Annual Report NTNU NanoLab 2013

NTNU – Trondheim
Norwegian University of
Science and Technology

NTNU NanoLab 2013

In 2013, NTNU NanoLab put focus on the development of international relations both on the research and the infrastructure side.

Nanotechnology research at NTNU is both growing in size and improving in quality. The number of publications in the area increased further and several papers in high-impact journals have been recognised.

This has led to increased international attention. Faculty members from NTNU participated in the "Japan-Norway Science Week 2013" in Tokyo, Japan and at a workshop with the nanotechnology researchers from the National Institute for Material Science (NIMS) in Tsukuba, Japan. In October, NTNU and RICE University (Houston, US) organized the first Norway-Texas workshop on nanoscience and -technology in Trondheim.

NTNU researchers contributed to the strategic important project initiative "Centre for Research-based Innovation (SFI) - Bridge" which was established on the base of the NorFab consortium.

In 2013, NTNU NanoLab got a new board that strengthens the implementation of NanoLab in NTNU's internal organization by including the deans of the involved faculties. The board has passed a market strategy for increasing the cleanroom use, an information strategy, and a staff plan to be realized in the coming years.


Photo: Sarah Gastinger

In 2014, the implementation of a reliable, long-term financing model and further development of international relations will be in focus. Horizon 2020 will publish the first calls and open enormous possibilities for research projects and through the infrastructure program. These arenas are essential for the realization of our long-term vision and we will intensify our efforts here. Also, the extension of the oversea relations will be important in order to increase the recognition of nanotechnology research at NTNU. Finally, the cooperation at the Nordic level, in particular with MyFab, the Swedish national infrastructure, and the continuation of the NorFab project will be of great importance.

These exciting results are founded on and would have been impossible without the cooperation and contribution of all "nano-researchers" at NTNU and the enormous efforts of the team at NTNU NanoLab.

Kay Gastinger
Director of NTNU NanoLab

NORWEGIAN MICRO- AND NANOFABRICATION FACILITY
NorFab

The Norwegian Micro- and Nanofabrication Facility, NorFab was established in 2010 by The Research Council of Norway as a national infrastructure with four partners; NTNU, University of Oslo, SINTEF ICT and Buskerud and Vestfold University College.

In 2013, NorFab has undertaken several networking initiatives. The common Myfab-NorFab User Meeting in Uppsala, April 17^{th}-18^{th} gathered 229 participants from Sweden and Norway. As a follow up of this meeting, a Nordic Expert Network within Micro- and Nanofabrication has been established. The first pilot meeting on dry etch techniques took place November 13^{th} -14^{th}, gathering 18 participants from 11 Nordic cleanrooms.

NorFab has also started an internal engineer forum for discussions and collaboration regarding operation and maintenance of cleanrooms. This forum had 4 meetings in 2013, which were open for all cleanrooms infrastructures in Norway.

On the national level, NorFab organized a Matchmaking Seminar for academic staff and industry representatives in Oslo, August 21^{st}.

Information regarding equipment, prices and options for support is given on NorFab's website: www.norfab.no

NanoNetwork
Norwegian PhD Network on Nanotechnology for Microsystems

The Norwegian PhD Network on Nanotechnology for Microsystems was established in 2009, with the objective to coordinate, integrate, and strengthen PhD programs in the field of nanotechnology and microsystems in Norway. The partner institutions are NTNU, University of Oslo, University of Bergen, Buskerud and Vestfold University College, and SINTEF ICT Microsystems and Nanotechnology. The network activities will continue with funding from the Division of Science, RCN, Program for National Graduate Schools, from 2014.

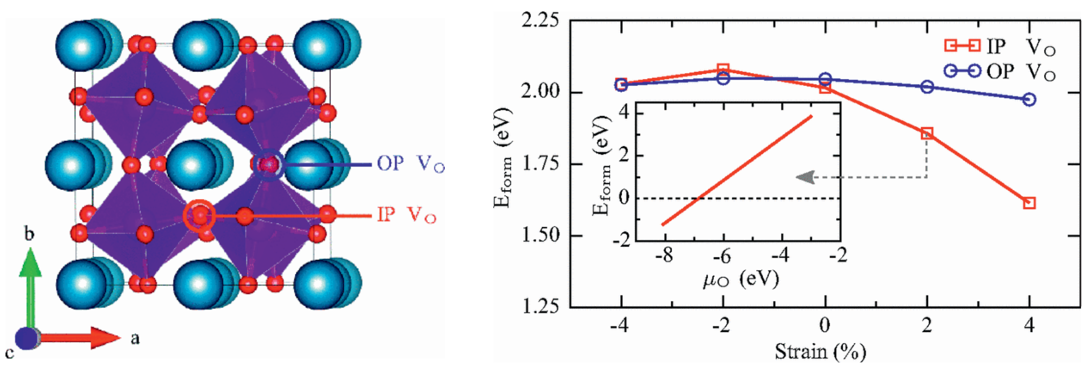
Last year, the Network offered 8 PhD courses on compact format and provided travel support for the candidates to attend these courses. The network has provided funding for laboratory and processing expenses to 20 PhD candidates and granted travel support to 36 candidates presenting their work at international conferences. Our 2013 annual workshop was held in Bergen from June 17^{th} to 19^{th}, gathering 83 participants from the Norwegian research communities in nanotechnology and microsystems.

Website: www.nano-network.net

Front page: Silicon optical ring resonator, Copyright: Leidulv Vigen and Astrid Aksnes, NTNU

Strain induced oxygen vacancy ordering by DFT

Epitaxial strain in oxide thin films can greatly modify the structure and properties. Strain is usually assumed to be accommodated by changes in bond lengths and angles. Density Functional Theory (DFT) calculations on the perovskite $CaMnO_3$-δ show that in-plane oxygen vacancies (IP VO) are strongly favoured by tensile strain. The equilibrium oxygen content, and concomitant charge carrier concentration, is thus sensitive to epitaxial strain. As out-of-plane vacancies (OP VO) are less sensitive, tensile epitaxial strain can yield vacancy-ordered interface structures which would not be possible in bulk material. Experimental observations of oxygen vacancies in a confined spatial area like epitaxial interfaces are very challenging, and "The DFT microscope" can greatly enhance the fundamental understanding of such interfaces.

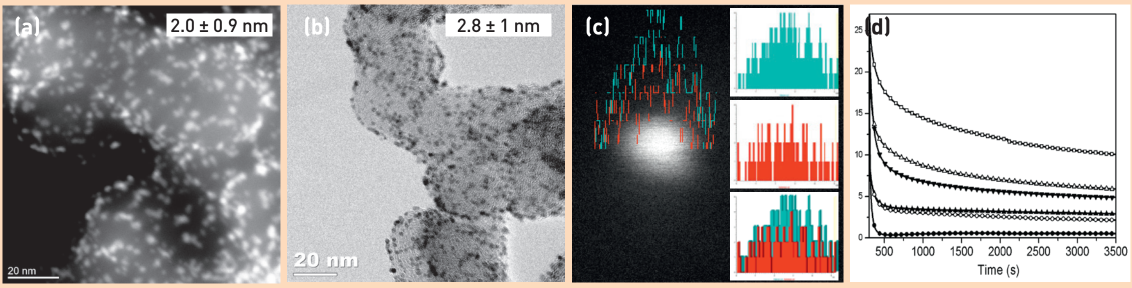


Biaxially strained $CaMnO_3$ with in-plane and out-of-plane oxygen vacancies and their formation energies from DFT.

"Strain-controlled oxygen vacancy formation and ordering in $CaMnO_3$" U. Aschauer, R. Pfenninger, S. M. Selbach, T. Grande, and N. A. Spaldin, *Phys. Rev. B* 88 (2013) 054111.

Ru@Pt core–shell nanoparticles for methanol fuel cell catalyst: Control and effects of shell composition

The cleanroom facility at NTNU Nanolab has been used to prepare carbon supported Ru@Pt core-shell nanoparticles (NPs). The Ru NPs with average size of 2.0 ± 0.9 nm are prepared initially by the polyol method. Then, the Pt is deposited on pre-synthesized Ru NPs to form a core-shell nanostructure of average size ~3 nm. By tuning the pH of the synthesis medium during Pt deposition, a shell with various chemical composition and structure can be obtained. In particular, an alloyed RuPt shell was obtained at a pH around 6 and a monometallic Pt shell was obtained at a pH around 10. The core–shell catalysts with alloy shells gave 10 times higher activity for the methanol oxidation reaction compared to the pure Pt catalyst.

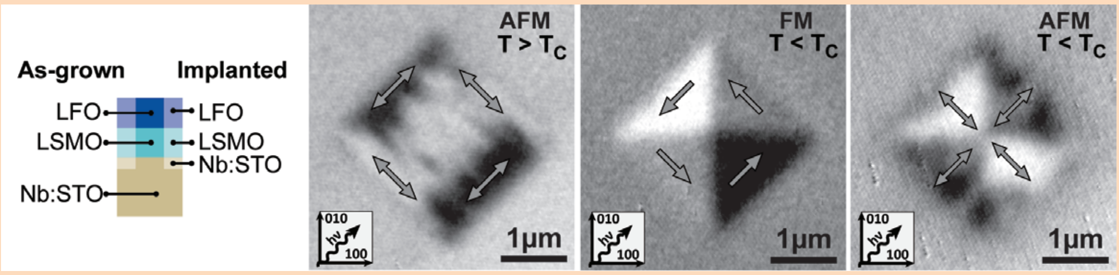


(a)HAADF-STEM images of Ru/C, (b)TEM image of Ru@Pt core-shell/C and (c)STEM-EDX line-scanning of single Ru@Pt core-shell particle, (d)Chrono-amperometric curves for methanol electro-oxidation on Pt_{shell} pH6/C (-□-), Pt_{shell} pH7/C (-Δ-), Pt_{shell} pH8/C (-▼-), Pt_{shell} pH 10/C (-▲-) and Pt/C (-●-). Conditions: 0.5 V, 0.5 M H_2SO_4 + 2 M CH_3OH solution, room temperature.

"Ru@Pt core–shell nanoparticles for methanol fuel cell catalyst: Control and effects of shell composition", N. Muthuswamy, J.L.G. de la Fuente, D.T. Tran, J. Walmsley, M. Tsypkin, S. Raaen, S. Sunde, M. Rønning, D. Chen, *Int. J. Hydrogen Energy,* 38 (2013) 16631.

Spin-Flop coupling and exchange bias in embedded complex oxide micromagnets

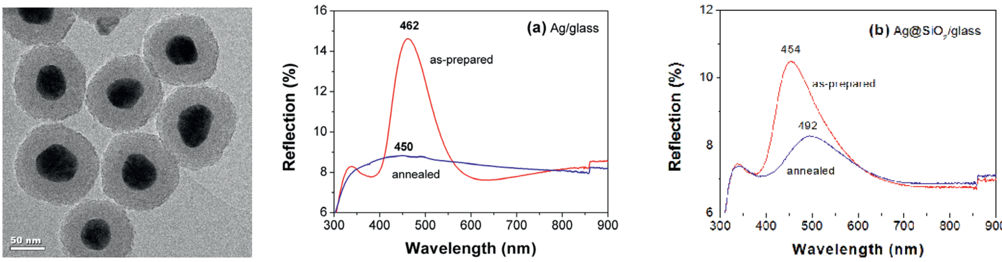
The magnetic domains of embedded micromagnets with 2µm x 2µm dimensions defined in epitaxial $La_{0.7}Sr_{0.3}MnO_3$ thin films and $LaFeO_3/La_{0.7}Sr_{0.3}MnO_3$ bilayers were investigated using soft x-ray magnetic microscopy. Square micromagnets aligned with their edges parallel to the easy axes of $La_{0.7}Sr_{0.3}MnO_3$ provide an ideal experimental geometry for probing the influence of interface exchange coupling on the magnetic domain patterns. The observation of unique domain patterns not reported for ferromagnetic metal microstructures, suggests the simultaneous presence of spin-flop coupling (i.e., perpendicular orientation of the ferro- and antiferromagnetic spins as shown for $T<T_C$ below) and local exchange bias in this system.



"Coupling and Exchange Bias in Embedded Complex Oxide Micromagnets", Y. Takamura, E. Folven, J.B.R. Shu, K.R. Lukes, B. Li, A. Scholl, A.T. Young, S.T. Retterer, T. Tybell, J. Grepstad, *Phys. Rev. Lett.* 111 (2013) 107201.

Nanoparticles as solar selective coating materials

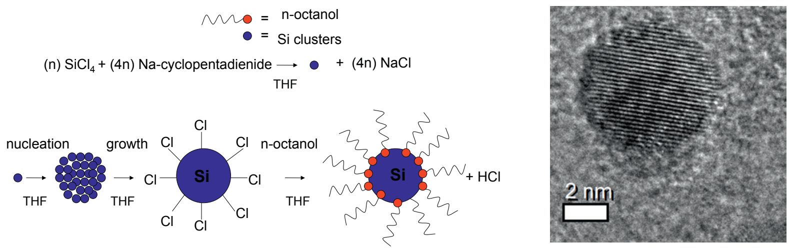
Glass can be colored by introduction of metal or metal oxide nanoparticles. The recent progress on synthesis and characterization of nanoparticles with controlled composition, size, and surface properties makes it possible to achieve advanced solar selective coatings for window glazing applications. In a recent work performed at *The Research Centre on Zero Emission Buildings (ZEB)*, NTNU, the potential of core-shell typed $Ag@SiO_2$ nanoparticles for such applications has been demonstrated. Incorporating Ag nanoparticles into an inert matrix such as SiO_2 not only maintains their predominant size- and shape-dependent optical properties, but also enhances greatly their stability and durability that are critical for applications in building and construction sector.



"Core-shell-typed $Ag@SiO_2$ nanoparticles as solar selective coating materials", T. Gao, B.P. Jelle and A. Gustavsen. *Journal of Nanoparticle Research,* 15 (2013) Article ID: 1370.

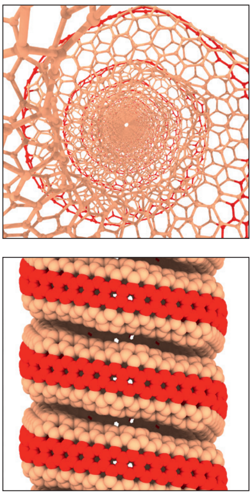
Solution based synthesis of fcc Si nano-crystals

Si nanostructures find application in various functional devices owing to their opto-electronic properties, including lasers and quantum dot devices, by utilizing their photoluminescence response over a range of wavelengths. We demonstrate for the first time, that simple face-centered cubic (fcc) silicon nano-crystals can be produced by a solution based bottom-up synthesis route at ambient conditions. Simple fcc Si nano-crystals (2-7 nm) were prepared at room temperature by using sodium cyclopentadienide as a reducing agent for silicon tetrachloride. The Si nanoparticles showed very fast PL decay times in the green region of the visible spectrum, indicating that such fcc Si nano-crystals have semiconductor properties. The work was carried out at Department of Materials Science and Engineering.



"Solution based synthesis of simple fcc Si nano-crystals under ambient conditions", M.H. Balci, R. Sæterli, J. Maria, M. Lindgren, R. Holmestad, T. Grande, M.-A. Einarsrud, *Dalton Transactions,* 42 (2013) 2700.

Tougher than the toughest

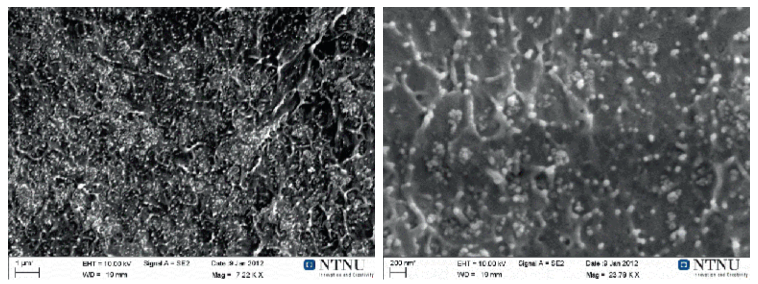


The ability of a material to absorb energy prior to rupture is a critical property called toughness. Graphitic nanomaterials are the world's toughest materials. Their toughness exceeds 1,000 J/g, much higher than high-strength steel wire (~ 37 J/g). Yet, the high toughness of nanomaterials such as carbon nanotubes (CNTs) is limited by their intrinsic low-ductility. Our atomistic simulations show that non-hexagonal defects distributed in coiled CNT nanosprings can yield *unexpectedly high toughness*, approaching 5,000 J/g. This comes from the plastic nano-hinge deformation caused by partial fractures, which are initiated and arrested by the defects required to coordinate the spring curvature. Multi-strand nanosprings offer an additional scalability in stiffness without breaking the high toughness. We thereby provide an atomistic principle for toughness enhancement by designing carbon-based nanomaterials. Furthermore, the CNT nanosprings would make an impact on future multifunctional energy applications, nanoscale- electromechanical or electromagnetic systems and devices because of their unique structural, electrical, thermal, magnetic and mechanical properties.

J.Y. Wu, J.Y. He, G.M. Odegard, S. Nagao, Q.-S. Zheng and Z.L. Zhang, *Journal of the American Chemical Society*,135 (2013) 13775; J.Y. Wu, S. Nagao, J.Y. He, and Z.L. Zhang, *Small*, 9 (2013) 3561.

Polyvinyl acetate/titanium dioxide nanocomposite membranes for gas separation

Membranes for gas separation have been commercial for around 30 years, but these polymeric membranes are simple materials such as polysulfones or polyimides, and have only been applied for very few gas mixtures. New insight the last decade has, however, documented that by embedding various types of nanoparticles in the polymeric membrane, the gas separation properties can be greatly enhanced. The nanoparticles influence the packing of the long polymer chains, and may thereby favour the diffusion of one gas component in a mixture of several – like promoting the transport of CO_2 through the nanocomposite membrane compared to N_2 (in flue gas) or CH_4 (in natural gas). In the research work featured here, we used nanoscale TiO_2 embedded in polyvinyl acetate, and were able to almost double the CO_2 transport through the membrane, while the increase in selectivity towards N_2 was 15%.

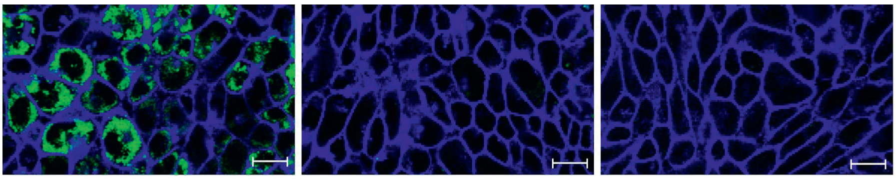


FESEM images of the Cross section of nanocomposite membrane, NCM-10

"Polyvinyl acetate/titanium dioxide nano-composite membranes for gas separation", J. Ahmad, M.-B. Hägg, *Journal of Membrane Science* 445 (2013) 200.

Chitosan-siRNA nanoparticles mediated improved drug delivery across the Blood-Brain Barrier

The molecular properties of the marine polysaccharide chitosan can be optimized for efficient silencing of specific genes (J. Controlled Release (2012) 158, 261–268), and chitosan-siRNA nanoparticles have been shown to mediate 90% gene silencing of the target gene without toxic effects. The blood-brain barrier (BBB) limits the availability of drugs to therapeutic targets in the central nervous system. One possible strategy to improve the delivery of drugs to the brain is siRNA mediated silencing of the efflux pump P-glycoprotein (P-gp), a membrane bound pump efficiently transporting specific xenobiotics back to the blood. We have investigated the potential of siRNA-chitosan nanoparticles in silencing P-gp in a BBB model and have shown that the transfection of rat brain endothelial cells mediated effective knockdown of P-gp with subsequent decrease in P-gp substrate efflux. This resulted in increased cellular delivery and efficacy of the model drug doxorubicin.



Intracellular delivery of doxorubicin visualized by CLSM. From left to right: cells treated with targeting siRNA nanoparticles (np), non-targeting siRNA np and untreated cells.

Nanoparticle Mediated P-Glycoprotein Silencing for Improved Drug Delivery across the Blood-Brain Barrier: A siRNA-Chitosan Approach. J. Malmo, A. Sandvig, K.M. Vårum and S.P. Strand, *PLOS ONE (2013) 8, e54182.*

8th Annual NTNU NanoLab meeting

A one-day seminar portraying research within nanoscience and nanotechnology at NTNU was arranged at Lian Restaurant in Trondheim on the 12th of November. The aim of the Annual NTNU NanoLab meetings is to facilitate cross disciplinary contacts between PhD-students, researchers and staff at NTNU and SINTEF. This year's program consisted of 25 presentations as well as social activities. All together, the meeting gathered 73 participants.

Norway-Texas seminar and workshop on nanoscience and nanotechnology

The event was organized as a follow-up of the Trans Atlantic Science Week in Houston i 2012 and took place at NTNU on the 14th – 17th of October. Eight selected professors from Texas were invited together with participants from all universities and relevant research institutes in Norway. The event started with a welcome reception hosted by the Rector of NTNU, Gunnar Bovim at Lerchendal Gård on Monday evening. The following days' program consisted of a one-day open seminar presenting the participating research institutions and their activities within nanotechnology. In addition, a representative from the Research Council of Norway gave an overview of the options for financial support for bilateral collaborations in Norway. After lunch, the participants gave short presentations of their research interests. The following day was allocated for targeted one-to-one meetings with the aim to seek out common project ideas. All together 37 meetings and 8 lab tours took place. Thursday was an open day for follow up of ideas and spontaneous meetings and local seminars at the departments at NTNU.

In addition to the scientific meetings, the program had ample time for informal talks in a social atmosphere. All in all, the arrangement had 56 participants. The seminar and workshop was arranged by NTNU NanoLab and RICE University in cooperation with the central administration at NTNU and the Vice Consulate of Norway in Texas. The event was sponsored by the Research Council of Norway and the Nanonetwork.

Myfab-NorFab User Meeting in Uppsala

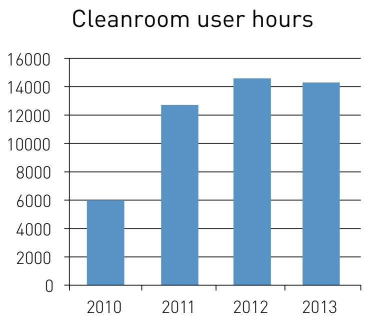
A common Myfab-NorFab User Meeting in Uppsala on the 17th-18th of April gathered 229 participants from Sweden and Norway. NTNU NanoLab participated in the planning of meeting and four of the lecturers came from NTNU. The main part of the meeting was allocated for sixteen thematic seminars within the following topics: Analysis, Thin Film technologies, Lithography and Etching technologies. In addition, four plenary speakers presented scientific highlights. The program also covered two inspiring presentations regarding academic and entrepreneurial exploitations. After the presentations on the the first day, there was a poster session in addition to organized lab tours. NorFab sponsored 45 Norwegian participants at the meeting.

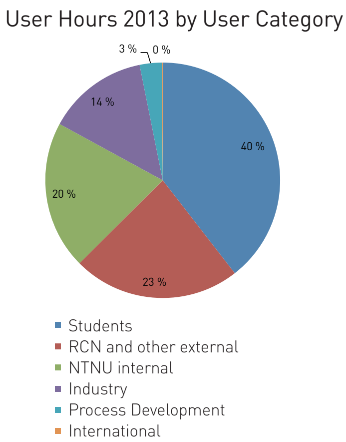
NTNU NanoLab's cleanroom

The cleanroom team has in 2013 focused on improved safety in the facility. New service routines for the gas system have been implemented to fulfill the new rules from the authorities. The procedures for evacuation alarm have been refined. Furthermore, service and maintenance of instruments and the cleanroom infrastructure have been improved to a professional level. A major task has been the reduction of the time for gaining access to the cleanroom and start-up of new activities. New this year, is a mandatory refresher course for active users who have taken the cleanroom course more than a year ago. Altogether, 164 people attended the cleanroom course in 2013 and 59 completed the refresher course.

Several new instruments have been installed, including a new mask aligner (SUSS MA6) with nanoimprinter (SCIL) and a T-CVD for synthesis of graphene. The FIB has been upgraded with micro manipulators and improved software to increase the user-friendliness.

In 2013, NTNU NanoLab had 207 active users in the cleanroom. The number of hours spent in the cleanroom was about the same as in 2012. About 40% of the user hours were generated by students at master level or below. Industry projects accounted for 14% and NTNU based research for 43%.





Training offered by NTNU NanoLab

- Cleanroom course
- Scanning (Transmission) Electron Microscopy (S(T)EM)
- Focused Ion Beam (FIB) with SEM
- Atomic force microscopy (AFM)
- Thin film deposition (PEVCD, sputter, evaporator)
- Dry etching (ICP-RIE)
- Wet etching (HF)
- Photolithography
- E-beam lithography
- Nanoimprinting
- Particle analysis

Module courses at Master level in NTNU NanoLab's cleanroom

- TFE4575 Physical Methods for Nanostructuring and Characterization
- TMT4515 Chemical Methods for Synthesis and Characterization of Nanomaterials
- TFY4525 Bionanotechnology
- TFY4335 Nano Life Science
- TFE4180 Semiconductor Manufacturing Technology
- TFY4265 Biophysical Micromethods
- TKP4190 Fabrication and application of nanomaterials
- FE8135 Nanostructuring

SINTEF: 3D studies of nano- and microstructures of cement using FIB-SEM

To enable efficient design of oilwell cements that can be placed with precision and that can withstand the harsh conditions often encountered downhole, it is important to understand how these materials are built up at smaller length scales. We have demonstrated how the nano- and micro-structure in the interior of set cement samples can be studied by using a Focused Ion Beam – Scanning Electron Microscope (FIB-SEM). This instrument performs nano-tomography, allowing small 3D cement volumes to be digitalized. In this way, chemical mapping of element distributions within the solid cement phases can be performed, and the 3D pore network can be reconstructed in great detail. This makes FIB-SEM an ideal experimental technique for investigating e.g. cement porosity, chemical attack on cements, cement-formation (de)bonding and structural or chemical effects of additives. The final output of FIB-SEM experiments, being digitalized microstructural features like grains or pores, can be used as input for numerical modeling schemes.

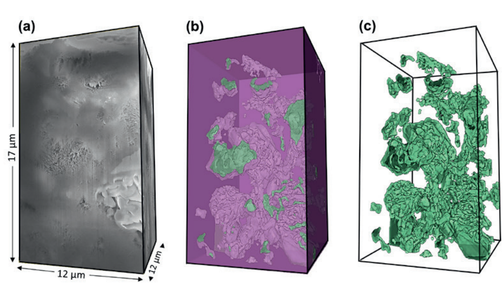


Fig. 6-(a) The reconstructed 3D volume based on the stack of SEM images recorded from the thick cement sample. (b) The segment volume (partly transparent) showing the complex nanoporous network inside the cement matrix. (c) The extracted 3D porous network. Copyright: Society of Petroleum Engineers.

Contact: Per Erik Vullum, SINTEF Materials and Chemistry.

SINTEF

Dissertations

The following candidates obtained a PhD degree at NTNU in fields related to nanoscience and nanotechnology in 2013. Highlighted candidates have carried out part of their work in NTNU NanoLab's cleanroom.

- Mercy Afadzi, *Delivery of Encapsulated Drugs to Cancer Cells and Tissue: The Impact of Ultrasound*
- ***Jamil Ahmad, Development of mixed matrix/nanocomposite membranes for gas separation***
- ***Magnus Breivik, Fabrication of mid-infrared laser diodes for gas sensing applications***
- Henrik Enoksen, *Quantum Transport in Hybrid Structures*
- Ming Gao, *High resolution characterization of responsive hydrogels for biomedical applications*
- Nitin Goyal, *Design and modeling of high-power semiconductor devices with emphasis on AlGaN/GaN HEMTs*
- **Sjoerd Hak**, ***Optimization of oil-in-water nanoemulsions for tumor targeting and molecular dynamic contrast enhanced MRI***
- Elisabeth Lindbo Hansen, *Soft Matter Physics of Clays and Clay suspensions: structural arrest, ordering, and host-guest interactions*
- Armen Julukian, *Nanofabrication and properties of gold and platinum nanostructures on graphite*
- **Fengliu Lou,** ***Aligned carbon nanotubes@ manganese oxide coaxial arrays for lithium ion batteries***
- **Hanne Kauko,** ***Quantitative scanning transmission electron microscopy studies on heterostructured GaAs nanowires***
- Kenji Kawaguchi, *Electrocatalysis and Novel Functions of Nano-Structured IrO2-Ta2O5/Ti Anodes Sourabh Khandelwal, Compact modeling solutions for advanced semiconductor devices*
- Tina Kristiansen, *Aerogels; a new class of materials for catalytic purposes*
- **Sina Maria Lystvet,** ***Emergent Properties of Protein-Gold Nanoconstructs for Biomedical Applications***
- Mohammad Alidoust Najafabadi, *Proximity Effects and Transport Properties of Nano-Scale Systems with Multiple Broken Symmetries*
- Tuan Anh Nguyen, *Polyhedral Oligomeric Silsesquioxanes: Effects on Adhesion, Water Resistance and Water Vapour Barrier Properties of Paperboard*
- Tor Nordam, *Scattering of light from weakly rough surfaces*
- Severin Sadjina, *Spin-Orbit-Induced Transport in Metals and Superconductors*
- **Eugenia-Mariana Sandru,** ***Polyene nanoparticles***
- Tor Olav Løveng Sunde, *Aqueous sol-gel processing of transparent conducting rare earth doped indium tin oxide*
- Guro Kristin Svendsen, *Electrooptical Modeling of Semiconductor Nanowires*
- Morten Tjelta, *Electrochemical and photoelectrochemical characterization of porous semiconducting electrodes*
- Haitao Zhou, *Nanostructured cathode materials for Li-ion batteries*
- Agnieszka Zlotorowicz, *Electrocatalysis for medium temperature PEM water electrolysis*

Board of NTNU NanoLab

- Prof. Bjørn Hafskjold / Prof. Anne Borg (from 01.08.2013), dean of the Faculty of Natural Sciences and Technology, NTNU (head of the board).
- Prof. Stig Slørdahl, dean of The Medical Faculty, NTNU.
- Prof. Ingvald Strømmen, dean of the Faculty of Engineering Science and Technology, NTNU.
- Prof. Geir Øien, dean of the Faculty of Information Technology, Mathematics and Electrical Engineering, NTNU.

Leader Group

- Director Kay Gastinger
- Prof. Nuria Espallargas / Associate Prof. Jianying He (from 01.07.2013)
- Prof. Tor Grande / Associate Prof. Fride Vullum-Bruer (from 01.08.2013)
- Prof. Øyvind Halaas
- Prof. Pawel Sikorski
- Associate Prof. Erik Wahlström
- Prof. Helge Weman
- Research manager Ragnar Fagerberg
- Coordinator Hanna Gautun
- Head of Laboratory Ida Noddeland

Awards

Sina Maria Lystvet was awarded the **Chorafas prize for the best PhD-thesis in 2013**. The title of her thesis was: «Emergent Properties of Proteins-Gold Nanoconstructs for Biomedical Applications».

Photo: Merethe Wagelund/NTNU Info

Management

Director: Dr. Kay Gastinger
Coordinator: Dr. Hanna Gautun

Permanent Technical Staff

- Head of Laboratory Ida Noddeland
- Senior engineer Mark Chiappa
- Senior engineer Espen Rogstad
- Senior engineer Trine Østlyng
- Senior engineer Espen Rogstad
- Staff engineer Ken Roger Ervik

Visit us at: www.ntnu.no/nanolab

Photo: Geir Mogen