

Overview of the NUS Nanoscience and Nanotechnology Initiative and its available facilities S. Ramakrishna and <u>T.C. Lim</u> National University of Singapore Nanoscience and Nanotechnology initiative

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•To develop research human capital and long-term research capabilities in the strategic field of nanoscience and nanotechnology

•To galvanize and coordinate multidisciplinary research effort (across departments, faculties and with the RIs) in nanoscience and nanotechnology

•To help set research priorities and directions for high impact nanoscience and nanotechnology research

NUSNNI began functioning in January 2002 and was officially opened in July 2004 by Acting Minister for Education, Mr Tharman Shanmugaratnam

Focus Groups





Nanobiotechnology



Objectives: Nanobiotechnology Focus Group is engaged to apply and further develop nanotechnology to solve problems in biology and medicine by investigating biological structure/function/mechanism and medical physiology/pathology/treatment in molecular and atomic scale.

Application: Cancer Nanotechnology

Using Nanobiotechnology to advance cancer diagnosis, treatment and prevention.

There has been no substantial progress in the past 50 years in fighting against cancer. The cancer death rate in US was 1.939‰ in 1950 and still 1.940‰ in 2001. <u>High technology, especially</u> <u>nanobiotechnology, will greatly improve and radically change the way we diagnose, treat and prevent cancer.</u> Novel nanodevices will be developed to have one or more clinically functions, including detecting cancer at its earliest stages, pinpointing its location within the body, delivering anticancer drugs specifically to malignant cells, and determining if these drugs are killing malignant cells. We aim to develop such "four-in-one" functional nanodivices. Nanotechnology can play a pivotal role by providing the technological power and tools based on the vast knowledge of cancer genomics and proteomics emerging as a result of the Human Genome Project. Nanobiotechnology will help meet the goal of reducing death and suffering from cancer by 2015.



Fig 3. Composition-tunable optical properties of alloyed ZnxCd1-xSe nanocrystals with high luminescence and stability across the visible spectrium.



Fig 4. TEM image of a double stranded DNA helically wrapped around a single walled carbon nanotubes.



Fig 1. Confocal microscopic images of Caco-2 cells after 1 hour incubation at 37°C with coumarin 6-loaded PLGA nanoparticles coated with vitamin E TPGS.



Fig 2.. The morphology of multi-walled carbon nano-tube, by SEM. Length: 10 µM, diameter: 100-250 nM.

Nanofiber Science & Engineering



An Overview of Electrospinning & Nanofibers Research The nanofibers range in size from 10 nm to about 1 micrometer

Features:

- 1 Increased Surface Area
- 2. Physical properties of the material can be altered or tailor made according to the application requirements

Areas of Research

- The areas in polymer nanofibers, which show potential to drastically, improve performance as Bio-Chemical Barrier, Sensors and Tissue Scaffolds are:
- 1) Patterned Nano Structure: Placement of nanofibers in a designed pattern.
- 2) Composite Nanofiber: Form composite nano fibers with polymer and nano particles
- 3) Surface Activation of nanofiber surface.
- 4) Super Hydrophobic: Plasma-induced polymer grafting of nanofiber surface.
- 5) Molecular Sensor
- 6) Polymer Nanofibrous scaffolds





1. Water Filtration and Purification – Tertiary Treatment

One such application is tertiary treatment and purification of industrial and drinking water. Nanofibers are functionalized with chelating ions can bind heavy metal impurities and salts from water and is removed by centrifugation or other settling techniques. Nanofilters also help in purifying water from trace organics. These filters can be designed and functionalized for specific treatment requirements



Nanofiber Science & Engineering



Cont'd - An Overview of Electrospinning & Nanofibers Research

2. Protection against C&B warfare agents

Nanofibers media offers **protection** by acting as an impermeable barrier for chemical and biological warfare agents and allows breath-ability

Large surface area of nanofibers can host dense functional groups and hence helps efficient **removal** of contaminants

Nanofibrous 3D material can also bind transition metal ions (Cu²⁺, Co²⁺, Ni²⁺, etc;) and act as **catalyst support** for many organic conversions

Nanofiber surface modified with enzymes act as **biosensors** for contaminants

Schematic Overview



3. Biotechnology – filtration /separation

Nanofibers Play a unique role in separation technology as the ONLY material that enables separation and purification of a biomolecule based on its biological function and chemical structure. This technique ideally suited for the isolation of a specific substance from complex biological mixtures.





Comparison of Gene expression among

- 1. SMC cultured on Polymer films
- 2. SMC cultured on Nanofibers
- 3. SMC cultured on Tissue culture plate



Objectives: Nanophotonics Focus Group is engaged to develop nanotechnology for solving the problem in Optoelectronics.

Applications:

(A) Epitaxial Chemical Deposition of ZnO Nanorods on GaN Substrate Using Aqueous Solution Method (B) Patterning of Two-Dimensional Photonic Crystal Structures Using Nanoimprint Lithgraphy

(A) Epitaxial Chemical Deposition of ZnO Nanorods on GaN Substrate Using Aqueous Solution Method

I. Introduction	Nanostructures have the potential applications in electronic and photonic devices due to some novel characteristics (the reduction of number and size defects in nanoscale , the enhancement of exciton oscillator and light emitting efficiency)
	 Nanostructure of wide band gap semiconductor ZnO is of particular interest Exhibits semiconducting and piezoelectric dual properties, Promising material for exciton based optoelectronic devices in the UV region direct band gap large exciton binding energy 60meV Tunable band gap by alloying with CdO and MgO the possibility of wet etching and the availability of large area substrate at relatively low cost material
II. Motivation	
	 Using aqueous solution method to grow the ZnO nanorods due to its advantages compared with other methods (CVD, MOCVD, MBE, pulse laser deposition) Involve a template less and surfactant free aqueous method Fabrication of wafer scale at low cost & low deposition temperature Simple growth technique Less hazardous No metal catalyst needed Using GaN as a template to obtain the ZnO nanorods GaN and ZnO have the same wurtzite crystal structure, and a low lattice constant misfit (~1.9%) Possibility to fabricate the n-ZnO/pGaN heterojunction New method to fabricate the III-nitride nanotubes by decomposing the ZnO at high temperature



Cont'd – Application (A)

III. Results & Discussion

Surface morphology of the obtained nanorods



□ The nanorods uniformly covered the entire surface with the hexagonal cross section and all the tips of the rods are contracted with six regular facets

□ From the cross section image, the rods grew vertically from the GaN substrate, having uniform thickness and length distribution

The diameter and length of the nanorods are 80-120nm and ~2 μm, respectively.

□ The hexagonal shape of the nanorods reveals that the rods grew epitaxially on the GaN film.

Growth parameters :

Zinc Acetate (CH₃COO)₂Zn.2H₂O 0.027M Ammonium Hydroxide 0.173M DI water Temperature : 100C Growth time : 5 hours



Top view and cross section SEM images of ZnO nanorods

IV. Conclusion

□ The ZnO nanorods were successfully obtained on the GaN substrate using the aqueous solution at low deposition temperature

□ The SEM and XRD analyses showed that the ZnO nanorods were vertically aligned with uniform hexagonal structure and of diameter 80-120nm and length of about 2µm. TEM characterization of the ZnO nanorods indicate single crystal having [0001] growth direction

□ The PL spectrum exhibited high optical properties of the ZnO nanorods



Application (B) – Patterning of Two-dimensional Photonic Crystal Structures Using Nanoimprint Lithography

I. Introduction

We report on the process development of nanoimprint lithography (NIL) for the patterning of 2-D photonic crystals, which allows high-throughput and low-cost production leading to commercial realization of photonic crystal devices. Ni moulds with 2-D photonic crystal pillar structures are fabricated by electron-beam lithography (EBL) and electroplating, and the patterns on the mould are transferred to a poly-methyl-methacrylate (PMMA) layer spin-coated on silicon substrate during nanoimprinting.

II. Experimental Details

- Seeding layer coating Au/Cr double layer deposited on Si substrate by sputter deposition
- EBL nanometer-size patterns of periodic holes defined in 400nm PMMA layer spin-coated on the seeding layer (JC Nabity Nanometer Pattern Generation System)
 - Ni electroplating nanometer-size pillars formed through the holes and overplated across the whole wafer to form a mould (M-O-T µGalv system)
- Mould release mould cut to desired size, released from the substrate, deburred and backside-polished
- NIL high pressure applied on the mould in contact with PMMA layer spin-coated on Si substrate above its glass transition temperature (Obducat NIL-4 nanoimprinter system)



Fig. 1: Process flow chart for Ni mould fabrication and nanoimprinting

III. Results & Discussion

Periodic holes arrayed in square and hexagonal lattices with diameters ranging from 100nm to 400nm are produced by EBL. The process parameters including beam current, exposure pixel, dose and writing mode are optimized to obtain good pattern shape as well as dimension fidelity. SEM pictures in Fig. 2 show an example of structure quality improvement by reducing the beam current.



Fig. 2: SEM pictures of PMMA after EBL; beam current = (a)120pA, (b) 50pA; smaller beam current gives a better circular shape fidelity



Cont'd – Application (B)

Two different recipes (Fig. 2a, 2b) are employed for electroplating. While both generate flat surface by optimizing current density and thus controlling the residual stress involved (Fig 2c, 2d), pillar structures with better quality were achieved by recipe II (Fig. 2f).



Fig. 3: Ni moulds fabricated by electroplating; (a), (b) electroplating recipes; (c), (d) surface profiles corresponding to Recipe I and II r e s p e c t i v e I y; (e), (f) S E M p i c t u r e s showing pillar structures corresponding to Recipe I and II respectively

Patterns on the mould are successfully transferred to the PMMA layer above the glasstransition temperature of PAMMA during nanoimprinting as is shown in Fig. 4. The main process parameters include embossing temperate, force, time and mould-substrate separating temperature. The PMMA thickness is kept around 250nm, which is slightly bigger than the pattern height on the Ni mould for the protection of the mould.



Fig. 4: SEM pictures of PMMA after nanoimprinting at 130°C and 40bar for 5min using Ni mould; pattern transfer well achieved

IV. Conclusion & future Work

- Ni moulds with designed photonic crystal patterns covering the area up to $20 \rm mm^2$ were fabricated; 200~250 \rm nm high pillars were

produced with the diameters ranging from 180nm to 400nm

- Nanoscale patterns on the mould were transferred to a PMMA layer on Si substrate by NIL, with periodic air holes formed in PMMA
- Pulse-electroplating and annealing will be employed for a better residue stress control, reducing the curvature across the mould

Nanomagnetics and Spintronics



Objectives: Fundamental research into engineering spin and magnetic properties in nanostructures, and applications in nanodevices and spintronics.

Focus Areas

- Semiconductor(SC)-based spintronics e.g. diluted magnetic SC, hybrid devices
- Metal-based spintronics e.g. spin transfer torque, spin dependent tunneling
- Theory and computer modeling of spin transport, dynamics and interactions
- Biomedical applications biosensors, nanoparticles for killing tumor.
- Molecular spintronics carbon nanotubes as templates

Research Achievements

- A. Unique 2D carbon "nanowall" structures formed from carbon nanotubes (a world first).
- B. Computer prediction of full logic function of proposed magnetic soliton logic
- C. Ultrasensitive sensors incorporating "castellated" or particle nanobridge structures potentially capable of single spin detection.
- D. E Patterned nanostructures and self-assembly of magnetic nanoparticles using polystyrene spheres as template for potential applications in Tb/in² information storage.
- F. Cytotoxic tests done using Co ferrite magnetic nanoparticles, as preparation for possible biomedical use.
- G. One potential biomedical use of magnetic nanoparticles is in antigen-specific GMR bio-sensors.





Nano/Micro Fabrication



Objectives: Nano and micro fabrication techniques are increasingly required to produce miniaturized components from hard-to-machine materials for applications in aviation, aerospace, medical instruments, communication systems, MEMS, etc. Tool-based nano/micro fabrication techniques have been developed to address these challenges. These techniques include ELID (*electrolytic in-process*) *dressing*) grinding and diamond turning for nano surface generation, and micro-machining processes such as micro-EDM, micro-turning, micro-milling, etc.

Objectives

To develop the machine tools and conventional/non-conventional material removal processes for tool-based nano/micro fabrication to generate nanometer level surface finish and 3-D micro features. The materials' portfolio includes hard and super hard materials such as silicon, glass, ceramics and diamond, various metals, and soft materials such as polymers.

Industrial Relevance/Applications

- Grinding of silicon wafer surfaces for semiconductor industries.
- Production of aspheric surfaces for lenses and molding dies for the optical industries.
- Production of nano surface finish on difficult to machine materials such as silicon, glass, ceramics and guartz with a wide spectrum of industrial applications.

Tool-Based Micro Machining











Micro-WEDG

A Multi Process Miniature Machine **Tool Developed for Micro Machining** Triangular hole (0.3mm) u-ÈDM

Thin walled fins (70 μ m) by μ -milling

Nano/Micro Fabrication



Cont'd – Tool-Based Nano/Micro Fabrication

Achievements

- Nano finishing technology using ELID grinding techniques to achieve surface quality of less than 10nm on hard-to-machine materials such as BK7 glass and silicon wafer.
- A desktop miniature ultra precision machine for diamond turning has been developed to achieve nano machining.
- An award-winning integrated machine tool with resolution of 0.1 micron and accuracy of +/-1 micron has been developed for multi-process micro machining such as μ-turning, μ-drilling, μ-milling, μ-EDM, μ-wire-cut EDM, μ- EDG and μ- ECM. Two patent have been awarded for the machine developed.

Tool-Based Nano Surface Generation by ELID Grinding

Tool-Based Nano Surface Generation by Diamond Turning



ELID Grinding setup for nano surface generation



Silicon wafer ground by ELID grinding



Concave lens on BK7 glass by ELID grinding



Mirror surface finish on electroless nickel plating by diamond turning



Mirror surface finish on silicon wafer by diamond turning



A high speed ultra precision lathe for diamond turning designed and developed in NUS

Conferences



- **1** Nanotech World Forum and Exhibition, 27th Oct to 1st Nov 2003.
- **2** International Conference on Materials for Advanced Technologies, 7th to 12th Dec 2003.
- **3** Seminar on Nanoscience and Nanotechnology, 27th Feb 2004.
- **4** Joint CNSI-NUSNNI-IMRE Workshop, 19th to 21st April 2004.
- **5** 1st Nano-Engineering and Nano-Science Congress, 7th to 9th July 2004.

6 Joint Japan Society for the Promotion of Science (JSPS) and NUSNNI Workshop, 1st to 4th Nov 2004.

7 International Conference on Materials for Advanced Technologies, 3rd to 8th July 2005 (Symposiums on: Magnetic Nanomaterials & Devices; Nano-Optics & Microsystems; Nanodevices & Nanofabrication; Polymer Nano-structured Materials).

8 Joint Korea-Singapore Symposium on Nanobioengineering, 11th to 12th July 2005.

- **9** Plans for Joint symposium with China.
- **10** Plans for Joint symposium with Australia.

Award





Gold Medal at the 4th Young Inventors Awards

Adapting the existing method of creating the scaffolds through a process called electrospinning, Ryuji designed a machine that can, for the first time, build 3-D scaffolds as well as closely align the nanofibres in 2-D and 3-D scaffolds, allowing cell growth on the scaffolds to be controlled more precisely.

Specific Programs



Multidisciplinary Scholarship



Graduate Research Scholarships:

- NUSNNI awards research scholarships for pursuing doctoral studies in nanoscience and nanotechnology.
- Candidates selected are based on stringent criterias and their supervisors must be from both Engineering and Science.

creativity innovation enterprise Towards a Global Knowledge Enterprise research education service

2 Books & over 20 Book chapters (FY 03 & 04)

An Introduction to Electrospinning and Nanofibers

Seeram Ramakrishna Kazutoshi Fujihara Wee-Eong Teo Teik-Cheng Lim Zuwei Ma Nanoscale Structure and Assembly at Solid-Fluid Interfaces



For FY 04 alone, over 200 international journal papers and over 100 conference papers.

High Impact Factor Journals





(a), (b), Statistical measurements of parameters of nucleation kinetics. (c), (d), The colloidal assemblies obtained under constant electric field and alternating electric field respectively. Scale bars, 10µm.

Ke-Qin Zhang, and Xiang-Y. Liu, "In situ observation of colloidal monolayer nucleation driven by an alternating electric field", **Nature**, 429 (2004) 739-743.

High Impact Factor Journals



Ref: Lay-Lay Chua, Jana Zaumseil, Jui-Fen Chang, Eric C.-W. Ou, Peter K.-H. Ho*, Henning Sirringhaus & Richard H. Friend*, *Nature* 434 (2005) 194





Int Journal Cover Selection





www.pss-a.com

applications and materials science

Review Article

Insights into OLED functioning (D. Berner et al., p. 9)

Original Papers



Editor's Choice AFM study of hexagonal BN film growth on 6H–SiC (0001) (Wei Chen, Kian Ping Loh et al., p. 37)

New in 2005: rapid research letters



Lu₂O₃: Yb³⁺ ceramics – a novel gain material for high-power solid-state lasers (K. Takaichi et al., p. R1)

202 + 1 + January 2005 WILEY-VCH ISSN 000318965, plays and (a) Marcel of the data state and (a)





www.iop.org/journals/nano Topical review: Labelling of cells with quantum dots WJ Parak, T Pellogrino and C Plank

Featured article: Redox-active monolayers on nano-scale silicon electrodes Q Zhao, Y Luo, S Surthi, Q Li, G Mathur, S Goueda, P R Larson, M B Johnson and V Misra



Special Issues







Spin-off Company in Nanotech





Mikrotools Pte Ltd 8 Prince George's Park Singapore 118407 enquiry@mikrotools.com

Spin-off Company in Nanotech







NUSNNI Laboratories at Faculty of Engineering

NUSNNI Labs: Faculty of Engineering



Nanotech Corridor 3

Nanobioengineering Lab Nano Biomechanics Lab

Advanced —— Manufacturing Lab

(Micro/Nano Fabrication Lab)

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Nanotech Corridor 4

MicroSystems Technology Initiative (MSTI) [Nano Electro-Mechanical Systems (NEMS)]

Nanotech Corridor 2

Centre for Optoelectronics (Nanophotonics) Nano Wafer Level Packaging Lab (Nanostructures and Nanomaterials)

Nanotech Corridor 1

 Silicon Nano Devices Lab (Nanoelectronics)
 Information Storage
 Materials Lab
 (Nanomagnetics)
 Zhao Lab (Nanomaterials
 & 3D Photonic Materials)



NUSNNI Laboratories at Faculty of Science

NUSNNI Labs: Faculty of Science







1. Silicon Nano Device Lab (SNDL)

2. Nano Biomechanics Lab

3. BIOMAT Lab

4. Nanobioengineering lab



Silicon Nano Device Laboratory

SNDL Clean Room



Cleanroom



- Industry standard cleanroom facility (class 1~100, 6 & 8 inch wafer processing)
- Total Area : 517 m²
 - Cleanroom Area : 420 m² (SNDL : 299 m², ISML : 90 m², yellow room: 31 m²)
 - Service Area : 97 m²

Utility Room & Chiller Yard







SNDL Equipment - I





Gate Cluster

- Vendor : Jusung Engineering
- Surface treatment chamber (I)
- High-K deposition chamber (II)
- ✤ Gate electrode chamber (III)

Etcher Cluster

- Vendor : Lam Research
- 2 Chambers: conductor and dielectric etch



ALD High-K System (Genus)







SNDL Equipment - II













- Full set of furnace tubes
 - Furnace stack : oxidation, annealing, alloy
 - LPCVD tubes : poly-Si, TEOS, Nitride
- 2 chamber UHV Epi system
- Nanowire growth system
- Magnetron Sputtering System
- 2 RTP systems (multi-zone controlled)
- 4 wet benches, IPA dryer
- E-beam evaporator
- ICP etcher
- Photoresist asher
- Optical lithography tools
- Characterization tools :
 - Spectroscopic Ellipsometer,
 - 4 point probe,
 - Surface profiler, Particle counter
 - XPS







SNDL Clean Room

Very Important Notice

The requested process <u>must</u> be CMOS IC process compatible







Nano Biomechanics Laboratory

Facilities at Nano Biomechanics Lab





DI Multimode AFM with PicoForce System



Atomic Force Microscope based Nanoindentation System



Leica DM IRB Inverted microscopy system with micromanipulators

Facilities at Nano Biomechanics Lab





Cell Robotics Laser Tweezers & Laser Scissors System



MTS Nano Tensile Tester



Instron Microtester



JEOL JEM-2010F FasTEM Field Emission Electron Microscope

NANO BIOMECHANICS LAB





Optical tweezers / Laser trap setup





Sample preparation area



Nano Biomechanics Lab



Micropipette aspiration setup



Atomic force microscopy system

Nano tensile tester

Mechanical Characterization of Nanomaterials





(Tan EPS & Lim CT, Appl. Phys. Lett., 2004)



BIOMAT Laboratory





Haptics Setup

This Haptics Setup system allows force feedback, thus enabling users to model spine operations in our laboratory.





Micro CT Machine

This Micro CT machine allows non destructive characterization and visualization of microarchitecture of structures like scaffolds and bones





Glow Discharge Plasma Machine

This Plasma Glow Discharge enables users to perform surface modifications and functionalization of polymers.





Glow Discharge Plasma Machine

Argon gas is used to functionalize the surface of polymers in BIOMAT LAB.





This 3-D printing machine enables users to produce 3-D scaffolds and models of bones.





Fume Hood

This Fume Hood allows users to carry out solvent related experiments without compromising the safety of other users.





3-D Rotational Moulding Machine

This 3-D rotational mould machine enable uses to mould polymers into their desired 3-D structures.





Spin Cast Machine

This self-developed Spin Cast machine enables users to fabricate ultra thin Polycaprolactone films (1-3 µm)





Two-Roll Milling Machine

This Two-Roll Milling Machine has allow us to produce solventfree Polycaprolactone thin films.





Heat Press Machine

This Heat Press machine make uses of heat pressing at high temperature, producing thin and uniform polymeric films.





Bi-axial Stretch Machine

This Bi-axial Stretch machine allows bi-axial uniform drawing of polymeric films into a flat sheet at elevated temperature.





Microscope coupled with Cold and Hot Stage

This Microscope is fitted with a cold and hot stage which allows users to observe microstructures of specimens at the desired temperature.





Vacuum Oven

This Vacuum Oven is used to dehydrate the specimens.



Nanobioengineering Laboratory



EPICS Altra Flow Cytometer

Specifications:

- Four color analysis Air cooled Argon , 15 mW, 488 nm operation
- Cell sorting system (25000 events/sec)

Application:

For detection and identification of microorganisms and particle & cell sorting











Confocal Microscope

Figure shows the confocal microscope is used to observe immuno-stained cellular constructs samples.







Attenuated Total Reflectance (ATR)/ Fourier Transform Infrared Spectroscopy



Applications:

To determine the type of functional groups present in a material. ATR enables the determination of functional groups at the surface of a material.

Single polymer beads, Single fibers, Carbon-filled materials such as O-rings, Liquids (including aqueous solutions, corrosive and caustics), Contaminants in paper **IR Microscope**

Allows the study of extremely thin samples which cannot be studied with the Ge crystal (very common materials studied using the microscope accessory is the PCL film). It is also equipped with ATR accessory





The JEM-2010F Field Emission Electron

Microscope is a multipurpose high resolution analytical electron microscope with high resolution image observation, microarea X-ray analysis, and with a wide range of capabilities.

Applications:

Characterization of structure, crystallography, and elemental composition of a large variety material, for instance, ceramics, metals, semiconductors, polymers, and biological samples





Planetary Ballmill

Applications:

Size reduction, mixing, homogenizing, mechanical alloying

Towards a Global Knowledge Enterprise

research education innovation enterprise

Thank you.

http://www.nusnni.nus.edu.sg/

