Those slides summarized several research projects I carried out at National Research Council (NRC) during 2003-2008

Nanostructure Fabrication by Nanoimprint Lithography and its Applications

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Part I: Introduction
What is nano? Why nano? How to reach nano?
Micro and nano-fabrication: lithography, etching, thin film deposition
One example of nanofabrication

Part II: Nanoimprint lithography (NIL) (hot embossing)
Hot embossing of several materials
• Polystyrene (biocompatible)
• Poly(lactic-co-glycolic acid) (biodegradable)
• MEH-PPV (semiconducting polymer for plastic solar cell)
• Metal (pulsed laser melting)
Replication of 100nm to mm-scale features by thermal NIL

Part III: Plasmonic chemical/biosensors based on metallic nanostructures
Introduction to plasmon
DNA hybridization detection by surface plasmon resonance (SPR)
Surface enhanced Raman scattering (SERS)
• Raman scattering and SERS fundamentals
• Fabrication of nano-prism array as SERS active substrates
• SERS detection of R6G molecules and peptides
• Fabrication of nano-pyramid, nano-ring/crescent and nano-star (brief)
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How small is nano?

**Things Natural**
- Ant ~ 5 mm
- Human hair ~ 50 micron wide
- Red blood cells ~ 2-5 micron
- Virus, ~100nm
- DNA ~2 nm diameter

**Things Manmade**
- Head of a pin 1-2 mm
- MicroElectroMechanical Systems (MEMS) 10 -100 micron wide
- Intel transistor
- Nanorobot
- Carbon nanotube ~2 nm diameter
- Au nanoparticles 13 nm & 50 nm

Adapted from office of Basic Energy Sciences Office of Science
Why go to nano?

L: length

- **Integrated circuit**: faster, more function, lower power consumption
- **Data storage**: higher capacity ($\propto 1/L^2$), 1Tbits/in$^2$ – 25nm×25nm/bit
- **Semiconductor**: quantum confined phenomena (quantum dots/wells…)
- **Magnetism**: single domain formation at $L<$magnetic domain wall thickness, superparamagnetism
- **Photonics**: new phenomena at $L<\lambda$
  - photonic crystal, negative refractive index, near field optics, plasmonics
- **Biomedical**: DNA sorting (nanofluidics), drug delivery (nanoparticles)
- **Chemistry**: higher surface area
  - higher reactivity for catalyst, higher sensitivity for chemical sensors
How to reach nano?

Bottom up (chemical): assemble small building blocks (atoms…) into larger structures
chemical synthesis, self assembly
nanoparticle, nanowire, nanotube…
low cost, high resolution (~few nm), 3D (sphere)

Top down (engineering): modify large object to give smaller features
lithography, thin film deposition, etching
expensive, precise control of size/shape and positioning

Top down approach: three components
Lithography: generate pattern in a material called resist
photolithography, electron-beam lithography, nanoimprint lithography
Thin film deposition (growth): spin coating, chemical vapor deposition,
molecular beam epitaxy, sputtering, evaporation, electroplating
Etching (removal): reactive ion etching, ion beam etching, wet chemical etching, polishing

→Semiconductor industry uses top down approach
Lithographies: a comparison

Photolithography: *duplicate* pattern on the photo-mask into photo-resist fast, low resolution (~\(\lambda\)), standard for micro-fabrication

Electron beam lithography: *generate* pattern by serial writing into a resist slow (1 wafer/day), high resolution (~10nm)

Nanoimprint lithography: *duplicate* mold pattern into a polymer resist fast, high resolution (2nm), low cost

→ NIL is the choice for nanoscale patterning over large surface area
Nanoimprint lithography (NIL)

**Thermal NIL**
(hot embossing lithography)
- Rigid resist at room temperature (RT).
- Softened and patterned at high T.
- De-emboss at low temperature.
- Simple and low cost resist polymers, for general applications.

**UV-curable NIL**
- Soft liquid resist, patterned at RT
- Hardened by UV-induced polymerization
- No thermal cycle, targeted for semiconductor industry requiring accurate alignment

UV-illumination
Nanofabrication: one example

Metal nanostructures

1. Thin film growth
2. Lithography
3. Deposition
4. Etching (dissolve resist)

Liftoff process
resist (polymer)
1. Thin film growth
2. Lithography
3. Deposition
4. Etching (dissolve resist)

Direct etch process
resist (polymer)
1. Thin film growth
2. Lithography
3. Etching
4. Etching (dissolve resist)

→3-5 steps to pattern each layer, computer chip has >15 layers
One more step: 200nm period pillar array with various diameters

1. Cr dots by liftoff

2. RIE silicon and remove Cr (RIE: reactive ion etching)
Goal: study how many antibody molecules are required to activate a cell. Activation represented by the upward slope of the fluorescence curve.

→ More antibodies, shorter cell activation time

Collaboration with MIT/Northeastern University (ongoing)
1. Nano-science studies the length range 1nm-100nm
2. New physics and applications appear at nanoscale, or just better performance
3. Two approaches for arriving at nano: “bottom up” and “top down”
4. Three components for “top down” approach: lithography, thin film deposition and etching.
5. Nanoimprint lithography is a molding process having high throughput and high resolution.
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Hot embossing of polystyrene (biocompatible)

Application: contact guidance of cell growth

- **Definition:** anisotropic topographic features induce cells to align along the direction of the anisotropy.
- **Importance:** in tissue engineering, if tissue is to be repaired, the new cells must be aligned and positioned correctly.

Collaboration with LOEX of Laval University
Corneal and dermal cell growth

- First layer: both cells aligned with the grating (as expected).
- Second layer:
  - Corneal cells - oriented at 60° relative to first layer, as in a native cornea
  - Dermal cells - no orientation

Guillemette, Cui… Nature Methods, to be submitted
Hot embossing of PLGA
(poly(lactic-co-glycolic acid))
(biodegradable, biocompatible)

Application:
Brain repair: grow neuron cells on PLGA gratings and transplant it into brain. The support PLGA will disappear in 1-2 months (as it is biodegradable).

Collaboration with NRC-Institute for Biological Sciences
Hot embossing of semiconducting polymers

Hot embossing at 120°C and 20bar.
MEH-PPV spun on a PEDOT/ITO/glass.

Collaboration with Organic Vision Inc. and McGill University
Application: nanostructured plastic solar cell

Classic planar p-n junction, low junction area, low efficiency

Nanostructured junction, high junction area, high efficiency

Layers: glass/ITO/PEDOT/MEH-PPV(p)/Alq3(n)/Al

- Plastic solar cells: flexible, light weight, tunable electrical properties, and potential lower fabrication cost.
- Limitation: low energy conversion efficiency due to low carrier mobility.
- Method to increase efficiency: increase the interface area by nano-patterning the p-n junction.

Cui…, NNT, 2004
“Hot embossing” of metal using pulsed laser

- XeCl pulsed excimer laser, $\lambda=308\text{nm}$, $\tau=20\text{ns}$, 0.2-0.5J/cm².
- Lines rounded due to surface tension and volume shrinking upon solidification

- Metals are generally more difficult to pattern due to lack of suitable RIE process (RIE: reactive ion etching)
- Here Ni and Cu were patterned and the step took only $\sim 100\text{ns}$ with minimal heating of the substrate

Cui..., manuscript in preparation
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NIL for small features (<10nm)

Press liquid into a nano-hole

Pressure (P)

F = \gamma \times 2\pi R

\[ P = \frac{\gamma \times 2\pi R}{\pi R^2} = \frac{2\gamma}{R} \propto \frac{1}{R} \]

- Pressure \( \propto \frac{1}{\text{diameter}} \).
- But for protruded mold features (pillars…), local pressure at the pillar is much higher than average - easy to imprint.

→ NIL not difficult for nano, especially for protruded mold features

UV-curable NIL, **2nm** carbon nanotube mold
Hua, Nano Lett. 2004

Thermal NIL into PMMA (**10nm** pillar array mold)
NIL for large features (>100 µm) - simultaneous pattern duplication of large and small features

• Application: large features needed to connect small ones to the outside world (electrodes...).
• Challenge: more polymer must be displaced over longer distances.
• A popular approach: two-step process - small features by NIL, large ones by photolithography.

• Previously, pattern size of 100µm duplicated by thermal NIL.
• Here, we will demonstrate pattern duplication of nm – mm features.

→ More challenging to duplicate large features than small ones.
Modeling of liquid flow for large features (>>pattern depth)

Assumptions: periodic mold structure (period 2L)
ignore inertial, gravitational forces and surface tension
resist film thickness = mold trench depth = \( h_0 \)

\[
L = \frac{2h_0}{3} \sqrt{\frac{p\tau}{\mu}} \propto \left( \frac{p\tau}{\mu} \right)^{1/2}
\]

\( L \): achievable feature size
\( p \): pressure
\( \tau \): imprinting time
\( \mu \): viscosity
\( h_0 \): film thickness

→ Achievable feature size depends on pressure, time and viscosity of molten polymer
Strategy to imprint large features (mm)

\[ L \propto \left( \frac{p \tau}{\mu} \right)^{1/2} \propto \frac{1}{\sqrt{\mu}} \]

For PMMA at \( T > T_g = 105^\circ C \) (glass transition temperature)

\[
\log \mu = n \log M_w - \frac{12.21(T - T_g)}{70.1 + (T - T_g)} + \text{const}
\]

- \( M_w \): molecular weight
- \( n = 1 \) for \( M_w < M_C \), un-entangled molecules
- \( n = 3.4 \) for \( M_w > M_C \), entangled molecules

Viscosity for PMMA \( (M_C = 30\text{kg/mol}) \)
- a) 12 kg/mol, 200\(^\circ\)C
- b) 12 kg/mol, 150\(^\circ\)C
- c) 120 kg/mol, 200\(^\circ\)C

\( \mu_a : \mu_b : \mu_c = 1:126:278 \)

→ Use low molecular weight PMMA and imprint at high temperature
Square (mm) imprinted into PMMA

Optical image

Profile

Schematic

mold wafer

resist need to etch

silicon wafer

Need excessive etch to remove the thick resist at the square center

But for nanoscale features…

Such a profile makes liftoff difficult. Solution: use tri-layer resist system

Strategy to imprint large features (mm)

→ Need excessive resist etch and tri-layer resist for large features
Fabrication process flow

1. Imprint
2. RIE PMMA (excessive etch)
3. RIE SiO₂ (CHF₃ gas)
4. RIE ARC (O₂ gas, etch little SiO₂)
5. Evaporate Cr
6. Liftoff Cr (dissolve ARC)
7. RIE Si (etch little Cr)

Fabrication is liftoff process using tri-layer resist plus etching

PMMA: Polymethyl methacrylate.
ARC: Anti-reflection coating, cross-linked polymer.
RIE: Reactive ion etching

→ Fabrication is liftoff process using tri-layer resist plus etching
For small features, line-width increased by ~18nm (acceptable).

For large features, 1.3mm squares faithfully duplicated (one order higher than previously achieved).

Cui…, Microelectronic Engineering, 2006
Summary for Part II

1. Hot embossing can pattern various functional polymer materials: biocompatible, biodegradable, semiconducting
2. It is not so difficult for NIL to achieve high resolution (<5nm)
3. It is more challenging to pattern large features (>100µm)
4. We have demonstrated simultaneous pattern duplication of nm to mm-scale feature
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Plasmon: "bulk", surface, localized

Plasmon: collective oscillation of free electrons (has nothing to do with plasma TV (ionized gas))

\[
E_{ext} = E_0 e^{i\omega t} \quad \sigma = \sigma_0 e^{i\omega t}
\]

Surface plasmon: plasmon confined to (metal) surface

Localized surface plasmon: plasmon confined to (metal) nano-structures

\[
\omega_p^2 = \frac{4\pi ne^2}{m} \quad \sim \text{frequency of visible light}
\]
Green = Reflected Light
Red = Transmitted Light

Color is due to resonant absorption of light, which excites localized surface plasmon when $\omega(\text{light}) \approx \omega(\text{plasmon})$
Surface plasmon resonance (SPR) biosensor

SPR: excitation of surface plasmon by light
Plasmon frequency is very sensitive to changes on metal surface
Application: biosensor (DNA, protein…)

- Excitation of surface plasmon by *evanescent* wave
- Resonant absorption occurs at certain angle $\theta$

→ SPR sensor detects change in thickness/refractive index on metal surface
Enhanced DNA hybridization detection by SPR on periodic Au nano-post array

Au nano-posts increase sensitivity by:
1. Coupling between localized (from nano-posts) and propagating (from underlying continuous film) surface plasmon.
2. Increasing the surface area for DNA binding.

DNA hybridization leads to a change in -
- refractive index by 5%
- thickness by 3.5nm

Collaboration with McGill University
Enhanced SPR detection of DNA hybridization

Sensitivity was enhanced by $5 \times$ when post diameter = 50nm

SEM image of Au nano-post array on 20nm flat Au film

→ Sensitivity was enhanced by $5 \times$ when post diameter = 50nm

Malic, Cui... Optics Letters, 2007
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SERS (surface enhanced Raman spectroscopy) fundamentals

Molecular vibration:
- Molecular vibrational energy quantized (phonon).
- Its frequency lies in infrared (IR) spectral range.
- IR spectroscopy is used to identify a material. (strong absorption when \( \nu(\text{photon}) = \nu(\text{phonon}) \))

Raman scattering:
- \textit{Inelastic} light scattering from a sample with a shift in frequency by the energy of its characteristic molecular vibrations (phonon).
- It detects \textit{symmetric} vibration, which complements IR spectroscopy.
- The vibration information is transferred from IR to visible, where brighter sources and more efficient detectors are available.

SERS effect: enormous enhancement (up to \( 10^{12} \)) of Raman scattering when a molecule is adsorbed on \textit{nanostructured} metallic surface.
This arises from two main mechanisms -
- Electromagnetic effect (dominant):
  \textit{localized} surface plasmon resonance. (Raman intensity \( \propto E^4 \))
- Chemical effect: electronic coupling between molecule and metal.
SERS advantages, approaches to generate SERS-active surfaces

Advantage of SERS: (3 “S”)
- Sensitivity - high, single molecule detection demonstrated
- Selectivity - high, fingerprint of each type of molecule
- Speed - high, few seconds

→ Nanofabricated metal structures - tunable resonant wavelength, homogeneous size/shape, more reproducible and reliable SERS detection
Motivation:

- Localized surface plasmon resonance can be tuned systematically by varying the size, shape and height of the nanoprism.
- Sharp corners of the nanoprism lead to a strong local electromagnetic field enhancement.

Previous fabrication method:

- Chemical approach: reduction of metal salts (difficult).
- Light-induced aggregation of small nanoparticle seeds (low yield and none-homogeneous)

→ Nanoprism: tune plasmon resonance, enhance local electrical field
Fabrication principle

PMMA pyramid by NIL with a mold having inverse-pyramid-shaped hole

1. NIL into PMMA
2. Evaporate Ti at large angle
3. RIE PMMA
4. Evaporate and liftoff metal

→ Again, it is a liftoff process

**PMMA:** Polymethyl methacrylate
**NIL:** Nanoimprint lithography
**RIE:** Reactive ion etching
Fabrication of mold with inverse-pyramid-shaped hole array

KOH etch Si (111) direction 100 times slower than other crystalline planes. So etching will “stop” at (111) crystalline plane, forming an inverse-pyramid-shaped hole.

→KOH etching of crystalline Si is anisotropic
SERS detection of rhodamine 6G (R6G) molecule

SEM image of 200nm period nanoprism

Raman measurement:
excitation laser $\lambda=785\text{nm}$, power 0.16mW/mm$^2$, beam spot size 25-30$\mu$m, collection time 3sec.

→Raman signal from ~monolayer R6G is nearly comparable to that from bulk silicon

Cui…, Nanotechnology, 2008
Effect of Cr (adhesion layer) on SERS

Why Cr is detrimental to SERS?
Electric permittivity $\varepsilon = \varepsilon_1 + i \varepsilon_2$
Strong SERS requires large $\varepsilon_1$ (strong charge oscillation) and small $\varepsilon_2$ (low loss)
But at $\lambda = 800$ nm $\varepsilon_1(\text{Cr})/\varepsilon_1(\text{Ag}) = 1/20$ $\varepsilon_2(\text{Cr})/\varepsilon_2(\text{Ag}) = 50/1$

→ Keep Cr adhesion layer as thin as possible (1nm)
Discrete Dipole Approximation (DDA):
• It computes scattering of light by wavelength-scale structures.
• The structure is represented by an array of dipoles located in a regular cubic lattice.
• Advantage: only the domain of interest (the metal nanostructure) is discretized.

For single Ag nanoprism, $\lambda_{\text{res}} \approx 720\text{nm}$.
Cr decreases absorption efficiency.
SiO$_2$ substrate red-shift $\lambda_{\text{res}}$ by 100nm.
Thicker Ag decreases $\lambda_{\text{res}}$
2×2 array has higher $\lambda_{\text{res}}$
SERS detection of bio-molecules

Challenges:
Bio-molecules compose of similar repeating units (base for DNA, amino acid for protein), hence their Raman spectra are very similar

Solution:
• Attach a tag molecule to the bio-molecule, but no longer label-free
• Collect large number of spectra and do regression date analysis

Application:
Detection of drug (e.g. recombinant proteins) abuse in sports (doping).

H: Human;  B: Bovine;  S: Sheep;
CRF: Corticotropin releasing factors

Collaboration with NRC – National Institute for Nanotechnology
Regression Data Analysis

Collected 798 spectra per sample

Partial least-squares regression
A. Score plot representing the first two principal components.
B. Contour plot showing the correlation for the three CRFs.
C. Contour plot for their mixtures ("contaminated" samples).
D. Contour plot for the three CRFs and their 12 mixtures (B+C).

→ SERS can distinguish bio-molecules, but need collect large number of spectra

Alvarez-Puebla, Cui... ChemMedChem, 2007
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Other types of nanostructures fabricated by NIL: nano-pyramid

Fabrication principle:

1. Triangular hole array
2. Deposit **thin** film
3. Deposit **thick** film
   Opening close-up gradually due to lateral deposition
3. Liftoff

Nano-prism (2D)  
Nano-pyramid (3D)
Nano-ring and nano-crescent

Fabrication principle:
Nanoring (red) defined by edge of nanodisk (blue)

1. Hole array with large undercut

Cr deposition at an angle

2. Deposit Cr to cover hole bottom

Reactive gas

3. RIE with lateral etch

Near field for nano-crescent

High field spots

200nm

Cui…, Microelectronic Engineering 2007; Li, Cui…Nanotechnology 2008
Fabrication principle:

1. Nano-hole array (blue color)

2. Enlarge holes due to lateral etching

3. Nano-stars (red color) formed when adjacent holes touch each other
Summary for Part III

1. Plasmon is collective oscillation of free electrons
2. Three types of plasmon: bulk, surface and localized
3. Surface plasmon resonance can be used for biosensors
4. We achieved $5 \times$ higher SPR sensitivity by patterning Au nano-post array on a flat Au film
5. SERS is an enormous enhancement of Raman scattering when a molecule is adsorbed on a nanostructured metallic surface
6. Using nanoimprint lithography, we fabricated arrays of nano-prism, nano-pyramid, nano-ring/crescent and nano-star.
7. We demonstrated SERS detection of R6G molecules, as well as peptides classification.
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- LOEX (Laval University): M. Guillemette, FA Auger, L. Germain
- McGill University: L. Malic, M. Tabrizian
- NINT: R. Alvarez-Puebla, H. Fenniri
- Organic Vision Inc.: S. Xiao
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