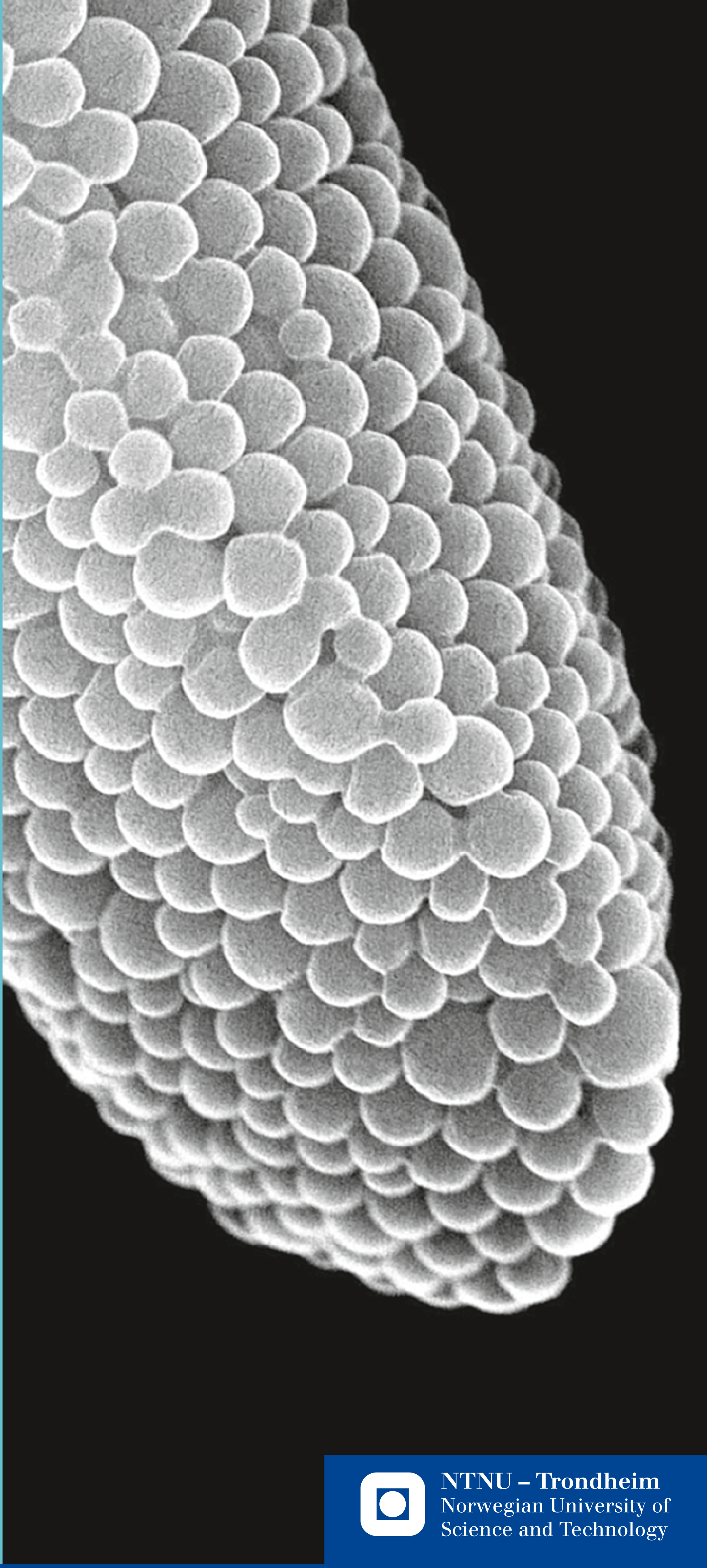
Annual Report NTNU NanoLab 2012



NTNU – Trondheim
Norwegian University of
Science and Technology

NTNU NanoLab 2012

NTNU NanoLab showed a positive development of the cleanroom infrastructure as well as the scientific production in 2012. The year was marked by the transition from the Research Council of Norway's main research program for nanoscience and –technology; NANOMAT to the new program NANO2021. Many research groups at NTNU were active participants in NANOMAT and are about to strengthen their role in the successor. Thus, when NANO2021 called for national coordinated projects in 2012, NTNU researchers led 7 out of 37 project proposals and were partners in at least 11 others. In addition, cooperation with SINTEF in threefold consortia between SINTEF, NTNU and industry can become a potential area of growth of nano-related research at NTNU in the next years.

Along with increasing research activity the use of the cleanroom is increasing. In 2012 we registered almost 15.000 user hours, while 8 PhD students with activity in the cleanroom received their degree. The cleanroom and all tools are now operational at a high scientific level and the efficiency of operations has been improved considerably.

Thus, the NanoLab organisation shifted its main focus during the last year. We have now been able to put more effort in coordination and support of nanotechnology research at NTNU. An important event was the match-making seminar for researchers from NTNU, SINTEF and industry carried out in November. At least 3 out of the 10 participating industry representatives contributed in the autumn call of NANO2021.

NorFab made significant developments as a national infrastructure during the last year. Together with our partners we published a common price policy and intensified the cooperation with Myfab, our Swedish sister organisation. However, the most important activity was the preparation of a new application within the Infrastructure program of the Research Council of Norway.

Finally, in 2012 our first start-up company based on activities in NTNU NanoLab's cleanroom, CrayoNano AS, was founded. Companies like this are important building blocks in a developing nanotechnology industry in Norway. NTNU NanoLab provides the fundament for this development with world leading research in nanoscience and -technology and the cleanroom infrastructure. We wish, and intend to play a leading role to contribute to a new industry adventure in Norway, in addition to oil and gas.

You are welcome to join us!

Kay Gastinger
Director

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 Vestfold University College. A milestone in 2012 was the official opening of the Research Centre at Vestfold University College on the 21st of September.

Minister of Trade and Industry, Trond Giske at the opening of the Research Center at Vestfold University College.

All together NorFab now offers 1240 m^2 cleanroom areas with common price list and booking system as well as user support. In 2012, NorFab supported 28 short term projects and 18 long term projects. Information regarding equipment, prices and options for support is given on NorFab's website: www.norfab.no

NanoNetwork

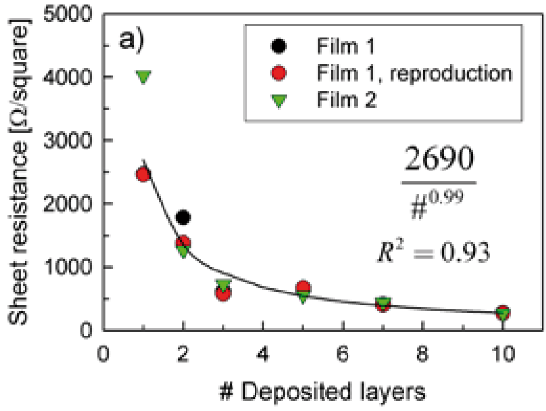
The Norwegian PhD Network on Nanotechnology for Microsystems was established in 2009, with the objective to coordinate, integrate, and strengthen PhD programs in the field of nanotechnology and microsystems in Norway. The partner institutions are NTNU, University of Oslo, University of Bergen, Vestfold University College, and SINTEF ICT Microsystems and Nanotechnology. The network activities will continue with funding from the Division of Science, RCN, granted in 2012.

In 2012, the PhD Network offered 8 PhD courses on compact format and gave travel support to 18 candidates attending these courses. The network has provided financial support for laboratory and processing expenses to 25 PhD candidates in the network, and granted travel support to 40 candidates presenting their work at international conferences and 5 candidates on extended research visits abroad.

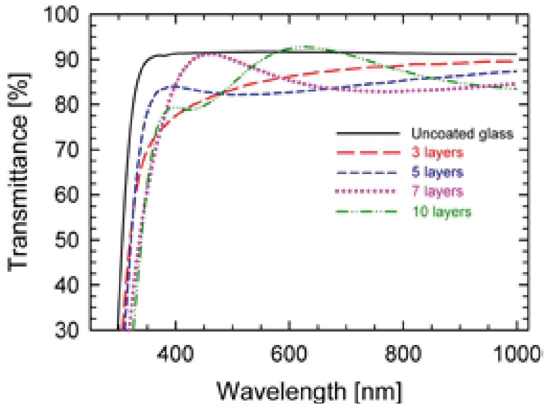
The 2012 annual workshop was held in Trondheim from June 11th to 13th, gathering 103 participants from the research communities of nanotechnology and microsystems. Website: www.nano-network

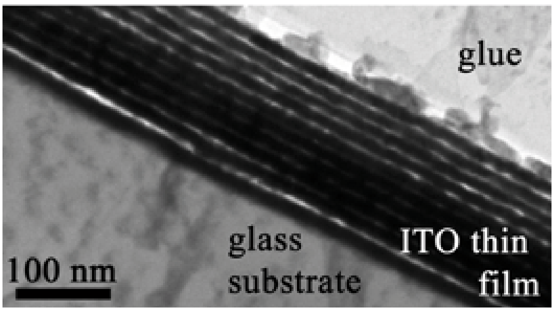
Front page: SEM-image of freeze dried polybutylcyanoacrylate (PBCA) nanoparticles to be used in drug delivery. The purpose of the imaging was to find out whether the particles were affected by the freeze drying process, a common way to store nanoparticles. Copyright: Andreas Gagnat Bø, Yrr Mørch and Catharina Davies, NTNU.

Transparent and conducting ITO thin films by an environmentally friendly spin coating technique



Transparent conducting oxides (TCOs) have a unique combination of properties and have found numerous technological applications. Indium tin oxide (ITO) is known as the state of the art TCO. By using the spin coater and the RTP furnace in NTNU NanoLab, ITO thin films have been deposited by a new water-based sol-gel process. The process is simple, inexpensive and environmentally friendly compared to other techniques and the prepared thin films demonstrate very good optical transparency and electrical conductivity.





Sheet resistance (top left), transmittance (bottom left) and TEM micrographs (right) of the deposited ITO films.

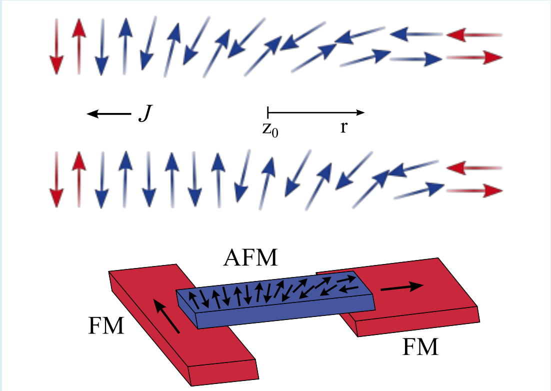
"Transparent and conducting ITO thin films by spin coating of an aqueous precursor solution", T.O.L. Sunde, E. Garskaite, B. Otter, H.E. Fossheim, R. Sæterli, R. Holmestad, M.-A. Einarsrud and T. Grande, J. Mater. Chem., 22 (2012) 15740.

Staggered Dynamics in Antiferromagnets by Collective Coordinates

Antiferromagnets (AFMs) can be used to store and manipulate spin information, and new developments have created opportunities to use AFMs as active components in spintronics devices. The dynamics of AFMs are described by equations which are very complex and with many degrees of freedom. The Brataas group at the Department of Physics, NTNU has presented a theory which is conceptually much simpler, using collective coordinates to describe staggered field dynamics in antiferromagnetic textures. The theory includes effects from dissipation, external magnetic fields, as well as reactive and dissipative current-induced torques. The following equations of motion for the collective modes have been derived:

$$M^{ij}(\ddot{b}_j + a\gamma G_2 \dot{b}_j) = F^i$$

where M^{ij} is the acquired mass of the effective particles described by the collective coordinates b_j, $a\gamma G_2$ parameterizes dissipation, and F^i represent external forces. In conclusion, the theory shows that at low frequencies and amplitudes, currents induce collective motion in AFMs by means of dissipative rather than reactive torques.



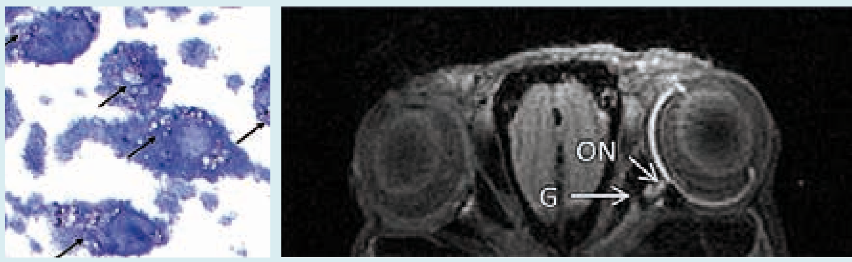
A one-dimensional antiferromagnetic texture pinned between two ferromagnets and driven by a current J.

"Staggered Dynamics in Antiferromagnets by Collective Coordinates", E.G. Tveten, A. Qaiumzadeh, O.A. Tretiakov and A. Brataas, Phys. Rev. Lett. 100 (2013) 127208.

Nanomedicine and image-guided Central Nervous System repair

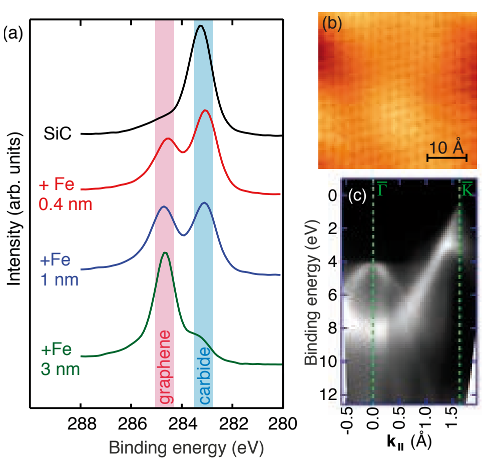
A major impact of nanotechnology in regenerative medicine is the facilitation of the design, application and assessment of multifactorial approaches to therapy with a view to clinical translation. Such approaches include the use of contrast agents for in vivo monitoring of cell transplants and neuronal pathways by MRI. We have incorporated T2/T2* contrast agents into cells for visualisation by MRI and combined them with T1 contrast agents for simultaneous assessment of neuronal circuit integrity and axon-regenerative responses to treatment. Having established the utility of this approach, we are currently designing and testing novel nanoparticles for multimodal molecular and cellular imaging as well as gene and drug delivery for repair of experimental CNS lesions. We are also utilising nanotechnology for the development of multifunctional biopolymers for in situ tissue engineering. This work represents a collaboration between the Department of Circulation and Medical Imaging, and the Departments of Biotechnology and Chemical Engineering at NTNU.

Left image: Cells after uptake of intracellular contrast agent (arrows). Right image: MRI of cell transplant and regenerating CNS axon tract after injury. ON: Manganese-enhanced optic nerve; G: cell graft at injury site.



"In vivo MRI of olfactory ensheathing cell grafts and regenerating axons in transplant mediated repair of the adult rat optic nerve", I. Sandvig, M. Thuen, L. Hoang, Ø. Olsen, T.C. Sardella, C. Brekken, K.E. Tvedt, S.C. Barnett, O. Haraldseth, M. Berry and A. Sandvig, NMR Biomed. 25 (2012) 620.

Controlled graphene formation on semiconductors



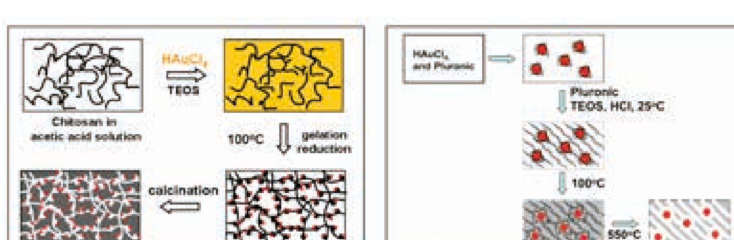
Graphene has excited much interest for its novel mechanical and electronic properties, and has been proposed as an ideal material for a wide range of applications. In order to capitalise on this, it is necessary to control its growth in an industrially realistic manner on poorly conducting substrates. In this project, we developed a method for growth on diamond and SiC at moderate temperatures, by making use of a chemical intermediate (in this case Fe/FeSix). This allows for graphene-on-semiconductor formation at industrially realistic temperatures, where the thickness and lateral distribution can be controlled by standard lithographies of the intermediate. The work was carried out at the Department of Physics in collaboration with Aberystwyth University, Aarhus University and the MAX IV laboratory.

(a) XPS measurements of the carbide to graphene reaction, showing its dependence on the initial Fe quantity. (b) STM image of the graphene layer formed and (c) ARPES measurement showing the bandstructure of the graphene with its characteristic pi-band reaching a Dirac-point at K̄.

"Iron-mediated growth of epitaxial graphene on SiC and diamond", S.P. Cooil, F. Song, G. T. Williams, O.R. Roberts, D.P. Langstaff, B. Jørgensen, K. Høydalsvik, D.W. Breiby, E. Wahlström, D.A. Evans, J. W. Wells, Carbon 50 (2012) 5099, Contacts: D. W. Breiby, E. Wahlström and J. Wells.

One-pot synthesis of gold nanoparticle functionalised mesoporous silica

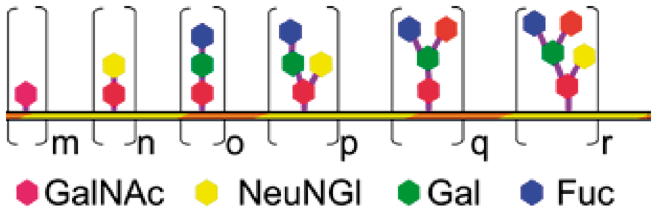
Gold nanoparticle functionalised materials are considered as good catalysts for many reactions, especially in liquid phase. The main challenge is to obtain materials in which the nanoparticles are homogenously dispersed, well bound to the support surface and still accessible for the substrates. To reach these goals for gold nanoparticle functionalized mesoporous silica, a one – pot synthesis comprising a tri-block copolymer and chitosan has been proposed. In this approach the polymers introduce mesoporosity into the silica material and simultaneously reduce the use of the gold precursor. After burning out of the template the nanoparticles reside in the cage like pores linked with a mesoporous network.



"One-pot synthesis of gold nanoparticle functionalised mesoporous silica - The double role of a tri-block copolymer and chitosan", B. Gawel, K. Lambrechts, K. Gawel and G. Øye, Microporous and Mesoporous Materials, 164 (2012) 32.

Mucin self-interactions at the nanoscale

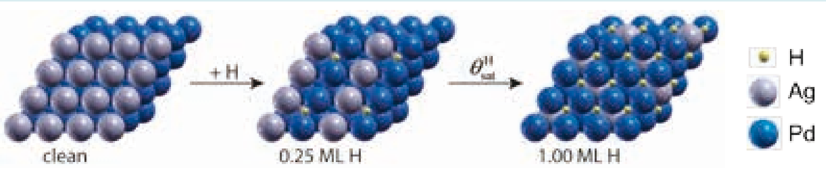
Determination of macromolecular interactions at the single-molecule level offer novel insight in their structure – function relationsships. We reported on the mucins self-interactions and how these properties depend on their saccharide decoration patterns. Mucins, being linear O-glycosylated glycoproteins involved in inflammation, cell adhesion, and tumorigenesis were investigated with emphasis on mucins expressing T and Tn cancer antigen. Using AFM and maintaining the mucins under near physiological conditions, distributions of unbinding forces and corresponding force loading rates were determined for force loading rates from 0.18 nN/s to 39 nN/s, All mucin samples investigated showed self-interaction, but mucins with only Tn-antigen or a mixture of Tn-, T-antigen, and another trisaccharide showed the largest tendency. These carbohydrate cancer antigens may, thus, play an active role in the disease by constitutively activating mucin and mucin-type receptors by self-association on cells.



"Enhanced Self-Association of Mucins Possessing the T and Tn Carbohydrate Cancer Antigens at the Single-Molecule Level", K.E. Haugstad, T.A. Gerken, B.T. Stokke, T.K. Dam, C.F. Brewer and M. Sletmoen, Biomacromolecules, 13 (2012)1400.

Adsorbate-induced segregation in a PdAg membrane model system

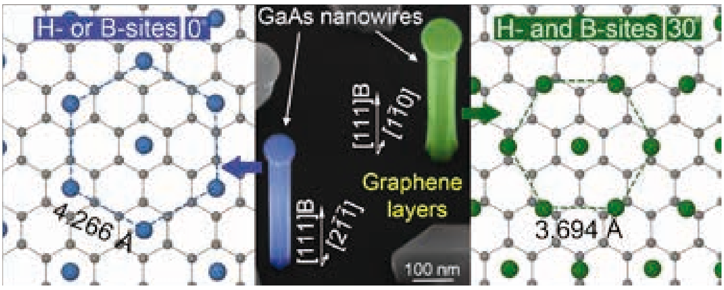
Palladium-silver (PdAg) alloys are suitable candidates for hydrogen separation technologies due to their high selectivity and permeability towards hydrogen. Poisoning effects due to CO and other molecules are well-known, whereas segregation effects of a reactive environment on the (surface) structure and composition of such membranes have been less emphasized. This work employs advanced calculations of the electron structure of PdAg crystals to provide a better understanding of how the adsorption of H_2, CO or O_2 on the surface is affected by the distribution between Pd and Ag in the topmost atomic layer and vice versa, using computational facilities provided by NOTUR. We find that the PdAg membrane surface is Ag rich in the absence of adsorbates. Pd atoms are pulled to the surface upon adsorption of O, H and CO, making the $Pd_3Ag(111)$ surface Pd-dominated at the corresponding saturation coverages. The research is carried out by the Dept. of Chemical Engineering, NTNU, in collaboration with the Dept. of Chemical and Biological Engineering at the University of Wisconsin-Madison, USA.



"Adsorbate-induced segregation in a PdAg membrane model system: Pd3Ag(111)", I.-H.Svenum, J.A. Herron, M. Mavrikakis and H.J.Venvik, Catalysis Today 193 (2012) 111.

Epitaxial growth of vertical nanowires on graphene

By utilizing the reduced contact area of nanowires, we have shown that epitaxial growth of a broad range of semiconductors on graphene can be achieved. A generic atomic model was established which describes the epitaxial growth configurations applicable to all semiconductor materials. The model has beem experimentally verified by demonstrating the growth of highly uniform self-catalyzed GaAs nanowires on graphite and graphene by molecular beam epitaxy as shown in the figure. Due to the catalyst-assisted growth technique used the nanowires were found to have uniform cross-sections. A prototype of a single GaAs nanowire photodetector demonstrated a high-quality material with no degradation due to the growth on the graphene substrate. We anticipate these semiconductor nanowire/graphene hybrid structures to be promising for various novel electronic and optoelectronic devices.



"Vertically aligned GaAs nanowires on graphite and graphene: Generic model and epitaxial growth", A.M. Munshi, D.L. Dheeraj, V.T. Fauske, D.C. Kim, A.T.J. van Helvoort, B.O. Fimland, and H. Weman, Nano Letters 1, (2012) 4570.

Kavli Nanoscience Symposium 2012

In conjunction with the Kavli Prize Lectures given in Trondheim on the 6th of September, NTNU NanoLab organized a half day international symposium with the following international speakers:

- Prof. Carlos Bustamante, Univ of California, Berkeley, USA:
  Discrete Steps and Intersubunit Coordination in a DNA-Packaging Ring ATPase
- Prof. Seeram Ramakrishna, National University of Singapore, Singapore:
  Electrospinning - A Means to Innovate for Funding Strapped Researchers
- Prof. Jochen Mannhart, Max Planck Stuttgart, Germany:
  Oxide Interfaces – A Fantastic World for Electrons; From MOSFETs to Novel Electron Systems
- Prof. Milena Grifoni University of Regensburg, Regensburg, Germany:
  Spin-dependent phenomena in carbon nanotubes

7th Annual NTNU NanoLab meeting

A one-day seminar portraying various activities within nanoscience and nanotechnology at NTNU was organized on the 12th of December, at Radisson Blu Royal Garden Hotel in Trondheim. The aim of these annual meetings is to facilitate cross disciplinary contacts between PhD-students, researchers and staff at NTNU and SINTEF. The program consisted of 21 lectures presenting ongoing projects at NTNU and SINTEF. The meeting gathered 60 participants.

NanoMatch-Making Seminar

A one-day seminar gathering participants from Norwegian industry and researchers from NTNU and SINTEF was held on November 13th 2012, at Radisson Blu Royal Garden Hotel. The aim was to promote collaboration between scientific groups from university and industry. At the same time, the opportunities offered by the Research Council and the national infrastructures NorFab, NorTEM and NICE were presented. Ten companies as well as staff members from NTNU and SINTEF were given 7 minutes to present their expertise and the challenges they were seeking collaboration to address.
Ample time was then allocated for mingling and networking. As a result, at least 3 of the participating industry representatives contributed in the autumn call of NANO2021 together with NTNU staff.

The meeting was organized by NTNU NanoLab and SINTEF Materials and Chemistry in cooperation with the Research Council of Norway and gathered 48 participants.

The NTNU NanoLab Lunch Seminars

NTNU NanoLab organized weekly lunch seminars in the spring semester of 2012. These seminars gathered 20-50 participants.

- *NORTEM- new TEM equipment to Norway – What is in it for you?* Randi Holmestad
- *Funding options for projects related to Nanotechnology - Nanoscience in 2012*, Kay Gastinger & Stefan de Graaf
- *Options at MiNaLab in Oslo – Available for users from NTNU*, Farbrice Lapique & Klaus Johansen
- *3D reconstruction by dual beam FIB/SEM*, Per Erik Vullum
- *The Langmuir technique: Solving your problems one monolayer at a time*, Wilhelm R. Glomm
- *Nanoimprinting in NTNU NanoLab's cleanroom*, Renato Bugge
- *Modification of diatom bio-nanostructures for enhanced light trapping properties*, Julien Romann
- *New opportunities for surface microscopy and analysis with electron and ion spectroscopy*, John Walmsley

NTNU NanoLab's cleanroom

Efficient start-up of new activities and improved working environment for all users have been in focus in 2012. The cleanroom course has been revised and it now takes only 4-5 hours of training to gain access to the cleanroom. Altogether, 129 people attended the cleanroom course in 2012.

A new lab information and management system (LIMS) was implemented in July. This is an instrument, user and project database which handles instrument booking and information flow. It is our anticipation that all new and experienced cleanroom users will find the necessary information in LIMS. Most of the information can be accessed without logging in. You may find LIMS at ntnu.norfab.no

Several new instruments have been installed, including a new sputter coater/evaporator and a Langmuir-Blodget trough. The ICP-RIE and the plasma etchers have been upgraded to increase the availability and reduce the risk for cross contamination. The table top SEM and the Veeco AFM have also been upgraded to be more user-friendly.

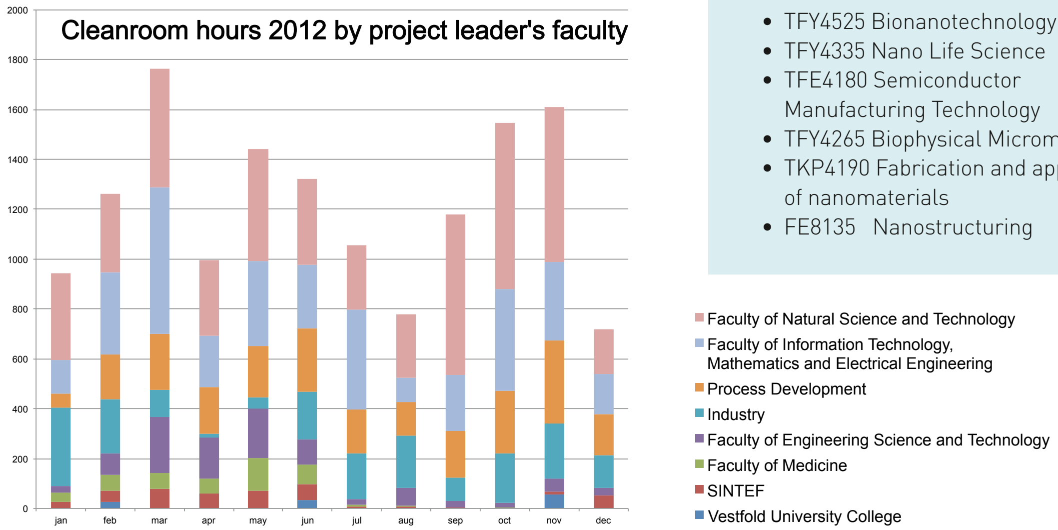
In June a new Head of laboratory was employed. In addition, the regular staff consisted of 4 engineers throughout the year. In 2012, there were around 160 active users in the cleanroom. The number of projects, as well as the user hours, increased compared to 2011, but there is still room for more activity. Like in 2011, just over 40% of the activity was generated by students. Industry projects accounted for 14% of the activity and university researchers for 27%. 17% of the cleanroom activity was related to process and course development.

Training offered by NTNU NanoLab in 2012

- 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

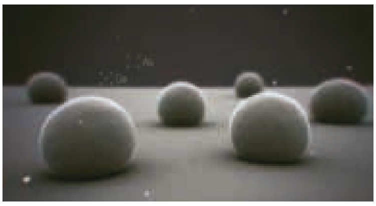
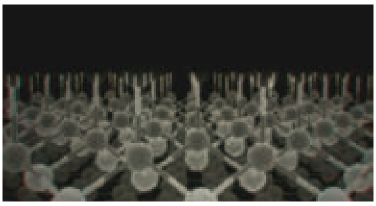
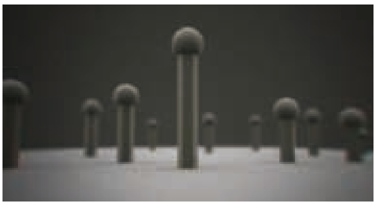
Module courses at Master level in NTNU NanoLab's cleanroom

- 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



CrayoNano to develop semiconductors on graphene

CrayoNano AS was founded in June 2012 in order to develop and commercialize a new hybrid technology based on semiconductor devices fabricated on graphene substrates. CrayoNano originates from semiconductor nanowire and graphene research done at the Department of Electronics and Telecommunications at NTNU. The technology has been patented by NTNU Technology Transfer of which CrayoNano is a spin-off. The founders, Professor Helge Weman and Professor Bjørn-Ove Fimland, are both responsible for important research groups and labs at NTNU focusing on synthesis and characterization of III-V semiconductors for use in photonics, solar cell technology and sensor applications. CrayoNano will be using NTNU NanoLab extensively in the coming years for both processing and characterization.

Contact persons: Morten Frøseth (CEO) and Helge Weman (CTO).

CrayoNano

Dissertations

The following candidates obtained a PhD degree at NTNU in fields related to nanoscience and nanotechnology in 2012. Highlighted candidates have carried out part of their work in NTNU NanoLab's cleanroom.

- **Inger Lise Alsvik, *Parameter Optimization in Preparation of Membranes for Osmotic Processes***
- Mohammad Haj Mohammadian Baghban, *Cementitious Nano-composites with Low Thermal Conductivity*
- Sigrid Berg, *Capacitive Micromachined Ultrasonic Transducers – Acoustic Challenges and Proposed Solutions*
- Jos Emiel Boschker, *Control of Surface Quality and Structural Distortion in Perovskite Oxide Thin Films*
- Stein Trygve Briskeby, *Carbon Nano-Fibre Supported Electrocatalysts for Fuel Cells*
- Roya Dehghan-Niri, *Advanced Transmission Electron Microscopy Studies of Cobalt Fischer-Tropsch Catalysts*
- Anette I. Dybvik, *Functional characterization of selected chitinases and chitosanases – a pathway to generate chitooligosaccharides from chitosans*
- Arnfinn Aas Eielsen, *Topics in Control of Nanopositioning Devices*
- **Sidsel Meli Hanetho, *Hybrid Aminopropyl Silane-Based Coatings on Steel***
- Ellinor Heggseth, *Enzymatic Degradation of Chitosans - A study of the mode of action of selected chitinases and chitosanases*
- Henrik Hemmen, *Experimental studies of smectite clays: colloids and nanoporous materials*
- Anders Lervik, *Energy dissipation in biomolecular machines*
- Paul Anton Letnes, *Optical polarization effects of rough and structured surfaces*
- **Jostein Malmo, *Chitosan-Based Nanocarriers for Gene and siRNA-Delivery***
- **Åsmund Fløystad Monsen, *On the properties of $La_{0.7}Sr_{0.3}MnO_3$ thin films on (001) $SrTiO_3$ substrates***
- **Morten Andreas Onsrud, *Carbon Cones as Negative Electrode Material in Lithium-Ion Batteries***
- Iver Bakken Sperstad, *Dissipative quantum phase transitions and high-temperature superconductors*
- Guttorm Ernst Syvertsen, *Synthesis and properties of electrolyte and cathode materials for proton conducting solid oxide fuel cells*
- Sedsel Fretheim Thomassen, *InAs/(Al) GaAs quantum dots for intermediate band solar cells*
- **Jelena Todorovic, *Correlated transmission electron microscopy and micro-photoluminescence studies of GaAs-based heterostructured semiconductor nanowires***
- Inga Ringdalen Vatne, *Quasicontinuum modeling of fracture in bcc materials*
- Mohammad Washim Uddin, *Durability of a PVAm/PVA blend membrane in natural gas sweetening*
- **Christian Carl Weigand, *ZnO Nanostructures and Thin Films Grown by Pulsed Laser Deposition***
- **Pjotr Öchal, *Carbon-supported Ru@Pt Core-shell Catalyst for Low Temperature Fuel Cells***

Awards

Espen Tjønneland Wefring was awarded **Bardal's scholarship 2012** for his master thesis: "Nano-structuring of oxygen permeable membranes by chemical etch techniques".

The Royal Norwegian Society of Sciences and Letters' prize to young scientists 2012 was awarded Associate Prof. Sverre Magnus Selbach for his studies of nanosized $BiFeO_3$ perovskite.

NTNU's research award for medical technology 2012 was given to Dr. Reinold Ellingsen, Dr. Sven Tierney, Prof. Dag Roar Hjelme, Dr. Ming Gao, Dr. Kamila Gawel and Prof. Bjørn Torger Stokke for the development of a technique for determining the thickness of hydrogels.

Photo: Oscar Stoltenberg Johansen

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- Prof. Zhiliang Zhang, Faculty of Engineering Science and Technology, NTNU.
- Prof. Jostein Grepstad, Faculty of Information Technology, Mathematics and Electrical Engineering, NTNU.
- Astrid Bjørnetun Haugen, PhD student, NTNU.
- Mira Thoen Feiring, student, NTNU.
- Frank Johansen, student, NTNU.
- Rudie Spooren, SINTEF Materials and Chemistry, Research Director.
- Ellen Dahler Tuset, Kongsberg Norspace AS, Commercial Director
- Randi Haakenaasen, Norwegian Research Defense Establishment, Principal Scientist.
- Bjørn Fuglaas, GE Healthcare, Director, Security and Internal Services (until 01.03.2012)

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Coordinator: Dr. Hanna Gautun

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- Staff engineer Mark Chiappa
- Senior engineer Espen Rogstad
- Senior engineer Trine Østlyng

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- Prof. Bjørn Ove Fimland / Prof. Helge Weman (from 01.07.2012)
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- Associate Prof. Marit Sletmoen / Prof. Pawel Sikorski (from 01.07.2012)
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- Research manager Ragnar Fagerberg
- Coordinator Hanna Gautun
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