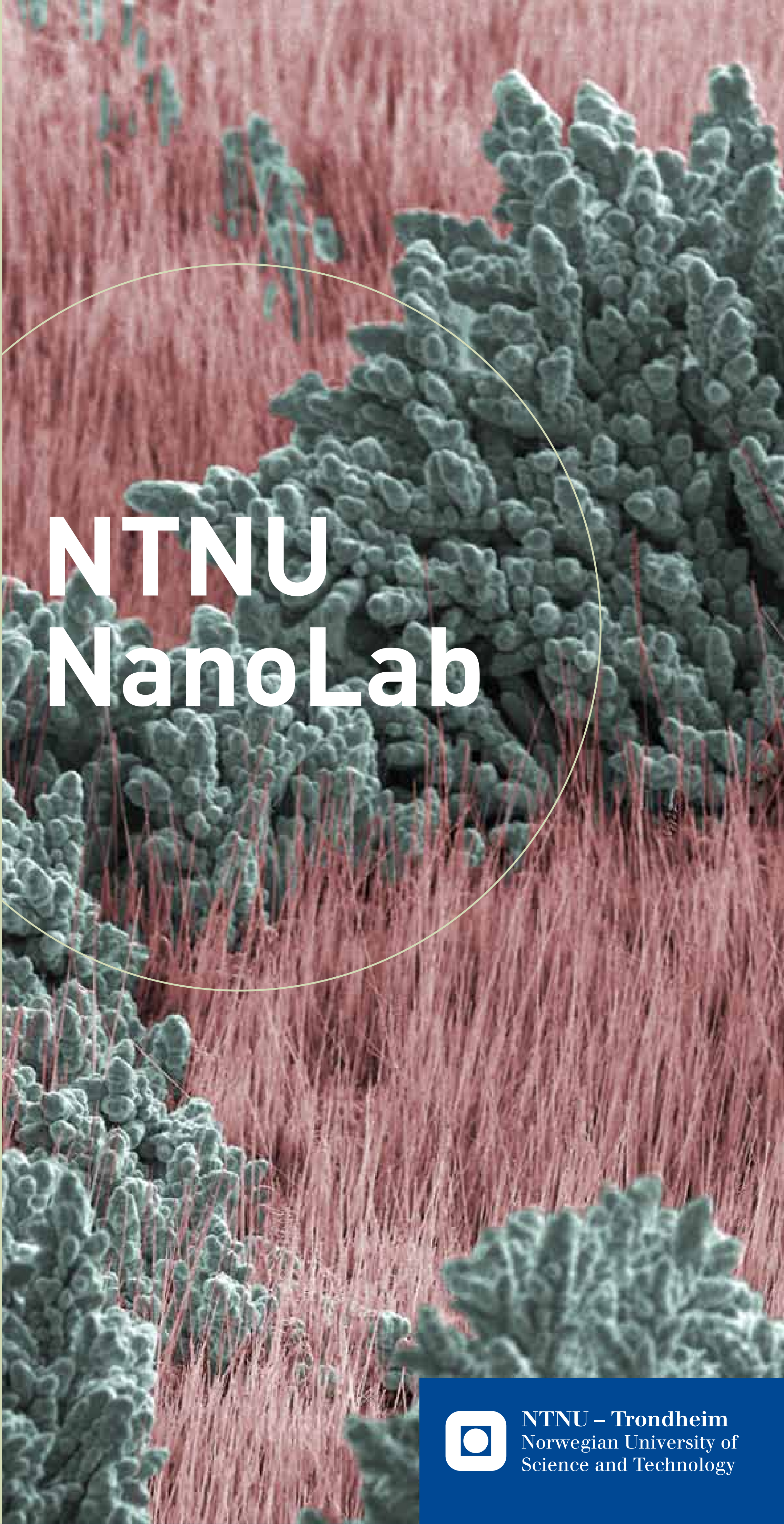
NTNU NanoLab

Annual report 2009



NTNU – Trondheim
Norwegian University of
Science and Technology

About NTNU NanoLab

NTNU NanoLab was established by the Board of NTNU in 2004 as the university's coordinating initiative within nanoscience and nanotechnology. The mission given to NTNU NanoLab was to:

- coordinate nanotechnological research and capital investments at NTNU
- build, equip and operate a new *state-of-the-art* laboratory for nanotechnological research and education
- promote a new MSc program in nanotechnology

The initiative currently comprises departments at five faculties: The Faculty of Natural Sciences and Technology, The Faculty of Information Technology, Mathematics and Electrical Engineering, The Faculty of Engineering Science & Technology, The Faculty of Medicine and The Faculty of Arts and is run in close cooperation with SINTEF.

Visit us at www.ntnu.no/nanolab/

Nanoscience and nanotechnology at NTNU

At NTNU, research related to nanoscience and nanotechnology takes place at 10 departments connected to 5 faculties.

Registered staff with running nanorelated projects in 2009:
Professors and Associate professors: 59
PhD students: 104
Post doctors: 42
Researchers: 11

Funding of nanorelated projects in 2009 (excluding salaries to staff members):
NTNU: 20 MNOK
The Research Council of Noway: 60 MNOK
EU: 12 MNOK

Annual Report published by NTNU NanoLab

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Editor: Hanna Gautun

Front page illustration: FEGSEM micrograph of CuO nanowires (red) grown around dendritic structures produced by copper electrodeposition under hydrogen evolution (turquoise). The nanowires only form if a several micrometer large layered system of CuO, Cu_2O and Cu is present at the sample surface. Thus, nanowire growth can be inhibited on areas covered with small structures. Such structures can be produced within the copper based system itself, e.g. by electrodeposition under hydrogen evolution. The method, which is suitable for decorating comparatively large areas with CuO wires with diameter of approximately 100 nm, has been developed in the Group of Bionanotechnology. Photo: Florian Mumm, Department of Physics, NTNU.

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NTNU NanoLab 2009

2009 was an exciting year at NTNU NanoLab. The construction of our cleanroom facilities was completed, and we saw how new processes and scientific tools were put to work for research and development projects. To be sure, there are still some challenges before the complex infrastructure is operated to its full potential, but we can confidently say that the year 2009 marked the transition of our attention from construction to research.

Operationally focus has been on establishing routines aimed at an open access cleanroom and procedures for safe and traceable use of the infrastructure with minimum extra administration. Parallel to these processes, installation of a large number of advanced scientific tools and instruments have taken place.
In line with this, the second significant effort has been to build a competent engineering staff and user community together to support strengthening of the competence. Thus, we are currently filling the positions to accommodate five qualified service engineers that can support the use of the infrastructure. To extend the dedicated process development, three post doctors are working on process building to provide a good basis for collaborations in the future.

At the national level, NTNU NanoLab has during the last year been instrumental in the application for establishing the Norwegian Micro- and Nano-Fabrication Facilities (NMNF), through a collaboration of the cleanrooms at NTNU, MiNaLab (The University of Oslo and SINTEF) as well as Vestfold University College. This initiative will provide for good value chains for outcomes from the research at NTNU. It will constitute one of the major means with which NTNU NanoLab can fulfil its purpose.

Another arena where NTNU collaborates with SINTEF, UiO and Vestfold University College is the Norwegian PhD Network on “Nanotechnology for Microsystems”, which was granted financial support by the Research Council of Norway from 2009 – 2012. NCE Micro- and Nanotechnology in Vestfold is an associated partner to this network. The goal of the “Nanotechnology for Microsystems” PhD network is to establish a strong national network for graduate studies in applied physics and microsystems through the introduction of nanotechnology. The leadership of the network is located at the Department of Electronics and Tele communications at NTNU.

NTNU NanoLab is also a strategic effort, with a role of supporting and merging of the related topics of the strategies of different faculties internally at NTNU. During 2009 the strategy of NTNU NanoLab was revised, to account for developments at NTNU in addition to the completion of the infrastructure. A result of this has been a marked increased activity in networking, with the regular seminar series and focused workshops that form starting points for fruitful collaborations.

NTNU NanoLab is a meeting place for interdisciplinary work in nanoscience and technology. It is intended to act as a catalyst for research at NTNU. We are developing arenas for collaborative efforts, ranging from seminar, work-shops and courses to an effective strategy well anchored within the university. At the end of 2009 NTNU NanoLab had approximately 120 registered users, and over 40 different approved activities, a promising start for vivid university collaborations.

Erik Wahlström
Director of NTNU NanoLab

Opening of NTNU NanoLab's Cleanroom

The official opening of NTNU NanoLab's entire cleanroom facilities took place on the 12th of May 2009. The ceremony was, for practical reasons, arranged in lecture hall R2 in the Natural Sciences building at NTNU where Rector Torbjørn Digernes greeted the 115 participants. State secretary of the Ministry of Education and Research, Åsa Nelvik, then formally declared the cleanroom open by cutting a nanoscale "ribbon" by operating the focused ion beam (FIB) machine. The process was followed by the audience through an on-line connection between the FIB and a screen in the auditorium. Acting leader of NTNU NanoLab, Erik Wahlström followed up by giving a presentation of the new facilities.

Acting director of NTNU NanoLab, Eric Walström observes as State secretary of the Ministry of Education and Research, Åsa Nelvik formally opens NTNU NanoLab's cleanroom.

The programme then progressed with presentations by invited speakers:

- Dag Høvik, Special advisor of the NANOMAT programme of the Research Council of Norway, *Nanotechnology in Norway - where are we and where are we going.*
- Knut Fægri, Dean of the Faculty of Mathematics and Natural Sciences, University of Oslo (UiO), *Perspectives on the future of materials science and nanotechnology at UiO.*
- Unni Steinsmo, Director SINTEF, *Nanotechnology - an enabling technology. Enabling what?*
- Hans Mooij, TU Delft, The Netherlands, *Coherent quantum processes in nanofabricated electronic circuits.*
- Eleanor Campbell, University of Edinburgh, Scotland, *Carbon nanotubes: past, present and future.*
- Roar Haugland, Scatec, *Development of Norwegian nanotechnology industry -the Scatec perspective.*

Acting director Erik Wahlström gives a guided tour of the cleanroom on the Opening day.

During the lunch break the invited guests were given a short guided tour of the cleanroom.

After the presentations everyone gathered in NTNU NanoLab's lecture area between chemistry buildings 1 and 2 for refreshments and inspection of the 20 posters presented by local contributors. The event was rounded off with a formal dinner at Lerchendal Gård in the evening.

NTNU NanoLab's cleanroom

The construction of the cleanroom facilities has been one of the core efforts of NTNU NanoLab since the start-up in 2004. The planning was carried forward by three committees in conjunction with NTNU's Technical Division. The first part to be built was the cleanroom area for chemical methods, which was opened for users in 2007. The planning of the larger clean-room area has been an ongoing process from 2005.

Great emphasis has been put on designing a flexible infrastructure that may easily be adapted to future demands. In addition to specialized equipment, the facility includes general support laboratories, offices and meeting areas. The construction work was finished in December 2008 and the complete facility will be commissioned during the spring of 2009.

The entire cleanroom area covers over 700 m^2 and is designed to be GMOII:2 compatible. The facility is organized in several zones equipped for various purposes:

- Area for chemical methods
- Area for physical methods
- Area for bionanotechnological methods
- Area for characterization
- Area for education

These areas will hold cleanness between class 10.000 and 100 and offer vibration free zones down to

VC-E. The laboratory will be furnished with state-of-the-art equipment for nanotechnological research in prioritized areas, complementing the facilities of SINTEF's MiNaLab in Oslo.

The processes available in the cleanroom include:

Chemical synthesis:
- Wet chemical methods (dip / spin / spray coating)
- Hydrothermic synthesis (high temperature ovens and autoclaves)
- Nanoparticle separation (centrifuges, ultrasound)

Nanoscale characterization:
- High resolution scanning electron microscope (STEM)
- High resolution focused ionbeam etch combined with scanning electron microscope (FIB)
- Atomic force microscope (AFM)
- Profilometer
- Scanning electrochemical microscope (SECM)
- Equipment for determination of particle sizes down to 2 nm
- Teaching STM and AFMs
- Particle size determination

Synthesis and characterization of thin films:
- Sputter deposition, Plasma enhanced chemical vapour deposition (PECVD), metallization by electron beam evaporation.
- Lithographic methods (FIB, UV-lithography, electron beam lithography (EBL), nanoimprint).
- Wet and dry etching (reactive ion etch (RIE), inductive coupled reactive ion etch (ICP-RIE), plasma etch.
- Teaching UV-lithography.

In addition to the cleanroom areas, the infrastructure includes supporting and technical facilities, laboratories for chemical and biological work, as well as offices, meeting rooms and areas for seminars. A fibre drawing tower will also be installed in conjunction with the laboratory.

The laboratory is open to all researchers interested in nanotechnology at NTNU, SINTEF and other Norwegian research organisations. In addition, masterstudents will be admitted in connection with project and diploma work. A half day "Introduction course in HSE and routines for work in the cleanroom" is mandatory in order to gain access. Approximately 120 users were accredited to the cleanroom and around 40 activities were approved in 2009.

Discussing the advantages of a cross-disciplinary cleanroom in the chemical area.

Highlights

Thin-film deposition by sol-gel technology

Thin films are frequently deposited onto bulk materials to change the mechanical, chemical, electrical, optical or other properties of the materials surface. Mechanical properties like wear and scratch resistance are increased by thin films e.g. to improve the energy efficiency of cars. The chemical properties can be altered by designing thin films with hydrophobic (water repellent), anti-fogging or antimicrobial properties. The optical properties that can be changed include anti-reflection, opaqueness, fluorescence or light trapping and hence thin films can be designed for electronic displays and solar cells in order to achieve better performance. Desired properties can be obtained by tailoring the composition, structure and morphology of the thin films. To achieve this, fundamental understanding of the physics and chemistry of the thin film deposition is essential.

The deposition method chosen often determines the properties of the deposited film. Cost, environmental effects, compositional purity and structural control are important criteria for the selection of the deposition method. The sol-gel method is widely used for preparation of thin films due to compositional homogeneity of the deposited film and the ease of up-scaling. Low cost and minimum environmental hazard make the method even more attractive. However, controlling the structure of the film as it builds up is more challenging. A schematic illustration of the dip-coating process for thin-film deposition is shown in Figure 1. A substrate is dipped into a sol composed of nanoparticles dispersed in an aqueous or non-aqueous solvent. The gel structure forms upon withdrawal of the substrate as a result of surface interactions between the nanoparticles when the solvent is evaporated. Interactions between the particles can be repulsive or attractive during deposition either rendering a dense arrangement of the particles or the formation of a network of aggregates. Porous or dense, smooth or rough films can be prepared by changing the chemistry of the sol and thus, the surface interactions between the particles. Evaporation of the solvent develops high capillary stresses in the nanosized pores between the nanoparticles. A major challenge using the sol gel method is to deposit thin films without any drying cracks. This can be accomplished by tailoring surface interactions to attain a strong structure that can withstand the drying stresses.

Figure 1. Illustration of thin-film formation by dip-coating from a sol of nanoparticles.

The aim of this study is to improve the fundamental understanding of the basic processes and forces that determine the success or failure of the preparation of defect free thin films based on nano-sized particles. In particular, the study will focus on the thin-film formation, i.e. a primary study of the build-up of the first nanoparticle monolayers on a substrate (silicon wafer) and a basic study of the interactions between the sol particles and the first adsorbed layers. The project is a collaboration between SINTEF Materials and Chemistry, The Inorganic Materials and Ceramics Research Group at the Department of Materials Science and Engineering (IMT), NTNU and Université de Franche-Comté (UFC), France. The work is performed by PhD student Hasan Güleryüz supervised by Professor Mari-Ann Einarsrud, Professor Tor Grande, Senior Scientist Ingeborg Kaus (SINTEF) and Professor Claudine Filiâtre (UFC). The Inorganic Materials and Ceramics Research Group at IMT has for about two decades been using the sol-gel method for various applications.

Initial stages of the thin film deposition of silica nanoparticles onto silicon wafers are monitored through *in situ* gravimetric measurements. The deposition behavior of the thin films is dependant on the sol chemistry. The effect of changing the sol pH from 6 to 10 is shown in Figure 2.

The deposition of the films shows that surface interactions have decisive influence on the transport characteristics and the deposition behavior of the particles. The conditions that promote aggregation (low pH) in the sol also enhance deposition on the substrate. However, aggregation reduces the mobility of the particles leading to a slower deposition rate.

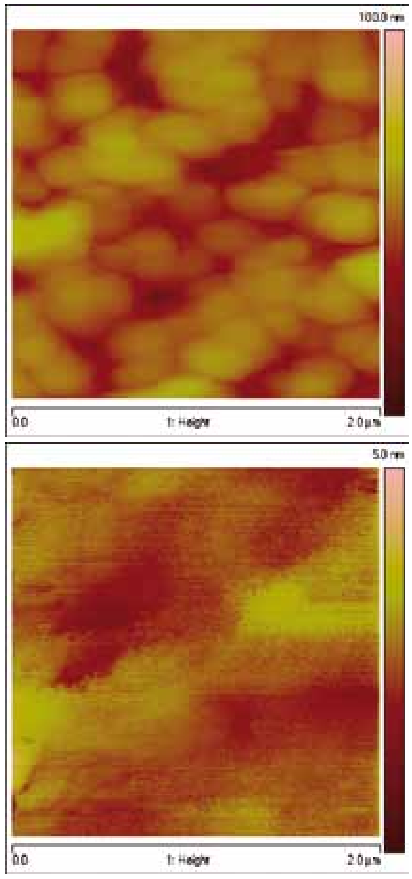


Figure 3. AFM images of silica thin films deposited from sols at (top) pH = 10 and (bottom) pH = 6

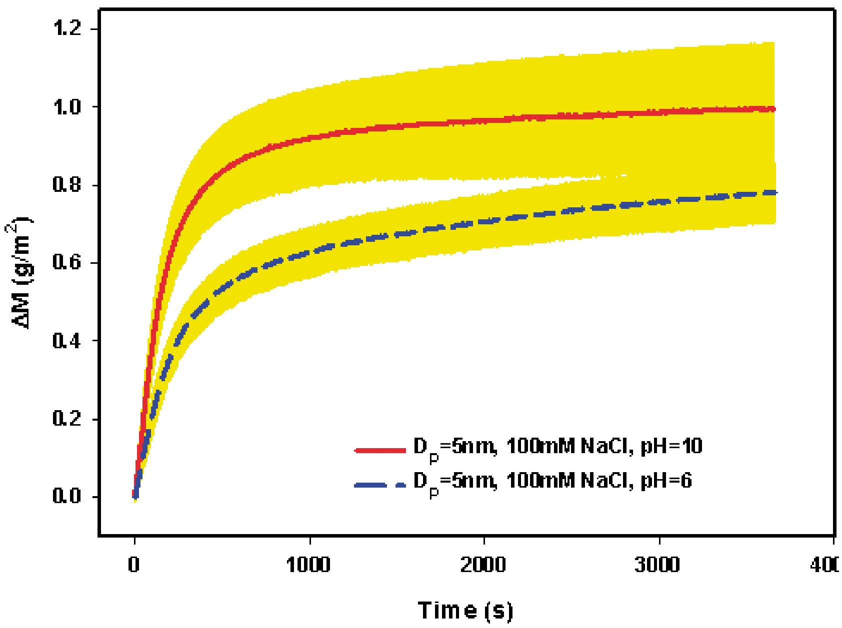


Figure 2. Mass change of silicon wafer immersed in silica sol measured as a function of deposition time at pH=6 and pH=10.

PhD student Hasan Güleryüz recording AFM images in NTNU NanoLab's cleanroom.

Atomic force microscope (AFM) is utilized for characterization of the deposited and dried films. Contact mode scanning carried out in a liquid cell reveals the fragile features dictated by the deposition conditions as shown from the topographic images in Figure 3.

The topography of the films is clearly affected by the pH of the sol. The rigid and rough structure formed at pH=10 is strong enough to withstand drying stresses. On the contrary, the soft and compliant structure formed at pH=6 collapses during drying and the film topography becomes smoother. The deposition kinetics is distinct for different deposition conditions imparting information on the film structure (Figure 2). Thus, analysis of deposition kinetics and characterisation of the sols and the films by means of associated chemistry makes it possible to estimate the structure of the deposited film.

Nanomechanics of Ugelstad polymer particles for novel applications

Thanks to the invention of the activated swelling polymerization method by the late Professor John Ugelstad, we are in a unique position for manufacturing micron-sized polymer particles with an extremely narrow size distribution. For a number of years the Ugelstad method has been successfully industrialized for use within pharmaceutical and biotechnological fields. Recently, there has been a renewed interest in exploiting this polymer particle technology towards new applications in the manufacturing of microelectronics and microsystems. One example of application of micron-sized polymer particles has been the development and manufacturing of ultra-thin Liquid Crystal Display (LCD) by means of Anisotropic Conductive Adhesive (ACA) technology.

In ACA application, metal coated polymer particles, which usually consist of a polymer core of micrometer size, a 30-50nm thick Ni inner layer and a 15-30nm Au outer layer, are used to replace the compact metal particles. To achieve a low contact resistance, the mechanical behaviour of the individual particles under large deformation is a key issue. However, due to small volumes, spherical geometry and the composite structure of the particles, methods for measuring the mechanical properties by both particle manufacturers and other R&D institutes have been highly unreliable, with a large scatter of results. The goals of this project have been to develop a nanoindentation based method suitable for characterization of polymer and composite particles, to establish a link between the mechanical properties and microstructures and further apply this knowledge to design particles of specific mechanical properties, as shown in Figure 1.

A unique nanomechanical methodology, nanoindentation based flat punch technology, has been developed at NTNU Nanomechanical Lab, including methods for individual particle dispersion and mechanical characterization. Using this technology we were able, to obtain for the first time, highly reproducible mechanical data for individual micron-sized polymer particles and determine the mechanical properties of a single particle with nanoscale accuracy.

Both polymer and metal coated composite particles (as shown in Figure 2) with various chemical compositions, crosslink densities, sizes and loading conditions have been studied using the nanoindentation based flat punch methodology. All the particles are amorphous and have a crosslinked structures. The contact load-displacement relationship of single micron-

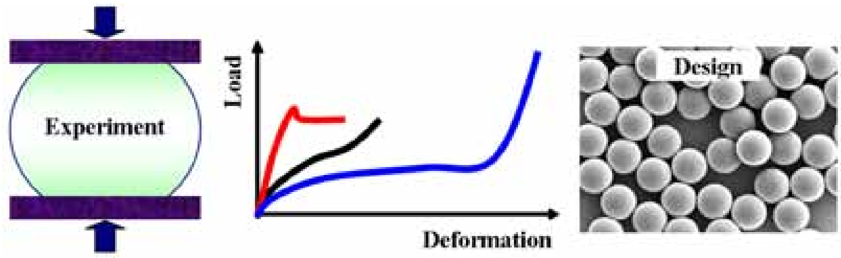


Figure 1. The three interrelated objectives in this work.

sized particles has been recorded and the stress-strain behaviour has been determined. It has been shown that the slightly crosslinked polystyrene particles display a yielding behaviour, and smaller surface cracks have been observed after deformation. However, the strongly cross linked acrylic and polystyrene particles show a brittle fracture behaviour.

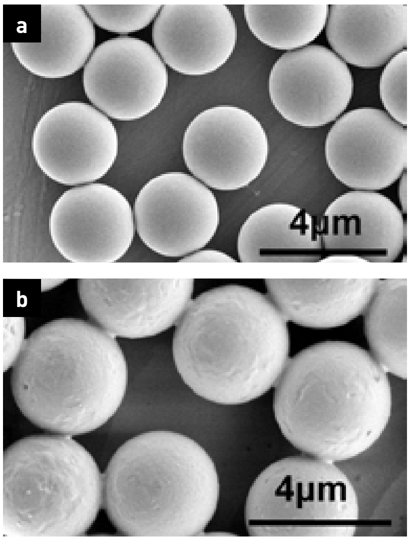


Figure 2. The micrographs of (a) polystyrene based particle and (b) Ni/Au coated acrylic based particles.

A striking particle size effect on the mechanical properties has been discovered. The slightly crosslinked polystyrene particles with same chemical composition but different sizes show that the smaller the particle diameter is, the harder the particle behaves. The compressive deformation rate also influences the particle size effect. For the larger strain rate the size effect is getting more pronounced, as shown in Figure 3. The corresponding mechanisms of the particle size effect have been analyzed and are mainly caused by a possible "core-shell" microstructure of particles. The accumulative pre-strain induced by the presence of pre-load and the adhesion between the soft particles and the silicon substrate or the rigid flat punch seems to be of secondary nature. Finite element analyses have been carried out to demonstrate this surface shell effect.

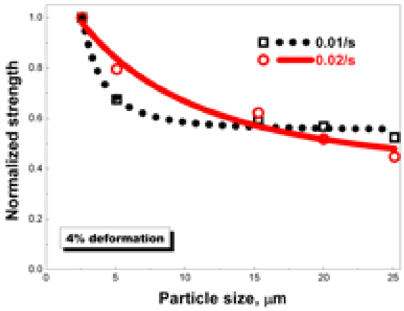


Figure 3. Particle size dependence of the normalized stress with strain rate 0.01/s and 0.02/s at deformation level 4%.

Another series of polystyrene particles with the same size but crosslink density varying from 2% to 55% has been characterized. It has been found that the increase of the crosslink density induces a significant ductile-brittle transformation of particle deformation behaviour. The highly crosslinked particles behave much stiffer than slightly crosslinked ones. The dominating mechanism is that the crosslinking restrict the possibility of the polymer chain to rearrange and hence redistribute the internal stress.

The influence of the nanoscale Ni/Au coating has been revealed through comparing the mechanical properties of metal coated polymer particles with that of identical size, but uncoated ones. It has been found that within a range of relatively small deformations the metal coating plays a prominent strengthening effect on the mechanical properties of the particles. Therefore, the deformation of metal coated particles can be identified in a three-stage process as shown in Figure 4. Initially the Ni/Au coating strongly strengthens the particles. The metal coated particle is much stronger than the uncoated one. Secondly, the effect of Ni/Au coating is significantly reduced when the cracking of the Ni/Au coating and the delamination between the Ni/Au coating and the polymer core occur. During this stage, the strengthening effect of the metal layer is reduced as the cracking and delamination of the metal continues. In the third and last phase with a relatively large deformation, the effect of Ni/Au coating vanishes completely and the coated particle shows nearly identical behaviour with the uncoated ones.

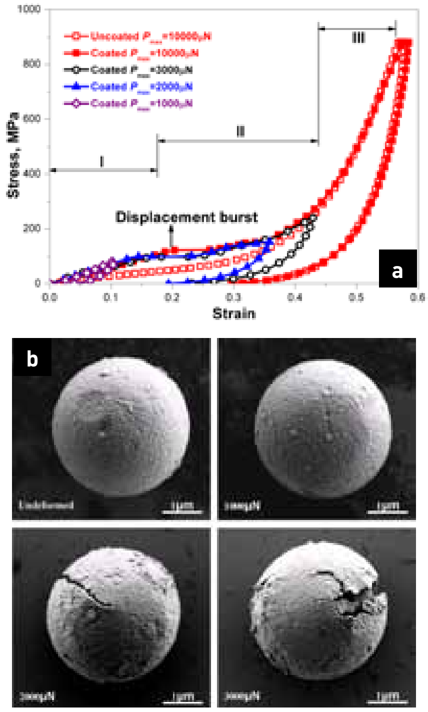


Figure 4. Compression stress-strain curves (a) and SEM images (b) of the Ni/Au coated acrylic based particles at different peak loads.

This work has been carried out by PhD candidate Jianying He under the supervision of Professor Zhiliang Zhang at The Department of Structural Engineering within the NANOMAT KMB project NanoPCP (2006-2009) financed by Research Council of Norway and Conpart AS. Until now, the nanomechanical technology developed in the project has initiated three joint R&D projects between NTNU Nanomechanical Lab and Conpart AS. The research has been focused on developing a multi-scale modelling methodology for Ugelstad polymer particles and establishing quantitative relations between nanoscale molecular structures and mechanical properties, as shown in Figure 5. Through this collaboration, the nanomechanical technology for the characterization of single particles has contributed

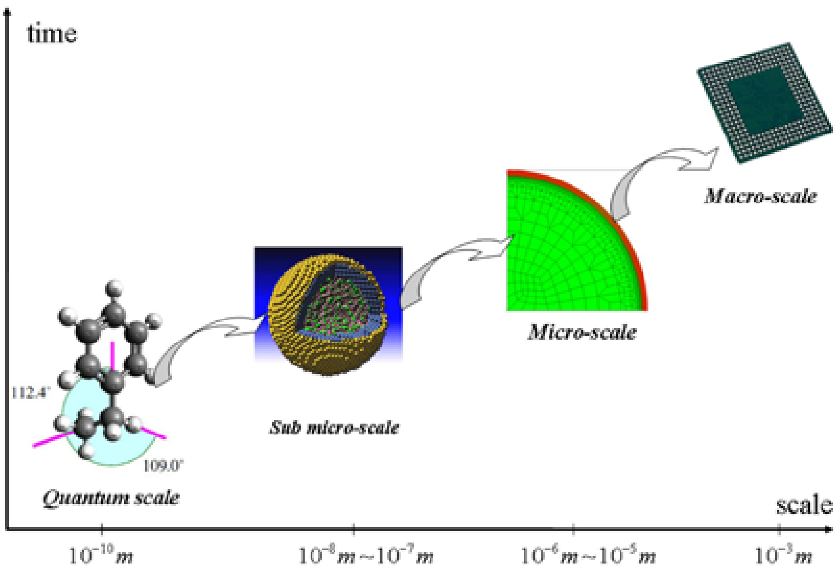


Figure 5. Multi-scale modeling methodology for Ugelstad particles.

significantly to Conpart's success as an advanced material supplier, entering into the international microelectronics market. The technology will also be critical for Conpart's ability to enter new segments of the microelectronics industry as well as the photovoltaic industry.

Surfaces for Digital Microfluidic Systems

Digital microfluidics is a variation of the idea of integrating a fluidic lab on a chip. In contrast to more commonly used microfluidics systems, which work with continuous flows in predefined channels, digital microfluidics is based on the manipulation and analysis of individually controllable droplets. One method of realizing such a system is based on self confined droplets on a superhydrophobic surface. Superhydrophobic surfaces, as known e.g. in the lotus leaf, show very high water repellency as a result of the combination of nanometer scale surface roughness and hydrophobic surface chemistry. Water or aqueous solutions in contact with such a surface will not wet it, but instead form highly mobile spherical droplets, which float on air pillows trapped between the surface features.

In contrast to other applications of superhydrophobicity, like self cleaning or corrosion protection, microfluidic systems are special because they require the droplets to stay controllably on the surface. Consequently, the advantage of high droplet mobility comes at the expense of high sensitivity to tilting and vibrations.

For our chip, we partially compensated this by introducing macroscopic features on the superhydrophobic surface, which could structurally confine the droplets which were placed within them. This was done by using copper as the base material for which low resolution patterning techniques adopted from printed circuit board fabrication were applied. To produce a superhydrophobic surface on the macroscopically (mm-scale) patterned chip, we used additional steps to create the necessary microscopic surface roughness. This may be done either by etching of polycrystalline samples along the grain boundaries, by electrodeposition of copper films with subsequent nanowire decoration based on thermal oxidization, or by combination of both methods (see Figure 1).

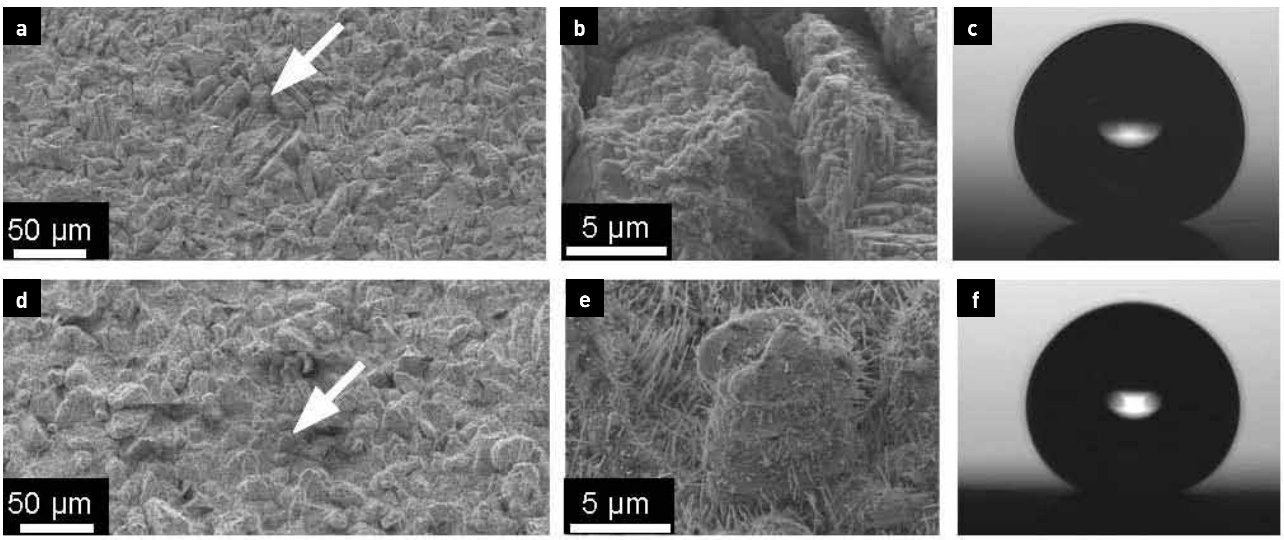


Figure 1: Top row: Copper surface produced by etching and a deposited water droplet. Bottom row: CuO surface with nanowires after oxidization and a deposited water droplet. Photo: Florian Mumm.

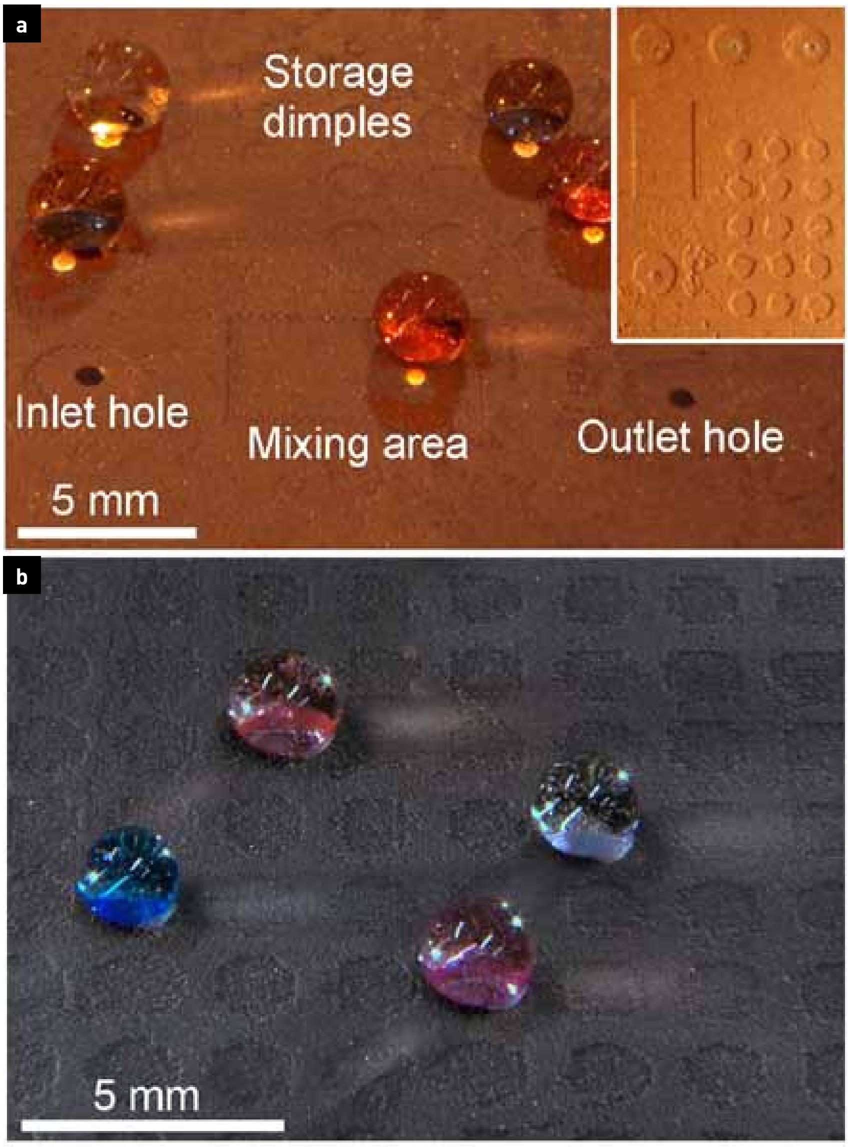


Figure 2: Cu based chip (a) and CuO based chip (b) with coloured water droplets. Photo: Florian Mumm.

Finally, the rough surfaces were hydrophobized using self assembled thiol-modified fluorocarbons, after which they showed a water contact angle as high as 171° ± 2°. An example chip consisting of inlet and outlet holes, a mixing area as well as storage dimples can be seen in Figure 2.

As the droplet mobility on super-hydrophobic surfaces is exceptionally high, novel techniques for moving and mixing the droplets become available. In combination with our chip, one such technique was demonstrated: An optical fibre stripped of its protective jacket was brought in direct contact with a droplet of water or different biologically relevant buffer solutions. As the adhesion between the buffer and the fibre was larger than between the buffer and the surface, the droplet simply followed the fibre when it was moved. When the fibre was lifted out of the droplet, it remained at its new position on the surface.

This work was carried out in the Group of Bionanotechnology, by PhD student Florian Mumm and Associate professor Pawel Sikorski, in collaboration with Associate professor Antonius T. J. van Helvoort at the Department of Physics.[1] The project also yielded insight into fabrication parameters for easy decoration of comparatively large areas with CuO nano-wires as illustrated by the cover picture of this report.

Reference:

1. F. Mumm, A. T. J. van Helvoort, and P. Sikorski. *Easy Route to Superhydrophobic Copper-Based Wire-Guided Droplet Microfluidic Systems.* ACS NANO 2009, 3, 2647).

Sub-50 nm nanostructures in perovskite thin films

Perovskite oxides exhibit a variety of interesting functionalities that can be employed in various device applications, ranging from sensors and actuators to active devices such as transistors and spintronics applications. One challenge of applying thin films of such materials is to define well-controlled nanostructures with sub-50 nm dimensions. At The Department of Electronics and Telecommunications, in collaboration with The Department of Physics, a research platform focused on suitable techniques for nanostructuring of different perovskite oxide thin film materials has been developed. The platform's main goal is to develop a combined top-down and bottom-up approach for fabrication of sub-50 nm perovskite nanostructures with controlled geometry, by epitaxial growth of nano-mesas on prestructured template materials.

First a scanning tunneling microscope (STM) was used to physically alter the surface of a template material. Scanning probes have been extensively studied for controlled surface modifications in a variety of material systems. The nanostructuring effort was focused on epitaxial thin films of $SrRuO_3$ and $La_{0.7}Sr_{0.3}MnO_3$. $SrRuO_3$ is interesting as template material, as it is inert and well lattice matched to a number of perovskite materials, providing the possibility to grow epitaxial nanostructures on the STM-structured template surface. Here the discussion is concentrated on $SrRuO_3$.

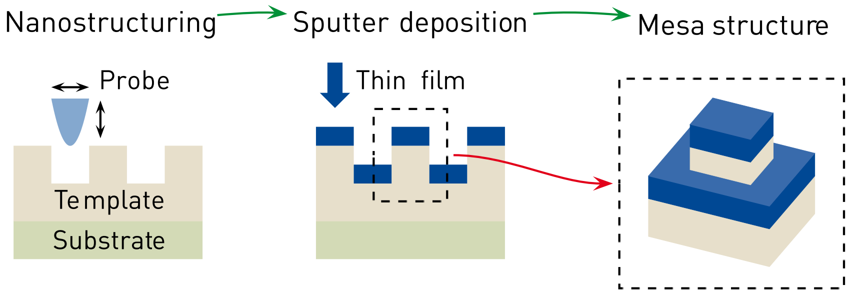
A significant focus has been the use of an STM to define nanostructures with high precision. Most of the work was carried out under ambient conditions, in order to simplify tip-exchange and multi-step work-flow. The depth of the etched structures was found to depend on the scan speed, the bias voltage, and the number of scan repetitions. In $SrRuO_3$, trenches with a lateral size of 5nm could be etched routinely. The high lateral and vertical control achieved provides the possibility to develop nanostructured templates for subsequent bottom-up synthesis of nanomesas.

In order to study the nanoscale thin film islands on top of these templates, ferroelectric $PbTiO_3$ nanomesas were deposited by off-axis RF magnetron sputtering. Ferroelectric nanostructures are interesting for various applications, including non-volatile memories and energy harvesters. Here the ability to grow $PbTiO_3$ nanomesas with lateral size down to 30 nm has been demonstrated. It is not only possible to synthesize individual nanomesas, the technique also permits preservation of the template surface topography at the $PbTiO_3$ surface for films thicker than the depth of the etched structures. Hence, this technique for definition of nanostructured perovskites can be used both to realize isolated nanomesas and to induce nanostructured surfaces in homogeneous films.

The combined top-down/bottom-up fabrication scheme for sub-50 nm perovskite nanostructures developed is general. Based on the fact that many perovskites are well lattice-matched, the present technique should work also for other interesting perovskite oxides, including multiferroics and strongly correlated systems.

References:

1. C. C. You, R. Takahashi, A. Borg, J. K. Grepstad, and T. Tybell, *Fabrication and characterization of $PbTiO_3$ nanomesas realized on nanostructured $SrRuO_3$/$SrTiO_3$ templates,* Nanotechnology 20, 255705 (2009)
2. C.C. You, N-V. Rystad, A. Borg and T. Tybell, *Nanoscale structuring of $SrRuO_3$ thin film surfaces by scanning tunneling microscopy,* Applied Surface Science 253, 4704 (2007)



Graphic presentation of the nanomensa production process.

Carbon-supported core-shell electrocatalysts for oxidation of small organic molecules

Electrochemical energy conversion and storage will remain an indispensable part of an inherently sustainable and environmentally friendly energy system.

The Electrochemical Energy Group at The Department of Materials Science and Engineering at NTNU focuses on studies of energy conversion in fuel cells and hydrogen storage by water electrolysis, as well as battery technology. One challenge is to limit CO poisoning of fuel cell catalysts and develop anode catalysts for Direct Methanol Fuel Cell (DMFC). This research has been carried out within the project "Carbon-supported core-shell electrocatalysts for oxidation of small organic molecules", funded by The Research Council of Norway through the NANOMAT programme, under the supervision by Professor Svein Sunde and Associate professor Frode Seland.

Bimetallic surfaces have long been known for their catalytic activity and selectivity, which often exceeds that of the individual components and have thus a wide range of applications. Some platinum alloys show a better CO tolerance and stability in comparison with pure platinum. In order to optimize the catalytic activity, the composition and architecture of the catalyst nanoparticles must be controlled. The ability to produce multi-component nanoparticles with optimized structures for other, selective or multifunctional reactions is expected to play a critical role in new energy conversion technologies. Monometallic, heterodimer, alloy and core-shell nanoparticles are examples of such structures.

There are still significant unknown features in the mechanisms of oxidation of CO and small organic molecules. Especially the balance between the bifunctional and pure electronic effects, as well as the role

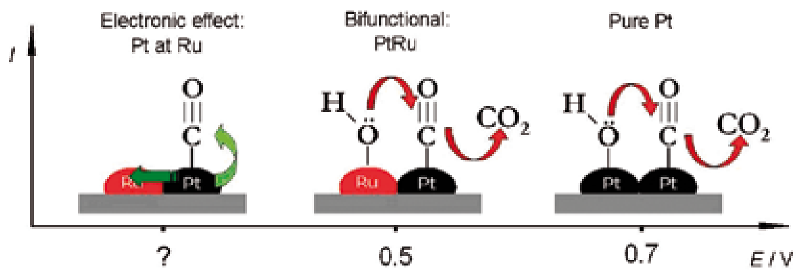


Figure 1. Contributions to the CO-stripping mechanism. From the left: Ligand effect, center: Bifunctional effect, right: the process at a pure Pt catalyst. The potential at which the processes occur are indicated below the figures, the "?" being currently determined in the core-shell project.

of metal-support interactions remain to be fully understood. The core-shell project aims *inter alia* at contributing to their discrimination, and is expected to lead to highly original results and fundamental insight relevant to the design of electrocatalysts for direct oxidation of methanol and other fuels.

The production of nanoscaled core-shell systems, and their characterization are challenging tasks. Professor Eichhorn's group at the University of Maryland, USA, with which our group at NTNU collaborates, has successfully established a procedure for the synthsis of Ru@Pt core-shell nanoparticles. The structures of these particles are comprised of essentially metallic, crystallographically disordered Ru cores with thin, 1-2 monolayer Pt shells.

Ru@Pt *Figure 2. Model of the Ru@Pt core-shell structure.*

As a result of the collaboration with The University of Maryland the synthesis of Ru@Pt was successfully carried out in NTNU NanoLab's cleanroom. The Ru@Pt 1:1 core-shell nanoparticles were prepared by using a sequential polyol process. $Ru(acac)_3$ (acac = acetylacetonate) was initially reduced in refluxing ethylene glycol (EG) in the presence of polyvinylpyrrolidone (PVP) stabilizers (MW = 55000). The resulting Ru cores were subsequently coated with Pt by adding $PtCl_2$ to the Ru/EG colloid and heating to 200 °C.

A physicochemical as well as an electrochemical characterization of the produced material has been performed, and the structure's identity has been confirmed. This successful synthesis allowed performing advanced electrochemical characterization of the novel electrocatalyst. We have recently verified that the homogeneity of the samples allows isolation of the ligand

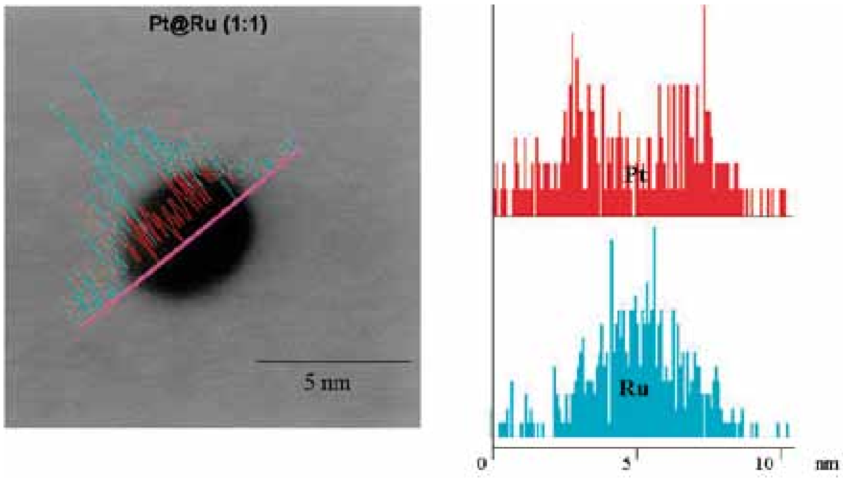
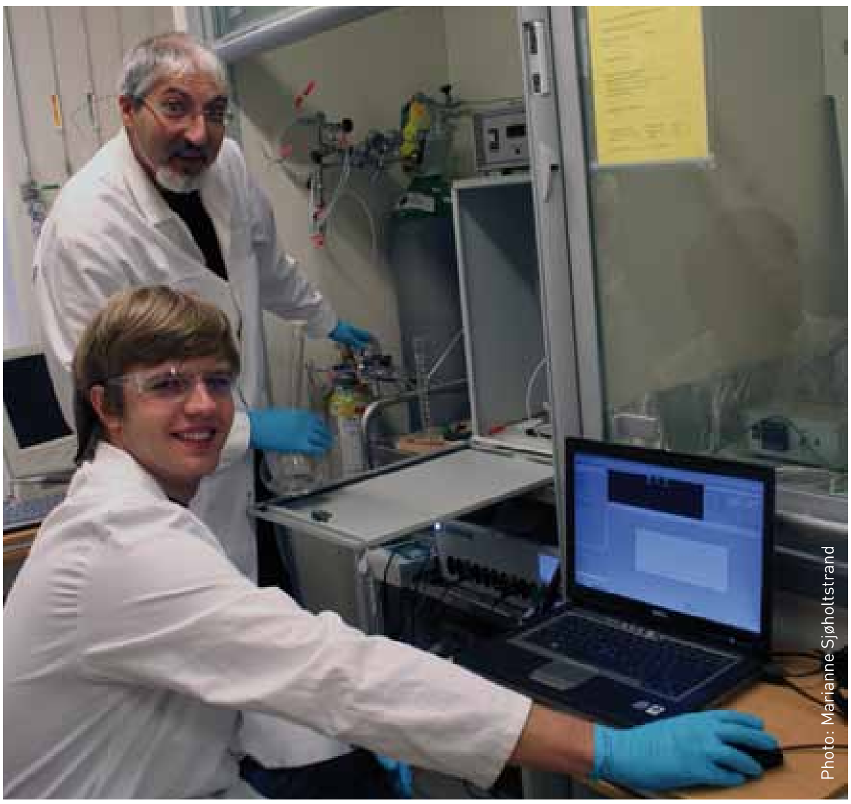


Figure 3. STEM-EDS line spectra of 4.5 nm obtained Ru@Pt (1:1) NP. Relative atomic % composition values (vertical axis) of Pt (red) and Ru (blue) are plotted against the line scan probe position (horizontal axis) and are given next to the STEM images.

effects from the bifunctional effects, and shown the former to be dominating in terms of the oxidation potential for CO on this core-shell catalyst.

This type of synthesis and control of bimetallic nanocatalyst architecture is crucial for mechanistic evaluation and rational development of heterogeneous catalytic transformations. From a practical point of view, the work may lead to new and more efficient catalysts for fuel cells anodes and other areas of applications.



PhD student Piotr Ochal and Dr. José Luis Gómez de la Fuente performing electrochemical characterization of the core-shell catalyst.

Meetings and Seminars

4th NTNU NanoLab User Meeting

The 4th NTNU NanoLab User Meeting was organized on the 13th and 14th of May 2009, in conjunction with the Opening of NTNU NanoLab's cleanroom. The first day of the meeting was devoted to scientific presentations by participants from NTNU and was opened by the Acting leader of NTNU NanoLab, Erik Wahlström. The programme consisted of the following lectures:

- Dheeraj Dasa, NTNU, *Tailoring of semiconductor heterostructure* nanowires for photonic applications by molecular beam epitaxy.
- Ton van Helvoort, *Semiconductor nanowire heterostructures characterization by dark field TEM.*
- Fervin Moses *Optical properties of axial and radial heterostructured III-V semiconductor nanowires.*
- Jacob Linder, *Pairing symmetry conversion by spin-active interfaces in superconducting junctions.*
- Sedsel Fretheim Thomassen, *Quantum dot density studies for quantum dot intermediate band solar cells.*
- Per Erik Vullum, *Transmission electron microscopy characterization of quantum dot intermediate band solar cell materials.*
- Roya Dehghan, *In-situ electron microscopy study of catalyst nanoparticle reduction.*
- Andreas Bertheussen, Anne Kirsti Noren and Alf Petter Syvertsen, *The nanotechnology students - Quick learners. Hard workers.*
- Annika Eriksson, *Interplay between structure and properties in perovskite-related materials.*
- Francesco Madaro, *Texturing of lead-free piezoelectric ceramics.*
- Per Martin Rørvik, *Hierarchical growth of ferroelectric $PbTiO_3$ nanorods.*
- Jianying He, *Failure of Nanostructured Composite Particles.*
- Shijo Nagao, *Molecular dynamics simulations of nanoindentation-induced incipient plasticity in binary semiconductors: GaAs and GaN.*
- Hans Krokan, *DNA-interacting proteins as molecular machines.*
- Marthe Thuen, *Nanoparticles in molecular imaging with MRI.*
- Florian Mumm, *Structured, copper based superhydrophobic surfaces for digital microfluidics.*
- Marit Sletmoen, *Single-molecule pair interactions of the α-GalNAc (Tn-Antigen) form of porcine submaxillary mucin with soybean agglutinin.*
- Øyvind Halaas, *Nanomedicine - possibillities and limitations for nanotechnology in future medicine.*

The meeting took place in Lecture Hall EL 3 in the Old Physics Building at NTNU and gathered 83 participants including MSc students and staff from 9 departments and 4 faculties at NTNU, as well as SINTEF.

The second day of the 4th NTNU NanoLab User Meeting offered a workshop on *Development of NTNU NanoLab's cleanroom* followed by guided tours of the infrastructure. The workshop was initiated by Acting director of NTNU NanoLab, Erik Wahlström who presented general information regarding NTNU NanoLab's cleanroom. The participants then split in three groups for discussions of the facility's options related to the following areas:

i) Physical methods, leader: Erik Wahlström
ii) Chemical methods, leader: Fride Vullum
iii) Bionanotechnological methods, leader: Pawel Sikorski

The NTNU NanoLab lunch seminars

The aim of these seminars has been to inform about available equipment and processes in NTNU NanoLab's cleanroom and to present ongoing activities within nanoscience and nanotechnology at NTNU and SINTEF. The seminars took place weekly on Tuesdays at 12.15 - 12.45, in NTNU NanoLabs meeting area and gathered between 30 and 50 participants.

Spring programme:

- Erik Wahlström: *NTNU NanoLab; Plans for start-up of activities.*
- Fride Vullum: *AFM options in NTNU NanoLabs's cleanroom.*
- Tron Arne Nilsen: *Short introduction to etching options in NTNU NanoLab's cleanroom*.

Photo: Ida Hederström

PhD student Tron Arne Nilsen giving a short introduction to etching options in NTNU NanoLab's cleanroom.

- Sophie Weber: *Options for surface coating in NTNU NanoLabs cleanroom.*
- Ton van Helvoort: *Short introduction Focused Ion Beam techniques in NTNU NanoLab's cleanroom.*
- Pawel Sikorski: *Application of nano-structuring in bionanotechnology.*
- Thomas Tybell: *Short introduction to lithography options in NTNU NanoLab's cleanroom.*

Autumn programme:
- Erik Wahlström (Leader of NTNU NanoLab): *Price policy and distribution of information regarding the clean-room.*
- Cecile Ladam (SINTEF): *Growth of ZnO nanorods by pulsed laser deposition for solar cell applications.*
- Helge Weman (Dept. of electronics and telecommunications): *Challenges and possibilities of heterostructured semiconductor nanowires for photonic device applications.*
- Magnus Rønning (Dept of chemical engineering): *Nanomaterials in catalysis.*
- Magnus Olderøy / Floriann Mumm (Dept of physics): *S(T)EM options in the cleanroom - with examples from the biophysics group.*
- Chaolin Zha (Dept of physics): *Spin torque oscillators.*
- Arne Brataas (Dept of physics): *Nano spintronics.*
- Trine Østlyng (NTNU NanoLab): *Particle size analysis by Nanosight*
- Åsmund Monsen (Dept of physics): *A short introduction to electron beam lithography.*
- Marit Sletmoen (Dept of physics): *Optical tweezers: Nanoscale positioning of micron and nanosized objects.*
- Spyros Diplas (SINTEF): *XPS, TEM and DFT studies of thin films and interfaces in ZnO/Si and ITO/Si structures.*

Workshop on oxide and interface magnetism

The first workshop within the STINT Institutional Grant for Younger Researchers, *Magnetotransport in strongly correlated electron systems,* was arranged at NTNU in 2009 by Associate prof. Erik Wahlström, and was a cooperation between the Department of Engineering Sciences, Uppsala University, Sweden, and the Department of Physics, NTNU. The workshop gathered 27 participants.

The aim of the workshop was to create a mutual understanding of the research aims of participating groups with the primary objective to find common ground to further the development of collaboration projects. The workshop focused on understanding thin film oxide magnetics, including the interaction and coupling between e.g. ferromagnetic and antiferromagnetic layers, focusing on the interplay between macroscopic and microscopic magneto transport phenomena.

- Roland Mathieu / Per Nordblad *Solid State Physics – Uppsala University, Sweden*
- Erik Wahlström / Anne Borg, *Condensed Matter Physics – NTNU*
- Tor Grande, *Inorganic Materials and Ceramics – NTNU*
- Thomas Tybell, *Physical Electronics – NTNU*
- Olof "Charlie" Karis, S*urface and Interface Science - Uppsala University, Sweden*
- Swarnail Bandopadhyay, *Theoretical Condensed Matter Physics – NTNU*
- Thomas Lottermoser, *Nonlinear Optics on Strongly Correlated Solid-State Systems - University of Bonn, Germany*
- Biplab Sanyal, *Materials Theory - Uppsala University, Sweden*
- Karl Fabian, *Geological survey of Norway*
- Dario Arena, *National Synchrotron Light Source, Brookhaven National Laboratory, U.S.A.*
- Aleksander Samardak, *Department of Electronics - Far Eastern National University, Vladivostok, Russia*
- Johan Åkerman *Spin Torque Oscillators - University of Gothenburg, Sweden*
- Biplab Sanyal (Uppsala University, Sweden) *Theory of improper multiferroicity in $HoMnO_3$.*
- Olof "Charlie" Karis (Uppsala University, Sweden) *Synchrotron based spectroscopic investigations of magnetic materials*
- Matthias Hudl (Uppsala University, Sweden) *Spin glasses: exchange bias without intiferromagnetic/ ferromagnetic interface.*
- Thomas Lottermoser (University of Bonn, Germany) *Nonlinear optics on multiferroics and beyond.*
- Dario Arena (Brookhaven National Laboratory, U.S.A.) *Ferromagnetic Resonance Probed with X-Ray Absorption.*
- Karl Fabian (Geological Survey of Norway) *Magnetic exchange bias in nano-structured ilmeno-hematite.*

Participants of the workshop Magnetotransport in strongly correlated electron systems.

- Sverre M. Selbach (NTNU) *Structure, phase transitions and defect chemistry of Mn-substituted $BiFeO_3$.*
- Chaolin Zha (NTNU) *Structural and Magnetic Properties of D022 MnGa films.*
- Åsmund Monsen (NTNU) *Towards LSMO-based TMR structures.*
- Jos Emiel Boschker (NTNU) *In-plane modulations in $La_{0.7}Sr_{0.3}MnO_3$ thin films: effect of substrate and initial growth.*
- Johan Åkerman (University of Gothenburg, Sweden) *Coexistence of localized and propagating spin wave modes in nano-contact Spin Torque Oscillators.*
- Aleksander Samardak (Far Eastern National University, Vladivostok, Russia) *Magnetic configuration feature of nanodisks.*
- Magne Saxegaard (NTNU) *A compact concentric scanning tunneling microscope for point contact investigations of magnetic nanostructure.*
- Erik Folven (NTNU) *Investigation of antiferromagnetic nanostructures by X-ray magnetic linear dichroism and photoelectron microscopy.*

Other nanorelated seminars at NTNU

- Prof. Gaetano Granozzi, University of Padova, *Titania at the nanoscale: what's at the bottom?*
- Prof. Signe Kjelstrup, Department of chemistry, NTNU, *Bringing thermodynamics to the nanolevel.*
- Prof. Gerrit Bauer (TU), *Onsager seminar: The Mechanics of Spintronics.*

Staff engineer Trine Østlyng demonstrates safe handling of HF in NTNU NanoLab's cleanroom. Photo Marianne Sjøholtstrand.

Courses

NTNU NanoLab has offered a number of courses in 2009 related to the activities in the cleanroom. All courses were given free of charge to staff and students at NTNU and SINTEF.

Introduction courses in HES and routines for work in the cleanroom
All users have to complete an introduction course in HES and routines for work in NTNU NanoLab's cleanroom before gaining access to the facilities. The course consists of a theoretical part (1.5 h) and a practical part in the cleanroom (1.5 h). Throughout 2009, staff engineer Trine Østlyng has given 10 introduction courses accrediting 127 users.

Courses in handling HF in NTNU NanoLab's cleanroom
A course in in safe handling of HF in NTNU NanoLab's cleanroom has been developed and offered three times in 2009 by staff engineer Trine Østlyng.

Introduction courses on Atomic Force Microscopy (AFM)
Staff engineer Ida Hederström has given introduction courses in application of AFM for 40 users in 2009. The course is required in order to receive access to NTNU NanoLab's AFM.

Introduction and specialization courses to Focused Ion Beam (FIB) techniques
In 2009, three basic courses in the use of NTNU NanoLab's FEI Helios-(DualBeam)-FIB were given. This course, which consists of a practical part and a theoretical part in the clean-room, is required in order to receive access to the instrument. In addition, three advanced courses focusing on specialized techniques were offered. The courses were taken by 10 users and were given by senior engineer Sören Heinze.

Introduction course to Scanning (Transmission) Electron Microscopy [S(T)EM]
Basic courses in the use of NTNU NanoLab's Hitachi S-5500-S(T)EM have been given to 15 users in 2009. The course, which consists of a practical part and a theoretical part in the clean-room, is required in order to receive access to the instrument and was given by senior engineer Sören Heinze and Mats Eriksson from Spectral Solutions. Altogether 15 users obtained training on the S(T)EM during 2009.

Short introduction to Energy Dispersive X-ray Spectroscopy (EDX)
Mats Eriksson from Spectral Solutions gave a 2-3 hour lecture with the title: "Introduction to EDX related to the Bruker SDD EDS installed on NTNU NanoLab's S(T)EM".

Dissertations

The following candidates have defended their thesis for a PhD degree at NTNU in fields related to nanotechnology in 2009.

- Liyuan Deng: *Development of novel PVAm/PVA blend FSC membrane for CO_2 capture.*
- Unn-Merete Fagerli: *Multiple myeloma cells and cytokines from the bone marrow environment; aspects of growth regulation and migration.*
- Brit Kathrine Frøyen Graver: *Effect of trace elements in indium, tin and lead on anodic activation of aluminium.*
- Eirik Glimsdal: *Spectroscopic characterization of some platinum acetylide molecules for optical power limiting applications.*
- Ole-Erich Haas: *Transport on a nano-scale; quasi-elastic neutron scattering and molecular dynamic studies.*
- Jianying He: *Nanomechanics of polymer and composite particles.*
- Ingvild Bjellmo Johsen: *Intracellular signallilng mechanisms in the innate immune response to viral infections.*
- Kristine Misund: *A study of the transcriptional repressor ICER.*
- Ronny Myhre: *Genetic studies of candidate tene3S in Parkinson's disease.*
- Tonje Strømmen Steigedal: *Molecular mechanisms of the proliferative response to the hormone gastrin.*
- Linda Tømmerdal Roten: *Genetic predisposition for the development of preeclampsia – candidate gene studies in the HUNT population.*
- Silje Marie Skogvold: *Development and properties of nontoxic solid electrodes for environmental surveillance. Application in automatic on site determination of metals in natural water and industrial solutions.*
- Hanne Martinussen: *Heteroyne Interferomety for Dynamic and Static Characterization of Micro- and Nanostructures.*
- Wakshum Mekonnen Tucho: *Self-supported, thin Pd/Ag membranes for hydrogen separation.*
- Silje Rodahl: *Adhesion of Organic Coatings on Aluminium.*
- Marius Sandru: *Development of a FSC membrane for selective CO2 capture.*
- Sverre Magnus Selbach: *Structure, stability and phase transitions of multiferroic $BiFeO_3$.*
- Hans Joakim Skadsem: *Transport and magnetisation dynamics in ferromagnetic nanostructures.*
- Ingeborg-Helene Svenum: *Interaction of functional groups with surfaces.*
- Sven Tierney: *Development and characterization of bioresponsive hydrogel materials.*
- Thea Kristin Våtsveen: *Genetic aberrations in myeloma cells.*
- Lars Erik Walle: *Surface science studies of TiO2 single crystal systems.*
- Jacob Wüsthoff Linder: *Quantum transport and proximity effects in unconventional superconducting hybrid systems.*

Awards

Teknas prize for young researchers for 2009 was awarded to Dr. ing Yrr Asbjørg Mørch, and PhD Jan Petter Morten. Mørch's PhD thesis was entitled *Novel alginate microcapsules for cell therapy - A study of the structure-function relationship in native and structurally engineered alginates, while Morten had studied Coherent and Correlated Spin Transport in Nanoscale Superconductors.*

The "ExxonMobil's forskerpris 2009" was also given to Dr. Jan Petter Morten for his PhD work.

The Faculty of natural sciences' prize for the best PhD thesis in 2009 was given to Dr. Jacob Linder from The Department of physics for his thesis entitled: *Quantum transport and proximity effects in unconventional super-conducting hybrid systems.*

Dr. Jacob Linder (left) receives The Faculty of Natural Sciences and Technol-ogy's award for Best PhD Thesis 2009 from Vice Dean Åse Krøkje (right).

Nanoscience and nanotechnology at NTNU in the media

- Forskning.no (13.04.09) *Leire i fritt fall.* http://www.forskning.no/artikler/2009/mars/215230
- Adresseavisen (11.05.09) *Åpner laboratorium for nanoteknologi.*
- NRK P1 (12.05.09) Åpning av NTNU NanoLab. http://www1.nrk.no/nett-tv/klipp/493458
- NRK Midt-Nytt (12.05.2009) *Åpning av NTNU NanoLab.*
- NRK Trøndelag (12.05.2009) *Åpning av NTNU NanoLab, Interview with prof. Helge Weman.*
- Universitetsavisen (13.5.09) *NTNU NanoLab offisielt åpnet,* http://www.universitetsavisa.no/ua_lesmer.php?kategori=nyheter&dokid=4a0aa29557c4a9.13328375
- Adresseavisen (13.05.09) *200 millioner til nanoforskning.*
- Gemini (27.05.09) *Tettpakket framtid.*
- Under Dusken (nr 8, 2009) *Satser mye på lite.*
- Adresseavisen (09.11.09) *Mikrobobler kan frakte medisin i kroppen,* http://www.adressa.no/forbruker/helse/article1408794.ece

Photo Ida Hederström

Publications

Nanomaterials

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Photo: Marianne Sjøholtstrand

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