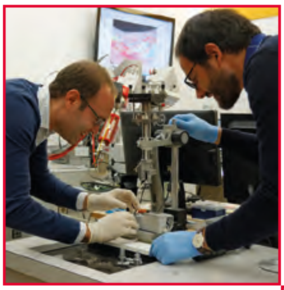
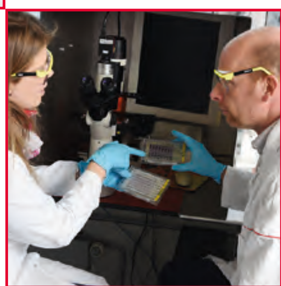
CCMX
Competence Centre for
Materials Science and Technology

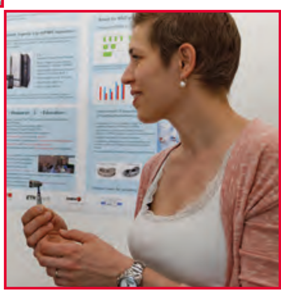
Annual Activity Report 2015










Annual Activity Report 2015

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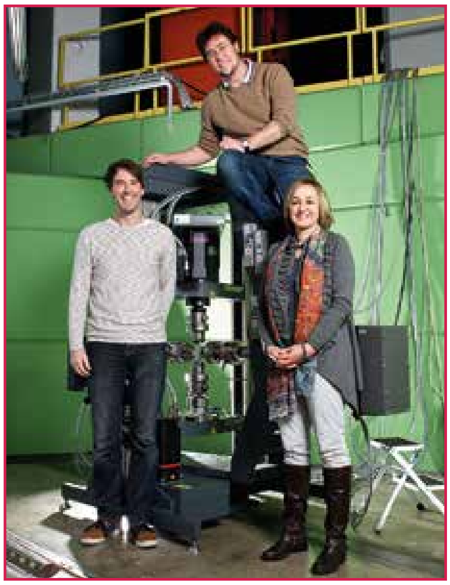
CCMX: Invigorating Materials Science in Switz

CCMX projects are a unique opportunity to address the needs of Swiss industry via collaboration with Swiss research institutes. The change from the ticket programme to the Challenges model increases the involvement of industry, fostering closer and enduring collaborations.

Peter Wick, Empa, NanoScreen Challenge



PHOTOS: NATHALIE JONGEN, COURTESY PSI

CCMX was established in 2006 to create new opportunities and closer connections in the field of materials science in Switzerland. The Centre's goal was to bridge the significant gap between the fundamental basic research conducted by Swiss academic institutions and the proprietary research performed in corporate laboratories.

CCMX's strategy has been to concentrate its efforts on focussed cutting-edge research, building strong industrial partnerships and establishing a dynamic network to encourage cross-disciplinary interactions.

In the last nine years, CCMX has introduced a new paradigm for collaborative research through its pragmatic and productive Public Private Partnership (PPP) programme, an approach that has evolved into a proven solution for funding pre-competitive materials science research.

Interdisciplinary collaboration is a core value at every level of the Centre's activity, fostering innovation through focussed, cutting-edge research, the results of which are published openly, creating potential for commercial applications. From funding to training to networking, CCMX has promoted increased interactions among stakeholders: through 119 formal interactions between the 5 research institutions, 57 companies and 2 Federal Offices.

In Phase 1 (2006–2009), CCMX allocated 10.5 mCHF to its Flagship Projects involving 9 companies. In Phase 2 (2008–2015), the strategy shifted to a 2-to-1 funding model, establishing more meaningful collaborations between academia and industry. CCMX also moved from short- to longer-term activities, continually increasing industry involvement with 23 companies actively participating.

The projects launched during Phase 1 and Phase 2 have created substantial return on investment, with offshoot projects amounting to more than 26mCHF in funding from industry, and from Swiss/international private and public sources.

The new funding model launched in Phase 2 has better aligned academia with industry—initiating collaborations at the pre-competitive stage of research has brought value by stimulating and sustaining Switzerland's long-term research efforts in materials science.

With Phase 3 launched in 2012, CCMX began to secure its legacy beyond the 2016 cessation of ETH Board funding. The Centre has continued promoting the kinds of collaboration that have become critical for innovation. The Materials Challenges research initiative, co-funded professor chairs and CCMX course offerings are expected to continue until 2020. To date, the first four Materials Challenges involve 32 companies, representing industry funding of 5.0mCHF. This structure should perpetuate industry support beyond 2020.

Another enduring aspect of CCMX's activities has been establishing openly available, unique characterisation apparatus, including:

- NanoXAS: unique combination of Scanning Probe and Scanning Transmission X-ray microscopes

2006
- CCMX launched in April

2007
- 16 Flagship Projects involving 9 companies
- 11 projects funded by the Analytical Platform involving 2 companies

2008
- Research Ticket Programme launched

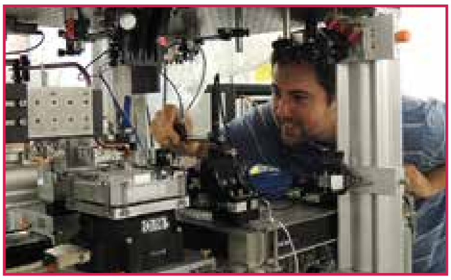
2009
- 4 Research Ticket Programme projects involving 10 companies
- 7 new projects funded by the Analytical Platform involving 3 companies

2010
- 2 new projects funded by the Analytical Platform
- MaCH2 analytical equipment database launched

2011
- 10 new Research Ticket Programme projects involving 13 companies and 2 Federal Offices
- 11 new projects funded by the Analytical Platform involving 13 companies

erland and Seeding the Future






PHOTOS: ALAIN HERZOG, NATHALIE JONGEN, TONY KUNZ, COURTESY PSI

- OMNY: Ptychographic tomography, unprecedented 3D resolution of 16nm
- Biaxial Deformation: unique rig improving *in situ*, realistic mechanical testing of industrial metallic alloys

CCMX training has offered opportunities to learn and network with peers and subject-matter experts through a recurring cycle of well-attended courses:

- CCMX Summer School: present comprehensive overviews in a given field
- CCMX Advanced Course: maximise practical experience
- Winter School: emphasise interdisciplinary collaboration

Since its launch, every aspect of CCMX's activities has advanced materials science in Switzerland. What began as a bridge has become a solid foundation that will support productive and innovative collaboration critical to the success of Swiss industry.

CCMX RESEARCH + TRAINING + NETWORKING ACTIVITIES SINCE 2006

CCMX PUBLIC PRIVATE PARTNERSHIP PROGRAMME

67 Total Projects (completed + ongoing), involving 52 companies and 2 Federal Offices

CCMX MATERIALS CHALLENGES RESEARCH INITIATIVE

MC1 Coating Competence Center for Sputter Deposition by HiPIMS, High Performance Coatings (with Oerlikon Balzers)

MC2 NanoScreen: Reliable and Rapid *in vitro* Safety Assessment of Nanomaterials, Understanding Nanosafety (with Cetics Healthcare Technologies, Midatech, KRISS, Federal Office of Public Health)

MC3 Additive Manufacturing and Metallic Microstructures (AM3), Re-thinking the Materials Science Behind Additive Manufacturing (with with General Electric [Switzerland] GmbH,Patek Philippe, Rolex, VV [Branch of Richemont International SA], Audemars Piguet & Cie, Swatch Group R & D Ltd, Heraeus Materials SA, Oerlikon Metco AG)

MC4 Self-Care Materials, Fibre Science for Controlled Release (with a consortium of 18 companies)

CCMX COURSES

6 Summer Schools, 388 participants
13 Advanced Courses, 270 participants
5 Winter Schools, 93 participants
11 other courses, 480 participants

CCMX DESSIMINATION ACTIVITIES

476 reviewed publications
805 conference talks and posters
21 patents

CCMX EVENTS

9 Annual Meetings, 1161 participants
19 Technology Aperitifs, 743 participants
11 other events, 413 participants

CCMX GRADUATES

64 PhDs completed since 2006

2012

- Tandem Approach launched: 4 co-funded Professor Chairs and Materials Challenges research initiative
- 2 new projects funded by the Analytical Platform involving 7 companies

2013

- First professor in place at ETHZ
- 2 professors in place at EPFL
- 1 new project funded by the Analytical Platform involving 1 company

2014

- Materials Challenge 1: Coating Competence Center, approved for funding
- Materials Challenge 2: NanoScreen, approved for funding
- 3 new characterisation facilities installed at ETHZ, Empa, and PSI

2015

- Materials Challenge 3: Additive Manufacturing & Metallic Microstructures (AM3), approved for funding
- Materials Challenge 4: Self-Care Materials, approved for funding

2016–2020

- Fourth professor in place at ETHZ
- 13 companies involved with Materials Challenges initiative
- Materials Challenge 5 proposal being evaluated
- Call for Proposals ongoing for Materials Characterisation Facilities

CCMX Co-funded Professors: Update

The new multichamber sputter deposition system, purchased with the support of CCMX, will allow us to grow more complex materials for our studies of magnetoelectric properties and train students on a state-of-the-art thin film deposition system.
Pietro Gambardella, ETH Zurich

Pietro Gambardella is Full Professor of Magnetism and Interface Physics at ETH Zurich. His work focuses on the synthesis and characterisation of magnetic systems with novel structural and electronic properties; this includes magnetic memories and sensors.

In 2015, his group of 13 researchers, which includes four PhD students, studied current induced spin torques in magnetic thin films for the realisation of an all-electrical non-volatile memory, as well as the anisotropic magnetoresistance effects in bilayer metal systems and metal/insulator heterostructures. Gambardella's group fabricated materials by molecular beam epitaxy and magnetron sputtering of bilayer and trilayer metal films, including heavy metals, ferromagnets, and oxides. He also installed a multitarget sputter deposition tool with substrate heating and cooling capabilities, separate metal and oxide deposition chambers, hard mask and wedge deposition capabilities. Gambardella's ongoing collaboration continued with a major supplier of sputter deposition equipment, and he initiated a research project in partnership with a major company active in his field.

2015 was an extremely fruitful year with concrete achievements that could be leveraged for new IP and publications, as well as to successfully apply for funding. This additional support will enable me to grow my group further and to work on exciting ideas that could impact both fundamental and applied materials research. Fabien Sorin, EPFL

Fabien Sorin, Tenure Track Assistant Professor in the Materials Science Institute at EPFL, leads the Laboratory of Photonic Materials and Fibre Devices. His expertise in multi-materials components and his unique experimental perspective continued attracting industry interest in 2015. This generated research contracts or the submission of joint proposals.

Sorin's custom-built draw tower started producing advanced multimaterials fibers, enabling his group of five PhD students and two Post-docs to achieve several breakthroughs in the past year. A major achievement was winning an ERC Starting Grant with the project FLOWTONICS. This research will involve innovative approaches based on viscous flow engineering to fabricate complex photonic architectures and will allow Sorin to recruit three more PhD students and two other Post-docs. Sorin was also granted a CCMX Materials Challenge on advanced fiber-based solutions for controlled release in collaboration with René Rossi at Empa. Training is an important aspect of Sorin's mission; he has taught the introduction to Materials Science class to Bachelor students, co-organised the Advances in Materials seminar series class with Michele Ceriotti and supervised ten semester projects completed in his laboratory.

PHOTOS: TONY KUNZ

Machine-learning can provide deep understanding of the structure-property relations of materials, and predict reliably the formation energy of molecules with unprecedented accuracy. Michele Ceriotti, EPFL

2015 has been a year of development and consolidation for Michele Ceriotti, Tenure Track Assistant Professor and head of the EPFL Laboratory of Computational Science and Modelling. His group has reached a critical size with five PhD students and three Post-docs, and he has secured funding for starting new projects and hiring new collaborators.

Ceriotti has engaged in intense dissemination activities advertising his lab's applications and methodological work on atomistic simulation of complex compounds. Among his successes is an ERC Starting Grant that will begin in May 2016. In collaboration with colleagues at EPFL, Empa and PSI, Ceriotti prepared and submitted a CCMX Materials Challenge proposal that was approved to study the materials science of additive manufacturing; eight industry partners will be involved with this research programme. Ceriotti has also secured an agreement with a major metal processing company that will fund the research of one PhD student. Beyond his active involvement in the National Center of Competence in Research MARVEL, he co-organised the Advances in Materials seminar series class with Fabien Sorin. Ceriotti successfully organised a CCMX Technology Aperitif with excellent attendence by the materials modelling community who traveled from all over Switzerland to participate in the event.

Professor Eric Dufresne, ETH Zurich

Whether between industry and academic research or materials science and biology, Eric Dufresne, recently appointed Full Professor of Soft and Living Materials at ETH Zurich, finds work most interesting at the interfaces.

Take the first example. After starting a company based on a technology developed during his PhD at the University of Chicago, Dufresne went on to work for a well-known management consultancy. His work was business focused, and "very far removed from technology." It did not feel right.

"The reason I originally wanted to start a company was because I felt that all my research was so abstract that it had no connection to anything real," Dufresne said. "Then I went to the real world of finance and consulting and I did not like the pure money side of things. During my post-doc I came to appreciate industry as a source of really good scientific problems."


PHOTO: TOM KAWARA

Dufresne was appointed to the faculty of Yale in 2004 and continued to pursue academic research in parallel with industry collaborations, including partners in the fields of chemicals and personal care, investigating topics as diverse as the physical properties of skin and the stability of electronic inks.

"There has always been at least one person in my lab at any given time working with industry and I've always found that it has been a very rewarding relationship," he said. "Some of the most interesting advances that we've made in the lab have been inspired by these partnerships—I'm keen to develop such relationships here in Switzerland."

In terms of science, while Dufresne's research focuses on both pure engineering of soft materials—including polymers, foams, gels and colloids—and pure biology, most of the lab's work is at the interface between the two.

The soft materials currently used by engineers are all in thermodynamic equilibrium or kinetically arrested. The systems in living devices though are not even close to equilibrium—the materials themselves usually have embedded energy sources and control systems enabling them to respond to their environments and change their properties. While there is a wide body of research being done on so-called active materials that have embedded energy sources, Dufresne believes that the other element is critical to successfully integrating these materials into devices.

"The embedded control systems are what we really need—that's what biology has that engineered materials in the lab don't have," he said. "'Soft and living materials' doesn't necessarily mean materials used in biology then, it means materials with embedded sources of energy and control systems. I'm really inspired to make better soft materials and that means moving towards these kinds of living materials."

One example of the work he will continue to pursue has to do with the optical properties of materials. Within soft matter physics, one of the longest running challenges has been figuring out how to grow materials that have nanoscale structures that interact strongly with light. The field has only had a limited number of tools and mitigated successes, mostly with a narrow range of crystal structures and optical properties.

The animal kingdom however exhibits much more diversity in the choice of materials and in actual structures and properties. While soft materials produced on the engineering side have been limited to a few simple lattices, nature offers a much wider range of structures and symmetries. Dufresne's work has included systematically characterising all the structures that produce colour in the natural world and then trying to understand how they work. Now, the lab is looking at how to make such soft materials. Industrial applications could include more efficient optical devices, including solar cells. Another example of the lab's work involves research into mechanics of soft materials, from human cells and tissues to pressure sensitive adhesives.

Dufresne, who will start full time at ETH in July 2016, says his decision to come to Zurich was largely based on the opportunity to pursue this vision of soft and living materials.

"The resources at ETH are amazing, and this completely changes the way I frame my research," he said. "Being able to articulate this vague but powerful idea about living materials and moving my lab towards that over next 25 years is something that I could only pursue here."

Innovative Coatings Centre Primed for Develop

Above: J. Patscheider, K. Thorwarth and M. Trant pictured next to the newly installed Oerlikon Balzers Igenia Sp3 coater at Empa, Dübendorf. Above right: M. Trant and K. Thorwarth holding coated medical implant samples. Right: The Ingenia Sp3 coater's samples holder.

We can now develop new deposition processes with the reliability and reproducibility of industrial conditions.

Jörg Patscheider, Empa

CCMX's first Materials Challenge, the Coating Competence Center for Sputter Deposition by HiPIMS, has attracted additional industrial partners before even opening its doors.

The Center, to be inaugurated on 7 April, 2016 at Empa's campus in Dübendorf, is the only one in Switzerland to offer high-power impulse magnetron sputtering with Oerlikon Balzers' Ingenia Sp3 coater. The 3000 kilogram machine, which produces thin films based on an advanced type of magnetron sputtering, allows for the deposition of dense coatings onto substrates with temperatures as low as 200°Celsius.

It features a wider range of power density compared to standard HiPIMS, independent adjustment of pulse duration and power level, pulse length beyond 100 msec, ultra-fast arc detection and high process stability. Put this all together, and you have an optimal instrument for deposition at both industrial and research and development scales.

"We can now develop new deposition processes with the reliability and reproducibility of industrial conditions," said Jörg Patscheider, head of the Thin Films and Coatings group at Empa.

This helps explain the appeal to industry. CCMX's partners at the Coating Competence Center were introduced to a new partner, SmartFish, at the poster session of CCMX's 2014 annual meeting in Bern. The company has already received funding for a joint project that involves preparing piezoelectric coatings for applications in the mechanical watch market. The center is also discussing projects with companies in the Medtech field, which have expressed interest in, among other subjects, coating three-dimensionally structured ing Industrially-viable Deposition Processes

PHOTOS: NATHALIE JONGEN

Left: Closing the door of the Ingenia Sp3 coater. Above: K. Thorwarth at the back of the deposition chamber.

polymeric implants with titanium. The researchers have run preliminary experiments producing promising first results.

CCMX-sponsored HiPIMS activities will also be used in another CTI R&D project with Oerlikon Surface Solutions AG to develop hard amorphous carbon coatings. The machine's capabilities will be used to provide coatings with high hardness, fully dense material as well as very flat surfaces. The various practical experiments being run on the Ingenia will be supported by numerical simulation of coatings' properties to develop suggestions on how they could be tailored for optimal properties such as residual stress states. The work will start in 2016. Another potential project involves adapting already established processes for making silicon-carbide based coatings to protect high precision tools for glass moulding of miniature lenses.

Education is an essential element of each Materials Challenge. In 2015, the Coating Competence Center team arranged a two-day short course presented by Prof. Joe Greene from the University of Illinois, Linköping University and Taiwan Technological University on Thin Film Nucleation and Sputter deposition. Thirty-four participants, including 14 from industry, took the course. Future projects will include PhD students working with the Ingenia coater.

Empa will also offer high precision printing, additive manufacturing as well as UHV sputter deposition of conducting oxides in addition to the HiPIMS activities.

Industry Takes Hold of Additive Manufacturing

The idea of this Challenge is to start investigating the materials science problems related to the theory and practice of additive manufacturing.

Michele Ceriotti, EPFL

PHOTO: TONY KUNZ

M. Ceriotti presenting his research activities in the field of materials modelling.

"Additive manufacturing" (AM) is used to build 3D parts layer by layer out of metallic, ceramic or polymer powders. The techniques involve depositing a coat of powder directly onto a substrate, melting it selectively and allowing it to cool and consolidate—the process is then repeated until the part is complete.

Advantages over traditional methods include the ability to create complex geometries; to mix materials during the layering process to produce structures with varying functions; to make new composite microstructures and maybe even new material classes. It is not straightforward though.

High heating and cooling rates lead to harder, less ductile microstructures with high residual stresses and residual porosity, affecting both function and aesthetics. The processing conditions are so different from those that have shaped standard approaches that researchers will need to develop new design principles. This will allow them to produce parts with specific mechanical and aesthetic properties and which meet high-quality specifications. The team behind CCMX Materials Challenge "Additive Manufacturing and Metallic Microstructures" wants to address these needs.

"From the materials science point of view, additive manufacturing presents a number of challenges in terms of both experiments and modelling due to the fact that this is effectively a very fast quenching process, very strongly out of equilibrium," said Michele Ceriotti, head of the Laboratory of Computational Science and Modelling at EPFL. "The idea of this Challenge is to start investigating the materials science problems related to the theory and practice of additive manufacturing."

The team—which includes Roland Logé, head of EPFL's Laboratory of Thermomechanical Metallurgy, Christian Leinenbach, head of Alloy Design and Processing Technologies at Empa and Helena van Swygenhoven at the Paul Scherrer Institute, as well as a number of industrial partners, will look at the fundamental materials science problems underlying the transition from traditional manufacturing techniques to additive manufacturing, focussing on metallic alloys and the correlation between process, microstructure and properties.

PHOTO: ALAIN HERZOG

H. van Swygenhoven, C. Leinenbach, M. Ceriotti and R. Logé preparing for the launch of their Materials Challenge.

Industry knows the additive manufacturing technology has now reached a stage where it can no longer be ignored.

Roland Logé, Empa

The researchers will tackle the problem from multiple angles, including optimising alloys and part production by additive manufacturing, multi-scale modelling of the out-of-equilibrium processes involved in the technique, *in-situ* and *ex-situ* characterisation of the microstructure, and thermo-mechanical treatments that may be used to enhance the properties of the material. They will look at the microscopic processes that control the formation of defects, the development of alloys specifically designed for additive manufacturing processing, the characterisation of the microstructure that arises because of the peculiar aspects of AM as well as the microstructural changes due to the thermal and mechanical fields, and the resulting properties.

The aim, ultimately, is to design alloys optimised for the process.

"Right now there are very few alloys that are processed by additive manufacturing, and in most cases these are alloys that have been tuned and optimised and designed for traditional manufacturing processes," said Ceriotti. "The idea would be to develop the insight that would allow us to design alloys specifically for additive manufacturing applications rather than using materials that have been around for quite some time and have been optimised having in mind the traditional way of making objects."

The work will initially focus on three classes of materials: solution-strengthened, precipitation-strengthened and two-phase alloys. Preliminary discussions with industrial partners suggest that the initial focus may be on types of stainless steel, red gold or other precious metals for watch manufacturers, and on nickel-based superalloys and titanium alloys for partners involved in heavy industry.

The researchers want to first carry out an extensive survey of materials' state of the art, discuss technological goals of partners, extensively test chosen materials and finally use this insight to design powders or manufacturing strategies that improve the properties of the final product. The work will also involve five doctoral students and specialised courses.

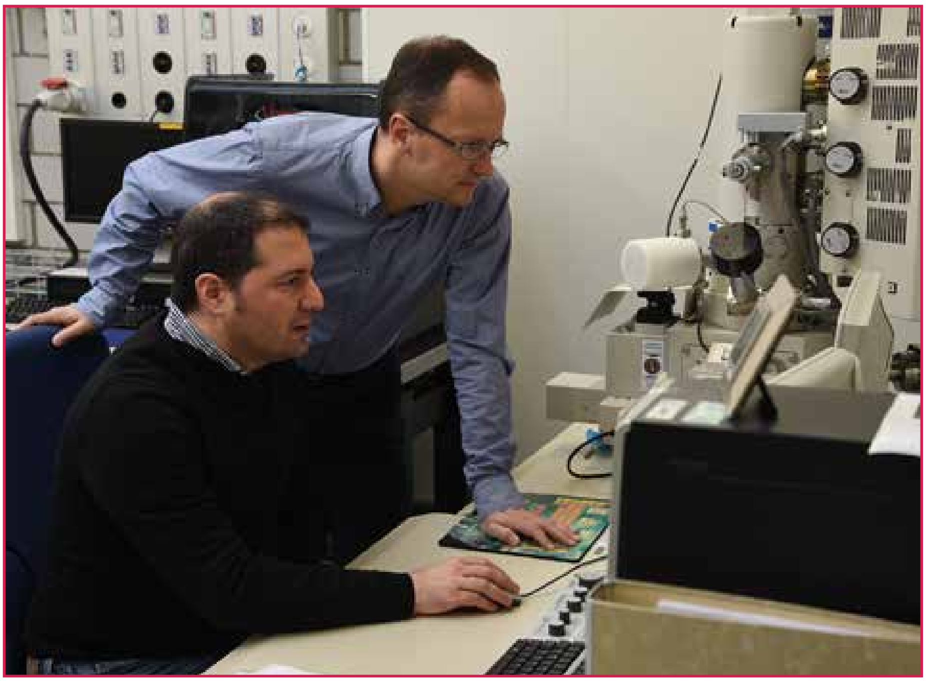
The interest in the topic is not just academic. This Materials Challenge involves eight industrial partners, covering the whole spectrum of metallurgical applications, from heavy-duty applications to micromechanical parts. The team has included powder supplier Oerlikon Metco to simplify investigation of new compositions, as well as to be able to control characteristics of the powders more precisely.

Companies may have been hesitant to invest in the technology because of several issues. The project will investigate some of the issues related to materials science, Logé said, while others are likely to be solved elsewhere within the next five years.

"By the end of 2020, there should be a good knowledge platform for all partners, from which we could envision future investments," he said. "Industry knows the additive manufacturing technology has now reached a stage where it can no longer be ignored."

Self-Care Materials Designed to Adapt and Rea

Illustration of polymeric optical fibre flexibility.



R. Rossi and F. Sparzio observing microscopy measurements of polymeric optical fibres.

PHOTOS: COURTESY EMPA

We really want to control the materials so that we can have delivery on demand. That's why we call the Challenge self-care materials—the materials should be able to react autonomously depending on changing conditions in the environment.

René Rossi, Empa

Fibre-shaped substances have large surface areas compared to their mass—this gives them considerable scope for interacting with the surrounding environment.

René Rossi, head of the Laboratory for Protection and Physiology at Empa, and Fabien Sorin, head of the Laboratory of Photonic Materials and Fibre Devices at EPFL—the principal investigators behind the CCMX Materials Challenge "Self-care Materials"—hope to exploit this property to develop fibre-based controlled substance delivery systems for use in fields as diverse as health care, textiles and packaging.

That is, teaming fibre-based systems with so-called smart polymers that react to external triggers such as temperature, pH, humidity or pressure could provide delivery on demand. This may result in items such as packages that can release antioxidants to preserve food longer; textiles that deliver drugs through the skin at specific locations, at a specific rate and specific time; in-body structures that can release anti-inflammatory drugs, or even fibrous tissues that can generate growth factors.

"When we look at the literature, there are a lot of polymeric delivery systems, but they're usually capsules or maybe some gels, and not many of them have really constituted a breakthrough in industry," Rossi said. "We think this is maybe due to a large part to processing problems. We have developed advanced fibre technologies and would like to use a different approach."

Today's delivery solutions are generally built around small passive capsules that release substances as they degrade or diffuse. There is little control over how the substances spread, where the delivery occurs, or at what time and rate. What's more, the systems are difficult to activate, there is limited scope for tuning different properties during processing, and they are not particularly well suited to the new types of surfaces with which they need to interact, notably in the fields of health care. Alternative processing approaches could help solve these problems.

The technologies in question involve Rossi's field of solution spinning and Sorin's field of multi-material thermal drawing. Solution spinning involves producing fibres by dissolving polymers in solvents and extruding them

ct to Changing Environments

PHOTOS: NATHALIE JONGEN

Y. Qu and F. Sorin examine multi-material fibres.

Standing next to the multi-material thermal tower.

inside coagulation baths containing non-solvents or into a heated chamber of air, depending on the type of spinning. It also includes the use of electrospinning technology enabling the generation of very small fibres with drug release abilities.

Fairly recent developments mean that the techniques can now be used to make hybrid structures that have controlled geometries that can impact the functionality of the material.

Sorin's "multi-material" fibre drawing technique refers to integrating metals, semiconductor materials, insulator materials, functional polymers or polymer nanocomposites with various properties into fibres using preform-based processing methods. Researchers can integrate multiple functional components into one fibre or assemble large-scale two- and three-dimensional geometric constructs made of many fibres.

These two approaches—scalable, low cost, and giving researchers the ability to tailor micro and molecular structures with high precision—may lead to a new class of multi-material micro-structured fibres integrating innovative biodegradable polymer architectures. Fibres have the additional advantage that they can be integrated into a variety of supports such as textiles, scaffold, tissues and even thin packaging films.

The team will investigate the interplay of viscosity, adhesion, surface tension and feature sizes, all of which are essential to achieving the right fibre performance and architecture. They will also look at how fibres interact with and behave in their environments through advanced characterisation techniques.

"There have been a lot of improvements in the way fibres are processed and in the types of materials, architectures and geometries that we can integrate inside them to have both active and passive control of delivery," Sorin said. "We want to leverage these new developments we've both been leading in our research areas for the field of controlled delivery."

The researchers say one important aspect of their work is simply bringing more materials science to the field of delivery. Until now, it has been dominated by pharmaceutical and medical research, that is, by biologists whose expertise does not lie in the materials science behind drug delivery or processing. Various elements of materials science can be used to integrate complex functionalities into the resulting systems and such expertise is also needed to investigate, for example, how electrospinning or thermal drawing processes affect the morphology and structure of the polymer fibre produced, and how these parameters affect in turn the kinetics of, say, drug release.

"We really want to control the materials so that we can have delivery on demand," Rossi said. "That's why we call the Challenge self-care materials—the materials should be able to react autonomously depending on changing conditions in the environment."

A number of industrial partners from the fields of health care, chemicals and textiles have joined the project, as have collaborators from the broader ETH domain. The consortium is a good indicator of the broadness of the interest in this field, which could develop products that monitor and release substances, preserve products or treat individuals.

Empa's Upgraded QuadProbe Enables Cross-Cor

PHOTO: COURTESY EMPA

Inside the QuadProbe instrument.

PHOTOS NATHALIE JONGEN

X. Maeder and J. Ast preparing the Raman spectrometer unit.

Researchers are able to get fast chemical and structural information at different length scales, all in one big instrument. Everything is integrated into one unique tool for the first time.

Johann Michler, Empa

The scanning electron microscope is the Swiss Army knife of materials science, the most used tool for analysing microstructures and the composition of samples. Scientists at Empa have figured out how to upgrade it with even more extensive capabilities.

Many methods of chemical and micro-structural analysis direct a beam of particles or radiation towards a sample to induce a response that is monitored by various detectors. The QuadProbe FIB instrument integrates ion, electron, x-ray and laser beams into a single, multi-purpose analytical platform. Combining these different probe beams and an array of detectors into a single instrument is set to improve imaging and the chemical characterisation of nano- and micro-scaled materials, offering advantages to researchers in a variety of fields.

"Researchers are able to get fast chemical and structural information at different length scales, all in one big instrument," said Johann Michler, head of the Mechanics of Materials & Nanostructures lab at Empa. "Everything is integrated into one unique tool for the first time."

The instrument is able to supply a range of data because of the different length scales on which the four beams operate as well as the various types of detectors that have been integrated into the machine. The QuadProbe's beams include X-rays, light, ion beams, and electron beams, which have effective spatial resolutions of about 50 microns, 1 micron, 50 nanometres and 1 nanometre, respectively. It incorporates a number of detectors, including an energy-dispersive and wavelength dispersive X-ray spectrometer, a secondary ion mass spectrometer as well as a Raman spectrometer and a camera for electron diffraction. A significant amount of sample information can be collected, including the major elemental composition, trace analysis, organic content, and crystal phase orientation mapping.

"It's easy to see the interest of a materials scientist who wants to maybe look at the composition of a material on a 50 micrometre length scale but then zoom down in different steps to examine, for instance, the crystal orientation of grain at the nanoscale," Michler said.

relation Analytics

J. Ast and X. Maeder using the QuadProbe FIB instrument, a multi-purpose analytical platform.

The tool has been under development for some time, with various techniques being added along the way with the help of large instrument companies and Swiss start-ups that provided detector technology. CCMX funding allowed the scientists to turn it into a real analytical platform by adding a range of additional detectors.

The instrument's analytical abilities have already drawn the attention of industrial users, who are attracted by the chance to investigate several different aspects of a sample at once. As Michler puts it, people from industry generally bring their problems to an analytical scientist, who applies his or her technique of expertise to try to solve it. The problem is not however fundamentally linked to a single particular technique. With the QuadProbe FIB, users can put the sample in and get information on different length scales and on different sensitivities, measuring for instance, parts per million of toxic cadmium or investigate on the nanoscale whether there is some organic impurity.

The instrument has been used to investigate hard coatings, which are produced by a number of companies in Switzerland. A certain deposition process results in droplets being integrated into the hard coating, resulting in micro-sized features. The researchers were able to determine the nature of these droplets by combining backscattered electron imaging with focused ion beam secondary ion mass spectrometry and superimposing the two types of images. The instrument has also been used in applications involving thin film multilayers, stainless steel coatings and the manufacturing of watch components.

"What's special here with all these industrial examples is that everything uses at least two techniques," Michler said. "Companies come to us if they need this sort of combination, which is not available in any other instrument. The special thing here is really this cross-correlation."

The instrument's analytical abilities have already drawn the attention of industrial users, who are attracted by the chance to investigate several different aspects of a sample at once.

ETH Zurich Inaugurates Switzerland's First Pla

PHOTOS: COURTESY ETH ZURICH

Switzerland's first plasma FIB-scanning electron microscope at ETH Zurich.

We always had people wanting to do larger things with the FIB and we had to either tell them it wouldn't work or that it would cost them a lot of time. We had a real need and we were lucky to be the first ones to get it in the end.

Joakim Reuteler, ETH Zurich

Focused ion beam systems can be used to analyse, modify or create structures in materials ranging from metals and ceramics to tissue from the brain and bone. While conventional FIB instruments relying on liquid-metal ion sources can investigate a wide range of materials, they face limitations when analysing larger samples or creating bigger structures.

That is, when sample dimensions reach a linear size of hundreds of micrometres, such instruments must run at the limits of their capacities for days. This is linked to a high risk of failure, requires that the whole instrument be exceptionally stable, and is too expensive in terms of cost and resources. On 16 June, the Scientific Center for Optical and Electron Microscopy at ETH Zurich, or ScopeM, inaugurated Switzerland's first plasma FIB-scanning electron microscope. It will allow researchers to do the same work within hours.

"We always had this trouble of people wanting to do larger things with the FIB and we had to either just tell them it wouldn't work or that it would cost them a lot of time," said Joakim Reuteler, a scientist at ScopeM. "We had a real need and we were lucky to be the first ones to get it in the end."

The instrument, funded in part by CCMX, is based on a new type of ion source. A microwave signal transfers energy to the electrons within the atoms of a dilute gas to generate a cold plasma ball. This allows for a virtual source size of about 50 µm from which focused ion beams with a current from pA to µA range can be created. The respective spot size ranges from sub micrometre for the small currents to 25 µm for the largest current of 2 µA. Xenon (Xe) is now the preferred ion species for plasma FIBs because it achieves the highest emission brightness. The sputter yield of Xe ions on crystalline silicon is about 30% larger than that of gallium, even though it is less destructive to the sample when

sma FIB-SEM

Presentations and demonstrations during the new instrument's inaugural event, June 2015.

milling at the same ion energy because the penetration depth is smaller. Though such instruments are well known in the semiconductor industry, Reuteler says ETH Zurich is an "early bird" in academia, with very few academic institutions in the world having such a machine.

The plasma FIB-SEM fills a critical gap in terms of machining accuracy and speed between conventional liquid-metal ion FIB milling and pulsed laser machining, which faces technical limitations that make it most suitable for length scales of tens of micrometres. Using chemically inert xenon rather than gallium as an ion source allows researchers to work the sample without forming intermetallic phases, grain boundary decoration or doping semiconductors. The plasma FIB system is currently the only technology that allows scientists to expose and polish an artefact-free, sub-micrometre flat cross section area of several 100 µm x 100 µm at a specific region of interest within an hour.

"The plasma FIB is the only technology that fills this gap, so in this sense it's the best technology," Reuteler said.

Reuteler has been working with colleagues from the Swiss Light Source at the Paul Scherrer Institute to test the machine, milling large sample pieces. The plasma FIB has been installed since the end of 2014, and the number of trained users is rising steadily. Though the machine is highly automated—loading the sample is simply a matter of pushing the load button, Reuteler says—it will still take time for users to use the system optimally in their applications.

"The trickier part is figuring out which currents, which scanning modes are most appropriate to achieve the desired sample prep or investigation," he said. "It always takes some work to figure out how to use a new technique best."

The plasma FIB system is currently the only technology that allows scientists to expose and polish an artefact-free, sub-micrometre flat cross section area of several 100 µm x 100 µm at a specific region of interest within an hour.

Neutron Microscope Getting Higher Resolution

PHOTOS: COURTESY PSI

E. Lehmann and P. Trtik with the neutron microscope prototype.

This improvement would be a boon to materials science fields ranging from electrochemistry, materials for nuclear safety, soft matter, and soil physics as well as to the life sciences in the imaging of various biological systems.

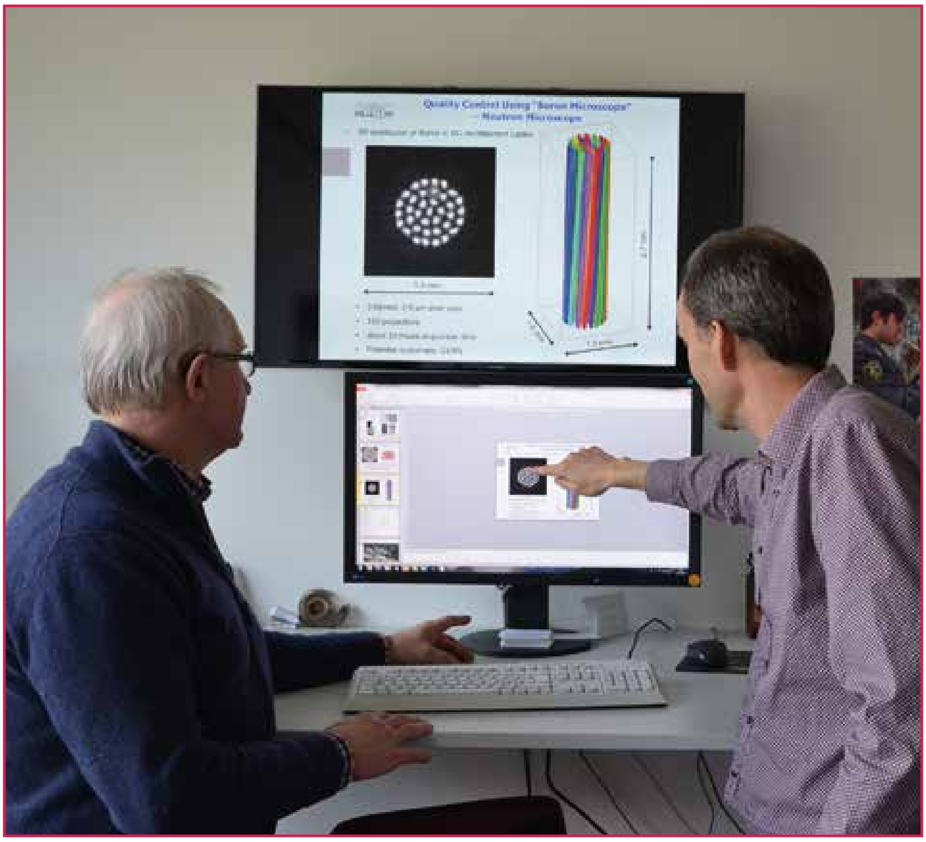
When Pavel Trtik was studying the distribution of water in concrete, he wished the instrument he was using allowed him to examine the phenomenon more closely through higher spatial resolution. Now, he is working on a project to improve resolution by more than a factor of four, to below five microns from 20 to 30 now.

This improvement would be a boon to materials science fields ranging from electrochemistry, materials for nuclear safety, soft matter, and soil physics as well as to the life sciences in the imaging of various biological systems.

The team behind the project, led by Eberhard Lehmann at the Paul Scherrer Institute, has built a prototype, already improving resolution significantly to about 7.6 microns. While quite an accomplishment itself, the next step, of increasing resolution to five microns or below, will be considerably more difficult. "What we have done is really a very nice step," he said. "We still have work to do though."

In order to reach this goal, the team will have to ensure that the performance of all the individual components of the microscope is optimised. This includes tailored magnifying optics, a high-resolution, high-performance neutron-sensitive scintillator, a high-resolution, high-sensitivity camera and neutron-sensitive spatial-resolution test objects and high-precision sample positioning stages.

None of this is particularly easy. The high spatial resolution requires, for instance, a thin scintillator, or material that luminesces when excited by neutrons. The scintillators required are not produced commercially—the team had to develop their own. The prototype uses a 4-micron- thin scintillator based on



Discussing quality control.

Standing next to the ICON beamline.

gadolinium oxysulfide. Making it thinner may seem the way to improve resolution, but the thinner a scintillator is, the less effectively it captures neutrons. So, the researchers had to find a way of keeping the device thin without sacrificing its capture efficiency.

The solution lies in isotopically enriched scintillator screens that allowed the scientists to produce even thinner scintillators or to make ones of the same thickness even more efficient. The team has recently accomplished this pioneering breakthrough, demonstrating the feasibility of production of such very thin isotopically enriched scintillators and showing that about four times more light output can be expected from them. They continue testing the devices and will soon publish their results.

"This is likely to bring us below five microns, so in principle, it's mission accomplished, at least for the primary goal of the project," Trtik said. "That said, there are a number of things that one can still improve."

These days, one of the main points limiting neutron imaging is available flux. For example, the world's strongest neutron source—the European Spallation Source (ESS)—now being built in Lund, Sweden, is set to deliver about the same number of neutrons as photons that are produced by a 60 watt lightbulb. That is, it is not a particularly strong source of light. Making images higher and higher in resolution, with smaller and smaller pixels, means having fewer neutrons available per given period of time. "At some point, you simply run out of neutrons," Trtik said.

The researchers are therefore working to optimise the set-up, looking to count every single neutron that comes from a good direction from the sample and, from that captured neutron, counting as many photons as possible. Part of this work involves a sub-project focused on developing specially engineered microstructured scintillators. The scientists are also considering using alternatives to the neutron source they are currently using, the ICON beamline at PSI.

Though the team plans to make the prototype more widely available to external users at the end of this year, the microscope has already attracted interest from other research groups, and some experiments have been run.

"There are prospective users who are interested in the microscope, and who are going to be using it to address their scientific or engineering issues," Trtik said.

CCMX Research Activity: Update

Nanoscreen: Understanding Nanosafety

First launched in October 2014, NanoScreen brought together partners from Empa, EPFL, Cetics Healthcare Technologies, Midatech and the Swiss Federal Office of Public Health to develop a more in-depth understanding of the interaction between nanoparticles and living systems. The aim was to identify potential side-effects for human health and quantifying these effects using *in vitro* methods.

Just a bit more than a year later, the group has already been joined by two more partners and has substantially increased funding.

"The level of interest demonstrates the urgency for a proper understanding of the nanomaterial-cell interactions," said Peter Wick, head of Empa's Laboratory for Particles-Biology Interactions. "A precise understanding of the correlation between the physical-chemical properties of the nanomaterials and their biological responses would enable an acceleration of the assessment of nanomaterials beyond the case-by-case approach."

Solid engineered nanoparticles are used in more and more products because they offer unique characteristics. While these exceptional properties offer potential benefits, they may also affect humans and the environment in unexpected ways. Although there are studies on the biological effects of engineered nanoparticles, the available results offer limited comparability and are often inconclusive or even contradictory.

NanoScreen, which now also includes the Computational Structural Biology group of Torsten Schwede at the University of Basel and the Korea Research Institute of Standards and Science, wants among other things to fill this gap by elaborating fundamentals for standardised methods. This will improve physico-chemical as well as biological characterisation of engineered nanoparticles in a robust, reliable and comparable way.

In addition to recruiting new partners, the group has also made solid progress in the research itself. NanoScreen management has decided to establish two libraries of engineered nanoparticles: one of silica nanoparticles of different sizes and porosities, and the other of differently sized gold nanoparticles with industrially relevant surface modifications. The particle libraries will be assessed in terms of biological response and interference with biological assays by Empa, in terms of dosimetry by EPFL and in terms of protein adsorption by the University of Basel. The researchers will then try to match the physico-chemical parameters of the engineered nanoparticles to their biological effects and evaluate potential new biomarkers together with appropriate measurement technologies for further characterisation. The end result should be a solid understanding of how engineered nanoparticles influence living systems. The approach will hopefully be acceptable by regulatory bodies and, in turn, useful to Swiss industry.

PHOTO COURTESY EMPA

S. May and P. Wick checking biological assays with optical microscopy.

EPFL's successful approach to synthesising different types of silica and gold particles and a technical meeting with the industrial partner Midatech—a company that designs, synthesises and manufactures biocompatible gold nanoparticles—mean that the first particle types have already been made available to all partners. A second set will be available soon. While biological tests with the nanoparticle libraries started towards the end of this year, Empa has already applied the methodology to running projects and was able to collect further details on the biological characterisation of engineered nanomaterials.

Nanoparticles are more challenging to study than bulk materials or classical chemicals because the engineered nanoparticle properties being evaluated may interact with assays or living systems in an unforeseen way. If the experimental design does not account for this, false positives of negatives may result. Empa is continuing to work on making the assays used reliable, reproducible and robust.

Though the partners have been focusing mainly on occupational health aspects, they would like to expand research activities to work with clinically relevant particles such as drug delivery systems and particles relevant to diagnostics. The first organ these types of particles are likely to come into contact with is blood, and the partners would like to develop relevant assays to address their specific needs and research questions.

"Establishing a blood compatibility assay would allow us to identify what happens when particles enter the blood stream," said Cordula Hirsch, NanoScreen project coordinator.

NanoScreen, the first programme funded under CCMX's Materials Challenges initiative, has also been active in education. Last summer the partners organised a workshop on "Cause and Effect Analysis: A new approach for developing robust nano-bio assays." This year's Winter School on "Nanoparticles: From Fundamentals to Applications in Life Sciences," co-organised with CCMX, was not only well-attended—it was oversubscribed.

CCMX Research Activity: Project Update

Dental Enamel Regeneration Improved by Stabilising EMD Proteins

PHOTOS NATHALIE JONGEN

Above: P. Heunemann and B. Simona at ETH Zurich preparing Quartz Crystal Microbalance.
Right: A. Apicella, M. Marascio and V. Colangelo during a TEM session in CIME, EPFL.

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

Straumann, Switzerland's dental implant maker, sells a product that can help tissue regenerate 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. Though the regeneration process is promoted by physiological conditions, neither the interactions between EMD, a mix of about 90% amelogenin and other proteins, and PGA, nor the product's mechanism of action, have been completely understood.

What has been clear is that altering the environment, pH or temperature can affect the shape of the EMD proteins and, in turn, have an impact on their stability and effectiveness. Researchers at EPFL and ETH Zurich have not only better characterised the protein over the course of CCMX Project "Structural evolution and rheological properties of carrier / protein complexes" they have also figured out a possible way of making EMD and the EMD/gel complex less susceptible to such changes.

"We managed to simulate the Emdogain molecule for the first time as far as we know, and this is the first step to understanding the more complex industrial systems," said Christopher Plummer, a senior researcher in EPFL's Lab of Composite and Polymer Technology. "We also now have strategies for improving the lifetime stability of the existing product, and that's what the project originally aimed at doing."

The researchers have found that incorporating arginine, a protein stabilizer that is well known but which has not to their knowledge been used for this purpose, may indeed stabilize EMD against irreversible aggregation with respect to variations in pH and temperature.

In a paper published in *Plos One*, the research team described how it analysed representative EMD-buffer solutions with and without arginine through 3D-dynamic light scattering, UV-Vis spectroscopy, transmission electron microscopy and Fourier transform infrared spectroscopy at different acidic pH and temperatures in order to simulate the effect of pH variations and thermal stress during manufacture and storage. The results suggested that arginine may improve the stability of EMD-products for the treatment of periodontal defects during manufacture and storage, without affecting the ability of current EMD-carrier systems to deliver the product to the site of the defect.

The project, in addition to the publication cited above, the first in a series currently in submission, resulted in intellectual property related to a similar system that can be applied in a slightly different way, potentially increasing the scope of medical applications.

"There are other things Straumann will develop, but I can't talk about them in any detail," Plummer said. "Globally, we are very satisfied with what we have achieved."

We managed to simulate the Emdogain molecule for the first time as far as we know, and this is the first step to understanding the more complex industrial systems.

Christopher Plummer, EPFL

CCMX Research Activity: Project Updates

NanoAir: A New Tool for Quantifying Nanoparticle Size, Distribution and Composition

With nanoparticles being integrated into an increasingly wide range of products, it is more and more important to evaluate their possible impact on humans, particularly through exposure by aerosol spray or incineration. Ideally, we should be able to monitor what is being released, both in terms of particle size distribution and chemical composition, and that in real time.

Researchers at Empa and the Paul Scherrer Institute have developed an instrument that can do just that.

While most of available techniques are offline methods and/or cannot provide physical and chemical information simultaneously, the combination of a Rotating Disk Diluter (RDD), a Scanning Mobility Particle Sizer (SMPS), and an Inductively Coupled Plasma Mass Spectrometer (ICPMS), RDD-SMPS-ICPMS, provides the information needed after scans of a few minutes in duration. The SMPS determines the size distribution of the airborne particles while the ICPMS allows the measurement of the content of specific elements. The RDD serves as a sampling interface, enabling the researchers to examine the aerosol as it is released and independently of its flow rate.

In an initial paper, the researchers including PhD student Adrian Hess and PSI post-doc Mohamed Tarik, tested the setup with a model aerosol containing airborne silver nanoparticles. The device successfully scanned particles in sizes ranging from 7 to 156 nanometres within 120 seconds, achieving similar sensitivities and efficiencies compared to state-of-the-art SMPS and ICPMS instruments. Another paper published by the team demonstrated the instrument's capabilities in more detail, giving the results of analysing uniformly composited metal aerosols, as well as an aerosol mixture containing smaller gold and larger silver nanoparticles.

In this latest study, the size-resolved ICPMS intensity curves correlated very well to the size distributions determined by the coupled SMPS as well as to those from a reference SMPS instrument. Particulate iron, gold and silver were successfully detected, and nanoparticles of different size and composition were distinguished. The researchers also took the opportunity to determine sensitivity and limits of detection related to particle number and mass concentrations for several elements and particle diameters.

PHOTO NATHALIE JONGEN

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

"The RDD-SMPS-ICPMS not only provided intensity curves, we could also directly relate the signals to element concentrations in the different particle size fractions," Hess said. "We learned to judge the quality of our signals and its noise."

The instrument has already been used to monitor aerosol particles from consumer spray products. As an example, the researchers have investigated and characterised tin nanoparticles emitted by a dirt repellent consumer pump spray product.

The spray bottle was placed in a glove box, where the spray experiments were performed in a defined and reproducible manner. The data showed that the tin content in the particles remained unchanged and that alterations in concentration and size distribution were caused by particle agglomeration rather than by particle loss and growth linked to condensation of volatile matter in the atmosphere. These results showed that the technique is able to dynamically observe nanoparticle behaviour with respect to both size changes and elemental composition.

The team has submitted another publication on the combustion of differently impregnated wood samples. "This application is just the beginning though," Hess said.

"Next steps involve characterising emissions from thermal processes which are closer to energy-related real applications," Hess said. "We also want to get the setup mobile in order to measure particles in real process gases or combustion emissions."

Other potential applications include the assessment of other consumer products containing engineered nanoparticles (ENP), research on metals in engine exhaust aerosols or monitoring of waste incineration emissions.

The instrument has already been used to monitor aerosol particles from consumer spray products.

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Research Activities Ongoing in 2015

PROJECT	PRINCIPAL INVESTIGATOR (PI)	PI'S INSTITUTION	OTHER INSTITUTIONS	ERU/PLATFORM
Liquid repellent wear resistant coatings (LIRE-WERE-CO)	Patrik Hoffmann	Empa	EPFL	SPERU
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
Ultrasensitive sensing transducer based on Fano interferences in plasmonic metamaterials—FANOSENSE	Olivier Martin	EPFL	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
QuadProbe FIB—an analytical FIBSEM microscope integrating ion, electron, X-ray, and laser beams for microanalysis in materials research	Johann Michler	Empa	-	NMMC
Neutron Microscope	Eberhard Lehmann	PSI	-	NMMC
Inductively coupled Plasma Focused Ion Beam (PFIB) system at ETH Zurich	Roger Wepf	ETH Zurich	-	NMMC

MATERIALS CHALLENGE	PRINCIPAL INVESTIGATOR (PI)	PI'S INSTITUTION	OTHER INSTITUTIONS
Coatings Competence Center for Sputter Deposition by HiPIMS	Jörg Patscheider	Empa	-
NanoScreen: Reliable and rapid *in vitro* safety assessment of nanomaterials	Peter Wick	Empa	EPFL, UniBasel

Research Activities Starting in 2016

MATERIALS CHALLENGE	PRINCIPAL INVESTIGATOR (PI)	PI'S INSTITUTION	OTHER INSTITUTIONS
Additive Manufacturing and Metallic Microstructures (AM^3)	Roland Logé, Michele Ceriotti	EPFL	Empa, PSI
Self-Care Materials	René Rossi, Fabien Sorin	Empa, EPFL	

2015 Peer Reviewed Publications [continued]

R. Gutzler, S. Stepanow, D. Grumelli, M. Lingenfelder, K. Kern, **Mimicking Enzymatic Active Sites on Surfaces for Energy Conversion Chemistry,** *Accounts of Chemical Research,* 48 (2015) 2132–2139.

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Fabien Sorin

M.A. Schmidt, A. Argyros and F. Sorin, **Hybrid Optical Fibers: An Innovative Platform for In-Fiber Photonic Devices,** *Advanced Optical Materials,* 4 (2016), 13–36.

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2015 Annual Activity Report Production

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Photos: Alain Herzog, Nathalie Jongen, Tom Kawara, Tony Kunz, Empa, ETH Zurich, PSI
Design & Layout: Sue Niewiarowski
Printing: Repro EPFL

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