NANOFABRICATION RESEARCH IN THE RESEARCH CENTRE IN NANOSCIENCE AND NANOTECHNOLOGY (CIN2)

Prof. Dr. Clivia M. Sotomayor Torres
Catalan Institute of Nanotechnology & ICREA
OUTLINE

• Facilities

• Examples of research work

• Perspectives
PEOPLE
Total 60
Including 13 non-researchers

PUBLICATIONS 2007 (08)
Nr of publications:
  59 (84)
Average Impact Factor:
  3.76 (3.96)

PROJECTS
11 National  1.2 M€
12 European  5.2 M€
4 SME’s  0.2 M€
Total  6.6 M€

INDUSTRIAL ACTIVITIES
1 Spin-off
5 Patents
ICN Work with industrial partners

With **NANÒNICA** incorporating nanoparticles in wools (2007)

With **ENDOR** on synthesis and medical applications of advanced nanomaterials. (2007)

With **BIOINGENIUM** in a feasibility study to produce hormones for veterinary use. (2007)

With **NANOINK** on a NDA-covered agreement concerning patterning and ink applications (2007)

With **LEITAT – ACONDICIONAMIENTO TARRASSENSE** on textiles suitable for UV protection (2007)
Catalysis
-Surface-supported Au nanoparticles for catalytic applications

Self-assembly and Nanofabrication (joint with VTT)
-Capillary-directed sedimentation on topologically patterned substrates

Drug and chemical compounds delivery
-Compounds encapsulation in Metal-Organic nanospheres

Nanobiomedicine
-Cell identification with Au nanoparticles by means of H ion reduction
-Au nanoparticles functionalized with peptide molecules
CIN2 COMMON EQUIPMENT

• **E-beam Metal Evaporators**
  • For non-magnetic materials
  • For magnetic materials (e-beam and sputtering)

• **SEM**
  • Environmental, 150 mm wafer – undergoing purchasing process
  • High resolution - undergoing purchasing process

• **TEM (200kV)** - undergoing purchasing process

• **X-ray Diffraction**
  • Powder XRD (for Co, Fe, Ni, … NPs)
  • Thin films XRD (High resolution, SAXS, reflectometry, texture, etc.)

• **XPS/UPS System**
• **SQUID**
• **Mid-far IR spectrometer**
• **AFM**
• **Nanoimprinter 50 mm wafer**
• **Focused ion beam (crossed-beam)**

Also
• **wide range of specialist equipment in each research group**
Research strands in CIN2

- Theory and Atoms on Surfaces
- Nanoparticles, NPs in matrices and energy research
- Nanofabrication and Nanometrology
- Physical Properties of Nanostructures
- Nanobiosensors
THEORY AND NANOSTRUCTURES MANIPULATION

P Ordejon, P Gambardella and J Fraxedas

- Atomistic simulations
- Magnetic atoms/molecules on surfaces
- Metal-organic nanostructures
- Small molecules
NANOPARTICLES, NPS IN MATRICES AND ENERGY RESEARCH

V Puntes, D Ruiz-Molina, P Gomez-Romero, M Lira-Cantu

- Inorganic nanoparticles
- Core-shell NPs
- Molecular electronics
- Metal-organic materials
- Energy storage and conversion
- Solar cells
C M Sotomayor Torres, D Ruiz-Molina, S Valenzuela and A Bachtold

- Self-assembly
- Focused ion and electron beam lithography
- Dip pen Lithography
- Nanoimprint lithography
- Nanometrology

Other teams using nanofabrication facilities are those of L Lechuga (plasmonics for biosensors) and J Fraxedas (synchrotron-based approaches)
Self-assembly of 3D colloidal crystals

W Khunsin, G Kocher et al, to be published

Quality improvement using acoustic noise
Concept of “opposite beads”

\[ p(r) = \frac{\sum_{A\neq B,C} \chi_r(\overline{AB}) \chi_\varepsilon(\overline{AB} + \overline{AC})}{\sum_{A\neq B} \chi_r(\overline{AB})} \]

\[ \chi_y(\bar{R}) = \begin{cases} 
1 & \text{if } |\bar{R}| < y \\
0 & \text{else} 
\end{cases} \]

\[ p(r) = \frac{\sum_A N_A(r)p_A(r)}{\sum_A N_A(r)} \]
Stochastic-resonance in photonic crystal growth

\[ D = 368 \text{ nm} \]

\[ \mu = 5.5 \mu m \approx 15D, \text{ and } \epsilon = 43 \text{ nm} \approx 0.12D \]
Particles self-assembled on patterned Silicon and SOI

Etched silicon substrate

PMMA opal self-assembled in basins in etched silicon substrate


The PHAT project
Molecular self-assembly assisted by nanoimprinting

3-(9-anthryl) pyrazole (ANP)

Evaporation

Physical vapor deposition

Microscopie de fluorescence

Intensité de fluorescence

Longueur d’ondes nm

V. Reboud et al., en préparation

Surface functionalisation

AFM Tip
Water Meniscus
Molecules, Biomolecules or Nanoscale Materials
Mirkin et al., Science, 1999, 283, 661
Writing Direction
Substrate

D Ruiz group
DPL Different writing modes

Static Writing

1 μm 16
925 nm 12
850 nm 8
450 nm 4
600 nm 2
725 nm 1
350 nm 1
225 nm 0.5
150 nm 0.25

Dynamic Writing

600 nm 16
250 nm 12
400 nm 8
150 nm 4

D Ruiz et al
Reverse Nanoimprint Lithography

Spin coated HSQ

Si Stamp

Pressure and heat

Reverse imprint, T ~ Tg

Substrate

Whole layer transfer mode

Inking mode

Reverse UV Nanoimprinting technique

- No residual layer
- No need for anti-adhesive treatment of the stamp
- The same photocurable polymer is used
- High resolution (stamp dependent)
- High throughput (<2 min)

N. Kehagias et al., Nanotechnology, 18, 175303, (2007)
Polymer double layer grating by RUV NIL

a/ SEM image of a large (>50μm) double layer grating. b/ Fourier transform of a/ showing good homogeneity of lines over the whole surface with limited dispersion in size and position c/ Far-field optical image of diffracted light by the 3D grating.

Modelling of the NIL process

Assume resist is an incompressible viscous fluid → non-stationary Navier-Stokes equations in velocity-pressure

\[
\text{Re} \left[ \frac{\partial \mathbf{V}}{\partial t} + (\mathbf{V} \cdot \nabla) \mathbf{V} \right] = -\nabla P + \Delta \mathbf{V},
\]

\[\nabla \cdot \mathbf{V} = 0, \quad \mathbf{V} = (v_x, v_y, v_z),\]

\text{Re} is the Reynolds number, \(V\) is the stamp velocity, \(P\) is the pressure. Also includes characteristic lateral size of the stamp and the density and the dynamic viscosity of the resist.

Stamp bending due to non-homogeneous stamp design.
Areas with high resist pressure result in a higher residual layer thickness.
Modelling of the NIL process

Optical microscope image.

Profilometer measurement, showing stamp bending during the NIL process.

Simulated “RGB” images of residual layer height distribution. White iso-lines indicate the stamp bending in nm (190nm-150 nm = 40nm of stamp bending).
Modelling of the NIL process

Simulated scan along the black line

Experimental scan along the black line

H, nm

Position (micrometer)

Height (nm)

- 58 nm
- 64 nm
Subwavelength diffraction

Sub-wavelength features within periodic test structure. Line-width, height, defects affect the relative efficiency of orders in far-field diffraction.

- Models (FDTD and Rigorous Coupled Wave Analysis) show sensitivity to dimension changes of <10nm
- Shows presence of defect – missing 50nm lines

Metrology with Photoacoustic spectroscopy

Physical parameters –
Young’s modulus,
Poisson’s number –
calculated from acoustic speed

Pump/ probe measurement
Pump: 70fs laser pulse
Probe: Optical reflectivity change at surface

Laser absorption / generation of thermal stress – acoustic wave

Partial Reflection from interfaces
Propagating acoustic waves

Collaboration with Juerg Bryner & Prof Dr Juerg Dual, Centre of Mechanics, ETH Zurich
Photoacoustic Metrology of Nanoimprint Polymers

- Good acoustic impedance difference
- Damping in polymer not excessive
- Strong signal from both interfaces
  Top interface: Al/polymer
  Bottom interface: polymer/Si

Physical parameters calculated from speed and thickness – Young’s modulus (E) and Poisson’s ratio (n)

\[ C_p = \sqrt{\frac{E(1-\nu)}{\rho(1+\nu)(1-2\nu)}} \]

PHYSICAL PROPERTIES OF NANOSTRUCTURES

J Nogues, A Bachtold, S Valenzuela, J Santiso, C M Sotomayor Torres

Carbon nanotubes & Graphene
Spin transport
Nanoionics
Nanoscale thermal conduction
Nanophotonics
Phonons in Nanostructures
Carbon nanotubes and graphene

Electro-Mechanics

graphene

nanotube motor

single-electron spectroscopy

Bachtold et al
mechanical properties of graphene

Garcia-Sanchez, van der Zande, San Paulo, Lassagne, McEuen, Bachtold
Nano Letters 8, 1399 (2008)
Transport properties of graphene

9 Tesla, 4 K

\[ \sigma_{xy} (4e^2/h) \]

\[ V_g (V) \]

mobility ~ 15’000 to 25’000 cm²/Vs

Moser, Barreiro, Bachtold, APL 2007
**Observations of confined acoustic phonons in silicon membranes**

C. M. Sotomayor Torres, A. Zwick, F. Poinsette, J. Groenen, M. Prunilla, J. Ahopelto, A. Mlayah, and V. Paillard

1 National Microelectronics Research Centre, University College Cork, Lee Maltings, Prospect Row, Cork, Ireland
2 Laboratory of Solid State Physics (LPST), UMR 5477, Paul Sabatier University, 118 route de Narbonne, 31062 Toulouse Cedex 04, France
3 VTT Centre for Microelectronics, Tietotie 3, 02150 Espoo, Finland

**Inelastic light scattering by longitudinal acoustic phonons in thin silicon layers: From membranes to silicon-on-insulator structures**

J. Groenen, F. Poinsette, and A. Zwick
Centre d’Elaboration des Matériaux et d’Etudes Structurales UPR 8011, CNRS-Université Paul Sabatier, 29 Rue Jeanne Marvig, F 31655 Toulouse Cedex 4, France

C. M. Sotomayor Torres
University College Cork, Tyndall National Institute, Lee Maltings, Cork, Ireland;
Catalan Institute of Nanotechnology, Campus de Bellaterra, Edifici CM7, E08193 Bellaterra (Barcelona), Spain;
and ICREA-Catalan Institute for Research and Advanced Studies, 08010 Barcelona, Spain

M. Prunilla and J. Ahopelto
VTT Micro and Nanoelectronics, P.O. Box 1006, FI-90044 VTT, Espoo, Finland
(Received 18 July 2007; published 23 January 2008)
3ω method and samples for nanoscale thermal measurements

- **Principle:** Temperature of the device lower if the substrate is more thermally conductive
- **Method:** Link the device temperature to the substrate thermal conductivity

Probes in contact with the pads of the chip fabricated at VTT

Schematics of a grease with nanoparticles and its nonplanar surface

P-Oliveir Chapuis
Diffraction Measurements

Patterns from triangular 2D defect layers inscribed on top of 3D PMMA opals with different periodicity

For these lattice constant and laser wavelength, unpatterned parts show no pattern

G Kocher, C M Sotomayor Torres et al to be published
Buried 2D Defect Layer in a 3D photonic crystal

Diffraction patterns from buried 2D defect layer inscribed in PMMA opals
How to increase the light extraction of a polymer film?

1. Modification of emission pattern: **micro-cavity**
2. Diffraction of trapped light: **Photonic Crystals**

\[ \frac{1}{4n^2} \approx 9.8\% \text{ of light extracted} \]
\[ (n=1.6) \]
\[ \approx 90.2\% \text{ trapped in the material} \]

Potential application: OLEDs
Emission of (CdSe)ZnS nanocrystals unaffected by NIL
Homogeneous distribution of nanocrystal in matrix essential
Improvement of light emission by factor of 2.4
Plasmon-exciton coupling in printed photonic crystals


PL spectra of the dye doped polymer film with (red) and without (black) Au nanorods, 39-fold enhancement in the PL intensity.
• A sound starting base for nanofabrication work to underpin research in CIN2.
• An enormous know-how pool on nanofabrication available. Logistics is a problem. “Viscous medium” is the other. Eg: 24 x 7.
• Procedures for efficient and timely work are needed to use access potential of CNM-ICTS Nanolithography clean room.
• Central pooling of information? Access via web?
• So far incomplete processing line for non-CMOS compatible fabrication. Access to CNM-ICTS Nanolithography covers part of processing needs. Many processes must be developed anew.
• Much work is carried out in collaboration with external partners through joint projects.
• Future looks much better with the new CIN2 building closer to completion.