thorwarth, **Invited Overview: An overview on diamond-like carbon coatings in medical applications,** *Surface and Coatings Technology, Special Issue: Biomedical Coatings,* 233 (2013) 119-130.

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Projects Ongoing in 2013

PROJECT TITLE	PRINCIPAL INVESTIGATOR (PI)	PI'S INSTITUTION	OTHER INSTITUTIONS	ERU/PLATFORM
Microtome4SIMS: Chemical tomography of biological material with 100 nm resolution	Beat Keller	Empa	Unispital Basel	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 TiAINb 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
VIGO: 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
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 (until 2012)	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 in 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
NanoAir – Online coupling of a scanning mobility particle sizer SMPS to an inductively coupled plasma mass spectrometer ICP-MS for size fractionated, elemental analysis of nanoparticles in aerosols	Heinz Vonmont	Empa	EPFL, PSI, FHNW	NMMC
Argon-cluster as primary ion source in time-of-flight secondary ion mass spectrometry (ToF-SIMS)—method development by ToF-SIMS and Atomic Force Microscopy (AFM)	Laetitia Bernard	Empa		NMMC
Failure and defect analysis of fibre composite materials by means of X-ray interferometry	Rolf Kaufmann	CSEM		NMMC
Structure-activity relationships of metal oxide nanoparticles-based gas sensors for non-invasive medical diagnosis by time- and surface-resolved XAS-IR	Davide Ferri	Empa	PSI	NMMC
Optical Detection and Investigation of Subsurface Features and Defects in Unpolished Transparent Materials and Films	Patrik Hoffmann, Yury Kuzminykh	Empa		NMMC

CCMX Team 2013

Management Team

Prof. Karen Scrivener, Chair
karen.scrivener@epfl.ch

Dr. Nathalie Jongen
Managing Director
nathalie.jongen@epfl.ch

Kathleen Collins
Website/Events/Courses
kathleen.collins@epfl.ch

Maria Delaloye
Administration/Accounting
maria.delaloye@epfl.ch

CCMX Management Board

Prof. Karen Scrivener, Chair (EPFL)

Dr. Nathalie Jongen
Managing Director

Prof. Heinrich Hofmann (EPFL)
Former Director, SPERU

Prof. Hans Josef Hug (Empa)
Director, Analytical Platform/NMMC

Prof. Harald Krug (Empa)
From October 2013

Dr. Katharina Maniura (Empa)
Former Director, MatLife, until March 2013

Prof. André Studart (ETH Zurich)
From October 2013

Chiara Corticelli (Empa)
Industrial Liaison Officer, Education & Administration

Analytical Platform Management Board

Prof. Hans Josef Hug, Director (Empa)

Prof. Cécile Hébert (EPFL)

Dr. Katharina Maniura (Empa)

Prof. Bill Patterson (PSI)

Chiara Corticelli (Empa)
Industrial Liaison Officer, Education & Administration

CCMX Steering Committee

Prof. Karen Scrivener, Chair (EPFL)
Dr. Jacques Baur (Rolex)
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Dr. Pierangelo Gröning (Empa)
Dr. Harry Heinzelmann (CSEM) (from July 2013)
Dr. Ernst Lutz (Sulzer)
Prof. Roland Siegwart (ETH Zurich)
Prof. Ralph Spolenak (ETH Zurich)
Prof. Friso van der Veen (PSI)

PHOTO TONY KUNZ

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CCMX
Competence Centre for
Materials Science and Technology

EPFL STI
Batiment MX
Station 12
CH-1015 Lausanne
+41 21 693 4656
www.ccmx.ch