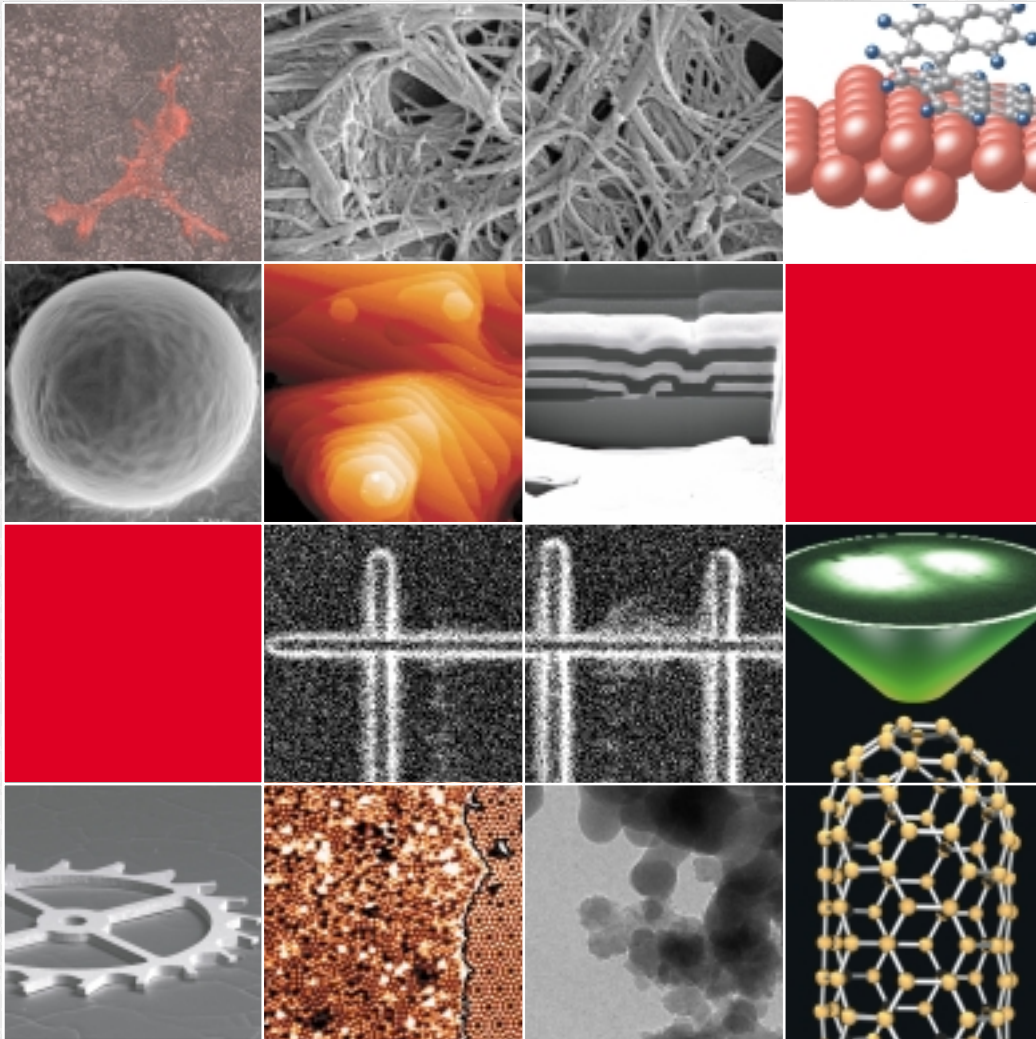


# ***Materials Design in the Nanometer Range***



100 nm

Dear Reader

The present leaflet provides a comprehensive summary of EMPA's activities in nanoscience and technology. Specific areas of research and development as well as our special equipment for the investigations of nanomaterials are portrayed.

Our labs in Dübendorf, St. Gallen and Thun, where collaborators engage in nanoscience, constitute EMPA's «Nanotechnology Competence Center» within the ETH Domain.

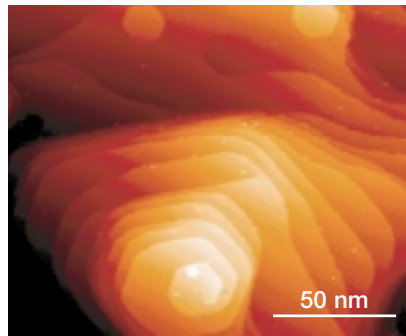
We wish you an interesting voyage of discovery.

With our compliments  
[Nanotechnology@empa](mailto:Nanotechnology@empa)

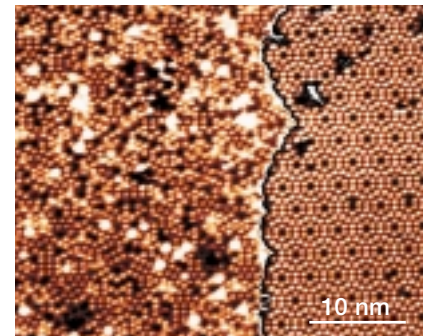
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Screw dislocation on Cu(111) surface



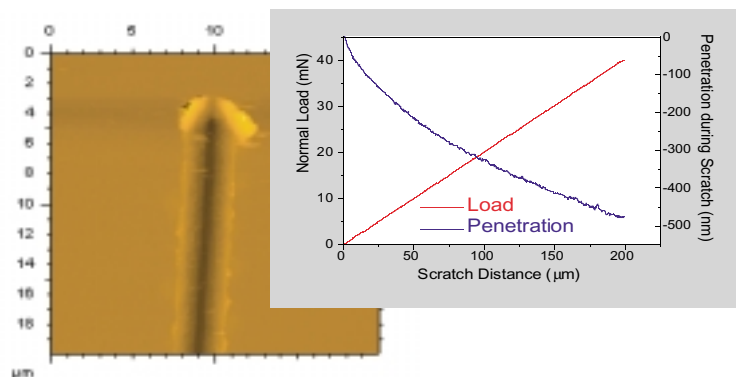
Ordered and disordered phases on Si(111) surface

Scanning Probe Microscopy (SPM) techniques have become important methods for the investigation of surfaces on the atomic level. In surface science and technology, EMPA makes use of different Scanning Tunneling Microscopes (STM) and Atomic Forces Microscopes (AFM) operating in ultrahigh vacuum, air or liquid. The most advanced instruments are the variable temperature STM (VT-STM) and the low temperature STM

(LT-STM). The VT-STM with an accessible temperature range from 25 to 1500 K is ideal for the study of dynamic processes on conductive surfaces (e.g. adsorption, diffusion and segregation). The LT-STM operates between 4 and 80 K and features the extremely high position stability (drift  $\sim 0.3$  nm/day) needed for the investigation of the local electronic structure on surfaces and single molecules.

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Scratch experiment on a 500 nm thin PZT-film on Si: Load and penetration as measured during the scratch (right) and image of residual scratch measured with AFM (left): No brittle failure is observed

Mechanical properties at small length scales differ from the properties on macroscopic scale. Within various research and PhD projects, EMPA puts special emphasis upon characterizing the mechanical properties of thin films (e.g. piezoceramics, hard coatings) and devices for micro-electromechanical systems (MEMS) using a nanoindenter (load range: 100 nN to 500 mN, load resolution 50 nN, depth resolution better than 1 nm). The

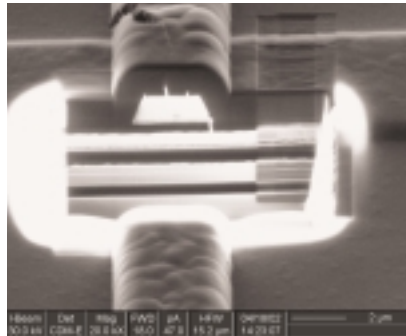
scratch option of the nanoindenter including lateral force measurement allows the study of film adhesion, tribological aspects as well as nanopatterning of surfaces.



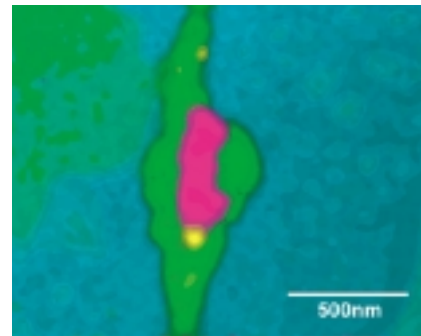
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Micro-cross section of a semiconductor device



STEM EDX mapping of precipitates in a Ni-base superalloy

The Focused Ion Beam (FIB) has become an important tool for micro/nanostructure modifications, materials analysis, and cross section analysis. It performs semiconductor device modification as well as local micro-cross sectioning. A variety of process gas chemistry allows local material deposition and selective milling. EMPA operates two state-of-the-art complementary FIB systems. One is especially suited for large, flat samples like semiconductor wafers, the other one operates

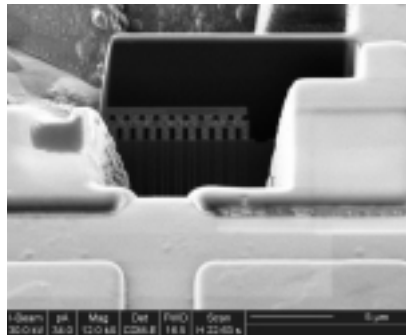
mainly for a precise TEM lamella preparation and includes a high-resolution electron microscope useful e.g. for post-FIB inspections of nanolayers.

The high-resolution imaging and analysis TEM is located close to the FIB systems. It can be operated in the scanning mode, too, and has a line resolution of 0.14 nm. Element information from B to U can be obtained by an energy dispersive X-ray analysis (EDX) system.

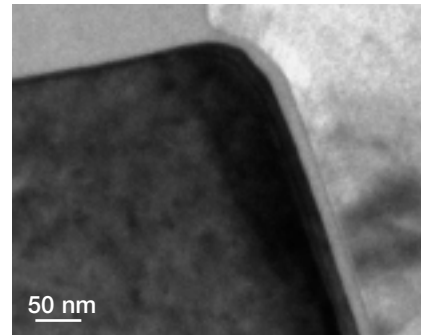
Reliability of Nanostructures

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Snapshot of a TEM lamella preparation with FIB



TEM bright field image of a 5 nm thick oxide layer

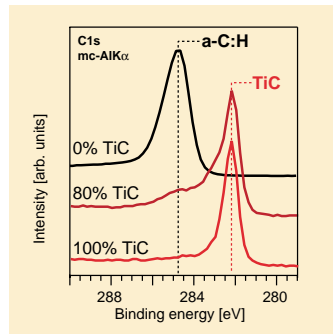
High reliability is often claimed for nanotechnology products although many projects are still exploratory where it seems difficult to apply reliability theory. But ignoring fundamental limits may lead to a dead end. Failures of nanosystems can be treated as stochastic processes or by their physical failure mechanisms. Reliability estimates of molecular, solid-state or any other system with nanosized functional elements have to consider

thermal fluctuations, quantum statistics and Heisenberg uncertainty relation resulting in contradictory requirements for minimum energy level separation of states, operation frequency and packing density. Failure analysis at the nanoscale is based on high-resolution equipment for preparation and imaging.

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XPS analysis of a nanocomposite coating consisting of nanocrystalline TiC with amorphous carbon (aC:H)



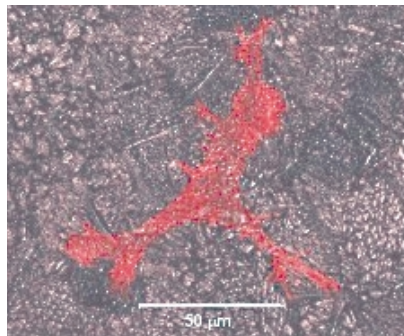
Scanning electron microscope picture of an anchor wheel coated with diamond like carbon containing ceramic SiC nanoclusters

Improved coatings are developed to fulfil the demand for lower friction, a longer lifetime, suppressed diffusion, desired biological behavior or a better thermal stability. Special emphasis is given to research on upcoming functional coatings, such as PZT, DLC, and new application fields, such as textiles and implants. The different properties of a coating are tuned to the desired range by alloying with adequate elements as well as by depositing nanoscaled multilayer coatings or isotropic nanocomposite coatings.

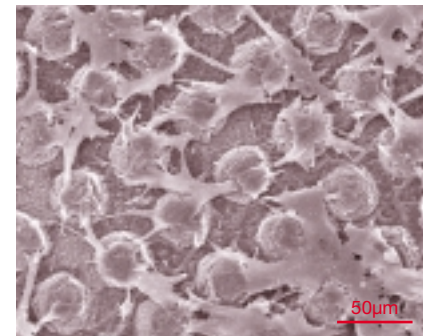
Thin films with specific properties are deposited by our different PVD and PACVD processes which include the possibility of band coating. Surface analytical methods such as XPS, XRD as well as TEM are used for thin film development and research.

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Confocal laser scanning microscope picture of an actin stained fibroblastic cell on a sandblasted surface



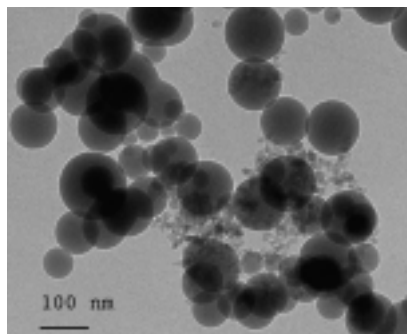
SEM-picture of a rat bone marrow cell culture on a nanostructured surface

Large efforts are performed to fulfil the clinical demand in replacing non-functioning tissues by implants or to support their regeneration. To meet these demands, we develop, design and improve implants and scaffolds by elucidating the surface features that steer cell behavior. Surfaces are characterized by their structure and protein binding capacity and patterns. Performance and behavior of primary human and

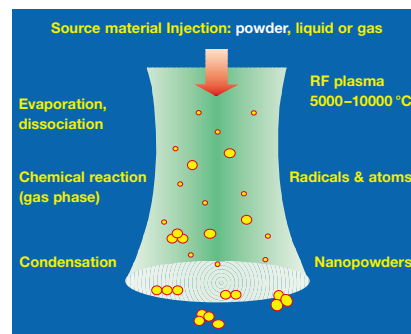
animal cells and of cell lines are assessed by biochemical and microscopical means. Part of our efforts is to engineer autologous tissues.

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Non-aggregated silica nanoparticles produced by flame synthesis



Condensation of nanoparticles in a radio-frequency plasma (RF plasma)

At Empa, oxide nanoparticles such as SiO<sub>2</sub> and TiO<sub>2</sub> are synthesized in flame reactors by high-temperature oxidation of metal-organic precursors. The plants allow scientific parameter studies as well as a powder production up to 2500 g/h. In addition, high-purity, non-oxide nanoparticles are produced in an inductively coupled high frequency plasma generator (30 kW). The materials investigated are Nitrides, Borides, and

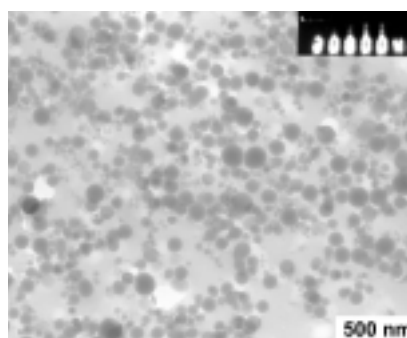
Carbides. Even pure nanometals for highly catalytic reactions (e.g. pyrotechnics) as well as ferrofluids (Co, Ni, etc) can be produced in industrially relevant quantities.

## Polymeric Nanocomposite Coatings and Microencapsulation

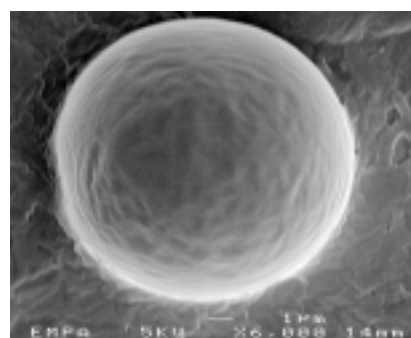
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Nanocomposite consisting of silica nanoparticles embedded in a methylacrylate matrix



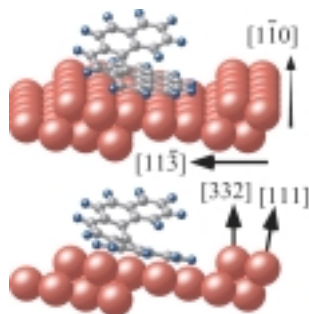
SEM-picture of a microcapsule made of biopolymer PHA

Nanoparticles embedded in a polymer matrix or coating provide added values like scratch resistance, easy-to-clean properties or tailor made optical properties. In contrast to microparticles, homogeneously and completely dispersed nanoparticles will improve the mechanical properties of their polymer host matrix; very interesting perspectives lie furthermore in electric, electrochemical as well as optical properties.

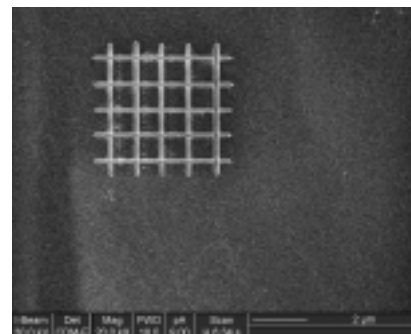
Polymeric microcapsules filled with active agents, ranging from <1 up to 1000 µm diameters, bring even more opportunities. Normally, they are spherical or roughly of the shape of the enclosed material. Shell materials are selected according to the physical properties of the core (solid, liquid or gas) respectively the application in mind (e.g. controlled release systems, masking, drug delivery).

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Alignment of helical aromatic hydrocarbons at mono atomic steps



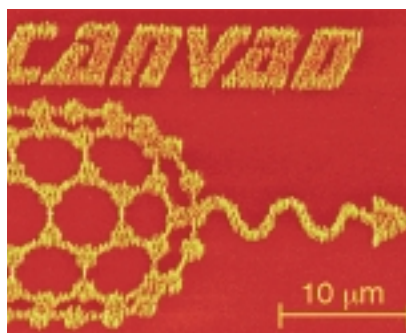
Structuring of Langmuir-Blodgett Films using the Focused Ion Beam equipment

Ordered organic thin films are central to the processes of living systems, catalysis, surface chemistry, and sensors. These films are routinely used in applications like inhibitors, binders, imaging media, adhesives and surfactants. EMPA has been involved in research and development in areas of supramolecular chemistry and organic thin film technology of highly ordered structures. Our research focuses on the bottom-up

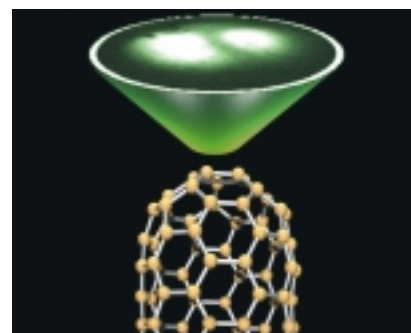
(self-assembly) approach to fabricate functional organic thin films with unique optical and physical properties, such as supramolecular functional materials and interactions of chiral molecules with surfaces.

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Microstructured carbon nanotube film



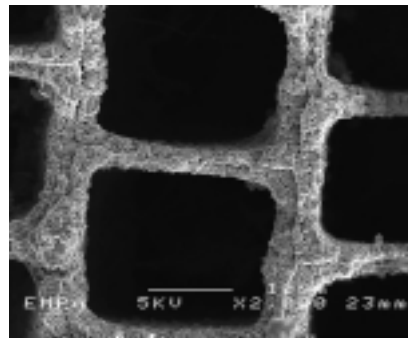
Electron emission spot from one single carbon nanotube

Information processing devices under the next paradigm will have switching frequencies of more than 1 THz and will integrate more than  $10^9$  devices/mm<sup>2</sup>, which indicates that the next device generation should be nanometer scale – the scale of molecules.

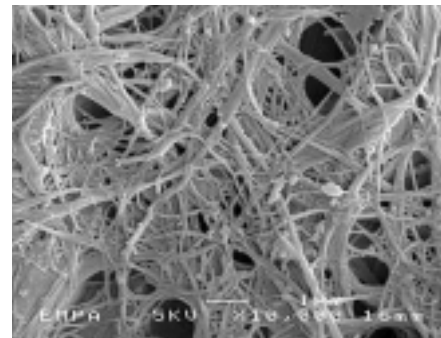
Carbon nanotubes provide an example of the challenges and opportunities associated with nanoscale materials. They show great promise for creating transistors or cold electron sources for vacuum electronic devices as traveling wave tubes or data storage devices.

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Ceramized pine structure



Separated cellulose fibril-bundles

The outstanding tensile strength of wood can be explained by cellulose-nanofibrils of the wood cell, embedded in a lignin matrix. Such separated single nanofibrils (10–100 nm) and fibril-bundles can also be used for polymer reinforced materials.

These wood structures can be converted by a pyrolysing process to carbon templates with micro- and nanopores and consequently with a high specific surface area. By infiltrating

the carbon templates with a silica sol they can be converted to a highly porous SiC ceramic by an in-situ carbothermal reaction, preserving the original pore structure of the wood. Due to the anisotropic and nanosized cellular structures, which so far cannot be initiated by artificial means, such materials are attractive for different applications like catalysts, filters or pre-forms for composite materials by infiltration with metals (MMCs) or pre-ceramic precursors (CMCs).



*For more detailed information on our activities please contact our home page or ask for a hand-copy of the reports «Activities 2001», «Annual Report 2001» (in German, English or French).*

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