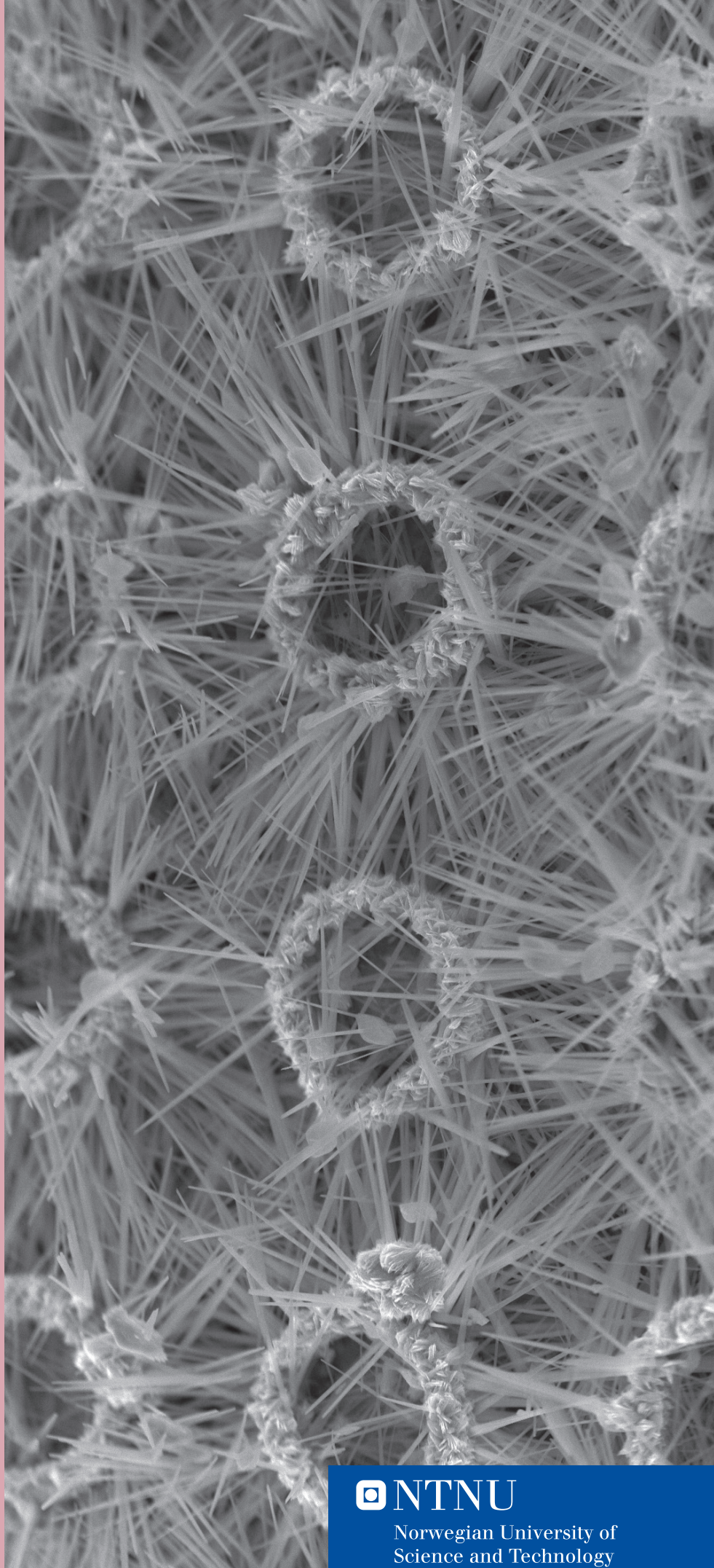
Annual Report NTNU NanoLab 2014



NTNU
Norwegian University of
Science and Technology

NTNU NanoLab 2014


Photo: Bente Rønning

NTNU NanoLab celebrated its 10 year anniversary in 2014 with a 2-days seminar, opened by the Minister for Education and Research, Torbjørn Røe Isaksen. Over the past 10 years the nano-community and -activity at NTNU has increased considerably. A state-of-the art cleanroom has been built and we have established a master study program in nanotechnology. Today, around 90 faculty members at NTNU work with nanoscience and nanotechnology. NTNU NanoLab has organised four significant meetings in 2014. Beside the 10 year anniversary and the Kavli Symposium on Nanoscience, two bilateral workshops took place. The NorTex Nano Summit gathered scientists in Texas while, the other workshop took place at NTNU with visitors from DTU Nanotech. The aim of these workshops was to foster common research proposals and promote exchange of graduate students.

The first phase of the national infrastructure project NorFab ended in 2014. Through NorFab, NTNU NanoLab received financial support for the cleanroom operation and new equipment from the Research Council of Norway (RCN). The NorFab consortium has applied for continued support from the INFRASTRUCTURE initiative of RCN. The outcome of this proposal will have a crucial influence on the future development of the infrastructure. In the meantime, the RCN program, NANO2021, has funded part of the operational costs to provide NorFab support in 2015.

I think we can be proud of NTNU NanoLab's development over the last 10 years. The strategic initiative on nanoscience and -technology at NTNU has been a great success. In an effort to strengthen the strategic coordination within nanoscience, nanotechnology and functional materials, NTNU has launched the initiative Nano@NTNU, starting 01.01.2015. A separate scientific leader will be hired to lead this activity. NTNU's cleanroom will continue as NTNU NanoLab with me as director. Thus, the framework for the next 10 years should be in place.

Finally, it was very motivating for the Nano community, to see Moser & Moser win the Nobel Prize in 2014. I hope one of the nanoresearchers at NTNU can follow their example one day.

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. With an initial grant for four years, 2014 was the last year of NorFab I. In this period 145 projects have been supported with 17,8MNOK in total.

In 2014 NorFab has undertaken several networking initiatives. The Nordic NanoLab Network (NNN) now includes Myfab (Sweden), DTU Danchip (Denmark) and VTT Micronova (Finland) and NorFab. The NNN had two meetings at the leader group level to exchange best practice experiences. In addition, the Nordic NanoLab Expert Network (NNEN) constitutes a forum for exchange of experiences within micro- and nanofabrication among engineers. In 2014, NNEN organized workshops in lithography, thin film and dry etch methods. All workshops had between 15-20 participants from 19 cleanrooms in the Nordic countries. In between the meetings the experts exchange experiences in an online forum, which has 79 members in the 4 different groups.

On the national level, NorFab and Vannklyngen organized a Matchmaking Seminar for academic staff and industry representatives in Tønsberg, the 14th of May.

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. Funding from NANO2021 ended in 2013, but the network activities continue on funding from the Division of Science, RCN, Program for National Graduate Schools, since January 2014.

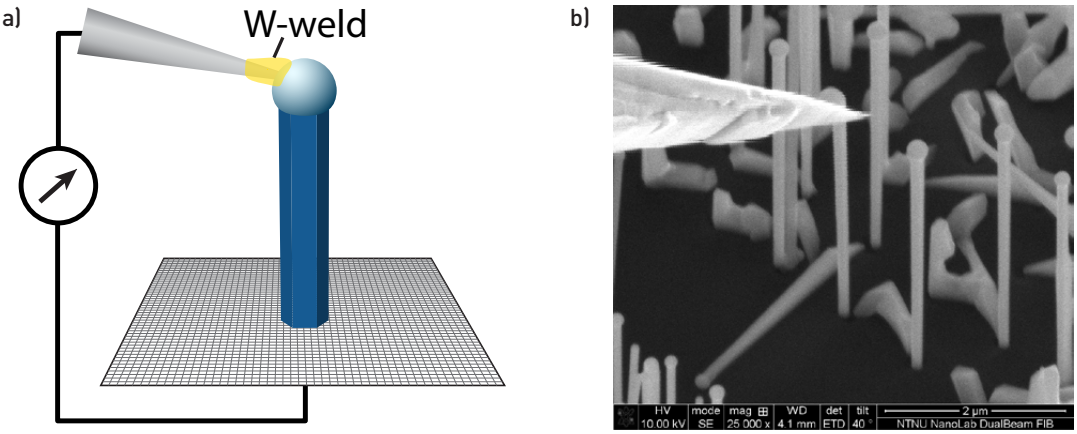
In 2014, the Network offered 8 PhD courses on compact format and provided travel support for the candidates to attend these courses. The network has provided funding for laboratory and processing expenses to 19 PhD candidates and granted travel support to 19 candidates presenting their work at international conferences. Our 2014 annual workshop was held in Tønsberg from June 16th to 18th, gathering 86 participants from the Norwegian research communities in nanotechnology and microsystems.

Website: www.nano-network.net

Front page: SEM image of CuO nanoneedles grown both inside and outside hollow copper micropillars on copper substrate as an attempt to develop a superhydrophobic, anti-icing surface. The image was taken using NanoLab's Hitachi S-5500 SITIEM. Photo: Zhiwei He.

In-situ electronic probing using focused ion beam

To optimize the electronic properties of nanostructures, their electronic characteristics have to be probed. Because of the dimensions, this is usually done in a scanning electron microscope. Applying a focused ion beam (FIB) system where the nanostructure or the contacts to it can be modified and regions of interest extracted for more detailed characterization offers new possibilities in this field [1]. In 2013, NTNU NanoLab installed an *in-situ* probing station for the FIB, consisting of four mobile micro-robots. The new system is stable, flexible and user-friendly. It has already been used to electronically probe single p-doped GaAs nanowires and was found to have a higher throughput than a conventional probing routine [2,3].

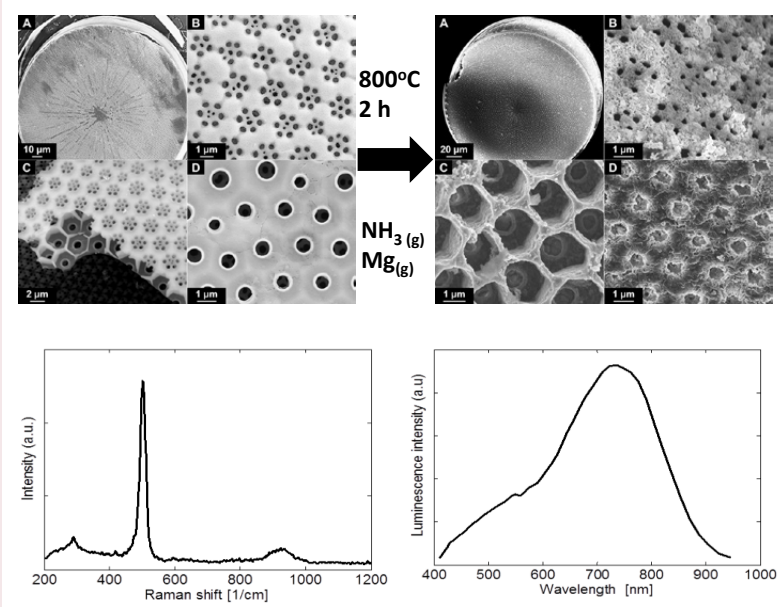


(a) Schematic and SEM image of probing a single GaAs nanowire on the growth substrate [1]. (b) SEM image of a 4-probe set-up where contacts are made in-situ by electron beam induced deposition [3].

[1] V. T. Fauske, D. C. Kim, A. M. Munshi, D. L. Dheeraj, B.O. Fimland, Weman, A. T. J. van Helvoort , J. Phys.: Conf. Series, 522 (2014) 012080. *[2] A. T. J. van Helvoort, Webinar Imina Technologies, 24 Sept. 2014. [3] M. Erlbeck, MSc Nanotechn. Thesis NTNU, 2014.*

Synthesis and properties of silicon/magnesium silicon nitride diatom frustule replicas

Diatom frustule replicas mainly consisting of magnesium silicon nitride and nanocrystalline silicon have been obtained by simultaneous metallothermic reduction and nitriding of silica diatom frustules at 800°C. The frustule replicas retained most of the complex nanoporous structures from the original frustules in the conversion. The optical scattering, transmittance and luminescence properties of the replicas have been investigated. Luminescence was observed and attributed to the presence of silicon nanocrystals. Wavelength dependent diffraction of light was observed in bio-silica frustules but not in frustule replicas, which was attributed to surface coarsening of the replicas during the reaction. The light transmittance was found to be lower in frustule replicas and was consistent with absorption of light by Si nanocrystals.

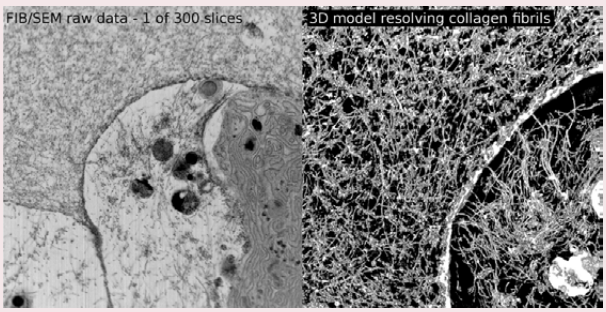


SEM images of diatom frustules before and after reaction along with corresponding observed Raman shift and luminescence (above).

I. A. Ødegård; J. C. Romann; A. Fossdal; A. K.Røyset; G.Tranell. Journal of Materials Chemistry A. 591 (2014) 175.

Structural characterization of neocartilage with FIB/SEM tomography

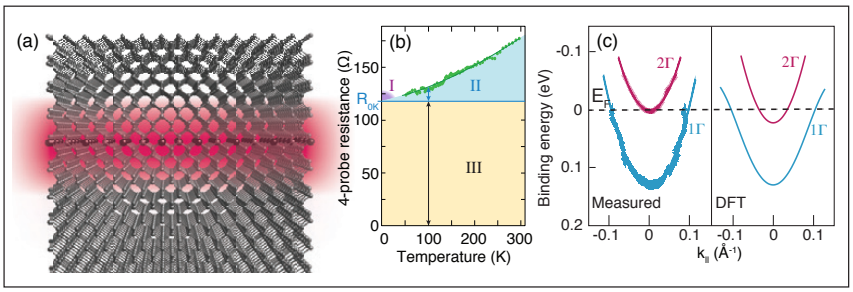
The Focused Ion Beam (FIB) instrument can be used to provide new insights into biological materials through 3D representation of nanoscale structures. We have used FIB/SEM tomography to study artificial cartilage (formed by chondrogenic differentiation of mesenchymal stem cells in alginate hydrogels), with the aim to understand ultrastructure and organization of collagen matrixes produced by the cells. Immunohistochemistry and second harmonic generation microscopy indicated that type II collagen was produced and distributed well within the material, but assembled into fibrils only in the pericellular space. FIB/SEM was used to obtain 300 serial SEM micrographs of extracellular microenvironment (The figure shows an area of about 20x20μm^2.), which were used to reconstruct 3D models of the collagen fibril network. The FIB/SEM data set had a resolution comparable with transmission electron microscopy allowing to resolve individual collagen fibrils, while at the same time providing full 3D information for investigated sample volume (20x20x7μm^3). This method showed that collagen assembled into fibrils both in pericellular space and further away from the cell when high cell density was used, and provides new insight into properties and formation process of this biomaterial.



Contact: P. Sikorski, Dept of Physics, NTNU. Ref: M. Olderøy et al. *PLoS ONE*, 9 (2014) e91662.

Silicon Quantum Devices

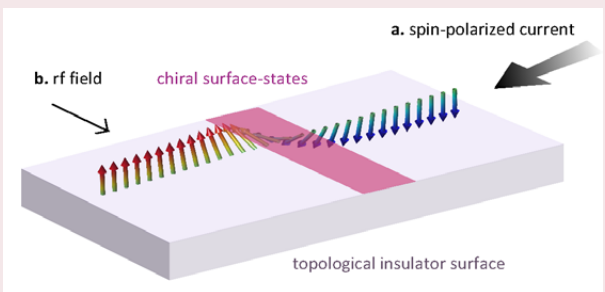
Silicon based quantum devices have advanced to the ultimate limit where individual dopant atoms dictate device behaviour. In this regime, device behaviour is dictated by quantum mechanics and an incredible range of new properties are possible. For example, silicon quantum devices attract interest for high performance quantum dot photovoltaics, LEDs and for quantum computation applications. In all cases, a key requirement is to understand, and to be able to manipulate, the electronic properties, especially the electronic bandstructure and many-body interaction. This has generally not been possible because of the difficulty of the experiment. In our work, we have created a number of silicon based quantum devices, and demonstrated that it is possible to directly measure their band structure and many-body interactions. This information will be of central importance in the ongoing development of silicon based quantum electronics.



Contact: J. Wells, Dept of Physics, NTNU. Ref: Miwa et al. Nano Letters, 14 (2014) 1515; Mazzola et al. *Appl. Phys. Lett*. 104 (2014) 173108; Miwa et al., *PRL* 110 (2013) 136801; Polley et al. *ACS Nano* 7 (2013) 5499

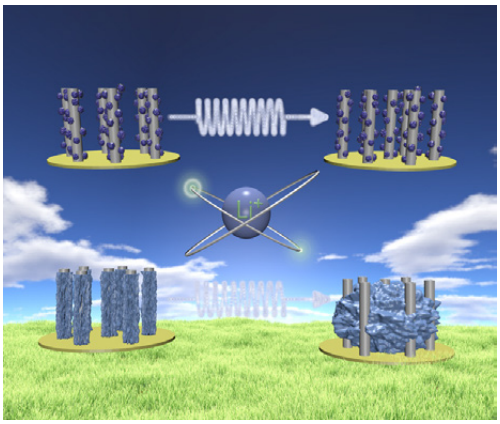
Improved domain-wall dynamics and magnonic torques using topological insulators

We have determined the magnetization dynamics that arises when a thin-film ferromagnet is deposited on a topological insulator (TI). Focusing on domain wall motion and the possibility of spin-wave torques, we show analytically that a number of interesting physical effects arise. The coupling between the domain wall and the TI stabilizes one particular chirality and topological charge of the magnetic texture. Moreover, the Walker breakdown threshold is substantially increased, allowing for higher attainable wall velocities. Finally, we show that spin-wave excitations in this system act with a torque even on homogeneous magnetization textures.



Ref: J. Linder, *Phys. Rev.* B 90 (2014) 041412(R)

Coaxial Carbon/Metal Oxide/Aligned Carbon Nanotube Arrays for Lithium Ion Batteries

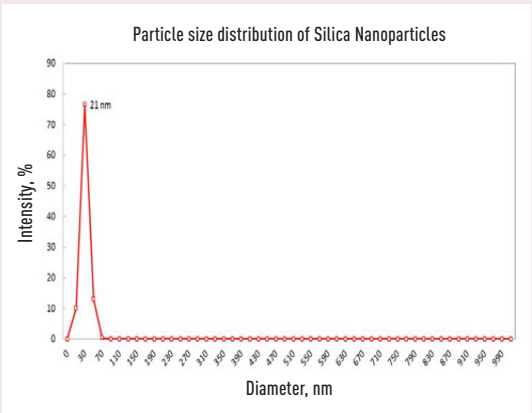


Coaxial carbon/metal oxide/aligned carbon nanotube arrays over stainless-steel foil are reported as high-performance binder-free anodes for lithium ion batteries. The external carbon layer was recognized as a key component for high performance. This layer increases rate capability by enhancing electrical conductivity and maintaining a low mass-transfer resistance and also improves cyclic stability by avoiding aggregation of metal-oxide particles and stabilizing the solid electrolyte interface. These coaxial nanotube arrays present a promising strategy for the rational design of high-performance binder-free anodes for lithium ion batteries.

Contact: D. Chen, Dept.of Chemical Engineering, NTNU,
Ref: Lou, F. et al., *ChemSusChem*, 7 (2014) 1335; Lou, F. et al., *J. Energy Chemistry*, 22 (2013) 78.

Effects of the initial rock wettability on silica-based nanofluid-enhanced oil recovery processes at reservoir temperatures

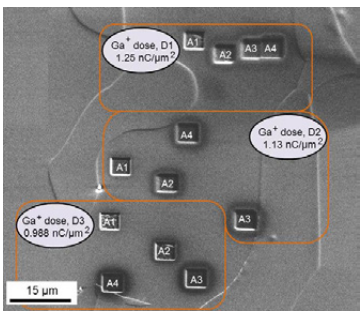
Nanoparticles offer several benefits in nanofluid-enhanced oil recovery, since they can easily be produced synthetically from abundant, non-toxic inorganic material such as silica (SiO_2). The surface properties of the particles can be controlled and manipulated. The nanoparticles are much smaller than typical reservoir pore throats and therefore have the ability to propagate in the reservoir rock. The precise size distribution of the particles dispersed in saline water (which represents the formation water in the reservoir) was studied with the NanoSight instrument at NTNU NanoLab. The result is shown to the right. Also, core flood experiments were designed to establish the relationship between reservoir wettability (water-, intermediate- and oil-wet systems) and oil recovery due to nanoparticles at reservoir temperatures. The results showed that the initial wettability affects oil recovery performance and showed greatest effect at higher temperatures.

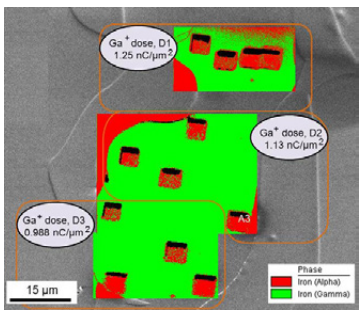


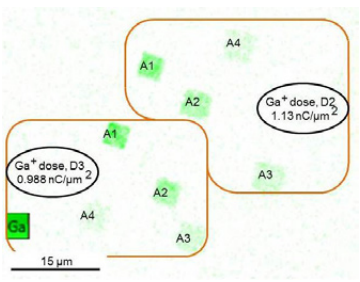
Ref: L. Hendraningrat and O. Torsæter, *Energy & Fuels* 28 (2014) 6228.

Chemically induced phase transformation in austenite by Focused Ion Beam (FIB)

Changing the chemical composition of a highly stable austenite phase in a super duplex stainless steel by gallium implantation resulted in a phase transformation. Different areas within the same austenite grain were subjected to a milling process using a combination of three different gallium doses and four acceleration voltages by the means of FIB at NTNU NanoLab. Further analysis of the investigated grain showed no evidence of shear or plastic deformation expected during a typical martensitic deformation. Thus, it was concluded that implanted gallium (green in the last picture), which is a ferrite stabilizer, was locally changing the chemical composition which resulted in the transformation of austenite (green phase in the middle picture) to ferrite (red phase).







Ref: A. Basa, C. Thaulow and A. Barnoush, *Metallurgical and Material Transactions* A (2014) 1189.

Kavli Prize Lectures and Kavli Nanoscience Symposium, NTNU 11.09.2014

The Kavli Prizes in nanoscience 2014 were given to Thomas W. Ebbesen, Sir John Pendry and Stefan W. Hell *"for their transformative contributions to the field of nano-optics that have broken long-held beliefs about the limitations of the resolution limits of optical microscopy and imaging."* After the celebrations in Oslo, the laureates came to Trondheim to give their prize lectures at NTNU on 11.09. In affiliation with the prize lectures, NTNU NanoLab organized the half day Kavli Nanoscience Symposium featuring the following international speakers:

- Sir Andre Geim, University of Manchester
- Bo Brummerstedt Iversen, Aarhus University
- Molly Stevens, Imperial College of London
- Ke Lu, Chinese Academy of Science, Shenyang

The prize lectures and symposium gathered more than 250 participants

Rector Gunnar Bovim together with the Kavli Prize Laureates: 1. row, left: Brenda Milner, Sir John Pendry and Thomas Ebbesen. 2. row left: Gunnar Bovim, John O'Keefe, Marcus E. Raichle and Stefan W. Hell.

Kavli Nanoscience Symposium (invited speakers and guests): 1. row left: Dean Anne Borg, Kavli Laureate 2012 Mildred Dresselhaus, Molly Stevens. 2. row left: Head of the scientific committee Pawel Sikorski, Bo Brummerstedt Iversen, Sir Andre Geim, Ke Lu.

NTNU NanoLab 10 Year Anniversary, Trondheim, 10.-11.11.2014

NTNU NanoLab was established in 2004 with the mandate to coordinate the research within nanoscience and nanotechnology at the university, build a cross disciplinary cleanroom and establish a 5-year master program in Nanotechnology.
The anniversary opened with greetings from the rector of NTNU, Gunnar Bovim and the Minister of Education and Research, Torbjørn Røe Isaksen, followed by Aase Marie Hundere, from the Research Council of Norway and Jack Ødegård, Executive vice president of SINTEF Materials and Chemistry.

From the left: Rector NTNU Gunnar Bovim, Minister of Education and Research Torbjørn Røe Isaksen, Director Nanolab Kay Gastinger, Dean Anne Borg and Dean Geir Øien.

The program featured 6 invited international speakers covering bionanoscience, nanomaterials and nanoelectronics, as well as focus on invention and development of robust infrastructures:

- Hans Mooij, Technical University of Delft
- Séverine Le Gac, MESA+ Research Institute for Nanotechnology, University of Twente
- Paula Vilharino, University of Aveiro
- Jeppe Lauritsen, iNANO – University of Aarhus
- Lars Samuelson, Lund University
- Miriam Luizink, Director of Strategic Business Development, University of Twente

Altogether the program consisted of 26 scientific presentations and a poster session exhibiting 18 posters.
A total of 145 PhD-students, post docs and scientific staff participated in the celebration.

NorTex NanoSummit in Houston, Texas 12.-14.10.2014

This seminar was a follow-up of the "Norway-Texas Seminar and Workshop on Nanoscience and –Technology" at NTNU in 2013 and the "Trans Atlantic Science Week" in Houston in 2012.The main goal was to promote collaboration between researchers in Texas and Norway. The Norwegian delegation counting 10 scientific staff members and 11 post docs and PhD-candidates, was headed by state the secretary of the Norwegian Ministry of Trade, Industry and Fisheries, Dilek Ayhan. The main focus of the summit was nanomedicine, nanomaterials and nano for oil & gas. The program lasted for 2.5 days, including plenary talks, thematic workshops, a poster session, and lab tours. In addition, ample time was allocated for informal talks in a social atmosphere. The summit gathered around 100 participants from Texas and was organized by Rice University and NTNU NanoLab. The summit was supported by the Research Council of Norway and the Royal Norwegian Consulate General in Houston.

DTU – NTNU NanoTech Seminar, 1.-2.12.2014

As part of the program for a rector led visit from DTU to NTNU, NTNU NanoLab organized a seminar/workshop focusing on nanotechnology and nanomedicine. The aim of the event was to seek out potential constellations for future cooperation, through exchange of students and common EU proposals. The program consisted of 15 presentations, 7 lab tours and 4 meetings, and involved 25 staff members from NTNU.

NTNU NanoLab's cleanroom

NTNU NanoLab acquired two new instruments in 2014. In June, a Micro-Raman Spectrometer was installed. It uses VIS excitation at 532 nm and NIR at 785 nm, and has a spectral range from 200 to 1050 nm. It is fully upgradeable to both UV and IR ranges. In addition, a Chemically Assisted Ion Beam Etch (CAIBE) using Ar and O_2 to etch all types of materials was installed in September. More information about these and all other tools in the cleanroom can be found on www.ntnu.norfab.no.

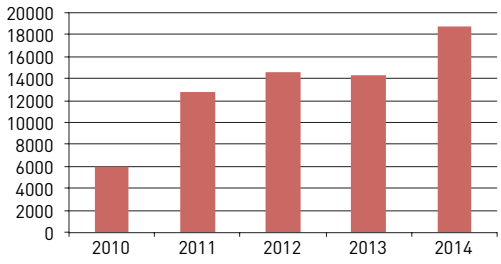
In cooperation with eager users, a process data base has been created on the URL lab.tips. Here, processes within lithography, dry etch, microfluidics, etc., are described and animated, and can be discussed. The data base is still in its infancy, but our hope is that it will become a useful tool for our users.

In August, we welcomed Mathilde Barriet as a new member of the staff, and we are now 6 engineers running the cleanroom. During 2014, the cleanroom staff has participated in the NNEN – Nordic Nanotechnology Expert Network, organized through NorFab. In addition, one of the engineers was part of a Norwegian Labour Inspection Authority project, which has collected knowledge about health issues regarding nanomaterials and embodied this in national guidelines for safe handling of nanomaterials at workplaces.

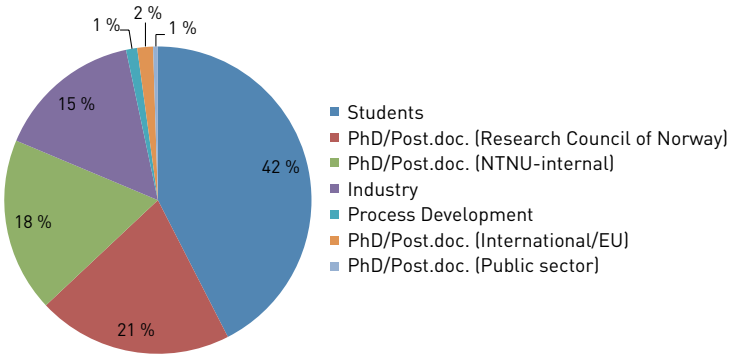
In 2014, we had more than 200 active users of the cleanroom. The number of user hours has increased with 43% since 2013, to over 18.600 hours in 2014. The distribution of user hours was the same as in 2013, with 43% of the user hours accounted for by NTNU based projects, 15% by industry and 42% by students at master level or below.

NTNU NanoLab is a service organization for researchers and students alike, and we continuously work with improving our services. An important tool is our user survey. In 2014, 68 users participated. With the cleanroom operation and the NanoLab staff.

User hours NTNU NanoLab



User hours by User Category



Training offered by NTNU NanoLab

- Cleanroom course
- Chemical handling
- Scanning (Transmission) Electron Microscopy (S(T)EM)
- Focused Ion Beam (FIB) with SEM
- Atomic force microscopy (AFM)
- Micro Raman Spectroscopy
- Thin film deposition (PECVD, sputtering, evaporation)
- Dry etching (ICP-RIE, CAIBE)
- Wet etching (HF, Piranha)
- 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
- TFY4330 Nanotools
- TFE4180 Semiconductor Manufacturing Technology
- TFY4265 Biophysical Micromethods
- TKP4190 Fabrication and application of nanomaterials
- FE8135 Nanostructuring

Grey Goo Symposium at NTNU, 12.02.2014

The Grey Goo symposium was arranged for the first time in 2014 as an effort to create a meeting place for companies and students enrolled in the master program in nanotechnology at NTNU. The aim of the symposium was to promote exchange of knowledge and ideas, and to prepare the ground for an increase in the number of collaborative master projects with the industry. Both students and industry representatives were thus given the opportunity to present their expertise, creating an atmosphere for further talks in the breaks. Around 80 students and 5 companies participated in the symposium, sponsored by NTNU NanoLab. The symposium was organised by the organisation of the nanotechnology students at NTNU, Timini.

Timini

Dissertations

The following candidates obtained a PhD degree at NTNU in fields related to nanoscience and nanotechnology in 2014. Highlighted candidates have carried out part of their work in NTNU NanoLab's cleanroom.

- **Mustafa Hasan Balci, *Solution based synthesis of Si quantum dots and downshifting layers for solar cell applications***
- Katrine Lie Bøyesen, *Reactivity and Synergism of Vanadium in Microporous Supports with Copper as a Co-cation*
- Mahdi Darab, *Dispersion, Performance and Durability of Pt Nanoparticles on Carbon for PEM Fuel Cell Catalysis*
- **Pål Gunnar Ellingsen, *Polarimetric and hyperspectral imaging methods for characterization of bio- and nanomaterials***
- Mari Helene Farstad, *Titania Thin Films on Gold and Palladium Surfaces – Structure and Properties*
- Vasco Rafael Póvoa Fernandes, *Palladium alloy model systems for understanding the surface properties of Pd-based membranes and catalysts*
- Jostein Bø Fløystad, *In situ hard X-ray ptychographic imaging for materials science*
- **John Olav Grepstad, *Patterned Dielectric Membranes Designed for Optical Sensing of Nano Particles***
- Astri Bjørnetun Haugen, *Synthesis and characterisation of textured lead-free piezoelectric ceramics*
- Astrid Marie Flattum Muggerud, *Transmission electron microscopy studies of dispersoids and constituent phases in Al-Mn-Fe-Si alloys*
- **Abdul Mazid Munshi, *Epitaxial Growth of Self-Catalyzed GaAs Nanowires by Molecular Beam Epitaxy***
- Vajiheh Nafisi: *Development of Mixed Matrix Membranes for Carbon Dioxide Capture*
- **Bjørn Rune Rogne, *Nanomechanical testing of iron and steel***
- Takeshi Saito, *The Effect of Trace Elements on Precipitation in Al-Mg-Si alloys – A Transmission Electron Microscopy Study*
- Tom Gøran Skog, *Development of Hollow Fiber Membranes and Membrane Modules Suitable for High Pressure Natural Gas Sweetening*
- Ragnhild Skorpa, *Hydrogen dissociation under equilibrium and non-equilibrium conditions*
- **Malin Sletnes, *Wet chemical synthesis of silicon nanoparticles and phosphorescent oxides for white light emitting diodes***
- **Ky-Nam Pham, *Potassium sodium niobate thin films by chemical solution deposition***
- Nicla Vicinanza, *An investigation of fundamental phenomena affecting the performance of sputtered Pd alloy thin film membranes for hydrogen separation*
- Daham Sanjaya Gunawardana Panditha Vidana, *Carbon formation phenomena and the initial stage of metal dusting corrosion – an experimental investigation*
- **Georg Voss, *Mesostructured alumina and the state of Ni as promoter for Co Fischer-Tropsch synthesis catalysts***
- Alexey Voronov, *Sensitivity enhancement of X-ray absorption spectroscopy applied to Co-based Fischer-Tropsch synthesis catalysts*
- Sigurd Wenner, *Transmission Electron Microscopy and Muon Spin Relaxation Studies of Precipitation in Al-Mg-Si Alloys*
- Min Zha, *Development and characterization of high solid solution Al-Mg alloys and an immiscible Al-Bi alloy processed by ECAP and annealing*

Kai Sandvold Beckwith's picture of a splitting cancer cell was selected as the **best image in 2014 by the national research school for medical imaging.**


Photo: Berit Myhre

The NanoNetwork's awards for best student presentation and best poster were won by Federico Mazzola and Jonas Ribe (photo), both from the Dept of Physics at NTNU.

Board of NTNU NanoLab
- Prof. Anne Borg,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
- Dr. Ellen Dahler Tuset, Kongsberg Norspace AS, Commersial director

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Coordinator: Dr. Hanna Gautun

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- Senior engineer Dr. Mathilde Barriet
- Senior engineer Mark Chiappa
- Senior engineer Ken Roger Ervik
- Senior engineer Espen Rogstad
- Senior engineer Trine Østlyng Hjertås

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- Associate Prof. Hilde Lea Lein
- Prof. Øyvind Halaas
- Prof. Pawel Sikorski
- Associate Prof. Erik Wahlström
- Prof. Helge Weman
- Research manager Dr. Ragnar Fagerberg
- Coordinator Dr. Hanna Gautun
- Head of Laboratory Linda Helander

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