

MaP Graduate Symposium

featuring the MaP Award 2015

The 10th Annual Gathering of Materials & Processes Researchers at ETH Zürich

Wednesday, June 3, 2015 at 9 a.m.

ETH Zürich, HG E5 and E-floor main hall

Rämistrasse 101, 8092 Zürich

Symposium Booklet

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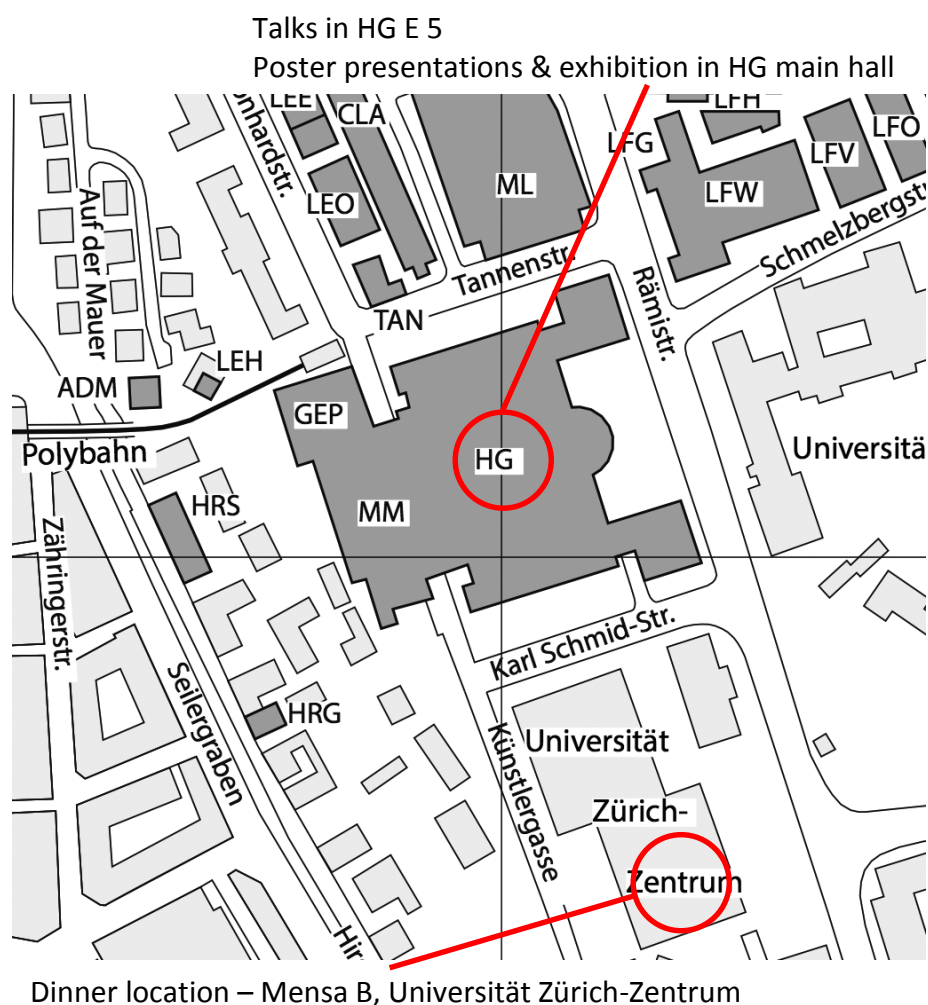


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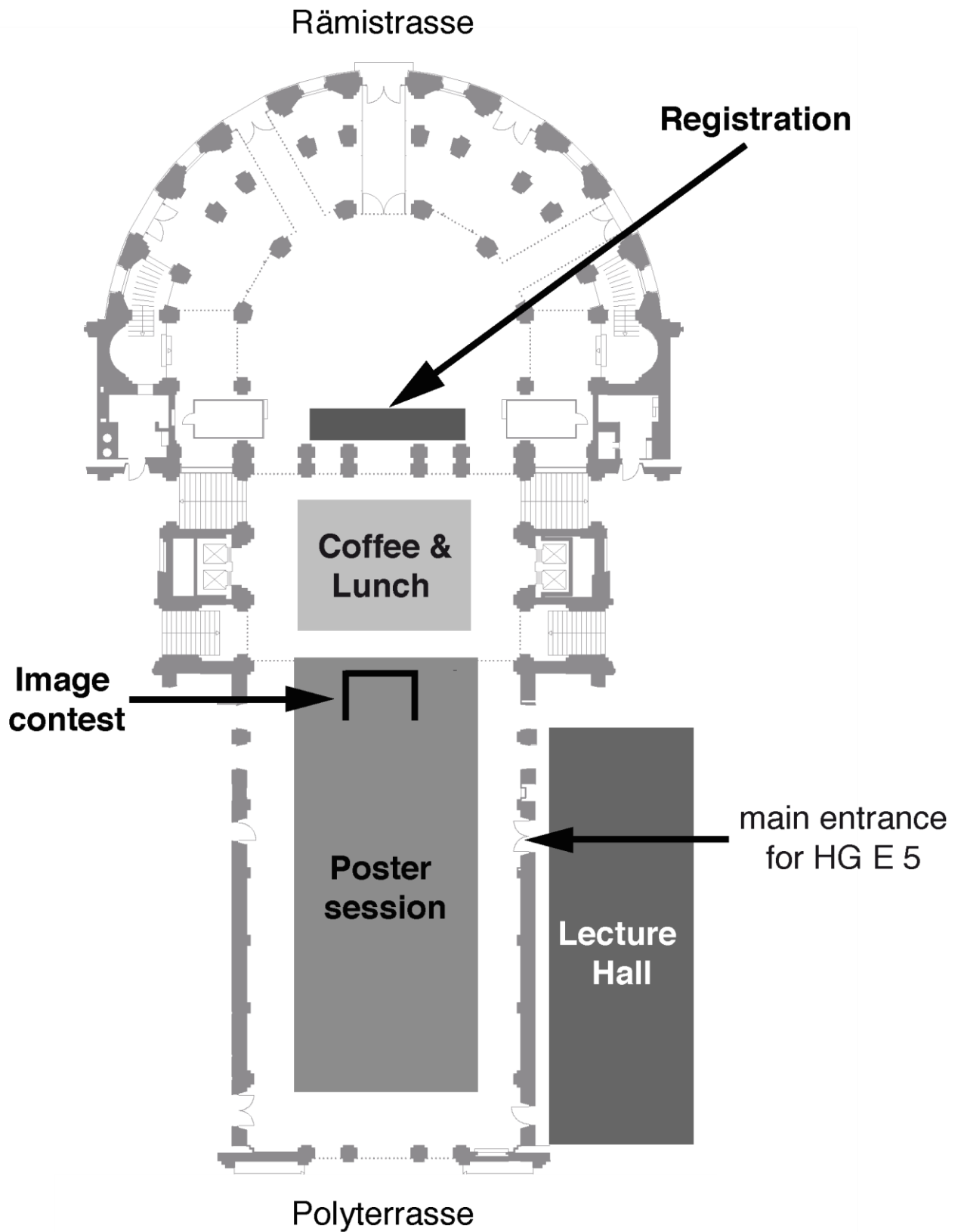
SITE MAP ETH ZENTRUM



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HG E-floor



PROGRAM

08:00	Registration
08:50	Prof. André Studart, MaP Director Opening remarks
09:00	Gregor Hofer, Polymer Chemistry, D-MATL Intriguing Structural Features and Properties of a Novel Two-Dimensional Polymer
09:15	Elia Schneider, Functional Materials Laboratory, D-CHAB Magnetic superbasic proton sponges are readily removed and permit direct product isolation.
09:30	Susanna Früh, Applied Mechanobiology, D-HEST Molecular Architecture of Native Fibronectin Fibrils
Live Demo by Eve Loiseau, Complex Materials, D-MATL	
09:50	Andreas Lamprecht, Mechanics & Experimental Dynamics, D-MAVT Direct 3D Measurements of Acoustic Forces within Microfluidic Channels and Chambers
10:05	Jana Segmehl, Wood Materials Science, D-BAUG Chemical Delignification to Tailor Wood-Derived Bio-Scaffolds for Multifunctional Materials
10:20	<i>Coffee Break & Poster Session</i>
11:00	Dr. Rupali Deshmukh, Multifunctional Materials, D-MATL Assembly of Crystalline Ultrasmall Copper Nitride Particles into Freestanding 3D Aerogel
11:15	Jan-Georg Rosenboom, Chemical Reaction & Separation Engineering, D-CHAB Ring-Opening Synthesis of Polyethylene Furanoate (PEF) as a 100% Renewable Resource-Based Substitute for Polyethylene Terephthalate (PET)
Live Demo by Andreas Frutiger, Biosensors & Bioelectronics, D-ITET	
11:35	Dr. Renata Negrini, Food & Soft Materials, D-HEST pH-responsive Lyotropic Liquid Crystals and Their Potential Therapeutic Role in Cancer Treatment
11:50	Francesco Robotti, Thermodynamics in Emerging Technologies, D-MAVT Guided Assembly Biolithography: Shaping Bacterial Cellulose
12:05	<i>Lunch & Poster Session</i>

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13:30	Dr. Sinead Griffin, Materials Theory, D-MATL From the Early Universe to the Hubbard Hamiltonian in the Hexagonal Manganite
13:50	Dr. Deniz Bozyigit, Nanoelectronics, D-ITET Understanding Performance Limitations of Semiconductor Nanocrystal-Based Optoelectronic Devices
14:10	Dr. Alexandre Larmagnac, Biosensors & Bioelectronics, D-ITET Novel Neuroprosthetic Devices for Spinal Cord Stimulation in Paralyzed Animal
14:30	Dr. Martin Süess, Nanometallurgy, D-MATL Highly Strained Si and Ge Micro- and Nanobridges for Micro- and Optoelectronic Applications
14:50	<i>Coffee Break</i>
15:30	Luisa Petti, Wearable Computing, D-ITET Large-Area Microelectronic Circuits on Plastic Foil
15:45	Stephan Kress, Optical Materials Engineering, D-MAVT Quantum Plasmonic Waveguides and Resonators
Live Demo by Lukas Böni, Food Process Engineering, D-HEST	
16:05	Hortense Le Ferrand, Complex Materials, D-MATL Magnetically-Assisted Slip Casting of Bioinspired Heterogeneous Composites
16:20	Mario Lenz, Bioanalytics Group, D-BSSE Controlled Synthesis and Functionalization of AuTTF Micro- and Nanowire Sensors
16:35	Flash Poster Presentations
16:45	Industries Presentations
17:15	Award Ceremony
17:45	<i>Industry Apéro</i>
19:00	<i>Dinner at Uni Mensa</i>

ABSTRACTS OF TALKS

Session 1: 09:00 – 10:20

Intriguing Structural Features and Properties of a Novel Two-Dimensional Polymer

Gregor Hofer, Thomas Weber, Max J. Kory and A. Dieter Schlüter

Polymer Chemistry, D-MATL, ETH Zurich

Traditional one-dimensional polymers (1DP) as used in plastics or rubber are made from basic bifunctional structure units (monomers), which can be connected to other bifunctional monomers via linear covalent bonding. This linear bonding results in one-dimensional, long-range ordered polymers with a variety of topologies. A breakthrough in the synthesis of sheet-like polymers was recently achieved based on the polymerization of a trifunctional monomer.^[1]

The chiral anthracene-based monomer is crystallized in the polar space group *R3* prior to their connection. The arrangement in the crystal allows the anthracene moieties from neighboring monomer molecules to face each other close enough to exploit the photodimerization effect. Continuous irradiation of the crystal with UV light initiates and propels polymerization, achieving a genuine two-dimensional polymer (2DP), which consists of sheets with thickness of a few atoms and theoretically infinite lateral extent. Single sheets of the 2DP can be gained through exfoliation. The polymerization is fully reversible by heat treatment. Both, polymerization and depolymerization processes can be frozen in at any time by simply removing the crystal from the triggering source thus creating a disordered and meta-stable monomer/polymer single crystal.

We were able to solve and refine the average structure of the monomer, the fully polymerized structure and various intermediate states using in-house X-ray diffraction experiments. A careful analysis of single 2DP layers and crystal-lattice parameters has revealed some interesting features. During polymerization, the layer itself does not shrink, but rather expands by 0.7%, a phenomenon uncommon for 1DPs.

With the information contained in the already observed diffuse scattering patterns, we expect to answer the question whether polymerization

propagates via a nucleation growth or a random percolation mechanism.

[1] M. J. Kory et al., *Nature Chemistry* **6**, 779 (2014).

Magnetic superbasic proton sponges are readily removed and permit direct product isolation.

Elia Schneider, Renzo A. Raso, Corinne J. Hofer, Martin Zeltner, Robert D. Stettler, Samuel C. Hess, Robert N. Grass and Wendelin J. Stark

Functional Materials Laboratory, D-CHAB, ETH Zurich

Organosuperbases, such as the classic proton sponge (1,8-bis(dimethylamino)naphthalene (DMAN) have become very important reagents in organic chemistry over the past years.^[1] Yet work-up such reactions remains challenging and tedious, particularly when using reagents of solubility similar to the desired products and a low tendency to crystallize. Thus, a chemically stable magnetic base with rapid capability for quantitative separation would be most interesting for industrial application and entail significant solvent savings, reduce time, expensive equipment and permit reagent reusability. Magnetic nanoparticles have fascinated scientists for several decades and were used in a plethora of applications.^[2] In chemical synthesis, magnetic nanoparticles have recently gained attraction for example in the field of catalysis since they combine high surface area with simple separation.^[3]

Here we present an organic superbases, coupled to magnetic nanoparticles, with stability amenable for use in challenging reaction conditions.^[4] We further show that such an easy-to-separate reagent is useful in a number of condensation reactions and simplifies work-up and product isolation. The immobilized magnetic superbases reagent was tested in the base-catalyzed Knoevenagel and Claisen-Schmidt type condensations and showed conversions up to 99%. High yields up to 97% of isolated product could be obtained by simple recrystallization without using column chromatography. Recycling of the catalyst was simple and fast with insignificant decrease in catalytic activity.

[1] T. Ishikawa, *General Aspects of Organosuperbases. In Superbases for Organic Synthesis*, John Wiley &

Sons, Ltd., 1 (2009). [2] A. H. Lu et al., *Angew. Chem. Int. Ed.* **46**, 1222 (2007). [3] A. Schätz et al., *Chem. Eur. J.* **16**, 8950 (2010). [4] E. M. Schneider et al., *J. Org. Chem.* **79**, 10908 (2014).

Molecular Architecture of Native Fibronectin Fibrils

Susanna Früh, Ingmar Schoen, Jonas Ries and Viola Vogel
Applied Mechanobiology, D-HEST, ETH Zurich

Fibronectin fibrils are important building blocks in the extracellular matrix and influence physiological processes like wound healing, tissue homeostasis or embryonic development. However, the hierarchical fibronectin fibers are also associated with pathologies like cancer or fibrosis. Elucidating the molecular structure of fibronectin fibrils and their hierarchical assembly in the extracellular matrix will help to understand the material properties and biological functions.

To determine the relative positions of various sites within native matrix fibrils, we combine site-specific protein labeling with single-molecule localization by stepwise photobleaching and direct stochastic optical reconstruction microscopy (dSTORM). Single end-labeled fibronectin molecules in thin fibrils revealed an average extension of ~133 nm. Using immunolabeling of specific antibody epitopes, thin fibronectin fibrils possessed characteristic punctuate labeling pattern. They showed a periodicity of ~95 nm in the thinnest fibrils, the protofibrils, and exhibited alternating C- and N-terminal regions. These results suggest that fibronectin molecules overlap by 30-40 nm between the N-termini in an antiparallel fashion. We propose a model for the intermolecular interactions mediated by the first five type I modules and type III modules of the adjacent molecule. Finally, protofibrils showed a random bundling to thicker fibers without a well-defined line-up. These findings might explain the extraordinary material properties of fibronectin fibrils such as their superb mechanical stability.

Direct 3D Measurements of Acoustic Forces within Microfluidic Channels and Chambers

Andreas Lamprecht, Stefan Lakämper, Iwan A. T. Schaap and Jürg Dual
Mechanics & Experimental Dynamics, D-MAVT, ETH Zurich

Spatial acoustic manipulation of objects within fluids and fluid-like materials is of fundamental importance in various fields of research and applications. The expanding interest on the manipulation of objects, like solid particles, functionalized beads, cells, etc., has formed new advances in life science, biophysical research and material science.

In the device optimization process it is necessary to fully characterize the acoustic properties by simulations and experiments. The experimental characterization is limited by the acoustic pressure prediction, due to the small physical dimensions of micro fluidic devices (10E-6m). A trivial pressure prediction is not possible and therefore new measurement techniques have to be developed and invented to describe the present physics experimentally. A novel technique is to use a calibrated high gradient laser trap to directly measure the total time-averaged acoustic force on a single dielectric silica micro particle.

Acoustic standing waves within a microfluidic device generate acoustic radiation and streaming forces on the optical trapped particle. These forces lead to a particle displacement in the optical potential which is measured in three dimensions by observing the particle shadow in the laser light in dependence of the excitation frequency and the global particle position within the device.

The acoustic forces (0.2-50pN) are proportional to the measured displacements and this allows us to calculate the unknown acoustic pressure distribution within an arbitrarily shaped acoustofluidic device. The three dimensional direct measurement opens up the possibility to quantify so far inaccessible acoustic small scale phenomena with the existing theory such as the effects of local and global acoustic streaming or close-by objects.

Chemical Delignification to Tailor Wood Derived Bio-Scaffolds for Multifunctional Materials

Jana Segmehl, Tobias Keplinger, Artem Krasnobaev and Ingo Burgert

Wood Materials Science, D-BAUG, ETH Zurich

Chemical delignification is widely used as a pretreatment in pulp and paper industry and biofuel production processes in order to improve the accessibility of the polysaccharide components through removal of the encrusting lignin. While in these common applications a disintegration of the wood structure is targeted, we applied delignification methods on bulk wood samples with the objective to retain the structural characteristics of wood at all length scales and to create a hierarchically structured and highly porous bio-scaffold with high mechanical integrity. Thereby it was aimed to enhance the cell wall accessibility of the wooden scaffold, in order to implement new properties and functions by insertion of nanoparticles or polymeric components, potentially resulting in the development of new materials with a broad property profile.

Here we report on the adaption of various delignification procedures (e.g. PAA, H₂O₂/HAc and NaOH) for the use on bulk wood samples. Detailed chemical analysis with high chemical and spatial resolution was applied not only to visualize the decrease in lignin content but also to gain information on the characteristics of the different delignification processes. Confocal Raman Spectroscopy revealed distinct differences between the different methods and the possibility to tune the lignin content by variation of reaction parameters, especially temperature and duration.

Session 2: 11:00 – 12:05**Assembly of Crystalline Ultrasmall Copper Nitride Particles into Freestanding 3D Aerogel**

Rupali Deshmukh, Elena Tervoort, Felix Rechberger and Markus Niederberger

Multifunctional Materials, D-MATL, ETH Zurich

Nanostructures of transition metal nitrides are often desired for improving their properties and further expanding their applications. Copper nitride is a potentially interesting material for resistive random

access memory chips, optical storage devices, high-speed integrated circuits and catalyst for oxygen reduction. Here we present a simple surfactant-free solution route for the synthesis of ultrasmall (about 3 nm in size) crystalline copper nitride nanoparticles. The formation of copper nitride nanoparticles, starting from two molecular precursors, occurs at temperature as low as 100 degree Celsius. The as prepared nanoparticles are highly dispersible in organic solvents. Therefore, the crystalline copper nitride nanoparticles are transformed into a colloidal solution that acts as the precursor for an integrated network (gel) of discrete copper nitride nanoparticles. The supercritical drying process is applied to remove the liquid phase from the gel giving a porous nanocrystalline copper nitride aerogel. The present invention offers the unique possibility to synthesize highly crystalline ultrasmall copper nitride nanoparticles via a novel solution method and to assemble them into high surface area 3D aerogel. Such a unique combination of composition and surface property is open for all possible applications particularly in catalysis.

Ring-Opening Synthesis of Polyethylene Furanoate (PEF) as a 100% Renewable Resource-Based Substitute for Polyethylene Terephthalate (PET)

Jan-Georg Rosenboom, Peter Fleckenstein, Giuseppe Storti and Massimo Morbidelli

Chemical Reaction & Separation Engineering, D-CHAB, ETH Zurich

Biomass-derived plastics can have a major impact on the anticipated shift of our societies from fossil-fuel based economies towards more sustainable civilizations. Furanic polymers such as polyethylene furanoate (PEF) are promising “green” substitutes for some of the world’s most dominant plastics, especially oil-based polyethylene terephthalate (PET). Those alternative polymers have already proven to possess favorable material properties over PET, e.g. reduced CO₂- and H₂O-permeability, which are essential for bottle or textile fabrication. While the recent efforts of other groups were invariably based on polycondensation, our aim is to explore ring-opening polymerization (ROP) to push furan-based polymer technology further towards industrially applicable processes.

In the first major process step, we are exploring 3 synthetic strategies for the synthesis of cyclic PEF

oligomers (cyOEF) for ROP from 100% renewable furanic resources: (1) rapid esterification of acid chlorides, (2) depolymerization of short PEF prepolymer under high dilution, and (3) reactive distillation of furandicarboxylic acid (FDCA) with excess ethylene glycol (EG) under EG removal. The latter is most promising due to high cyOEF yields (85%) and almost complete recyclability of the side products. Purification using precipitation and adsorption processes allowed us to deliver cyOEF at high purity from linear species for ROP (> 99% by HPLC area).

In the second step, high molecular weight polymer synthesis via ROP was shown feasible at conversions and purity above 95% with M_w up to 56,000 g/mol (PMMA equivalent) and $PDI < 2$. Reactions were employing tin and titanate initiators in melt at up to 300°C. DSC analysis showed superior thermal properties over PET, specifically significantly higher glass temperature and lower melt temperature, allowing for higher thermal stability and more facile bottle fabrication, respectively. Currently, the process intensification and catalyst systems are being optimized before deeper material properties analysis for comparison with PET will be undertaken.

pH-responsive Lyotropic Liquid Crystals and Their Potential Therapeutic Role in Cancer Treatment

Renata Negrini, Wye-Khay Fong, Ben J. Boyd and Raffaele Mezzenga

Food & Soft Materials, D-HEST, ETH Zurich

Cancer cells are typically characterized by a pH significantly lower than physiological conditions, which paves the way to possible cancer therapies exploiting pH as a trigger. A pH responsive Lyotropic Liquid Crystal (LLC) matrix is developed in this work suiting ideally pH-responsive controlled drug release for tumor treatment. To this end, a weak base, Pyridin-4-ylmethyl linoleate (PML), was synthesized and combined with a neutral lipid monolinolein (MLO), in order to obtain lipid mesophases exhibiting a change in symmetry when PML is protonated in response to pH changes around its pK_a (≈ 5.5). The system was optimized by testing different amounts of PML and following the structure evolution at different pH by small angle X-ray scattering (SAXS). The final design composition yielded a bicontinuous cubic phase (Pn3m symmetry) at pH 5.5 and a reversed hexagonal columnar phase (H2) at pH 7.4. By exploiting the different intrinsic transport rates of Pn3m and H2,

diffusion studies at two different pHs simulating physiological and cancer conditions were conducted by employing glucose and doxorubicin (DOX) as model drugs. Both molecules were shown to diffuse significantly faster in the Pn3m phase at acidic conditions. In vitro drug release studies of DOX conducted at pH 7.4 and 5.5 from the above-designed bulk mesophases coexisting in excess buffer with cancer cell cultures confirmed the potential of the system in cancer therapy, by yielding a three-fold increase in the efficiency by which cancer cells are killed at pH 5.5 versus the physiological pH 7.4. This work provides the first example of a responsive lipid mesophase system in which a spontaneous change in symmetry at pHs characteristic of tumor growth, enhances the release of anticancer molecules, standing as a new drug delivery system for cancer therapy, in which the lower extracellular pH typical of tumor is the only endogenous triggering stimulus.

Guided Assembly Biolithography: Shaping Bacterial Cellulose

Francesco Robotti, Bottan Simone, Dimos Poulikakos and Aldo Ferrari

Thermodynamics in Emerging Technologies, D-MAVT, ETH Zurich

Guided Assembly-based Biolithography (GAB) is a powerful replica-molding technology that allows the transfer of on-demand functional topographies to the surface of bacterial cellulose films. The approach is based on standard fermentation of glucose into cellulose in a static culture of *Acetobacter Xylinum*. In particular, at the interface of a microstructured PDMS mold preferential nanofiber patterns are created by the physical confinement of bacterial motion. In this way, the cellulose nanofibers are assembled in a three-dimensional network which closely replicates the geometry imposed by the mold. Additionally, GAB yields directional alignment of individual nanofibers and memory of the transferred geometrical features upon dehydration and rehydration of the cellulose substrates.

The fidelity of this facile and affordable method is assessed by scanning electron (SEM) and atomic force microscopy (AFM). The interaction of surface-structured bacterial cellulose substrates with human fibroblasts and keratinocytes is reported to illustrate the efficient control of cellular activities which are fundamental in wound healing and tissue

regeneration. Additionally, the deployment of surface-structured bacterial cellulose substrates in model animals as skin wound dressing or body implant proved the high durability and low inflammatory response to the material over a period of 21 days, demonstrating beneficial effects of the surface structure on skin regeneration. Altogether, the possibility to control specific cell and tissue responses by a non-inflammatory, micro-structured material has huge potential in the field of soft tissue repair.

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13:30 – 14:50

Dr. Sinead Griffin

Materials Theory, D-MATL, ETH Zurich

From the Early Universe to the Hubbard Hamiltonian in the Hexagonal Manganite

Jumping from the expanse of galactic scales to land in the laboratory might seem a gargantuan task. Common to both, however, is the concept of symmetry breaking and in particular the formation of topological defects. This work displays the simplicity and power of applying a topological description to a multiferroic material. Their formation and understanding is central to describing both the functional and exotic properties of these materials. It demonstrates an early-universe theory – the Kibble-Zurek mechanism – for the first time in a crystal. This mechanism, which remains an open question in cosmology, predicts a scaling law for the number of defects formed during a phase transition. Extending the description to the whole class of hexagonal multiferroics provides a universal description of defects, domains, and domain walls. With this knowledge both the density of defects and the average domain size can be tuned for these functional multiferroic materials. Finally, using the same crystal structure, we design a material obeying the Hubbard Hamiltonian using a novel top-down approach that harbors possible exotic superconductivity.

Dr. Deniz Bozyigit

Nanoelectronics, D-ITET, ETH Zurich

Understanding Performance Limitations of Semiconductor Nanocrystal-Based Optoelectronic Devices

Semiconductor nanocrystals (ncQDs) are one of the promising material systems for next-generation light emitting devices (LEDs) and solar cells. While improved fabrication methods have recently increased the device performances, a detailed understanding of the opto-electronic processes, which determine the operation of such devices, is still being developed. The goal of this thesis is to characterize and identify the performance-limiting electronic processes in these devices.

To understand efficiency limitations in ncQD-based LEDs, we have investigated the role of the electric field by combining time-resolved photoluminescence experiments and 3D-quantum mechanical calculations. We show that at high fields (>1 MV/cm), which occur in all common LED architectures, ncQDs will typically lose up to 90% of their luminescence efficiency due to the quantum-confined Stark-effect. Based on this finding, we develop guidelines for the design of the ncQDs band-structure and improved device architectures.

The performance in ncQD-based solar cells is limited by the presence of electronic defect states acting as charge carrier traps and recombination centers, which have proven themselves to be notoriously difficult to characterize. In this thesis work, we implemented three complementary trap characterization techniques and applied them for the first time successfully to ncQD solar cells. The comparison between the techniques allows (1) to validate the underlying model for the interaction between charge carriers and trap states and (2) to show that the existing models begin to fail by grossly underestimating the trap state density (>10 fold) when a high density of traps is present. Finally, we use the findings to develop a quantitative model of the charge transport in ncQD-based solar cells, which allows the rational optimization of these devices.

Dr. Alexandre Larmagnac

Biosensors & Bioelectronics, D-ITET, ETH Zurich

Novel Neuroprosthetic Devices for Spinal Cord Stimulation in Paralyzed Animal

Traumatic injuries of the spinal cord have long-term health, economic and social consequences, giving a sense of the urgency to the development of ways to treat them. Worldwide, an estimated 2.5 million people live with a chronic spinal cord injury (SCI). Consequently, there is a critical need to improve rehabilitative strategies to help these patients to regain the ability to stand or step. Courtine and colleagues recently demonstrated the impressive capacity of pharmacological and electrical spinal cord stimulations to promote full weight bearing walking in paralyzed rats when combined with rehabilitation. Specifically, they showed that epidural electrical stimulation (EES) applied at S1, L4, or L2 spinal segments could each promote unique patterns of locomotion, which were biased toward flexion when stimulating upper lumbar segments and toward extension when stimulating the sacral level. Next, they revealed that the combination of two, and even more efficiently three, sites of EES promoted clear synergistic facilitation of stepping in paralyzed rats. Together, these results suggest that multi-site EES strategies would enable a finer control of locomotion after a SCI than currently possible with existing stimulation paradigms. However, no systematic studies on the potential benefit of multi-site EES have been conducted so far, largely because of the lack of interfaces for the concurrent stimulation of multiple spinal cord locations. In this thesis, we aim at developing a novel neuroprosthetic multi-electrode array (MEA) for chronic multi-site EES in vivo and a dedicated wireless implantable electrical stimulator in collaboration with the team of Prof. Courtine.

Dr. Martin Süess

Nanometallurgy, D-MATL, ETH Zurich

Highly Strained Si and Ge Micro- and Nanobridges for Micro- And Optoelectronic Applications

Strain engineering is an interesting approach to optimize device performances or access physical

properties that are not present in the unstrained materials. E.g. strain can be used to boost the mobility in silicon, which allows for faster switching transistors. Another example is germanium, where strain can convert the indirect bandgap to a direct one, giving rise to the possibility of an on-chip Ge laser. Commonly, strain engineering approaches used in semiconductors to enhance device properties are based on epitaxy methods, which limit the thickness of the strained layer. Here we show a method how a layer with small strain can be patterned in a way to multiply the initial strain by more than a factor of 20. This method is benchmarked on those semiconductors on two different length scales. In Si we demonstrate a record strain of 4.5% in a merely 30 nm wide wire, which is a suitable size for a transistor channel, and in Ge we achieve a strain of 3.1% and are able to demonstrate that with this strain the conversion from indirect to direct band gap is well underway. Due to its universality the method has also potential applicability on other materials and length scales.

Session 3: 15:30 – 16:35

Large-Area Microelectronic Circuits on Plastic Foil

Luisa Petti, Niko Münzenrieder, Giuseppe Cantarella, Christian Vogt, Lars Bütthe, Alwin Daus, Mathieu Chanson, Giovanni A. Salvatore and Gerhard Tröster
Wearable Computing, D-ITET, ETH Zurich

Thin-film transistors (TFTs) and microelectronic circuits on plastic promise to enable new large-area electronic applications, such as rollable displays, electronic skins or smart textiles. To realize such systems, metaloxide semiconductors, especially Indium-Gallium-Zinc-Oxide (InGaZnO₄), are promising materials, since they possess high electron mobility even if deposited at RT. InGaZnO₄TFTs fabricated on plastic at temperatures <150 °C exhibit state-of-the-art mobility around 15 cm²V⁻¹s⁻¹ with a process yield of 93%. This enables the realization of digital circuits, such as unipolar inverters, NAND gates, and ring oscillators, which are bendable down to 5 mm. The good DC performance of InGaZnO₄ devices, together with their high transit frequency of up to 135 MHz also allows fabricating high-performance, fast, and flexible analog circuits. For example, flexible InGaZnO₄-based single-stage cascode amplifiers yield a gain of 11 dB and a cut-off frequency

of 2.6 MHz. On one side, vacuum-deposited InGaZnO_4 -based circuits open the way to short-wave receiver applications and radio frequency identification tags (RFIDs). On the other side, low-cost and large-area applications (e.g. sensoric systems) call for scalable solution-based manufacturing techniques. Among the possible solution-processed semiconductors, spray-coated Indium-Oxide (In_2O_3) is particularly interesting. By employing In_2O_3 spray-deposited at 250 °C, we demonstrated flexible unipolar inverters with gains >5 V/V. High gains, together with simple circuit design, low power consumptions and noise are only possible with complementary logic. Nevertheless, the realization of flexible CMOS circuits suffers from the lack of p- and n-type organic or inorganic semiconductors yielding similar TFT performance. Recently, we integrated spin-coated highly-selected semiconducting p-type (7,5) single-walled-carbon-nanotubes (SWCNTs) with both vacuum-deposited InGaZnO_4 and spray-deposited In_2O_3 . Flexible inverters based on SWCNTs and InGaZnO_4 exhibit gains of 85 V/V. Additionally, flexible inverters based on fully solution-deposited SWCNTs and In_2O_3 yield gains of 22 V/V. These results pave the way towards large-area and high-performance flexible microelectronic applications.

Quantum Plasmonic Waveguides and Resonators

Stephan Kress, Felipe Antolinez, Patrizia Richner, Sriharsha Jayanti, David Kim, Maximilian Fischer, Andreas Riedinger, Stefan Meyer, Kevin McPeak, Dimos Poulikakos and David Norris
Optical Materials Engineering, D-MAVT, ETH Zurich

Quantum optics involves the coupling of quantum emitters to their electromagnetic environment. Because this coupling is related to the concentration of the optical field, it is typically constrained by the diffraction limit of light. One way to circumvent this limitation is by moving to quantum plasmonics, which uses surface plasmon polaritons (SPPs) instead of photons. However, despite the advantages of this approach, quantum plasmonics has not yet been fully explored, largely due to the difficulty of creating the necessary structures. We address this problem by combining state-of-the-art quantum emitters with plasmonic structures (waveguides and reflectors) that approach theoretical performance limits. We synthesize highly luminescent colloidal quantum dots

(photoluminescence quantum yields $>90\%$) and precisely place them on template-stripped silver wedge waveguides. We demonstrate efficient coupling of the quantum-dot emission into guided plasmonic modes in the waveguide. In addition, by adding efficient reflectors, high-Q plasmonic resonators are obtained. More generally, the flexibility and fidelity of the resulting quantum plasmonic systems indicates that they will enable a broad set of nanoscale quantum optical measurements and devices.

Magnetically-Assisted Slip Casting of Bioinspired Heterogeneous Composites

Hortense Le Ferrand, Florian Bouville, Tobias P. Niebel and André R. Studart
Complex Materials, D-MATL, ETH Zurich

Heterogeneous composites are widely used in nature to fulfill functional demands imposed by the environment but remain difficult to fabricate synthetically particularly at microscopic length scales due to the lack of adequate easily accessible processing tools. We report on an additive manufacturing platform to fabricate complex-shaped parts exhibiting bio-inspired heterogeneous microstructures with locally tunable texture, composition and properties within a wide range of organic-inorganic volume ratios. The technology combines an aqueous-based slip casting process with magnetically-directed colloidal assembly to create programmed microstructural designs using anisotropic stiff platelets in a ceramic, metal or polymer functional matrix. Using quantitative tools to control the casting kinetics and the temporal pattern of the applied magnetic fields, we demonstrate that this robust approach can be used to design and fabricate heterogeneous microstructures with unprecedented periodic patterns of micro-reinforcement orientation and tooth-like bilayer parts with intricate shapes displaying fully programmable site-specific composition and texture.

**Controlled Synthesis and Functionalization of AuTTF
Micro- and Nanowire Sensors**

Mario Lenz, Yanlong Xing and Petra S. Dittrich

Bioanalytics Group, D-BSSE, ETH Zurich

Conductive nano- and microwires are very promising sensing elements. In the past years, various approaches to form such wires have been presented. However, most of them are based on reactions in bulk with little control on the reaction conditions of individual wires, and require post-processing such as purification. In addition, it is still challenging to integrate the created structures into a functional analytical device. Recently, we showed the advantage of microfluidic devices for the creation of nanowires made of metal organic compounds or coordination polymers. We exploited the laminar flow conditions providing a well-defined interface between two

streams, or used small reaction volumes to allow defined diffusion of the precursors.

Here, we demonstrate an improved microchip designs for the site-specific formation of single nano- and microwires. The geometry of the channels provides physical constraints with respect to number, orientation and localization of the created wires.

We utilized such wires before for sensing volatile compounds. In addition, we successfully achieved the functionalization of nanowires for label-free sensing of biomolecules. Binding of dopamine to the functionalized wires could be detected by using Raman spectroscopy. Moreover, we showed in a proof-of-concept experiment that we could immobilize antibodies on the nanowires to enable the binding and detection of analytes with a high specificity. Future work focuses on the realization of conductivity measurements for sensing biomolecules.

POSTER PRESENTATIONS

Name	Poster Title	Affiliation	Poster No.
Marta Airaghi Leccardi	Design, Fabrication, and Characterization of AFM Tips for Tensions Measurement and Manipulation of Fluid Interfaces	Interfaces, Soft Matter & Assembly, D-MATL	20
Anton Bauhofer	Modeling of Microstructured Muscle Cell Actuated Sheets	Mechanics & Materials, D-MAVT	33
Manuel Baumgartner	Ultrafast Magnetization Switching by Spin-Orbit Torques	Magnetism & Interface Physics, D-MATL	44
Christoph O. Blattmann	Thin, Flexible and Electrically Conductive Ag-PMMA Nanocomposite Films	Particle Technology Laboratory, D-MAVT	2
Lars Büthe	Cavity and Pendulum Challenges for Flexible Tilt Sensor	Wearable Computing, D-ITET	31
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ABSTRACTS OF POSTERS

1 Biomineralization of Binary and Ternary Nanoparticles in Apoferritin for Controlled Carbon Nanotube Synthesis

Laura Jenni, Ruizhong Lin, Wei Liu, Tobias Süss, Miroslav Haluska, Yucheng Zhang, Rolf Erni and Christofer Hierold

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For the synthesis of single walled carbon nanotubes (SWNTs) with targeted characteristics the catalyst properties are crucial. Especially for sensor applications long nanotubes are desired as they allow fabrication of multiple devices with a single SWNT. It was demonstrated that by using bimetal and ternary alloy nanoparticles the yield and the average length of the grown tubes can be enhanced. In this study, nanoparticles were synthesized inside the protein cage apoferritin, which had been extracted from the equine spleen. Specifically, Fe-Mo, Fe-Pt, Fe-Ni and Fe-Co as well as Fe-Mo-Pt and Fe-Mo-Ni nanoparticles were prepared. Carbon nanotubes were both grown with methane and acetylene. For the nanotube synthesis with methane the average carbon nanotube (CNT) length grown from Fe-Mo-Pt particles was enhanced significantly (CNTs longer than 100 μm were observed) in comparison to pure Fe catalysts (which typically do not exceed 15 μm). Under the same conditions Fe-Ni and Fe-Pt resulted in a more than 300 % increase in yield, with respect to iron nanoparticles. Executing the synthesis inside the biological nanoreactor apoferritin allows for almost independent control of parameters such as the elemental composition, size and the deposition density on the growth substrate. Therefore this approach can be used to synthesize SWNTs with targeted properties to be later integrated in chemical or mechanical sensors, such as a CNT resonator.

2 Thin, Flexible and Electrically Conductive Ag-PMMA Nanocomposite Films

Christoph O. Blattmann, Georgios A. Sotiriou and Sotiris E. Pratsinis

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Electronics keep decreasing in size and weight, while simultaneously increase in flexibility. Electrically conductive polymer nanocomposites are highly prized for such applications, however, the particle agglomeration tendency within the polymer matrix inhibits them from broad employment because particle agglomerates result in inhomogeneous composite properties. Specifically for electrical conductivity, a higher filler fraction is necessary to overcome the percolation threshold. Metallic nanoparticles, as alternative to carbon-based nanomaterials, are beneficial to achieve high conductivities. Here, flame-made silver nanoparticle-PMMA composite films are synthesized based upon flame-aerosol deposition and polymer spin-coating. It does not require any additional sintering process or nanoparticle surface functionalization steps. By carefully tuning the deposition parameters, Ag nanoparticles with high degree of sinter-neck formation can be achieved within seconds. Sinter-necks are beneficial for good electrical properties. Finally after embedding the nanoparticle film within PMMA, sub-micrometer thick nanocomposite films are made with bulk-metal equivalent electrical conductivity. The flexibility of such films was demonstrated after releasing from the substrate by repetitively bending. Throughout the bending the electrical resistance remained unchanged.

3 Single-Crystalline W Thin Films Achieved by Ion Beam-Induced Selective Grain Growth

Ma Huan, Matteo Seita and Ralph Spolenak

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Ion-beam irradiation can be a useful tool to athermally engineer the microstructure of refractory metal films. Our results show that vapor deposited, fiber textured tungsten films can be converted into single crystals under specific irradiation conditions. This transformation occurs when the ion beam is set parallel to one of the possible channeling directions of the textured film, such that only one subset of grains is orientated for ion-channeling. As a result, a selective grain growth (SGG) process leads the overgrowth of such grain subset until a single crystal film is obtained. SGG is driven by the minimization of the free volume energy stemming from the ion-induced defect content, which is lowest in channeling oriented grains.

4 Localized Fluorescence Excitation with the Guided Second-Harmonic in Lithium Niobate Nanowires

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The ability of nanowires to guide light has been shown in many materials. In particular, chemically synthesized nanowires can generate second-harmonic of the guided near-infrared light and as a result provide light in the visible range.^[1,2] However, the chemically synthesized nanowires suffer from low crystallinity resulting in low second-harmonic that has not been quantified yet.

Here, we quantitatively investigate lithium niobate nanowires that were fabricated by a top-down method combining ion beam irradiation and wet etching.^[3] We demonstrate the second-harmonic generation and propagation in the nanowires. We excite a fluorescent dye with the guided second-harmonic signal. We define experimentally the power limit for detectable fluorescence excitation and, by modeling, we find the smallest NW cross-section to achieve this second-harmonic power. Finally, we measure an increase of the guided SH by adjusting phase matching through phase matching.

[1] Y. Nakayama, *Nature* **447**, 1098 (2007). [2] R. Grange, *Appl. Phys. Lett.* **95**, 143105 (2009). [3] H. Hartung, *Opt. Mater.* **33**, 19 (2010).

5 Large-Area Alignment of Tungsten Oxide Nanowires over Flat and Patterned Substrates for Room-Temperature Gas Sensing

Wei Cheng, Yanrui Ju, Payam Payamyar, Darinka Primc, Jinyi Rao, Christoph Willa, Dorota Koziej and Markus Niederberger

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Alignment of metal oxide nanowires over a large area of flat and patterned substrates is a prerequisite to use their collective properties in devices. As n-type semiconductors, tungsten oxides (WO_{3-x}) have promising applications in gas sensors, electrochromic devices, or as photocatalysts. Ultrathin (diameter less than 10 nm) tungsten oxide nanorods and nanowires have already been synthesized and assembled for electrochromic applications. However, up to now, no successful attempts to align them for gas sensing applications have been reported.

In this work, we report the synthesis of colloiddally stable, ultrathin $W_{18}O_{49}$ nanowires, their alignment with high directional order over a macroscopic area and their H_2 sensing properties at RT. The nanowires are prepared by a facile solution method. They are single crystalline, with diameters of about 1.7 nm and aspect ratios larger than 100. Most importantly, they are highly dispersible in chloroform and chlorobenzene, enabling the use of these dispersions for the alignment experiments by Langmuir-Blodgett technique. The flexible ultrathin nanowires can be successfully oriented over a large scale and transferred to Si/SiO₂ substrates patterned with Pt interdigitated electrodes. The aligned $W_{18}O_{49}$ nanowire thin films exhibit excellent sensing performance towards hydrogen in humid air, at RT, indicating their high potential for gas sensors. The strategy developed here represents one of the few examples, where nanoscale building blocks are successfully integrated in a macroscopic device.

6 Enhanced Ag⁺ Ion Release from Aqueous Nanosilver Suspensions by Absorption of Ambient CO₂

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Nanosilver with closely controlled average particle diameter (7 - 30 nm) immobilized on nanosilica is prepared and characterized by X-ray diffraction and transmission electron microscopy. The presence of Ag₂O on the metallic nanosilver surface is confirmed by UV-vis spectroscopy and quantified by thermogravimetric analysis and mass spectrometry. The dissolved Ag as Ag⁺ ions [Ag⁺] from nanosilver in de-ionized water exposed to ambient laboratory air containing 580 ppm of CO₂ and CO₂-free synthetic air (79% N₂ and 21% O₂) is monitored electrochemically. For all air compositions, the [Ag⁺] instantly reaches the level that corresponds to the dissolved preexisting Ag₂O layer on the nanosilver surface. Under CO₂-free synthetic air, the [Ag⁺] levels for all nanosilver sizes do not change for seven days indicating that the remaining metallic Ag. In contrast, under laboratory air (CO₂ 580 ppm), the [Ag⁺] of all three nanosilver suspensions slowly increases indicating the additional release of Ag⁺ ions from the metallic Ag core that depends on solution pH. The pH rapidly increases during preexisting Ag₂O dissolution by the released OH⁻ species along with Ag⁺ ions resulting in basic pH solutions. This prevents further Ag⁺ ion release by metallic Ag dissolution resulting in the constant [Ag⁺]. When these solutions are exposed to CO₂-containing atmospheres, absorption of CO₂ in the host solution leads to its acidification and facilitates metallic Ag dissolution. So the pH of nanosilver solutions increases first rapidly by dissolution of preexisting Ag₂O and release of OH⁻. Slow absorption of atmospheric CO₂ gradually acidifies these solutions promoting metallic Ag dissolution that allows additional release of Ag⁺ ions. Thus, metallic Ag dissolution in water exposed to CO₂-containing ambient air during its prolonged storage is enhanced by acidification resulting from atmospheric CO₂ absorption in solution, which significantly increases the risk to environment.

7 [AuMes]₅ as a Precursor for Halide-Free Deposition of Au Nanoparticles on SiO₂, Al₂O₃, and TiO₂

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The easily prepared [AuMes]₅ halide-free organometallic precursor was used to generate small Au nanoparticles on SiO₂, Al₂O₃ and TiO₂. Incipient wetness impregnation of [AuMes]₅ on SiO₂ followed by reduction under H₂ leads to the formation of 1.4 nm Au nanoparticles. In contrast to SiO₂, Au nanoparticles are directly formed upon contacting [AuMes]₅ with Al₂O₃ or TiO₂, at RT Organic residues can be removed via reduction under H₂ without sintering. On Al₂O₃ particle size and weight loading is a function of dehydroxylation temperature, attaining sizes down to 0.9 nm, while 0.6 nm particles are obtained on TiO₂.

8 Mie Scattering of Single BaTiO₃ Nanoparticle for Enhanced Second Harmonic Generation

Flavia Timpu, Michal Wojdyr and Rachel Grange
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Nanomaterials with a high refractive index (usually above 2) and a diameter comparable to the excitation wavelength exhibit multiple resonances in the optical range that can be used for imaging applications. Contrary to plasmonics occurring in metallic nanostructures, those resonances take place in dielectric nanoparticles and they are well described by the Mie scattering theory.

Here we investigate dielectric nanocrystals as BaTiO₃ that are noncentrosymmetric and, in addition to the Mie scattering, they exhibit nonlinear optical effects such as second harmonic generation (SHG). In this work the Mie scattering spectrum and the SHG of a 200 nm BaTiO₃ nanoparticle are measured. Two resonances in the visible range are observed in the scattering spectrum. They correspond to the electric and magnetic dipoles Mie resonances. These two resonances correlate with the peaks in the measured SHG power of the same nanoparticle. Thus a SHG enhancement of 5.85 is demonstrated.

Since the power of a second-order effect as SHG decreases quadratically with the volume of the

nanoparticle, achieving a significant SHG enhancement is essential to have very bright emission. Therefore, one can obtain better contrast for multiphoton bioimaging or other optoelectronics applications.

9 A Comparison of the Microstructural Evolution of Binary Refractory Metal Systems W-Cu vs. W-Al

Franz T. N. Vüllers, Wilhelm Hüttenes and Ralph Spolenak

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Tungsten has outstanding properties unmatched by other pure materials. In combination with a second constituent, this property vector can be extended even further. Two promising partners for interesting applications are Cu and Al. As properties are no mere function of composition, but also a function of microstructure, we aimed towards full microstructure control of these two binary refractory systems from nanocrystalline solid solutions to completely phase separated, interconnected networks with feature sizes on the nanoscale.

10 Aliovalent Ni in MoO₂ Lattice – Probing the Structure and Valence of Ni and Its Implication on the Electrochemical Performance

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Here, we present a synthesis of MoO₂ nanoparticles doped with 2 at% of Ni in a mixture of acetophenone and benzyl alcohol at 200 °C. Based on in situ X-ray absorption near-edge structure (XANES) and ex situ extended X-ray absorption fine structure (EXAFS) measurements at Ni K-edge and Mo K-edge, we discuss scenarios on how the “doping” reaction, that is, the incorporation of Ni in the MoO₂, proceeds. We can clearly exclude the formation of NiO or Ni nanoparticles. Moreover, within the resolution of our in situ XANES experiments, we observe that the ternary compound Ni:MoO₂ nucleates directly in the final composition. Although the local structure around the Ni ion adopts the MoO₂ crystal structure pointing at the substitution of tetravalent Mo by Ni, we find that Ni remains divalent. This aliovalent substitution results in the relaxation of the local structure, which is additionally reflected in the slight shrinking of the total volume of the unit cell of

Ni:MoO₂. Interestingly, such a small amount of divalent Ni has a tremendous effect on the performance of the material as anode in Li-ion batteries. The initial discharge capacity of Ni:MoO₂ based anodes almost doubles from 370 mAh/g for MoO₂ to 754 mAh/g for Ni:MoO₂ at 0.1 C (1 C = 300 mA/g). Additionally, we observed an atypical increase of capacity for both MoO₂ and Ni:MoO₂ anodes upon cycling with increasing cycling rate.

11 Aggregate Characteristics Accounting for the Evolving Fractal-like Structure during Coagulation and Sintering

Eirini Goudeli, Maximilian L. Eggersdorfer and Sotiris E. Pratsinis

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Coagulation and partial coalescence (or sintering) frequently results in fractal-like aerosol structures in natural and industrial processes. Aerosol methods are used routinely for commercial synthesis of nanostructured particles (e.g. flame reactors: fumed SiO₂, pigmentary TiO₂, carbon black; hot wall reactors: ferrites, ZnO, Ni) for several decades. The asymptotic form of such structures is described reasonably well with the so-called fractal dimension, D_f . Little is known, however, for its evolution, from spheres to fractal-like particles and, in particular, its effect on aerosol primary particle and collision diameters that determine the environmental impact or manufactured product performance. So the effect of a variable or constant D_f on product crystalline (TiO₂) and amorphous (SiO₂) aerosol particle characteristics is elucidated over their process synthesis parameter space (cooling rate $10^3 - 10^6$ K/s, maximum temperature 1600 – 2000 K and precursor molar fraction $10^{-4} - 10^{-1}$). So aerosol dynamics by coagulation and sintering are simulated accounting for the evolving fractal-like structure by either a linear interpolation or detailed mesoscale simulations (Goudeli et al., 2015) from spherical to asymptotic fractal-like structures. In addition, two sintering rates for SiO₂ as well as expressions for the effect of particle structure on sintering rate are compared in terms of product particle characteristics. Neglecting the evolution of D_f hardly affects the product primary particle and soft-agglomerate diameters but overestimates the agglomerate collision diameter growth rate during the hard- to soft-agglomerate transition. This underpredicts the hard-agglomerate

diameter by 25 – 30% at high cooling rates ($10^5 - 10^6$ K/s). The sintering rates significantly affect both the transient and final primary particle and hard-agglomerate diameters that determine the final particle properties, especially at low maximum temperatures (≤ 1800 K).

E. Goudeli et al., *Langmuir* **31**, 1320 (2015).

12 Wooden Sunscreen

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UV degradation of wood is a topic building materials such as facade elements of houses suffer from every day. The state of the art is to reapply a coating regularly, to prevent the facade from color change and degradation. As wooden elements approach in the high class building sector, there is a need for less maintenance intensive, longer lasting, and invisible coatings to prevent UV degradation. To develop such a coating, UV absorbing nanoparticles shall be deposited on the wooden surface. The main challenge is to build a good bridge between the wooden surface and the coating in a way that the coating is homogeneously distributed on the heterogeneous wooden surface. To deposit the nanoparticles on the wood the Layer-by-layer (LbL) technique is used. Thereby, a thin coating is build up due to electrostatic charges. Several layers with opposite charges are applied on the wood. Atomic Force Microscopy images can be used as a characterization technique to reveal the interaction of the coating components with the wooden surface, and thereby help to increase the understanding of the LbL-parameters influencing a good coating.

13 An Ultrastrong, Ductile and Stable Metallic System: Help from Small Size and High Entropy

Yu Zou, Huan Ma and Ralph Spolenak
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Refractory high-entropy alloys (HEAs) are a class of emerging multi-component alloys, showing superior mechanical properties at elevated temperatures and being technologically interesting. However, they are generally brittle at RT, fail by cracking at low compressive strains and suffer from limited formability. Here we report a strategy for the

fabrication of refractory HEA thin films and small-sized pillars that consist of strongly textured, columnar and nanometer-sized grains. Such HEA pillars exhibit extraordinarily high yield strengths of approximately 10 GPa — among the highest reported strengths in micro-/nano-pillar compression and one order of magnitude higher than that of its bulk form— and their ductility is considerably improved (compressive plastic strains over 30%). Additionally, we demonstrate that such HEA films show substantially enhanced stability for high-temperature, long-duration conditions (at 1100°C for 3 days). Small-scale HEAs combining these properties represent a new class of materials in small-dimension devices potentially for high-stress and high-temperature applications.

14 Conserving Natural with Artificial: A Thermal Stress Analysis of Repair Mortar for Swiss-Built Heritage

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Repair of altered stone using mortar is an interesting strategy, since it avoids replacement and extends the lifetime of the original stone. However, the stresses that could arise due to a mismatch of dimensional changes between the repair layer and the stone substrate can significantly reduce the durability of the repair and affect the historical material. In this study we analyze the magnitude of the thermal stresses that originate in an acrylic-based artificial stone used for the reprofiling of natural sandstone. This kind of artificial stone has been developed in the late 70's in the Ecole Polytechnique Fédérale de Lausanne (EPFL, Switzerland) for its peculiar property of reversibility in an organic solvent. To evaluate the thermal stresses we propose an analytical solution that considers the viscoelasticity of the repair layer. The temperature profile used in the numerical evaluation has been measured at the interface between the two materials in the Catholic Church of Notre-Dame de Vevey (Switzerland), where the artificial stone has been used in a restoration campaign. The viscoelastic behavior of the artificial stone has been characterized by stress relaxation experiments in the laboratory. The analysis reveals that, on the temperature range

found on-site, the viscoelasticity of the repair material allows relief of most of the stresses built by the thermal expansion coefficients mismatch and accounts for the durability of this particular repair material, as observed by the practitioners.

15 Secondary Crack Formation as Toughening Mechanism in Nanocomposites of Epoxy and Inorganic Fullerene-like WS₂

Dietmar Haba and Andreas J. Brunner

Composites, Mechanical Systems Engineering, Empa

Epoxy is a thermosetting polymer known for its high strength and modulus, but also for its inherent brittleness. In this work, low loadings of inorganic fullerene-like WS₂ nanoparticles were used to improve the epoxy's toughness considerably. Dynamic light scattering was found to be useful for measuring the dispersion quality within the epoxy before curing provided that the nanoparticles' polydispersity was low. The dispersion quality was found to be a major factor determining the toughening effectiveness, while selected surface functionalization of the nanoparticles had no measurable benefit. Crack lines on the fracture surfaces have earlier been interpreted as a sign of modulus inhomogeneity, but nanomechanical atomic-force microscopy indicates that the epoxy's modulus did not depend on the distance to the nanoparticles. Instead, the crack lines might arise from secondary cracks that were initiated by the nanoparticles; these result in an increased fracture surface as well as in shear fracture, which might be the main toughening mechanisms.

16 Adhesive Bonding of Structural Hardwood Elements

Mohammad Masoud Hassani, Samuel Ammann, Falk K. Wittel, Peter Niemz and Hans Jürgen Herrmann
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Nowadays, a number of important innovations are based upon the application of either pure hardwoods, or hardwood in combination with softwood. Due to the much more excessive hygric behaviour of hardwood, residual stresses arise that are much higher than those in softwood and more demanding

for the mechanical performance of bond-lines. It was our aim to advance adhesive structural bonding with hardwood by gaining more knowledge on the physical processes. For this reason the project was split into two topics, i.e. (i) experimental investigations and (ii) numerical modelling.

A rheological model for the mechanical behavior of wood under mechanical loadings and varying environmental conditions was introduced. The considered deformation modes were elastic, plastic, hygro-expansion, visco-elastic creep, and mechano-sorption. All corresponding constitutive formulations were implemented into a user-material subroutine within the environment of the Finite Element method.

Following the verification of the proposed model, delamination of hardwood bonding and fracture of hardwood adherends were studied on configurations that are much closer to real applications than typical delamination tests. Three-layered European beech panels of aligned and cross-wise configurations were subjected to cyclic climatic variations. Although all adhesive bonds were produced in a moist condition, calculations showed that also samples bonded at the dry state or lamellae with moisture differences at bonding can be covered to extend the reach of this study as an alternative delamination testing of hardwood bonding.

It was indicated that due to high moisture gradients across the bond-line, significant inelastic deformations and local stresses build up under moisture cycling that lead to inter- and intra-laminar damage. By performing moisture-stress FE calculations with elaborated wood and adhesive material models, changes in the strain field in the region of the crack tips were captured. Moreover, numerical simulations of delamination under successive de-moistening steps were also carried out based on the concept of the cohesive zone model.

It is believed that the numerical model has a large potential for optimizing even hybrid CLT with respect to the lamella orientation, thickness, and other parameters such as moisture differences among lamellae.

17 Tailored Materials Design

Jan Rys

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Metamaterials are emerging rapidly due to their exotic properties, such as negative Poisson's ratio, negative refraction or extremely low mass density. These unusual properties arise from the arrangement of smaller units within the bulk rather than from its composition.

In our work we present a complete framework for the fabricating of deformable objects with spatially tailored mechanical properties.

First, we precompute a database of tiled structures indexed by their mechanical properties, e.g. elastic modulus or Poisson's ratio. To achieve a large and ideally coverage in the mechanical property space, we introduce a numerical optimization method for sampling structures with desired properties. Second, we synthesize the interior microstructure of a specific object by tiling microstructures drawn from the prior computed families.

Our results indicate a good fit between the predicted and the measured mechanical properties. Furthermore we show the possibility to change the non-linear behavior of the base material to a linear behavior only by choosing a proper set of microstructures.

18 Step Emulsification for Droplets and Double Emulsions

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Microfluidic droplets are a novel paradigm for the handling of fluids in a miniaturized form. Fluid handling is required from biological to pharmaceutical and material science, and all of these fields profit from the format of monodisperse droplets. Recent years also led to the format of "double emulsions", where a fluid droplet is encapsulated in a second fluid shell. Such double emulsions find applications e. g. to encapsulate detergents or drugs and release them by controlled shell breakup.

In our work, we study the generation of such droplets and double emulsions by a method called "step emulsification". Compared to other droplet generation methods, step emulsification is suited for parallelization and results in very monodisperse droplets. A geometric step of the microfluidic channel height induces the breakup of water-in-oil and water-in-oil-in-water droplets. From a fluid dynamic perspective, the mechanism of step emulsification was analyzed, depending on fluid viscosity, surface tension and device design. A combination of step emulsification with a flow-focusing design resulted in a stable method for the generation of double emulsions. Considerations of hydrophilic/-phobic channel coating, surface tension measurements and surfactants are discussed as well as the method for microfluidic device fabrication by soft lithography.

19 Size- and Phase-Dependent Mechanical Properties of Ultrathin Silicon and Ge₂Sb₂Te₅ Films

Franziska Schlich and Ralph Spolenak

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Ultrathin semiconductors or phase-change materials (5-30 nm) on metals constitute color filters, which selectively absorb wavelength ranges of the incident light. Recently, it was demonstrated that these coatings are attractive for tunable color devices. Color change of ultrathin Si and Ge₂Sb₂Te₅ was induced by reversible switching between the amorphous and crystalline phase. These structures hold significant promise for optical data storage and for flexible display applications. During operation thermal stress caused by temperature gradients are introduced and could lead to failure of the material. The mechanical properties of Si and Ge₂Sb₂Te₅ have been investigated before. However, crack formation in ultrathin semiconductors and phase-change materials have yet to be reported.

Here, the mechanical properties of these ultrathin films are determined by uniaxial tensile tests in dependence of the film thickness. Si and Ge₂Sb₂Te₅ films in the thickness range between 10 nm and 100 nm are sputter deposited on polymeric substrates. The onset strain of fragmentation is measured and the crack formation is investigated for amorphous and crystalline films. Different methods like resistance measurements, optical microscopy, and scanning electron microscopy are applied to observe

the crack development in the films. Generally, cracks occur later for amorphous films in comparison to crystalline films. Moreover, the results follow the trend of increasing onset strain of fragmentation with decreasing film thickness.

Besides optical data storage and display applications these results are of great interest for MEMS applications and ultrathin solar cells.

20 Design, Fabrication, and Characterization of AFM Tips for Tensions Measurement and Manipulation of Fluid Interfaces

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Dynamic wetting phenomena lack of a proper qualitative and quantitative characterization, especially at the micro and nanoscale. The project focuses on the design and fabrication of AFM tips capable of measuring forces and energies involved in wetting phenomena, differentiating between contact line sliding and pinning effects of a fluid interface. The tips geometry consists of micro-sized pillars with a groove all around at middle height. Different fabrication processes were investigated and optimized for the success of the tips preparation. These structures were finally fabricated with smooth surface walls and sharp groove edges. After fabrication on wafers, the tips were glued to tiptless cantilevers, detached from the substrate, and exploited for fluid AFM measurements. Strong pinning at one edge of the groove was detected and adhesion forces in the μN range were measured. This successful and stable pinning allows effective and reproducible manipulation of interface curvature, which can be also used for targeted migration and assembly of nanoparticles trapped at fluid interfaces. The assembly outcome can be different depending on the tips cross-section.

21 Stress Wave Propagation in SiO_2 -Colloidal Crystals

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Stress wave propagation in discrete materials, such as in lattices of particles in direct contact (i.e., granular crystals), has been extensively studied, because of the interesting dynamic responses that arise from the Hertzian interactions. Colloidal systems in liquid environments are a common example of discrete systems at the micro-scale. The dynamics of colloidal systems are expected to present interesting phenomena, owing to the diversity of interaction forces governing the stress propagation. In addition to elastic Hertzian interactions, electrostatic, van der Waals, entropic, hydration and steric forces are expected to play an important role. All these interactions can be modified by environmental parameters and surface treatment of the colloids. Moreover, the motion of colloids in fluid environments suggests that non-conservative hydrodynamic forces should be considered. In this research, we analyze stress wave propagation in SiO_2 colloidal crystals, assembled in fluid media with different viscosities. Experimentally, we excite stress waves in the colloids using a ns-pulsed 532nm Nd:YAG laser with different power values. The laser is pulsed on gold-coated striker spheres, which gain momentum from the ablation of the gold layer. We capture the motion of the surrounding SiO_2 particles using a high-speed camera. The experimental results are then compared to discrete element simulations, which consider hydrodynamic and elastic forces, in addition to the repulsive electrostatic force. The results suggest that the hydrodynamic forces control the stress wave propagation and overcome the role of repulsive, electrostatic forces, when the inertial effects of colloids cannot be neglected.

22 Development of a Novel Adhesion System between Timber and Concrete

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Wood-concrete structures are gaining importance in the construction industry. As opposed to traditional building materials, wood represents a more sustainable solution, and has great advantages, such as its lightweight. Up to date, connections between timber and concrete have been achieved through mechanical fastenings such as screws and bolts, while adhesive connections were largely overlooked. The main benefits of glued wood-concrete structures are to obtain an equal distribution of force concentration and a more rigid interface, and its main drawback is the challenge associated to low compatibility in between wood and concrete. Our goal is therefore to modify the wood surface in order to promote adhesion at the interface wood-concrete.

An elegant method to promote adhesion between dissimilar materials is the use of organofunctional silanes (alkoxysilanes). Upon hydrolysis, alkoxysilanes can undergo a condensation reaction with the free hydroxyl groups at the surface, hence forming a sol-gel surface modification. In this project we intend to use alkoxysilanes with amino functionalities: this approach will provide novel functionalities and act as a priming system which may enhance crosslinking between amino functionality and epoxy glue, guaranteeing an improved adhesion system between wood and concrete.

23 Insights into the Mechanisms of Capillary Assembly

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Capillarity – assisted particle assembly trapped on topographically structured templates has been widely explored in micro- and nanofabrication, for nanoelectronics,^[1] bioanalysis^[2] and plasmonics.^[3] The advantages of capillary assembly include a broad range of applicable particle sizes, accurate placement with nanometric precision and little limitation on particle material. However, scarce knowledge has been gathered to date concerning the impact of the

trap geometry, and in particular trap depth, on assembly results. Here we show that the assembly results vary as a function of the trap depth, when the length of the traps allows for more than one particle to be assembled. By increasing the depth of the traps, the results change from trapping one particle only to complete trap filling. Confocal microscopy is employed to extract the local motion of particles during trapping. A systematic investigation is carried out to map the results obtained with a series of trap depths and surface tensions. A qualitative model supported by numerical simulations will be used to elucidate the underlying mechanism of the observed assembly results. Trapping of one particle is favored with a shallow trap and a moderate surface tension. A subsequent refilling step enables assembly of another type of particle next to the assembled one. The new finding opens up a new strategy to fabricate anisotropic and heterogeneous particle clusters.

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24 Soft, Thin, Mucin-like Graft-Copolymer Adsorbates as New Biolubricants for Tissues

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Osteoarthritis is a degenerative condition in which a loss in the glycoaminoglycans content of synovial joints causes reduction of wear resistance in the cartilage tissue. To develop a treatment that could restore the lubrication properties of injured joints, we took inspiration from one of the natural responsables for friction reduction in the synovial fluid, i.e. lubricin. PEG-based surface modifiers presenting graft-copolymer structures were studied as synthetic analogues of natural lubricants.^[1,2] Nevertheless, they undergo thermal degradation and enzymatic oxidation in physiological media.^[3] To overcome these disadvantages we have developed new biomimetic graft-copolymer adsorbates featuring a biodegradable polyglutamic acid (PGA) backbone with poly-2-(methyl-2-oxazoline) (PMOXA) and aldehyde-bearing side chains. Specifically, PMOXA presents enhanced stability compared to PEG^[4] and forms a biopassive and lubricating brush film at the interface. Furthermore, the aldehyde functions

provide selective anchors for aminolized natural tissue surfaces (such as cartilage) via Schiff-base formation. A series of PGA- α -PMOXAx- β -aldehyde copolymers with different sidechain lengths (x) and PMOXA(α)/aldehyde(β) grafting densities were successfully synthesized. Graft-copolymer adsorption on aminolized surfaces was investigated and their dry thickness was measured through ellipsometry. Quartz crystal microbalance with dissipation (QCM-D) and colloidal probe atomic force microscopy (CP-AFM) were used respectively to study the antifouling and the lubricating properties. Depending on the adsorption conditions and on the graft-copolymer architecture, different film thicknesses were achieved and very low coefficients of friction were observed for the thicker hydrated coatings. The combination of selective anchoring to aminolized surfaces with high lubricating properties, biocompatibility and protein resistance makes these PGA- α -PMOXAx- β -aldehyde graft-copolymers promising biolubricants for natural tissues.

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25 Study of Wetting and Tribological Properties of Ceramic Matrix Composites through Liquid Infusion Approach

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The wetting properties of a surface are known to be influenced by surface chemistry and surface roughness. In nature, low surface energy materials like polymers can repel liquids but are not wear resistant. Contrary, hard materials such as ceramics, metallic alloys are wear-resistant but not liquid repellent. However, there are varieties of applications that require combination of liquid repellency and wear resistance. To this affect, we synthesized mesoporous α -Al₂O₃ bulk samples through slip casting and subsequent sintering to obtain 70-99.5% densities. The open porosity of samples are infiltrated with different liquids like hexadecane, fomblin oil, water etc. that were hypothesized to form a thin film of liquid always on the ceramic surface through capillarity, surface diffusivity that can repel other

class of liquids (termed test liquid) and also maintain low friction and wear. Alumina composite produced was evaluated for wetting characteristics and observed that test liquids replace the infiltrated liquids in most cases due to varying magnitude of van der Waals' forces of attraction between ceramic and liquid/s. Friction coefficient values between 0.2-1 have been observed in dry self-mated condition for 70-99.5 % dense samples. The infiltration of the mesoporous specimens with different liquids resulted in reduction of both wear and friction coefficient. The best results were attained when fomblin oil was the infiltrating liquid, which dropped from 0.2 (dry condition) to 0.065 for 99.5 % dense sample. The general measured order of the effectiveness of decreasing the wear rate and friction coefficient as a function of the infiltrating liquid is: fomblin oil > liq. eicosane > hexadecane > dry.

26 Static and Dynamic Wetting of Rough Particles

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Wetting is one of the most discussed and studied phenomena in surface science. It has fascinated researchers since the early decades of 20th century and still nowadays presents great perspectives for further investigations. Particularly, wetting of rough colloids has recently gained increasing interest due to the potential of rough particles to stabilize more effectively emulsions and foams, as well as from a more fundamental point of view in relation to the generation of capillary forces.

The understanding of wetting at the nanoscale and its characterization is a challenge, especially at the level of individual nanoparticles. Freeze-fracture shadow-casting combined with cryo-SEM imaging of the oil/water interface is nowadays the state-of-the-art for the characterization of nanoparticles of different shape and nature at liquid-liquid interfaces. It could be extended to include the systematic study of other parameters of fundamental importance for particle wetting, namely surface roughness and surface topography.

Starting from a different perspective, atomic force microscopy (AFM) has been increasingly used in the literature to investigate wetting phenomena. It gives the opportunity to monitor dynamically the wetting

of single particles, adding important information to static electron microscopy data obtained with FreSCa.

In this work, we aim to combine those two complementary techniques in order to gain a more general and exhaustive perspective on the role of roughness on single-particle wetting.

27 Tumor Targeted Paclitaxel Nanocrystals

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Preparation of nanosuspensions of poorly soluble drugs is particularly attractive due to high drug loading and minimal use of excipients. In this work, we aim to obtain long circulating nanoparticles of hydrophobic anticancer drug paclitaxel, capable to accumulate passively in the tumor tissue *via* the enhanced permeability and retention effect after intravenous administration. Paclitaxel nanocrystals were obtained by wet milling of the drug powder with a solution of sodium poly(styrene sulfonate). These template negatively charged nanocores were coated layer-by-layer with poly(L-arginine) (PLR) as polycation and poly(styrene sulfonate) as polyanion control the dissolution of the drug. It was found that the amount of the drug released from the nanoparticles was reduced after deposition of 5 layers of polyelectrolytes by ca. 30% after 8 h for PLR terminated particles. A PEGylated terminal coating layer was then added to provide steric stabilization and decrease the opsonization after intravenous administration. This last layer consisted of the graft copolymer poly(L-lysine)-*g*-PEG/alkyne-PEG (PLL-*g*-PEG/alkyne-PEG) with ~40% of lysine residues functionalized with methoxy-PEG and ~10 % with alkyne-PEG. The alkyne groups will allow the further conjugation of a “clickable” azide-bearing DARPIn, which is binding to EpCAM molecule, overexpressed by many tumor types. The PEGylated nanoparticles had a diameter of 250±33 nm, zeta potential of around -7 mV, and were stable for at least 24 h when stored in cell culture medium at 37°C. *In vitro* cytotoxicity assays on human colon carcinoma cells (HT-29) showed that the “non-targeted” coated nanocrystals exhibited an activity similar to that of the commercial nanoparticulate paclitaxel formulation Abraxane®. PLL-*g*-PEG/alkyne-PEG polymer was conjugated to a “clickable” DARPIn, and

will be used to decorate the nanoparticles. The activity and the cellular uptake of the “targeted” formulation will be compared with the “non-targeted” version.

28 Bioprinting of Three-Dimensional Sulfated Polysaccharide Structures for Local Growth Factors Delivery

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INTRODUCTION: The ultimate goal of bioprinting is the creation of organs with precise 3D architectures which, one day, will overcome the shortage of donors for transplantation. The success of 3D bioprinting relies on the development of engineered bioinks capable of mimicking the complex nature of native tissues. Freeman et al.^[1] determined the interactions between sulfate groups and several heparin-binding proteins. Inspired by this, we developed a sulfated polysaccharide capable of growth factor binding and local delivery.

METHODS: Gellan gum sulfate (GG-S) was prepared via a chemical reaction with chlorosulfonic acid in formamide.^[2] The outcome of the sulfation reaction was assessed via elemental analysis, NMR and FTIR. The molecular weight (Mw) distributions after reaction were measured using SEC-MALLS. Sulfated gellan gum was added into our previously-developed bioink and the printability of the mixture was assessed via rheology. The sulfated bioink was printed into a standard dumbbell-shaped construct using the Biofactory® bioprinter (RegenHU, Switzerland) to further characterize its mechanical properties. Cytotoxicity of GG-S was assessed with Live/Dead assay on hASCs encapsulated into 3D hydrogels over 21 days.

RESULTS: C-NMR spectra confirmed that sulfation is achieved preferentially on the secondary alcohols of the glucose units, followed by the primary alcohols on the rhamnose units. Mw distribution measured with SEC-MALLS reveals a considerable degradation of the polysaccharide chains comparable to that found in literature.^[3] Rheology data collected from cation-crosslinked hydrogels containing different amounts of several sulfated polysaccharides shows highly tunable mechanical properties without

considerably affecting the printability of the original bioink. The Live/Dead assay of hASC encapsulated into bioink and bioink+GG-S (5% w/v.) hydrogels reveals good viability comparable to the 1% alginate control.

DISCUSSION and CONCLUSIONS: Gellan gum is a natural, FDA approved polysaccharide which allows a wide range of chemical modifications. In this project we present a highly-reproducible method to prepare sulfated gellan gum with precise DS and Mw distribution. This material was designed to improve our previously-developed bioink by adding the ability to bind growth factors in order to better mimic the native ECM.

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29 Microinjection of Liposomes in Living Cells

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Microinjection is a powerful cell manipulation technique, so far mostly used in biological sciences to study the function of macromolecules that are unable to cross the plasma membrane of living cells. Compared to common *in vitro* incubation methods, microinjected materials can bypass the endosomal pathway, thereby avoiding final breakdown in the lysosomes. By employing microinjection in the field of drug delivery, it may become possible to modulate the characteristics of cells and use them as vehicles for drugs and enzymes. In this study, liposomes were microinjected in HeLa cells, providing them with an external self-contained vesicle, which may carry biologically active enzymes. Four different liposome formulations were studied for their intracellular stability as well as their potential cytotoxicity. The formulations tested differed in terms of transition temperature of the main lipid and the presence of poly(ethylene glycol) (PEG) chains at the liposomal surface. The intracellular stability of the formulations was monitored over time after injection using a fluorescence dequenching assay with the dye/quencher pair pyranine/p-xylene-bis-pyridinium bromide. The integrity of the intracellular lipid vesicles containing the dye/quencher pair was verified by exposing the cells to Triton X-100, which

causes the rupture of the liposomal membrane, and a corresponding increase in fluorescence intensity. The cytotoxicity of the injected formulations was evaluated employing a propidium iodide exclusion assay. The PEGylated formulation composed of lipids with a high transition temperature (41°C) was the most stable, remaining intact even after two days within the cell. Interestingly, all liposome formulations appeared to be well tolerated by the cells, since the mortality did not exceed that of the buffer injected cells. The microinjection of liposomes is therefore a promising, non-toxic, easy-to-perform method to provide cells with new organelle-like entities, which, depending on the formulation, may show sufficient stability to be used in drug delivery applications.

30 BaMgF₄ as a Nonlinear Optical Ferroelectric Material for Whispering Gallery Resonators

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The present work investigates the suitability of monocrystalline barium magnesium fluoride (BaMgF₄) for nonlinear optics in whispering gallery resonators. The great potential of this material lies in the extremely large transparency range from deep ultraviolet (UV-C) to mid-infrared and in the possibility to phase match nonlinear optical processes due to alternating ferroelectric domain structures. The small nonlinear coefficient limits the efficiency of conventional continuous wave nonlinear optics. This drawback can be compensated by the concept of whispering gallery resonators.

A single crystal of BaMgF₄ was grown, oriented and characterized. An increasing optical extinction was found from the near infrared to the UV-C spectral range. This behavior was interpreted as volume scattering and was investigated further by using structure and element sensitive methods and scattering based calculations.

Whispering gallery resonators of c-cut BaMgF₄ were fabricated by hand and characterized optically. The intrinsic quality factors were measured in the blue and near infrared spectral range and lied at 2×10^6 and 10^7 as it would be expected from the extinction measurements.

A significant reduction of volume scattering can be expected by optimization of pulling rate and rotation speed at the Czochralski growth process. This would lead to higher quality factors of whispering gallery resonators and thus to the possibility of high efficient frequency conversion in the UV-C.

31 Cavity and Pendulum Challenges for Flexible Tilt Sensor

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Smart textile applications call for new technological developments in the area of flexible circuits and sensors in order to allow an unobtrusive integration of electronic functionality into everyday garments. Here, we present a novel mechanically flexible tilt sensor fabricated on a polyimide foil where conductive microspheres (acting as the pendulum) form an electrical connection between adjacent contact pads. In this way, a digital output signal, indicating the orientation of the sensor, is extracted without the need of additional evaluation electronics close to the device. The sensor is based on a sandwich structure where a cavity is formed between two polyimide foils structured with gold contacts and microspheres placed inside the cavity.

For the definition of the sensor cavity, SU-8 – an epoxy-based photoresist – is used to create a pillar structure (60 μm edge length, 40 μm spacing, 150 μm height) on the plastic substrate with the patterned contact pads. The pillars limit the sphere movement to the area of the contact pads while also acting as a spacer between the two substrate layers of the sensor. The pillar structure allows a mechanical bending of the substrate down to a tensile radius of 4 mm. As a pendulum, which follows the orientation of gravity inside the cavity, commercially available solder spheres with a diameter of 250 μm are used. These are sputter-coated with nickel/gold (2 nm/15 nm) in a custom-made vibration device to achieve a homogeneous coating to mitigate the thin oxide layer on the microsphere's surface. The surface resistivity of a single microsphere is thus reduced from $> 1 \text{ k}\Omega$ to around 30 Ω .

32 Brush-Hydrogels with Graded and Gradient-Like Properties

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The fabrication of organic coatings with well-defined structure and physico-chemical properties is of particular interest in several disciplines. Bio-organic layers on different length-scales, featuring variable composition, structure and modulus have been observed in several biological systems, such as human cartilage, mammalian skin and the nacre of oyster shells.^[1-3] These complex materials consist of mechanically graded structures that resist and respond to the external normal and shear forces, protecting underlying tissues from incurring damage. Among the many “natural” coating systems, human epidermis is constituted by different layers of cells which present diverse properties to confer resistance against abrasion.

In order to mimic these natural structures, materials scientists have been made numerous efforts to fabricate coatings with graded and gradient-like mechanical properties within a single film. Despite this, the fabrication of a full-organic, polymer-based coating architecture featuring nano-scale variations of properties still represents a challenging task.

In order to fabricate polymer films presenting discontinuous and continuous variations of mechanical and chemico-physical properties we applied sequential surface-initiated polymerization (SIP) of different monomer compositions to create polymer brush/hydrogel films with vertically defined structure. Specifically, poly(hydroxyethyl methacrylate) (PHEMA) brush and brush-hydrogel layered films were synthesized by surface-initiated atom transfer polymerization (SI-ATRP) in the presence of different concentrations of diethylene and tetraethylene glycol dimethacrylate (DEG/TEGDMA). Sequential SI-ATRP of monomer mixtures was employed to fabricate brush-hydrogel films presenting vertically graded properties. Alternatively, continuous variation of monomers during the SI-ATRP resulted in brush-hydrogels featuring gradient-like variations of polymer architecture through the film thickness. The chemical, mechanical and tribological properties of the films

were characterized by ellipsometry, FT-IR and colloidal probe atomic force microscopy (CP-AFM). All these measures confirmed the influence of polymer architecture on the properties of the films at specific depths.

Additionally, graded and gradient-like brush-hydrogels were used as reactors for the synthesis of polymer-inorganic hybrids presenting metal nanoparticles (NPs) embedded within the films. Depending on the vertical crosslinker content different NPs morphologies were obtained at determined depths. Through this multi-step fabrication polymer-inorganic hybrids with vertically defined structures and variable NPs loading were obtained. In conclusion we believe these methods will represent an easy and effective mean to form coatings with defined mechanical properties and tunable optical characteristics.

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33 Modeling of Microstructured Muscle Cell Actuated Sheets

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Muscle cell actuation of bio-mechanical systems has only recently been explored as approach for creating functional, biomimetic devices. Modern micro-scale fabrication techniques allow for the creation of complex structures, imitating the function of extracellular matrices and thus enable controlled cell growth and functioning. The combination of active biological parts with passive mechanical structures opens prospects for applications in myocardial tissue engineering.

This work investigates microstructured 3D printed sheets, optimized for unidirectional muscle cell actuation. Different unit cell geometries, capable of supporting growth, adhesion and contraction of adjacent cells, were characterized. Finite element simulations, homogenization technique, and analytical modeling were utilized to assess the structural response to adhesive muscle cell contraction. Periodic sheets with strongly directional Young's Moduli in ranges of 20 - 50 kPa and 800 - 850

MPa, suitable for muscle cell actuated deformation, were identified and fabricated.

34 Stimuli Sensitive Microcapsules with Macroporous Polymer Shells

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We demonstrate a novel method to obtain microcapsules with polymer shells of controlled macroporosity and mechanical properties that can be tuned within a wide range, with maximum loads between 800 and 2 mN. These capsules contain two liquid compartments: an aqueous core and an oil phase that permeates the macroporous polymer shell.

Microcapsules are produced by microfluidics, using a co-flow flow-focusing glass capillary device to make water-oil-water (W/O/W) double emulsion templates. A mixture of acrylate monomers (glycidyl methacrylate and ethylene glycol dimethacrylate) and porogens (phthalate-based, alkane or linear alcohol) is used as oil phase. Heterogeneous polymerization of the acrylate monomers leads to a biphasic structure in the capsule shell: a network of polymer beads permeated by the liquid porogen and eventually covered with a thin, tight polymer skin.

The size of the polymer beads and the presence or absence of the skin are adjustable by varying the composition of the monomer mixture, resulting in either open or close controlled porosity.

The presence of two thin polymer skin seals the interstitial liquid porogen providing a supplementary compartment that can be exploited as carrier for coencapsulating chemicals. Porogens with low boiling point, such as a short alkane, lead to thermosensitive capsules that can explosively release their content within seconds when exposed to a thermal shock.

Incorporation of glycidyl methacrylate monomers results in polymer capsules with epoxy-functionalized surfaces, which can be further reacted with amine-based functional compounds. We exploited such epoxy groups as anchors for grafting pH sensitive polymers and for creating magnetic capsules by covalently binding dopamine-coated iron oxide nanoparticles and polymer latex to the polymer shell.

35 Contact-free Probing of Electronic Transport Phenomena at the Nanoscale

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The observation of anomalous electronic transport at ferroelectric domain walls and its significance for nano-electronics triggered tremendous scientific interest. To date, the transport behavior and potential barriers at domain walls have been predominantly scrutinized by scanning probes. This, however, convolutes the intrinsic electronic properties with contact resistance and inhomogeneous probe fields, so that the detailed origin of the behavior remains obscured.

Here, we report on the capability of high-resolution X-ray photoemission electron microscopy (X-PEEM) to image and characterize ferroelectric domain walls contact-free and with nanometer resolution. In the ferroelectric semiconductor ErMnO_3 , we visualize ferroelectric domain walls by exploiting photo-induced charging effects and generate an electronic conduction map by analyzing the kinetic energy of photoelectrons. With this we open a pathway for non-destructive and element-specific studies of electronic and chemical domain-wall structures bypassing previous experimental limitations and significantly expanding the accessible parameter space.

36 Reaction of ^{29}Si -Enriched Ca_3SiO_5

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Hydration of silicates is widely studied in many different fields and, in particular, in cement chemistry, where tricalcium silicate (C_3S) reacts with water to produce a poorly crystalline calcium silicate hydrate (C-S-H) and calcium hydroxide (CH). For this industrially important mineralogical phase, we have studied step-by-step the mechanism of its hydration as well as the effect of aluminate ions on it, which amount is always increased in low CO_2 cements

where clinker is replaced by supplementary cementitious materials.

The use of ^{29}Si -enriched C_3S allowed relatively rapid ^{29}Si MAS NMR experiments, which together with the ^{27}Al NMR yield new insights into intermediate structures. In particular, it was possible to reveal the initial surface hydroxylation and to precise the following condensation mechanism. This work showed that solid state NMR can be used successfully not only to follow qualitatively the hydration of C_3S but also quantitatively. All experiments were done in-situ without perturbing the C-S-H structure by any drying technique.

As a source of aluminate ions NaAlO_2 solutions of various concentrations were used. Retardation of C_3S hydration was detected at early age of reaction but followed by increase in the amount of the hydrates that are formed at later stage. By ^{27}Al NMR we could observe in situ formation of different phases containing 4-fold (Al in C-S-H), 5-fold (Al substituting Ca in the interlayers of C-S-H) and 6-fold aluminum (AFm and TAH) at different time of reaction. Furthermore, NMR studies were complemented with isothermal calorimetry experiments.

37 Element-Specific X-Ray Resonant Phase Tomography at the Nanoscale

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Recent advances in fabrication techniques to create mesoscopic 3D structures have led to significant developments in a variety of fields including biology, photonics, and magnetism. Further progress in these areas benefits from their full quantitative and structural characterization. We present resonant ptychographic tomography, combining quantitative hard x-ray phase imaging and resonant elastic scattering to achieve ab initio element-specific 3D characterization of a cobalt-coated artificial buckyball polymer scaffold at the nanoscale. By performing ptychographic x-ray tomography at and far from the Co K edge, we are able to locate and quantify the Co layer in our sample to a 3D spatial resolution of 25 nm. With a quantitative determination of the electron

density we can determine that the Co layer is oxidized, which is confirmed with microfluorescence experiments.

38 Direct Net Gain Measurements in Organic Microcavities

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Microcavity lasers based on small organic molecules provide the opportunities for both applications, like for instance broad band tunable laser light sources in the visible range of the electro-magnetic spectrum and fundamental research, e.g. probing quantum effects that arise when light is confined to structures that are comparable in size to its wavelength.

For designing energy efficient microcavity lasers, knowledge about the gain spectrum of the optically active material is crucial. Unfortunately, the well-established gain measurement techniques are hardly applicable to microcavity lasers, where light is confined to length scales smaller than the light wavelength. In this regime, the environment influences the spontaneous emission properties of the gain material and therefore, the optical gain is changed.

In this work, a new gain measurement technique is presented that accounts for all small-size effects by measuring the gain directly in the cavity device. In contrast to all established techniques, the lineshape of the microcavity emission spectrum below lasing threshold is evaluated instead of the intensity of the output power. The obtained knowledge about the gain can be used to lower the lasing threshold by optimizing the cavity structure.

39 Mapping of Flow-Induced Nanostructures of Complex Fluids by Combining Microfluidics and Scanning-SAXS

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In situ measurements with high temporal and spatial resolution are necessary to understand the dynamics of macromolecules under flow. Variations in molecular shape and dimensions due to degradation/aggregation/self-assembly under shear, molecular alignment with flow and shear-banding are

some examples of transitions under flow. By combining microfluidics and scanning-SAXS, the nanostructure of complex fluids like polymeric solutions, surfactants, proteins and biological fluids can be observed and quantified. A flow-map of wormlike micelles measured with SANS shows the dependence of molecular alignment on shear and extension rates as a function of the volumetric flow rate. However, to gain in spatial resolution, the beam diameter had to be reduced, which increases the acquisition time of each pattern to about 10 minutes. On the other hand, the use of SAXS for flow-mapping allows measurements with higher spatial and temporal resolution. A newly developed microfluidic device (based on polyimide and glass) generated very low background scattering, resulting in strong signal from the samples, even in very thin channels (100 μm). The acquisition time was 0.1 s with a beam size of $40 \times 40 \mu\text{m}^2$, leading to much better resolution and data collection efficiency. One example is a wormlike micellar solution undergoing shear-banding at increasing volumetric flow rates. A color-code mapping of anisotropic scattering angle confirmed that the micelles align with flow in high shear/extension areas. Based on the evolution of the vortex formation (upstream), the influence of the fluid viscoelasticity can be determined as a function of confinement. The outlet region also has important information on the alignment of macromolecules in expansion flow.

40 Optimization of Redox-Stability and Electrochemical Performance of Ni-YSZ Anodes Based On Detailed 3D Microstructure Analyses

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Understanding the relationship between microstructure and effective materials properties is a key to purposeful design of electrodes for solid oxide fuel cells (SOFC). The electrode performance is influenced by the complex interplay between the different electrochemical and transport processes. Optimization of various electrode properties (i.e. high electrochemical activity, low charge transport resistances and high durability/long service lifetimes) is a challenging task for materials processing. In this

study we focus on the optimization of performance and redox stability of Ni-YSZ anodes.

Ni-YSZ anodes of varying microstructure (fine, medium, coarse) are fabricated and exposed to 8 redox cycles at 950 °C. Detailed image analysis of 3D microstructure obtained from FIB-tomography and the correlation with experimental data (i.e. polarization resistance, electrical conductivity) results to a complex phenomenological pattern of microstructure-performance relationship upon redox degradation. The initial microstructure has a strong influence on the performance and degradation. In the coarse sample redox cycling imposed a significant loss of percolation of YSZ, which can be attributed to lower sinter activity and weaker bottlenecks (compared to fine). In contrast, in the fine sample a strong alteration is observed for the Ni phase (i.e. loss of percolation, increase of tortuosity). This is explained by a higher density, which is reached during sintering of the fine material. Obviously the low porosity of the fine anode leads to a lower tolerance for Ni rearrangements.

New fabrication guidelines for improved redox stability of Ni-YSZ anodes can be proposed based on the findings of this study. Our investigations show that optimized anode microstructures result from a combination of fine and coarse powders in well-defined proportions, and addition of pore former. Moreover, 3D imaging and thorough topological analyses are necessary to capture the complex picture of microstructure degradation and its relation with effective properties and electrochemical activity.

41 Mechano-transduction through Soft Materials - High Precision 3D Analysis of Flow Patterns in Liposomes

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We present a novel method to study the transduction of external shear forces through liposome membranes and their effect on the enclosed lumen. Defocusing fluorescence microscopy is used in combination with high-precision 3D analysis of fluid flows outside and inside the liposome trapped inside a microfluidic channel.

Endothelial cells constitute the inner layer of our blood vessels. They are in direct contact to the blood

stream and thereby exposed to a variety of chemical and physical stimuli. The most distinguished physical stimuli is mechanical strain induced by external (blood) flow, hence shear stress. Its effect on the lipid membrane and its transfer into the lumen, a process referred to as mechanotransduction, remains unclear.

In this study, we used giant unilamellar vesicles (GUVs) as a model system for endothelial cells. Our method reveals the three-dimensional dynamics of membrane and luminal flows through the use of flow tracer particles in combination with fluorescence widefield defocusing microscopy. GUVs are trapped inside microfluidic channels using a biochemical cross-linking system.

Our analysis reveals bead movement along the GUV membrane in a bi-hemispheric pattern including rare crossing events between the hemispheres, independent of external flow speed. Bead movement in the direction of external flow occurs along the GUV top before it sinks down and moves back along the equator. At increasing external flow the luminal flow velocity increases linearly. Our results show that the mechanotransduction of shear forces through a GUV takes place at an efficiency of below 5%.

The addition of fluorescent tracers to the inner and outer side of the lipid bilayer may further elucidate the mechanisms of force transduction. Also, our system is applicable for investigations of the surface flows of other fluid interfaces e.g. in droplets microfluidics.

42 Design Principles of Perovskites for Thermochemical Oxygen Separation

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The separation and concentration of O₂ from gas mixtures is of great importance for various sustainable energy technologies, such as solar fuel production via thermochemical splitting of CO₂ and H₂O into CO and H₂, the feedstock for the synthesis of hydrocarbon fuels and chemicals. A rationale is introduced to design perovskites for oxygen separation via “thermochemical pumping” of O₂ against a pO₂ gradient with low-grade process heat. We show that the ideal oxygen exchange capacity of

perovskites can be determined from the activity of oxygen vacancies quantified with electronic structure computations. The predicted material properties are validated by thermogravimetric analysis and high-temperature X-ray diffraction for SrCoO_{3-δ}, BaCoO_{3-δ} and BaMnO_{3-δ} perovskites and Ag₂O and Cu₂O metal oxide references. These measurements confirm that the performance of SrCoO_{3-δ} - having an oxygen exchange capacity of 44 mmol O₂ mol⁻¹ SrCoO_{3-δ} and an oxygen exchange rate of 12.1 μmol O₂ min⁻¹ g⁻¹ at 600-900 K - surpasses the performance of state-of-the-art Cu₂O at the same conditions. We show how the presented redox trends can be understood due to lattice expansion and the magnitude of electronic charge transfer.

43 Probing Ferroelectric Domain Engineering in Thin Film Heterostructures by Optical Second Harmonic Generation

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The evidence of the electric field control on the antiferromagnetic ordering in multiferroic bismuth ferrite (BiFeO₃)^[1] increased interest in low energy consumption logic and memory devices. The use of solely an electric field to manipulate magnetic bits is indeed promising towards energy dissipation reduction.

However, to exploit such functionality for devices it is essential to attain deterministic control of ferromagnetism at the single domain scale. Therefore a ferromagnet/multiferroic heterostructure has been designed based on the combination of magnetoelectric coupling in BiFeO₃ (BFO) and exchange coupling between magnetic materials thus offering a new pathway for the electrical control of magnetism.^[2,3]

Here we show that second harmonic generation (SHG), can detect the distribution of ferroelectric domains in BFO thin films non-invasively and unimpeded by transport properties. We use epitaxial strain for engineering different types of BFO domain patterns that are characterized by SHG, showing a unique relation between the domain distribution and the film symmetry. We then manipulate the BFO film by voltage poling and demonstrate the sensitivity of the SHG process to this manipulation.

The concept applied to BFO is transferable to other multiferroics compounds thus indicating the general feasibility of SHG as a characterization technique for heterostructures in which buried ferroelectricity plays a key role in the emergence of magnetoelectric coupling.

[1] Zhao et al., *Nat. Mat.* **5**, 823 (2006). [2] Heron et al., *Phys. Rev. Lett.* **107**, 217202 (2011). [3] Trassin et al., *Phys. Rev. B* **87**, 134426 (2013).

44 Ultrafast Magnetization Switching by Spin-Orbit Torques

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Current-induced spin torques are of great interest to manipulate the orientation of nanomagnets without applying external magnetic fields. They find direct application in non-volatile data storage and logic devices. Recent demonstrations of perpendicular magnetization switching induced by in-plane current injection in ferromagnetic heterostructures have drawn attention to a class of spin torques based on orbital-to-spin momentum transfer (SOTs), which is alternative to pure spin transfer torque (STT) between non collinear magnetic layers and amenable to more diversified device functions. We will present advance made to build first perpendicular SOT-MRAM devices and ultrafast sub-ns magnetization switching by spin-orbit torques in 100 nm wide Pt/Co/AlOx dots with perpendicular magnetic anisotropy. By measuring the switching probability as a function of current pulse amplitude and bias field for pulses ranging from 180 ps to 10 ms, we show that deterministic bipolar magnetization switching is achieved down to the shortest current pulses without evidence of precessional magnetization switching. This makes SOT-based heterostructures a promising candidate for ultra-fast recording applications such as MRAMs and cache memories.

45 Impact of Chemical Doping on Domain Structure in Hexagonal Manganites

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The unique properties of magnetoelectric multiferroics are, to a large extent, determined by the coexistence and interaction of magnetic and electric domains. A major challenge towards future applications is to optimize the properties of these domains, such as their transport, without weakening or even losing the existing multiferroic order. Here, we present our study of ferroelectric and antiferromagnetic domains in chemically doped hexagonal manganites. We show that the electronic conductance of ErMnO_3 can be enhanced or suppressed by introducing either divalent (Ca^{2+}) or tetravalent (Zr^{4+} , Ti^{4+}) ions into the system. Using piezoresponse force microscopy (PFM) and optical second harmonic generation (SHG) we monitor the corresponding changes on the level of domains. We find that the RMnO_3 -characteristic domain topography, as well as the multiferroic transition temperature, are robust against the applied ionic alteration (up to 1%), which demonstrates the usability of chemical doping for non-perturbative property-engineering of multiferroic domains.

46 Magnetic Flux Pattern at Surfaces and Impurities in Chiral p -Wave Superconductors Revisited

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In recent theoretical work, Bouhon and Sigrist [PRB 90, 220511 (2015)] have investigated the band structure dependence of the edge states and currents in Sr_2RuO_4 using a self-consistent Bogolyubov-de Gennes approach. They have found that the relation between the current direction and the orientation of the surface, in combination with the position of the Fermi surface, is not as straightforward as assumed previously. For some situations, there may even be a current reversal.

Within a Ginzburg-Landau approach, we analyze in full detail the reversed current flow using the modified Ginzburg Landau coefficients calculated by

the microscopic theory, and including all screening effects. We consider both the geometry of a disk and a point impurity in the bulk. We also compare different surface scenarios, in particular diffuse and specular scattering.

We recover the previously presented results of reversed current flow. In addition, we discuss the detailed structure of the arising flux pattern. For the edge current around a disk with specular scattering, while the current changes direction between the 0° to the 45° direction for the case of high filling, its peak magnitude increases both compared to the case of low filling, and to the case of diffuse scattering.

47 Deterministic Control of Multiferroic Order at the Level of Domains

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Spin-spiral multiferroics exhibit a strong coupling between the electric and magnetic subsystems which is of potential interest for technological applications. Although these systems have been investigated for more than a decade, the magnetoelectric domain evolution under external fields is still largely unknown. Using optical second harmonic generation we resolve how electric and magnetic fields affect the multiferroic domains in the archetypal spin-spiral multiferroic TbMnO_3 . In consecutive electric switching cycles, varying multi-domain patterns emerge before a single-domain state is obtained. This observation reflects that the domain walls can easily move without being pinned by, e.g., structural defects. In striking contrast to the electric-field response, multi-domain patterns persist when the polarization direction is flopped by applied magnetic fields. Here, a uniform polarization rotation is observed within all domains, which incorporates a transformation of neutral into nominally charged domain walls. Our results are explained based on numerical Landau-Lifshitz-Gilbert simulations and provide first evidence for the scalability of macroscopic magnetoelectric properties onto the level of domains.

48 Magnetic Diffuse Scattering in Thermally Active Artificial Kagome Spin Ice

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Artificial kagome spin ice consists of mesoscopic single domain magnetic islands placed on the nodes of a kagome lattice and coupled together via magnetostatic interactions. This system has attracted considerable interest due to its complex magnetic phase diagram^[1] and moment excitations which resemble emergent magnetic monopoles.^[2] Up to now, static systems or systems with slow island moment dynamics were investigated using microscopy techniques.^[3,4] In contrast, by using scattering techniques, magnetic moment correlations can be studied in a much wider range of temperatures and moment fluctuation rates. Here, we apply X-ray Resonant Magnetic Scattering (XRMS) to look at zero-field magnetic correlations in thermally active artificial kagome spin ice with sub 100 nm islands. Using electron beam lithography and evaporation of Permalloy (Ni₈₀Fe₂₀) we produce in-plane ferromagnetic nanomagnets which exhibit superparamagnetic behaviour. Pure magnetic diffuse scattering, originating from kagome spin ice correlations, was observed at zero-field and RT in a thermally active kagome array. Experimental data can be understood using Monte Carlo simulations and subsequent numerical calculation of scattering patterns using kinematic scattering theory.^[5,6]

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49 Optical Investigation of Ferroic Domains beyond the Resolution Limit

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In recent years optical second harmonic generation (SHG) has been shown to be a versatile, non-destructive tool to investigate the often complex domain structures of ferroic and multiferroic materials. Ferroic domains vary broadly in structure and size, depending on the nature of the ferroic ordering. So far, however SHG was restricted to domains larger than the optical resolution limit of 1 micrometer.

Here we present a method by applying a numerical model and simulation to overcome this limitation and to analyze ferroic domain structures some orders of magnitude smaller than the optical resolution limit.

The method is based on the relation between the orientation of the ferroic order parameter and the phase of the nonlinear optical signal. It gives a relation between domain size and density, optical resolution and the intensity of the SHG signal. To show the reliability of the model, we applied it to several simulated domain structures. The simulation of the domain structures is based on an iterative geometrical algorithm, which allows us to generate complex domain patterns like the ferroelectric vortex structures or the irregular bubble like antiferromagnetic domains in hexagonal YMnO₃. The numerical calculations were compared with experimental data and found to be in excellent agreement.

50 The Bulk Monopolization in Diagonal Magnetoelectrics

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The magnetoelectric monopole - a term appearing in the expansion of the interaction energy of the magnetic field H with the magnetization density - is formally allowed to exist in materials with broken time-reversal and inversion symmetry and is related

to the response of diagonal magnetoelectrics. By exploiting similarities to the modern theory of polarization, we develop a theory of bulk monopolization and implement a Wannier function-based calculation in first-principles simulations. We compare the values obtained in the bulk picture to values obtained in a local moment picture and point out the relevance of each description.

51 Advanced Characterization of Helical Spin Structures and Domains in Skyrmion Systems

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Magnetic whirls, so-called skyrmions, emerge in various chiral magnets and attract tremendous attention due to their exotic properties. Skyrmions can, e.g., be moved at ultra-low current densities and give rise to the topological Hall Effect. Much less is known about the magnetic phases that surround the

skyrmion state, including fundamental aspects such as the domain formation and spin-defect interactions. The fragmented knowledge is partially due to the challenging experimental access and the general difficulty to image antiferromagnetic order at the nanoscale.

Here, we show how helical magnetic structures in the skyrmion system FeGe emerge and transform as a function of temperature and magnetic field. We discuss the magnetic field-driven response of multi-domain states, the influence of defects, as well as fingerprints of spontaneous jump-like movements of the periodic spin arrangement. The results are gained near RT using magnetic force microscopy (MFM) and diamond nitrogen-vacancy center microscopy. Besides providing new insight to the physics of skyrmion materials, our results foreshadow a promising pathway for measuring complex spin textures with high spatial and temporal resolution.

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