Nanometrology: *enabling applications of nanotechnology*

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S Pullteap and H C Seat, ENSEEIHT, Toulouse

Outline

1. Introduction

2. Nanometrology for Nanoimprint lithography:
   a. Sub-wavelength diffraction
   b. Photoacoustic metrology

3. Nanometrology for Self-assembly
   a. Opposite partners/ elements
   b. Rotational diffraction

4. Conclusions

Nanometrology and Nanotechnology

- Critical to enable the industrial uptake of nanotechnology
- Necessary to measure:
  - Product characteristics, device performance, toxicology (potential public health risks), product lifetime, security
- Requirements for manufacturable technology
  - Standardisation, regulation – repeatable and universal
  - Easy to operate
  - Developed in coordination with manufacturing techniques
    - Integrated, in-line, real-time, advanced process control
    - Relevant measurands

Nanometrology Challenges

- Miniaturisation – things are getting smaller
- Heterogeneous integration – things are getting more complex
  - 3rd Dimension increasingly used
  - Dimensions and material properties
- Insufficient standardisation of techniques or reference samples
- Existing methods are slow, often destructive and not optimised for 3D

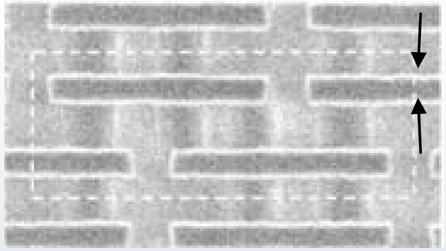
Metrology challenges for Nanofabrication

- **Critical dimension measurement < 50 nm**

  Semiconductor lithography node 32 nm (2011)

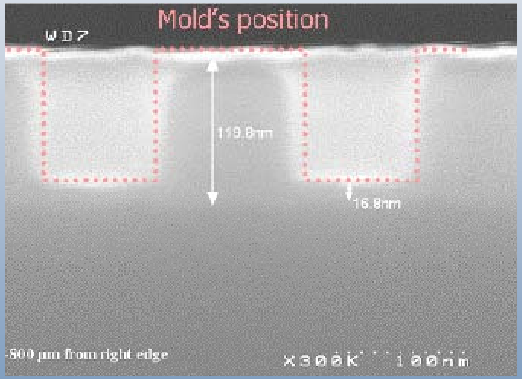
  Critical dimensions and physical properties

Intel SRAM

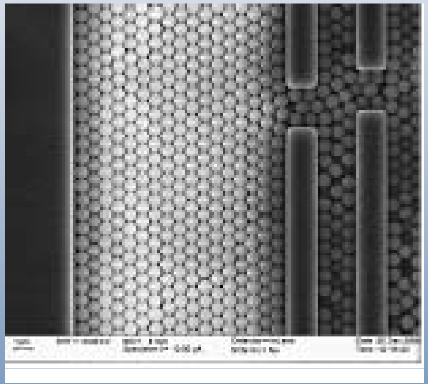


- **3D Structure**

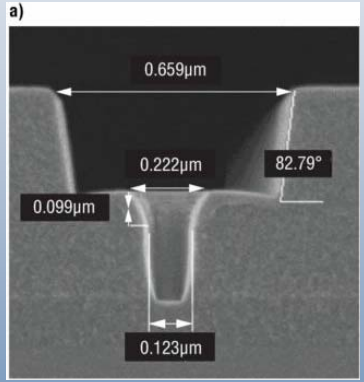
  Complex: Typical of heterogeneous integration, interconnects



S. Landis et al, Nanotechnology (2006)



Ye et al, Langmuiir **22** 7378 (2006).3D Photonic crystal in Si



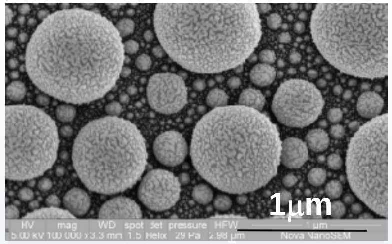
B.Chao, Proc SPIE 6921 (2008)

- **In-situ, inline, real-time**

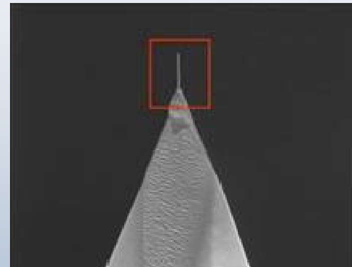
  Advanced Process Control of systematic drift and process behaviour

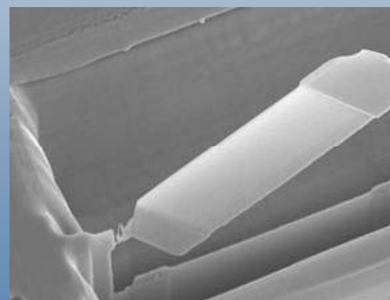
Metrology techniques for nanoscale – limitations

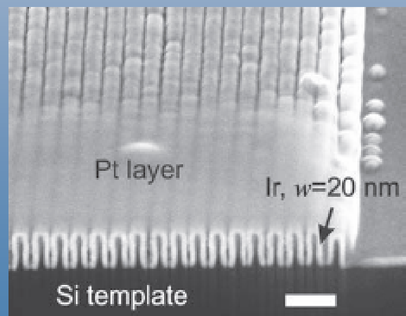
- **SEM** → For height, slope, profile, requires destructive cross-section.
- **AFM** → Difficult to access sidewalls, corners, relatively slow
- **TEM** → Resolution ~ 0.1 nm.
  → Destructive, slow
- **X-ray Imaging** → Requires synchrotron x-ray source
- **Optical Scatterometry**
  → Requirement of wavelength, polarization or angle variability.



A.E. Vladar et al, Proc SPIE 69220H

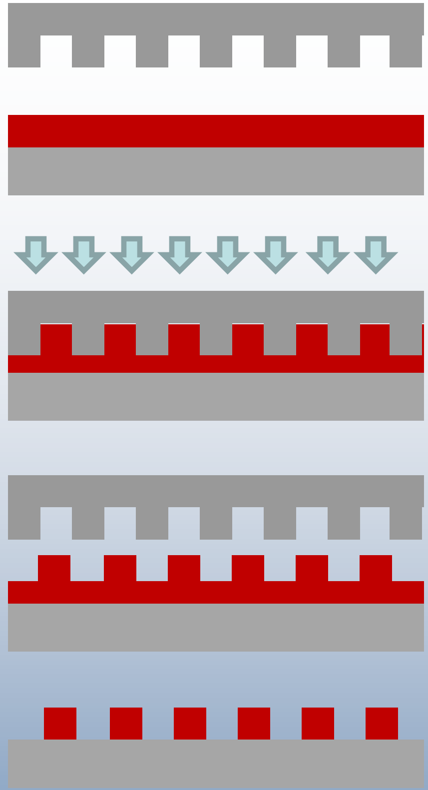

B.C. Park et al, Proc. SPIE 651819


Wintech Nano-Technology Ltd



C. David et al, Proc. MNE 2008

Nanoimprint Lithography (NIL)



Stamp (Si, Quartz, etc)

Resist (polymer, monomer)

Substrate

Imprint
(Pressure +heat or UV light)

Release
(cool down)

RIE of residual layer

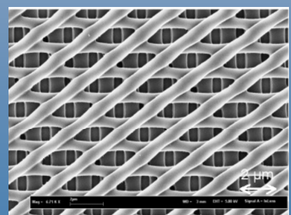
Advantages

- Resolution (sub 10 nm)
- Fast (sec/cycle)
- Low cost ($0.2M vs $25M)
- Simple
- Flexible (UV, heat)

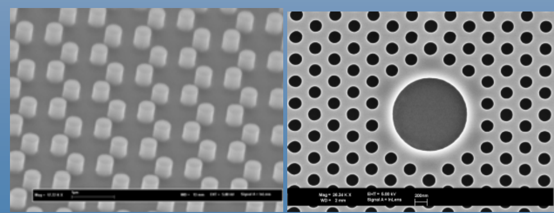
Applications

- Semiconductors
- Optics
- Bio
- Organic electronics
- Sensors

High resolution ·········· Complex patterns ·········· Functional devices



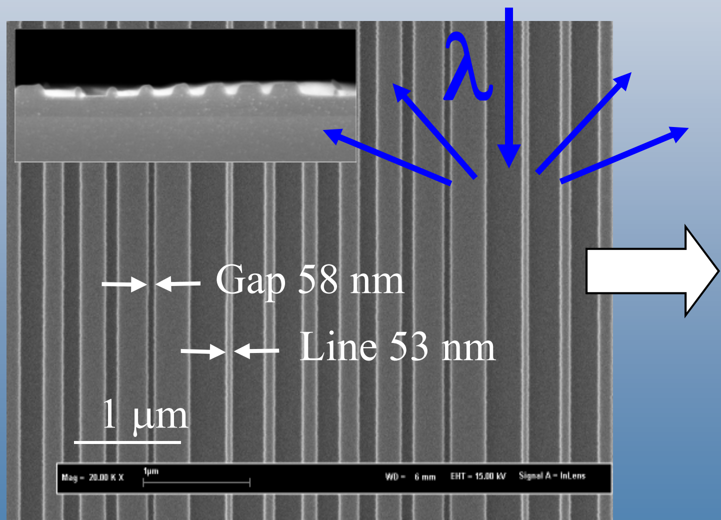
N.Kehagias, Nanotechnology **18** (2007)

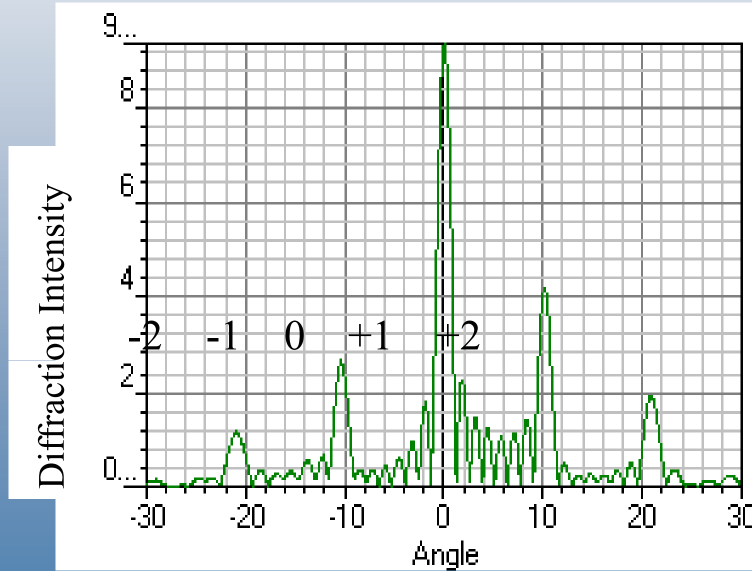


V.Reboud, Jpn. J. Appl. Phys., **47** (2008)

Sub-wavelength diffraction metrology

- Test structures of blazed gratings → asymmetric diffraction pattern
- Individual lines < diffraction limit. Groups of lines > diffraction limit
- Sub-wavelength features in grating eg Line-width, height, defects, sidewall angle, curvature. Linewidths: 50, 100, 150, … 350nm
- Defects affect relative intensity of diffraction orders in far-field
- Suitable for transparent or opaque structures
- Non-destructive, Fast collection of data

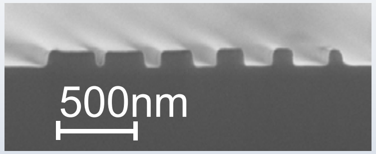


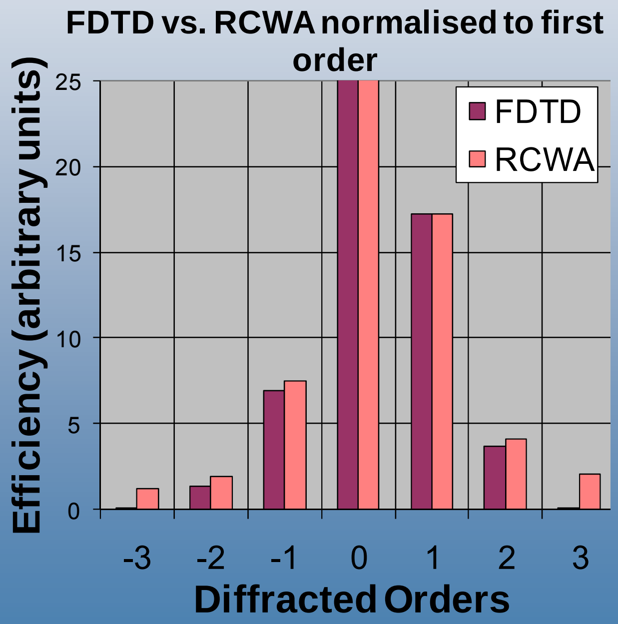


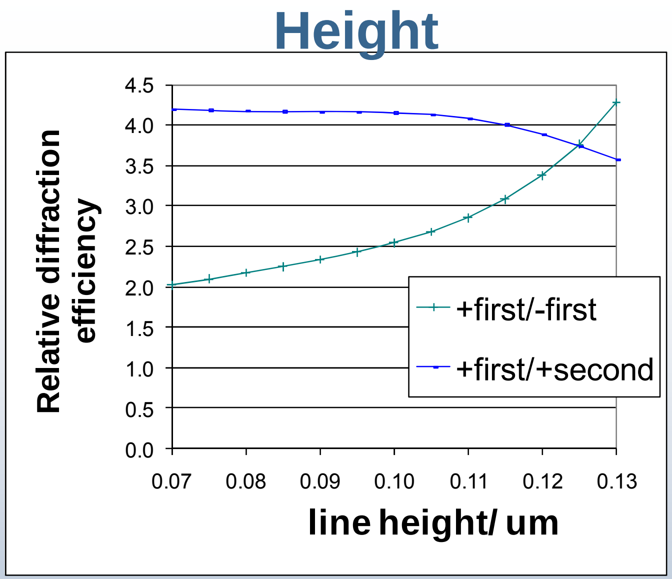
T. Kehoe et al, Proc. SPIE 69210F, 2008

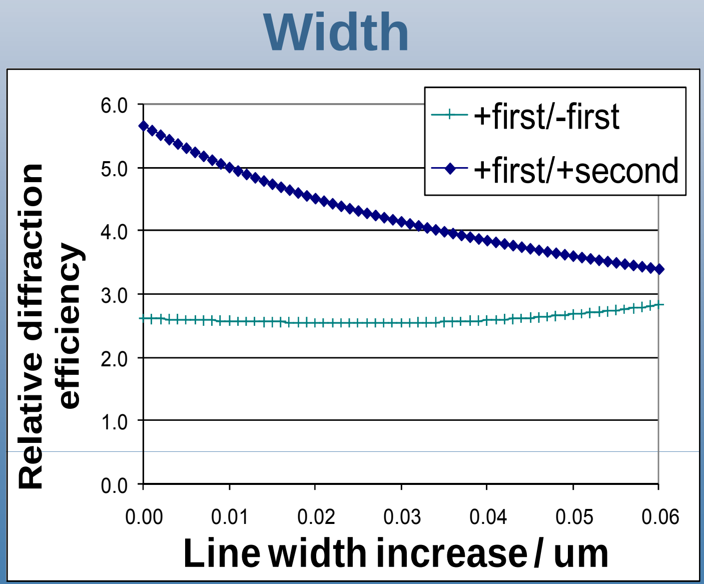
Modelling of Sub-wavelength diffraction

- **Rigorous Coupled Wave Analysis (RCWA)**
- **Finite difference time domain (FDTD)**



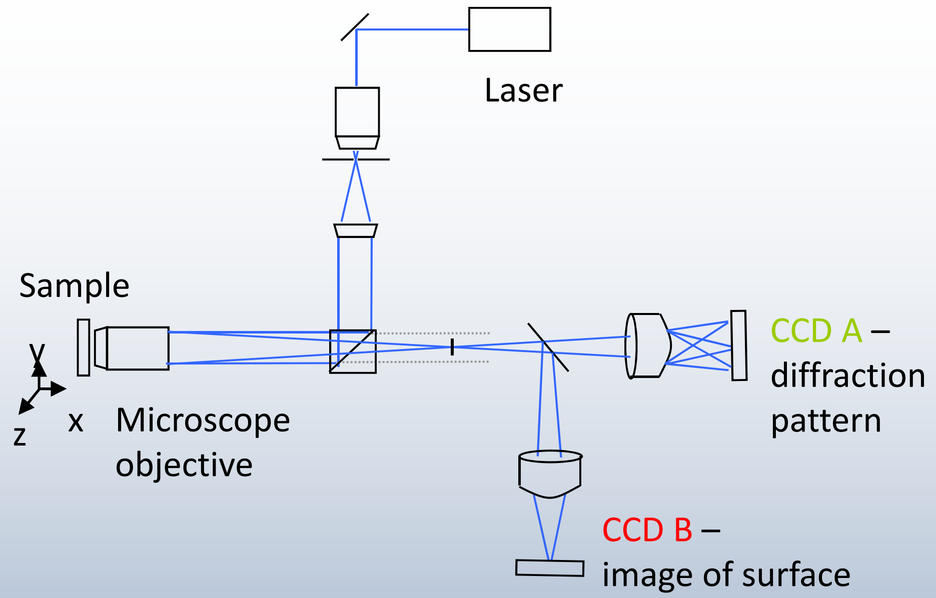


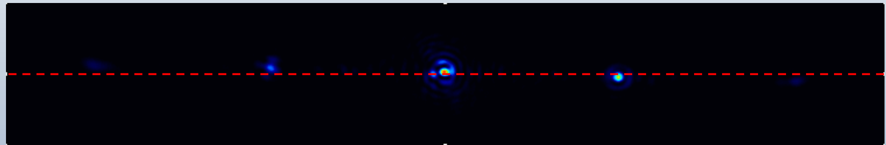


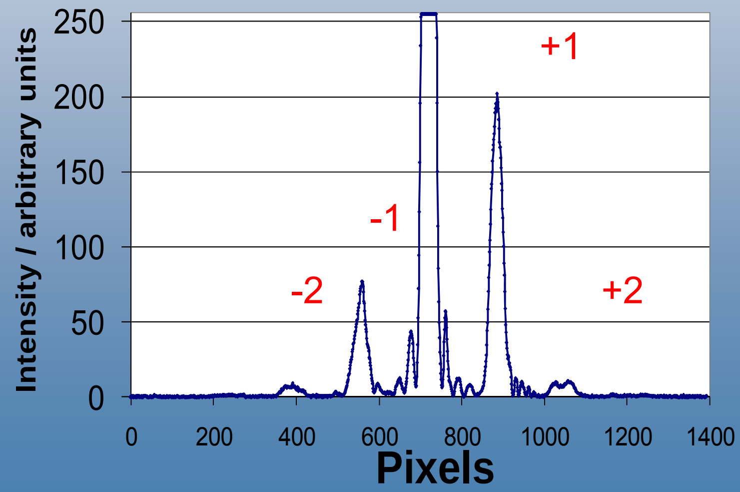


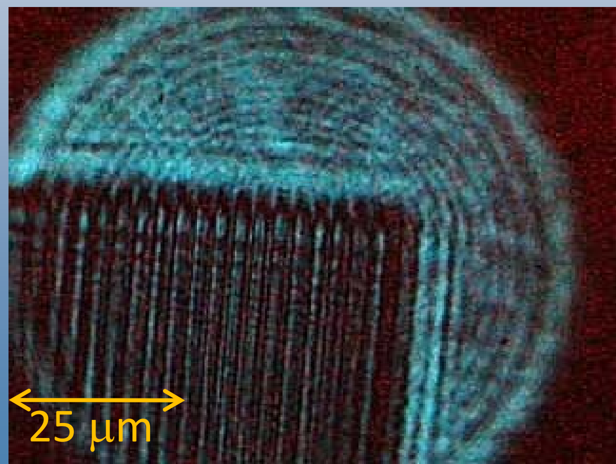
Sub-wavelength diffraction measurement system

- In-line design
- Sub-wavelength diffraction metrology with surface imaging by microscope optics
- Enables centring of the laser spot on the gratings





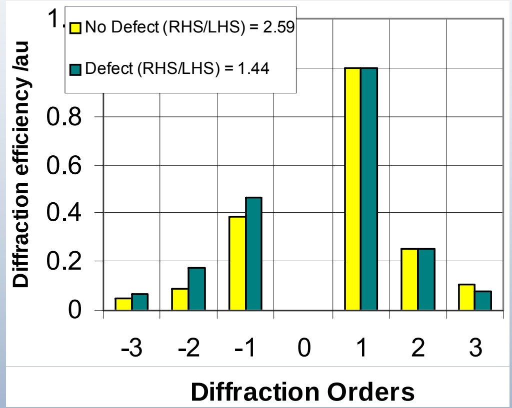




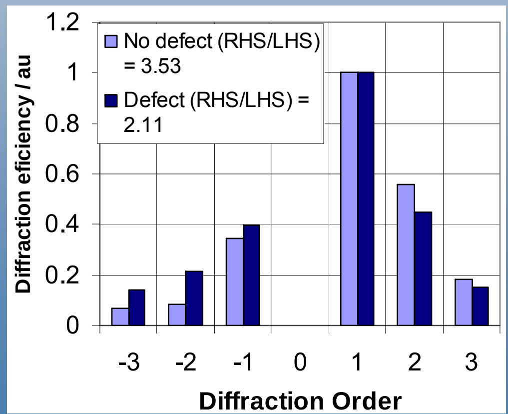
Sub-wavelength – Detection of defects

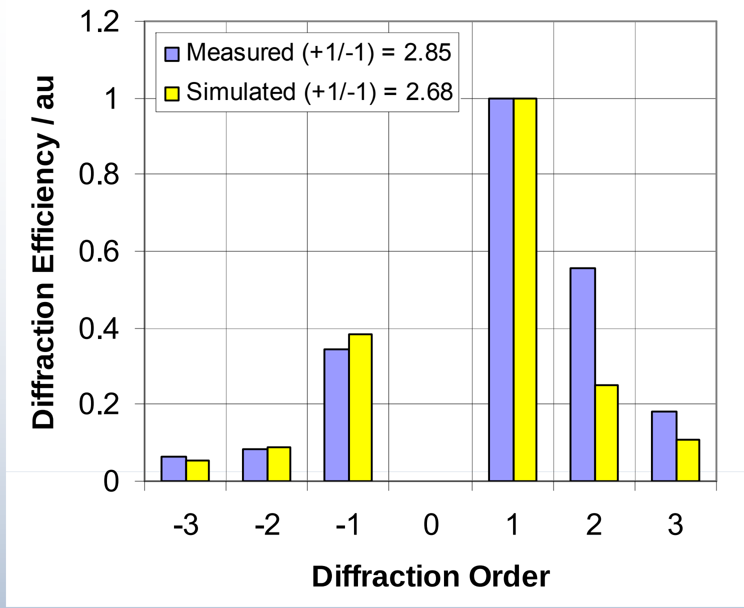
- Defect detection
  - – missing 50 nm & 100 nm lines
- Agreement between simulated and measured diffraction efficiencies

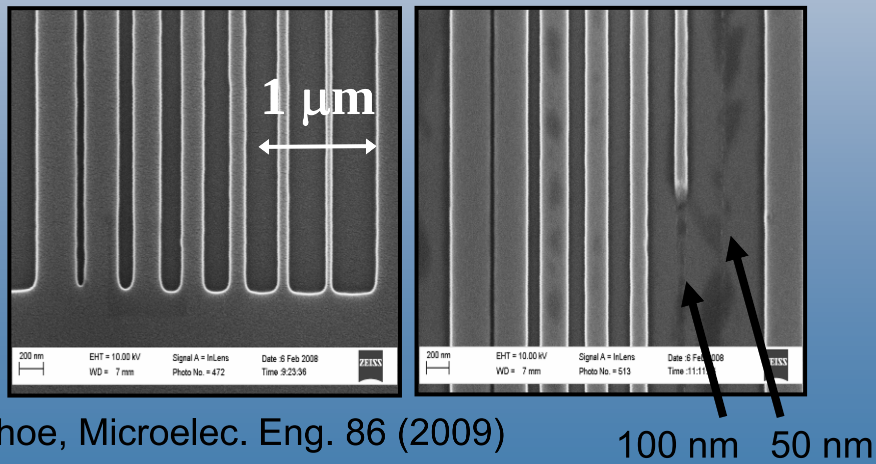
Simulated



Measured

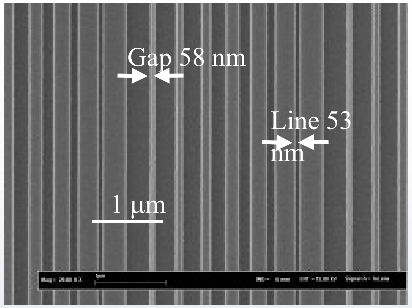




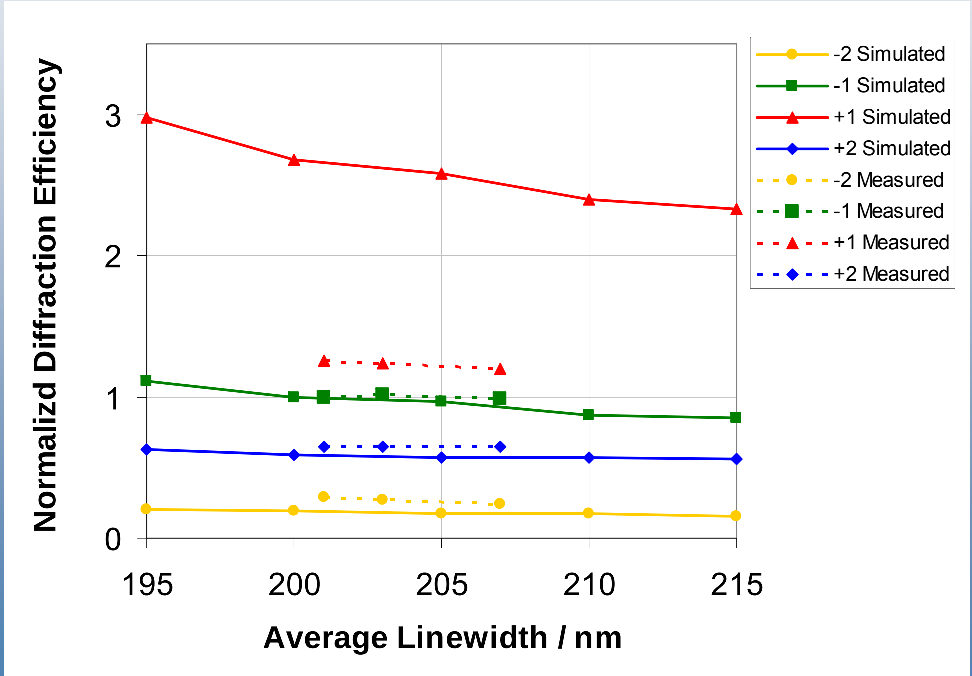


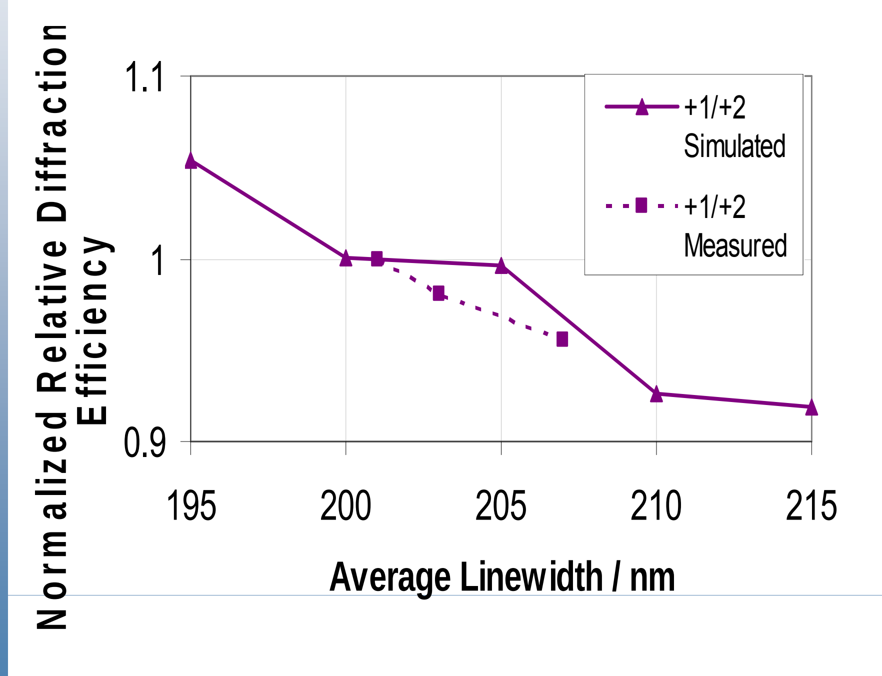
T. Kehoe, Microelec. Eng. 86 (2009)

Sub-wavelength – Detection of defects



- Grating stamps made with average line-width from 193 – 214 nm
- Measured and modelled diffraction efficiencies (1st & 2nd order) decrease with increasing line-width, by approximately 5% per 5 nm
- +1/+2 relative diffraction efficiency decreases at approximately 4% per 5 nm

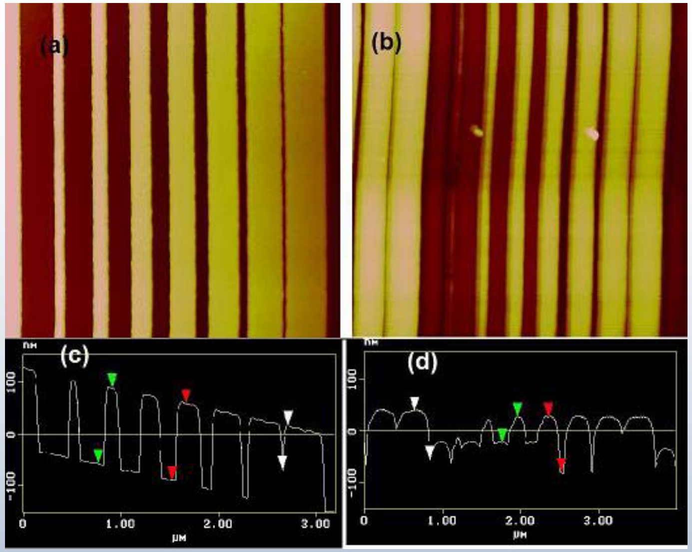




T. Kehoe, NNT 08 conference, Kyoto, Japan

Diffraction from 3D Structures

- Polymer relaxes and partially reflows, creating rounded line profiles
- Measured diffracted order intensities before and after reflowing of the lines



Comparison of measured and simulated diffraction intensities for imprinted lines and reflowed lines

T. Kehoe, NNT 2010, Copenhagen, Denmark

Key steps for NIL – Polymer physical properties

1. Stamp fabrication

2. Imprinting process: Temp, Pressure, UV

   → Glass transition temperature, Viscosity

3. Demoulding: Mechanical strength

   → Young´s modulus, Poisson’s ratio

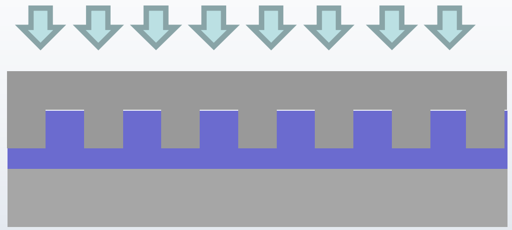
   Adhesion / anti-sticking coating

   → surface energy

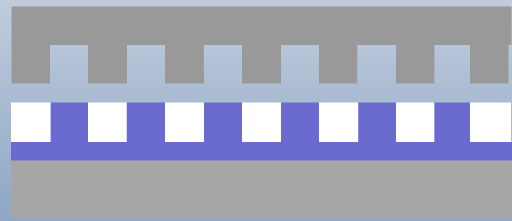
4. Etching: Polymer etch resistance

At nanoscale values may change

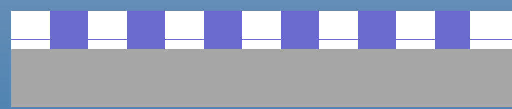
Imprint
(Pressure +heat or UV light)



Release (cool down)

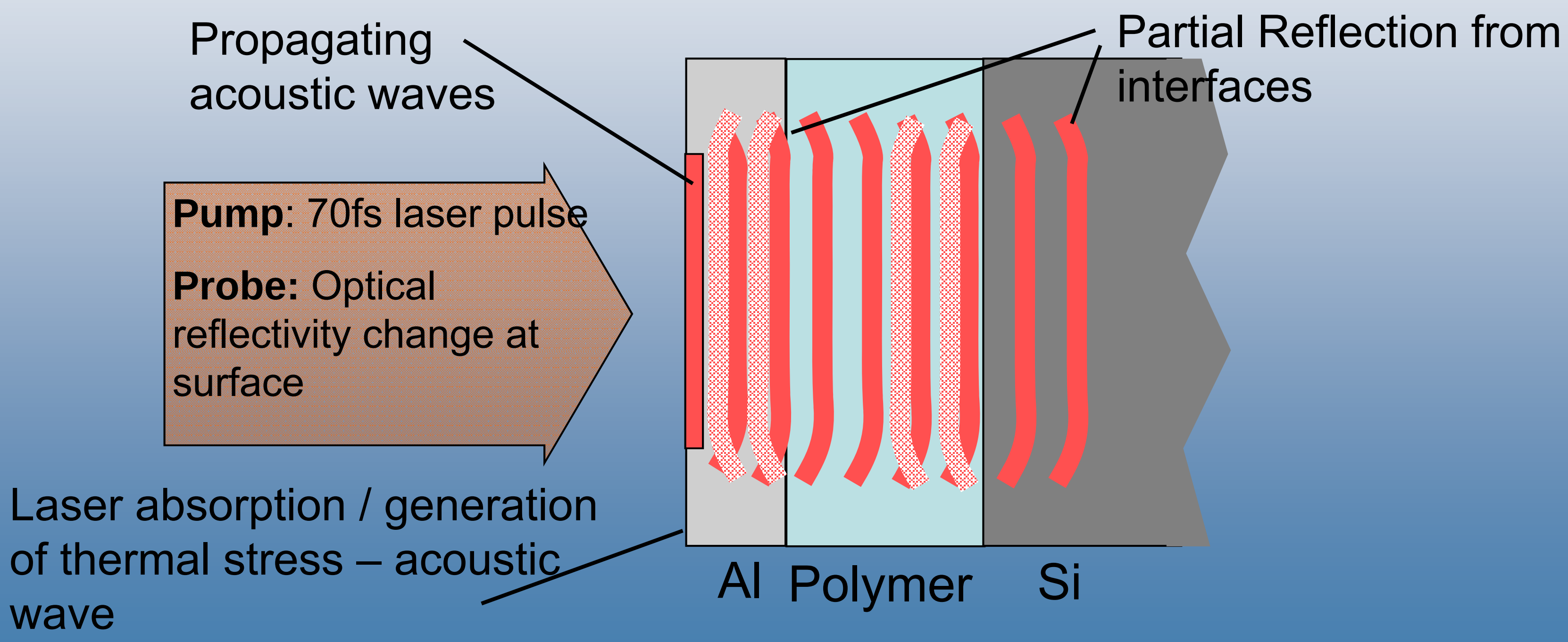


RIE of residual layer



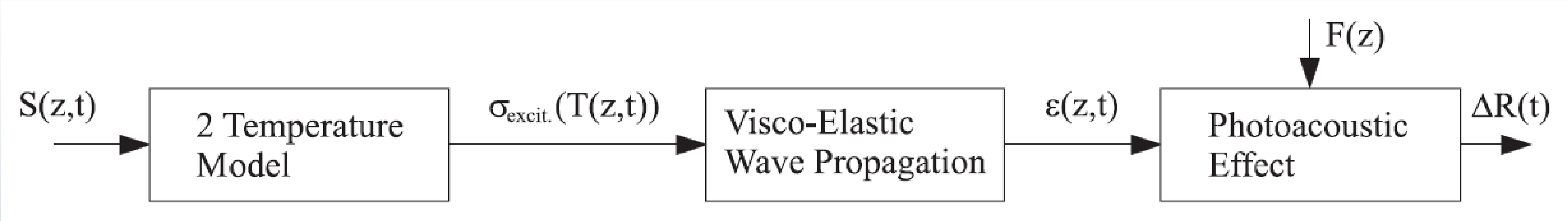
Photoacoustic Metrology

- Thickness measurements, resolution ~10 nm
- Acoustic scattering from interfaces → changes surface reflectivity
- Acoustic speed → Physical parameters – Modulus, Poisson's
- Pump-Probe Laser, 70 fs, λ = 810 nm, Time resolution 0.1 ps



One-dimensional photoacoustic model

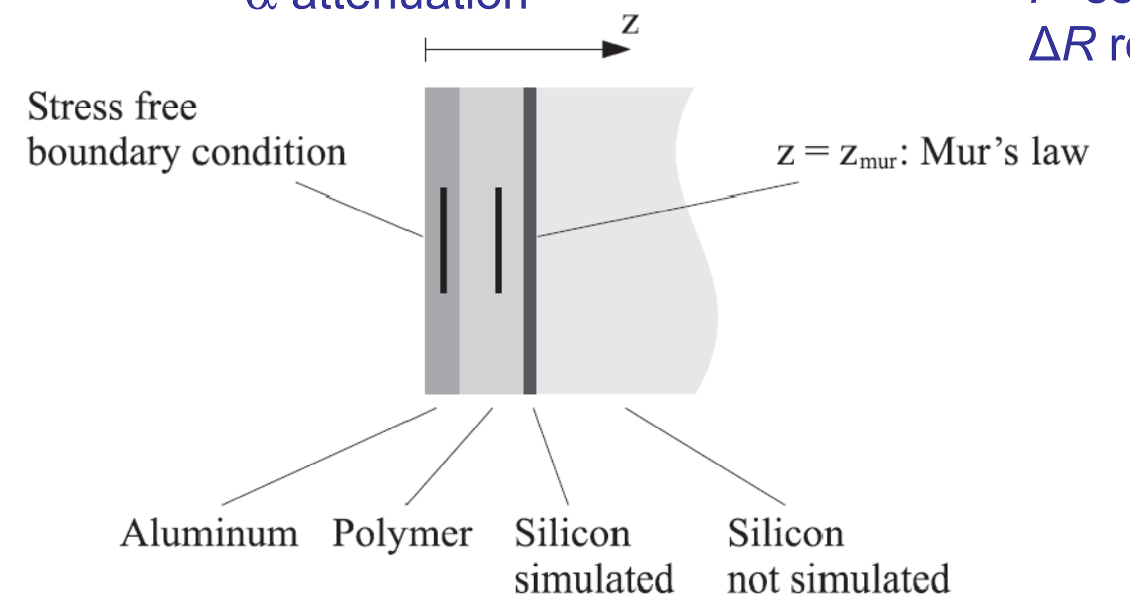
- Finite Element Simulation, including viscoelastic damping
- Measurement ΔR + Thickness (Ellipsometry) + Optical / Physical properties (absorption, density) → Thermomechanical model



S laser power
z depth
t time

σ_{excit} stress, *T* temperature,
α attenuation

ε strain
F sensitivity
Δ*R* reflectivity change



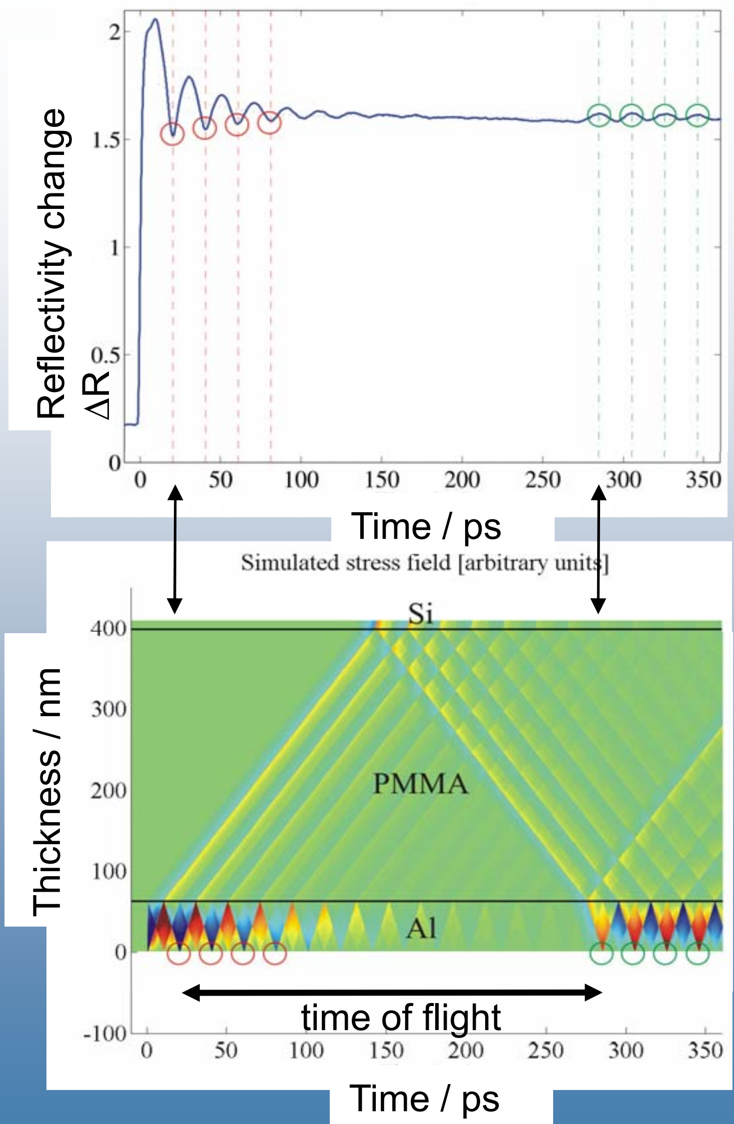
Photoacoustic Metrology of Nanoimprint Polymers

- Nanoimprint polymers
  13 – 586 nm thick layers
- Damping in polymer not excessive
- Good acoustic impedence difference → Strong signal
- Top interface: Al/polymer
- Bottom interface: polymer/Si
- FilmThickness compared to ellipsometry and profilometry
- Physical parameters calculated using Finite Element model

	c_p (m/s)	E (GPa)	ν *	ρ (kg/m^3)
mr-I PMMA	2603	3.2	0.4	1012
mr-NIL 6000	2504	2.95	0.4	1008

J. Bryner, 2007 IEEE Ultrasonics Symposium, 2007 p 1409

336 nm PMMA

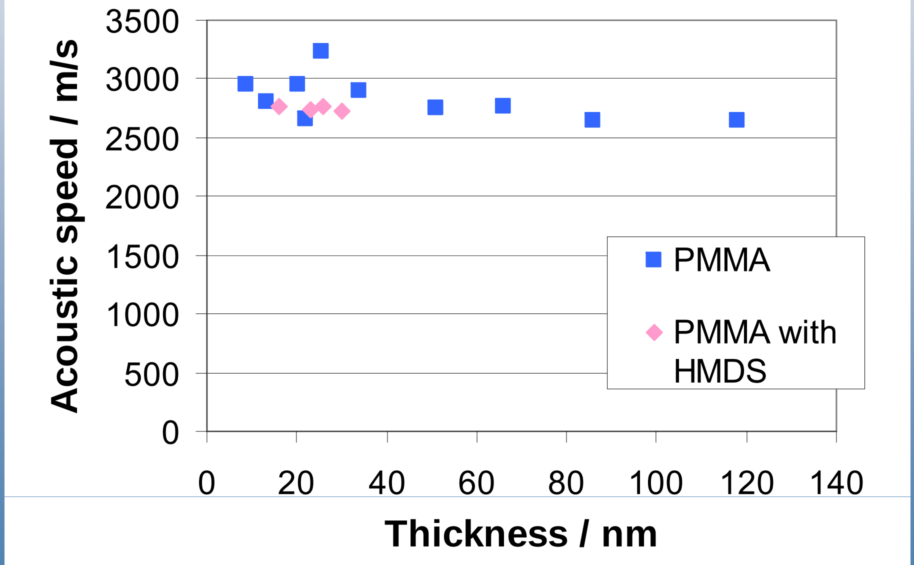


Nanoscale Effects

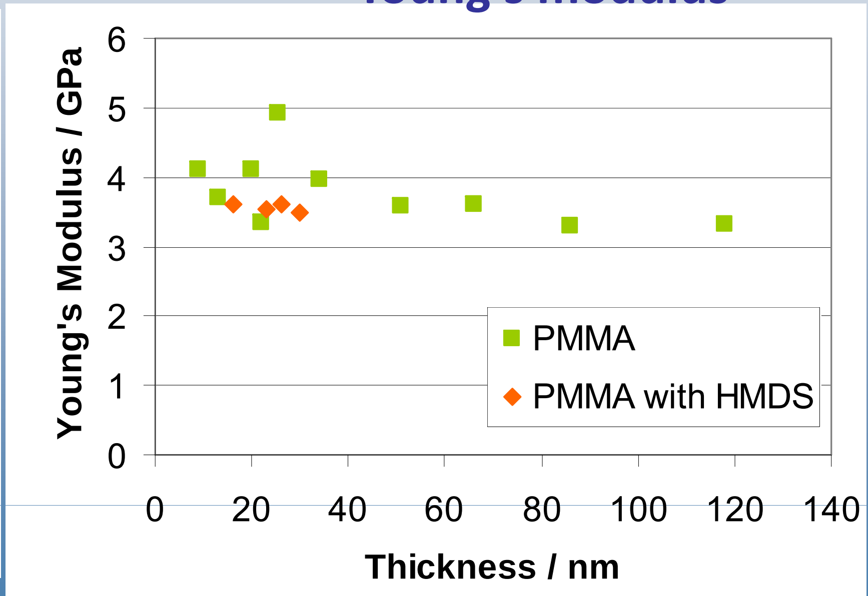
ICN Institut Català de Nanotecnologia

- Acoustic speed and Young's modulus increase below 80 nm
- Acoustic speed (c_p) increases by 12%
- Young's modulus (E) increases by 26% at 13 nm.
- Primer layer of HMDS (Hexamethyldisilazane) added → smaller increases
- Increase probably due to interface effects rather than confinement

Acoustic speed



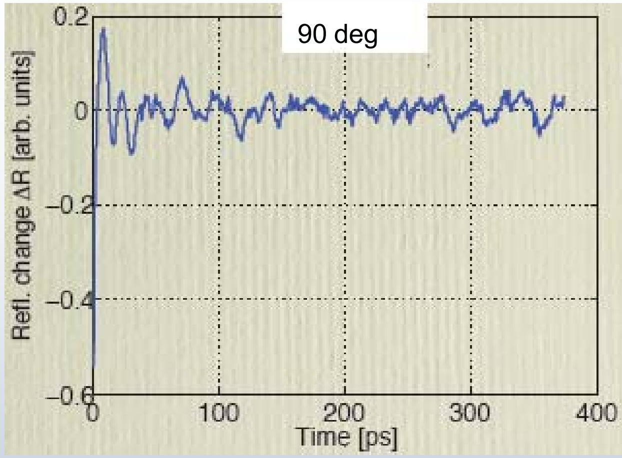
Young's modulus

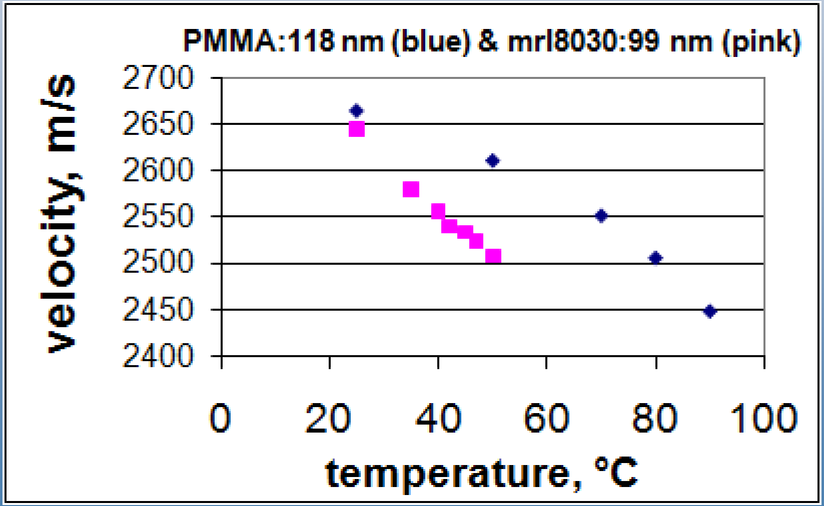


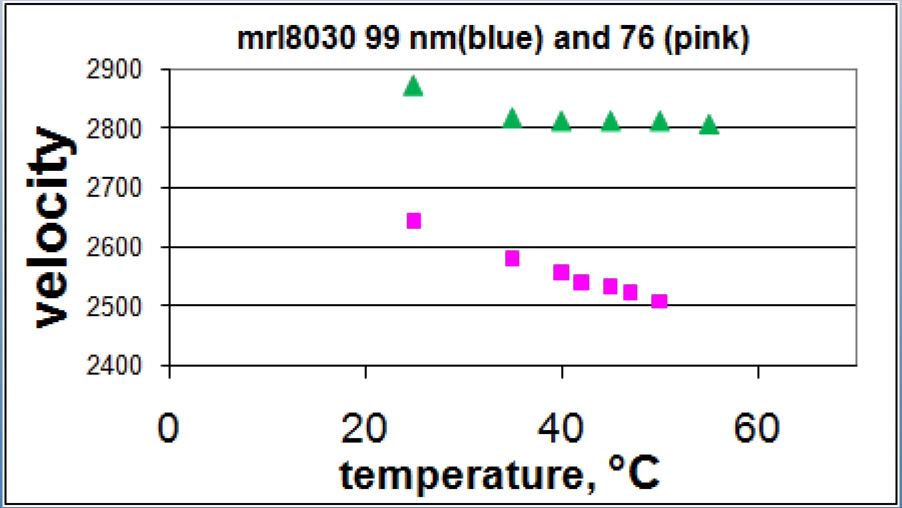
T. Kehoe, Proceedings of SPIE Vol. 6921 (2008)

Raised Temperature Measurements

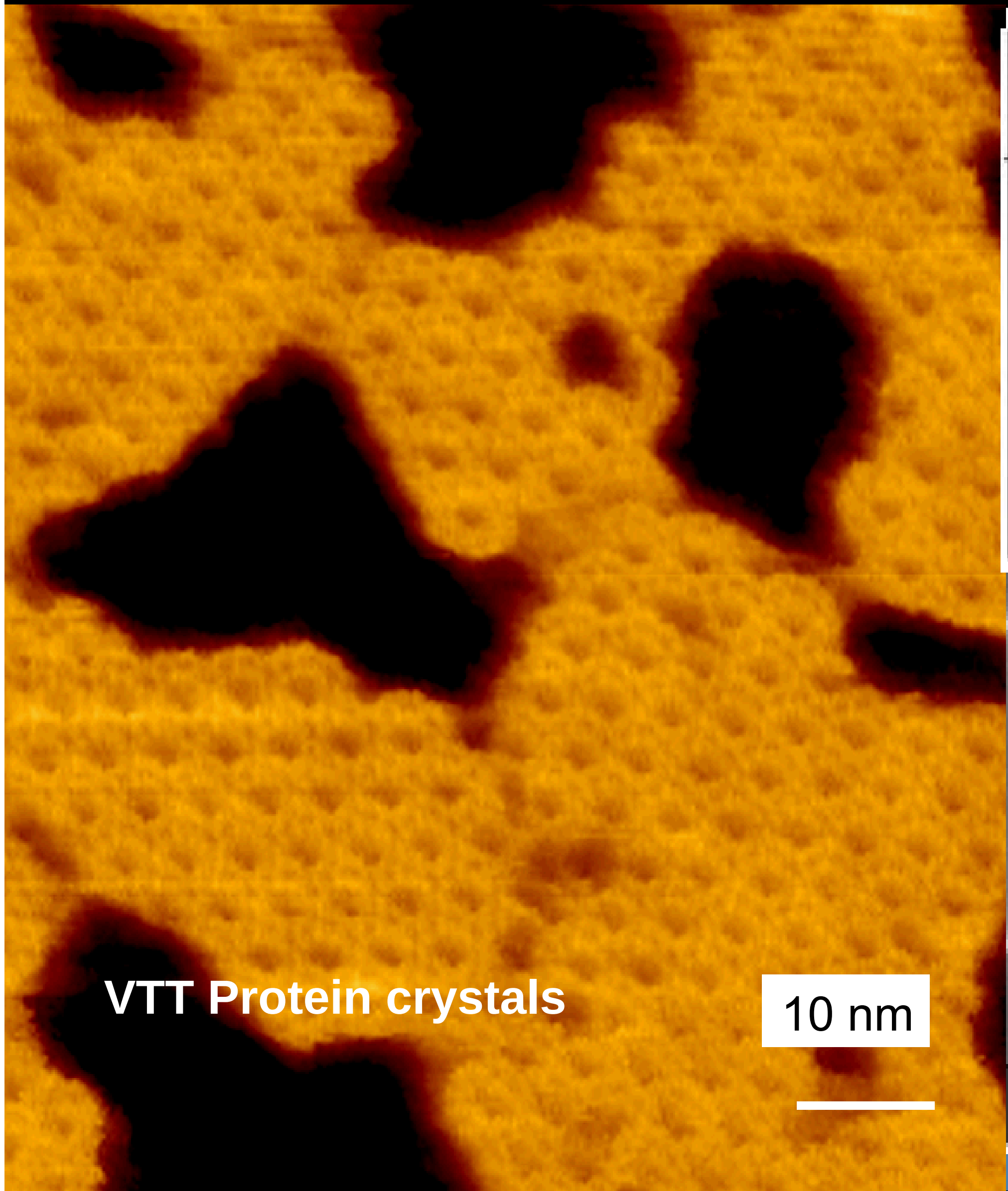
- Investigation of physical properties approaching glass transition temperature, T_g
- Acoustic speed inversely proportional to thickness
- Close to T_g increase of noise due to buckling of aluminium

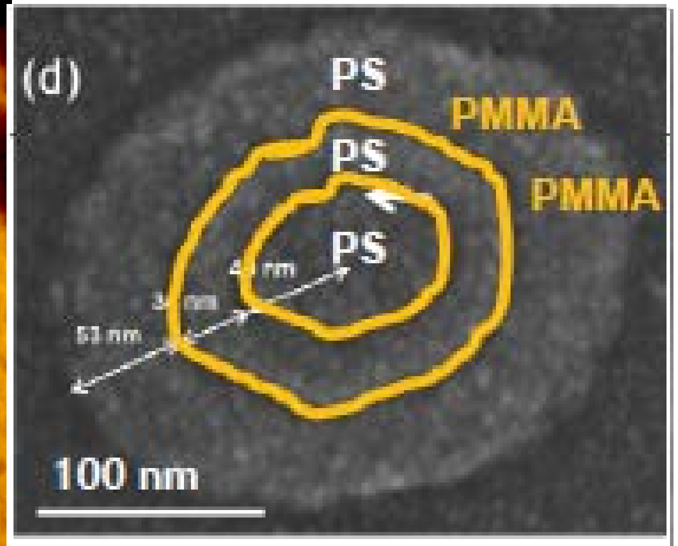


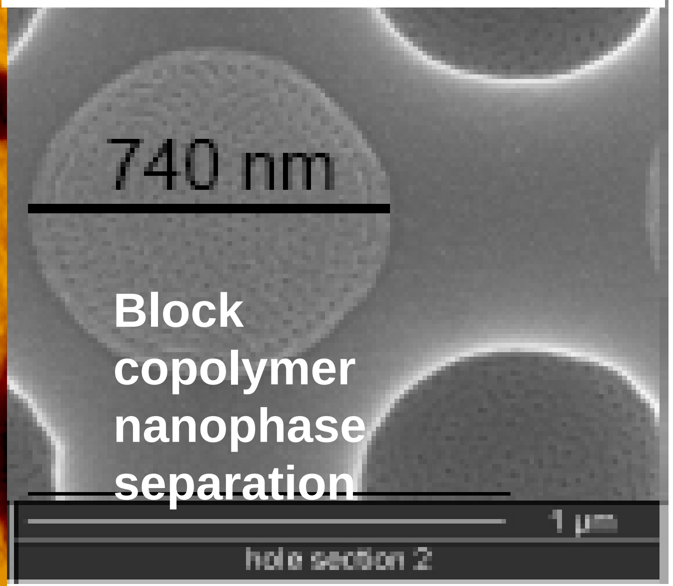


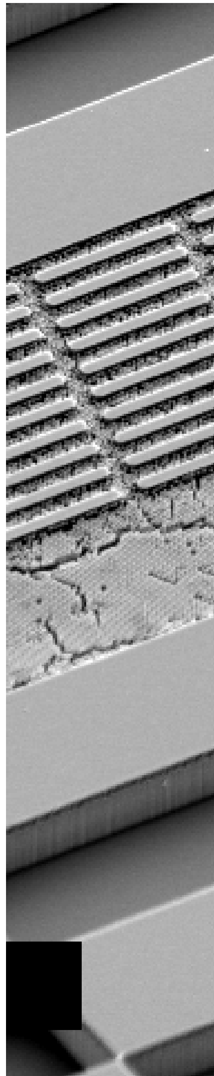


Examples of self-assembled structures



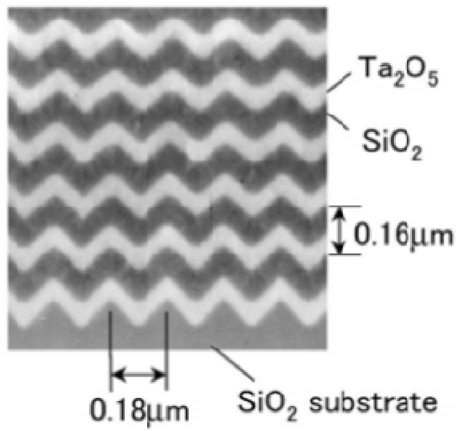






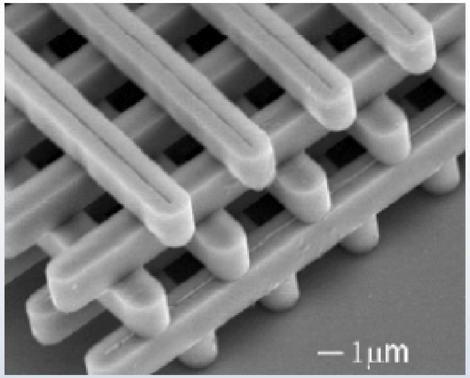
3D periodic structures: eg. photonic crystals

Autocloning



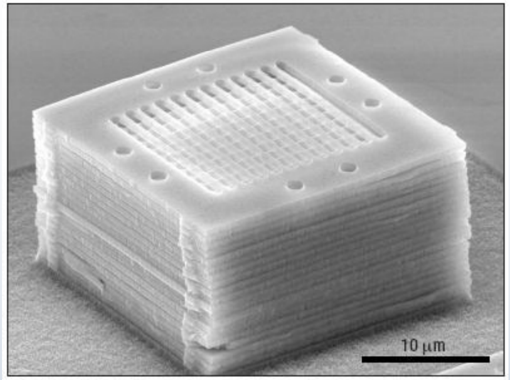
S Kawakami, Tohoku, 1997

Layer-by-layer



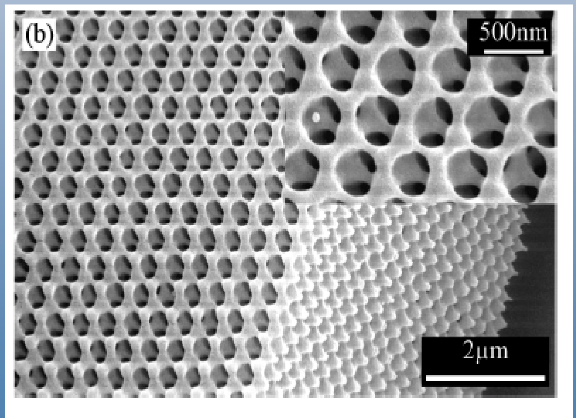
S Y Lin, Sandial Lab, 1998

Microassembly



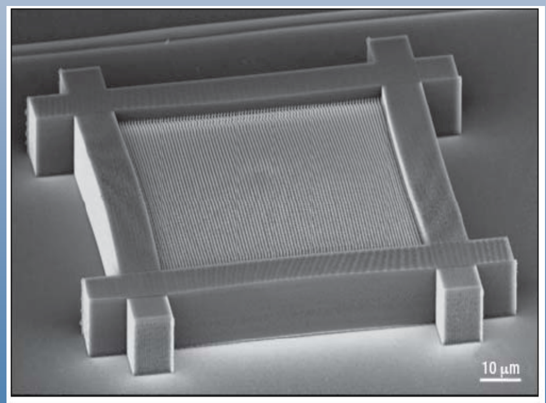
K. Aoki, Semicon Lab, 2003

Holography



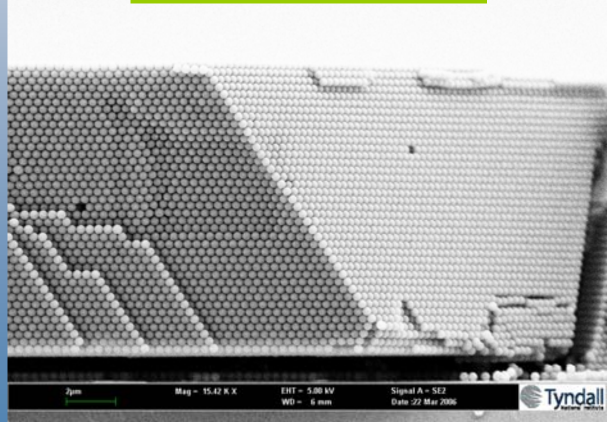
K. Aoki, Semicon Lab, 2003

Direct laser writing



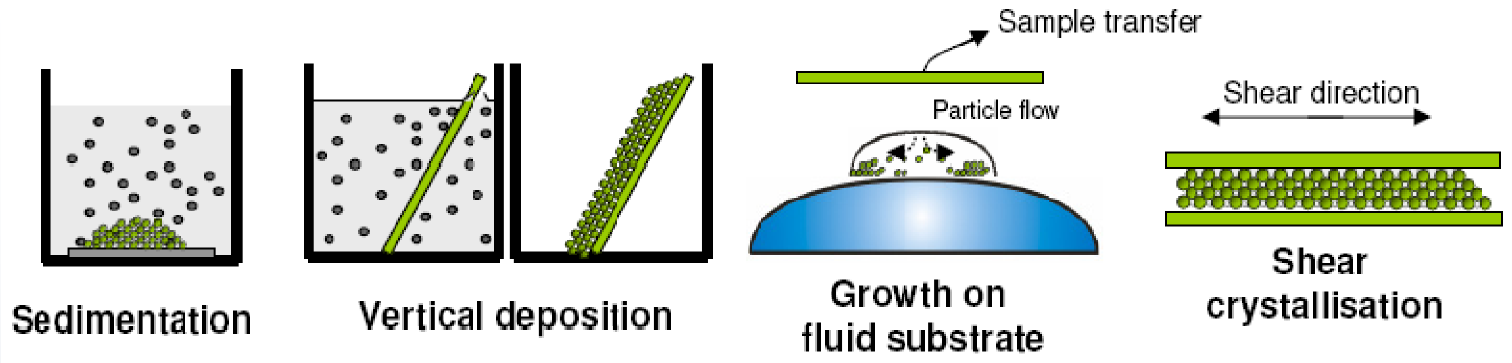
M. Deubel, Karlsruhe, 2004

Artificial Opals

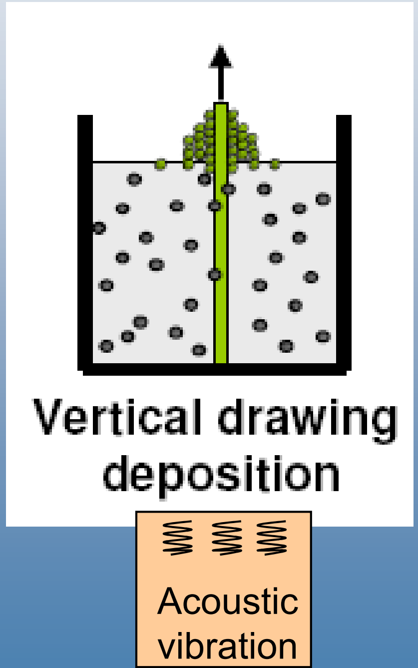


Tyndall/ICN-CIN2

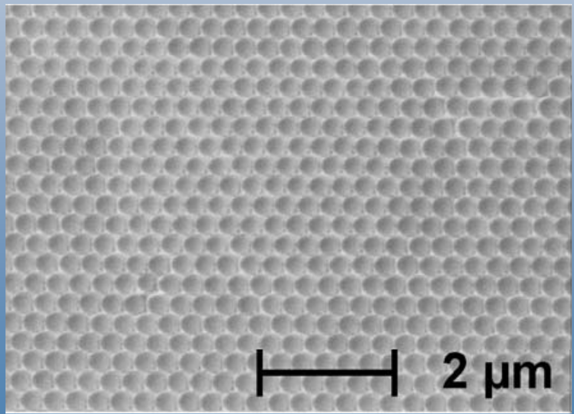
FCC colloidal crystals: Improving structural order

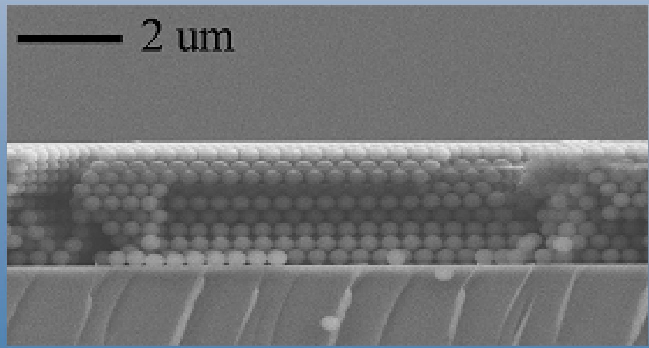


Equilibrium crystallisation → Fast crystallisation → Reduce interaction with substrate -> reduce crack → Shear force to improve crystal ordering



Improved quality using acoustic noise



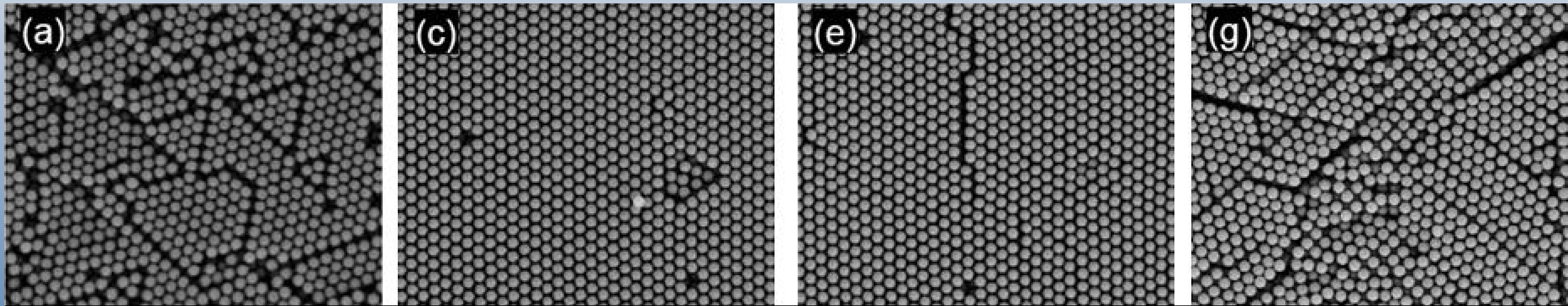


A .Amman et al Proc. SPIE Vol. 6603, 660321 (2007)

Quantifying order in self-assembly

- Define scale
- Make approach compatible with existing methods or at least acceptable
- In-line or a posteriori?
- Reliable?
- Suitable for a standard?

2 µm

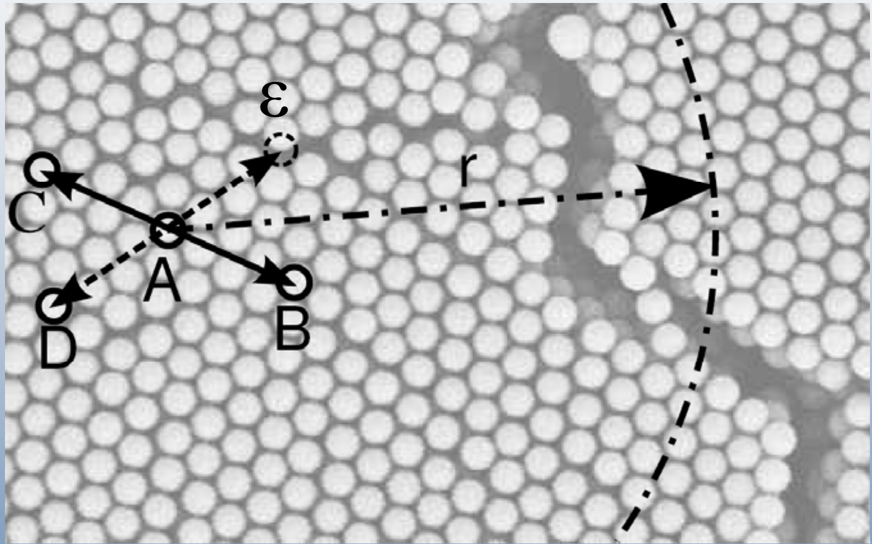


L = 0 dB	L = 20 dB	L = 30 dB	L = 40 dB

→ L = 20 dB is calibrated to water displacement of 2.5 µm

Concept of "opposite beads"

p(r) – probability of finding an opposite beads within a radius r, for a given tolerance parameter ε for the exact location of the spheres



At sphere 'A'

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

$$\chi_y(\vec{R}) = \begin{cases} 1 & if \quad |\vec{R}| < y \\ 0 & else \end{cases}$$

Global sum:
weighted average

$$p(r) = \frac{\sum_A N_A(r) p_A(r)}{\sum_A N_A(r)}$$

Conditions met by p(r)

- Scalar quantity (dependence on certain predefined orientation undesirable)
- Integral measure of a locally observable quantity
- Based on actual position of sphere (not on pixel representation of SEM image: contrast & focus dependent)
- Robust against missing spheres.

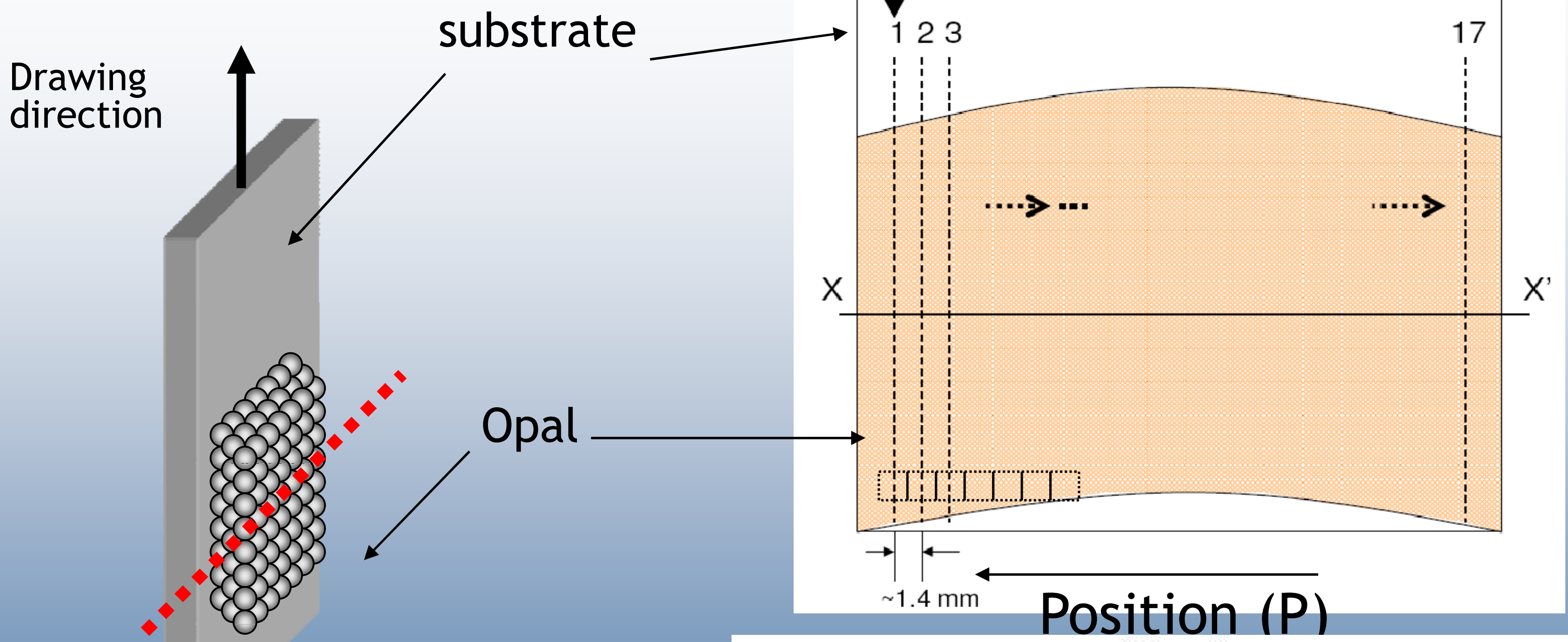
Perfectly ordered system

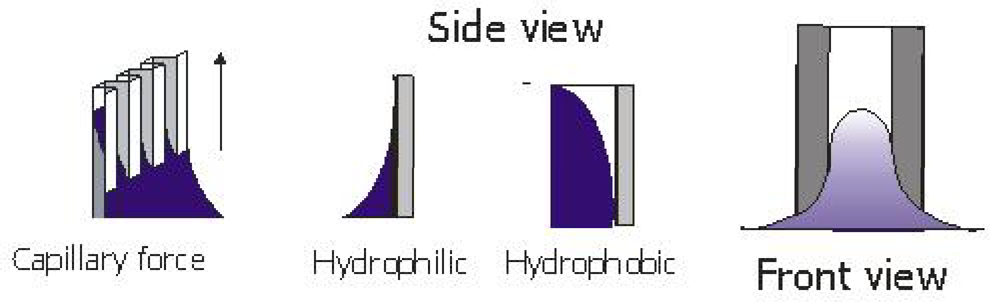
$$p(r) = \frac{\sum_A N_A(r) p_A(r)}{\sum_A N_A(r)} = 1$$

Theory	$0.04 \leq p(r) \leq 1.00$
Experiment	$0.06 \leq p(r) \leq 0.46$

SEM Characterisation

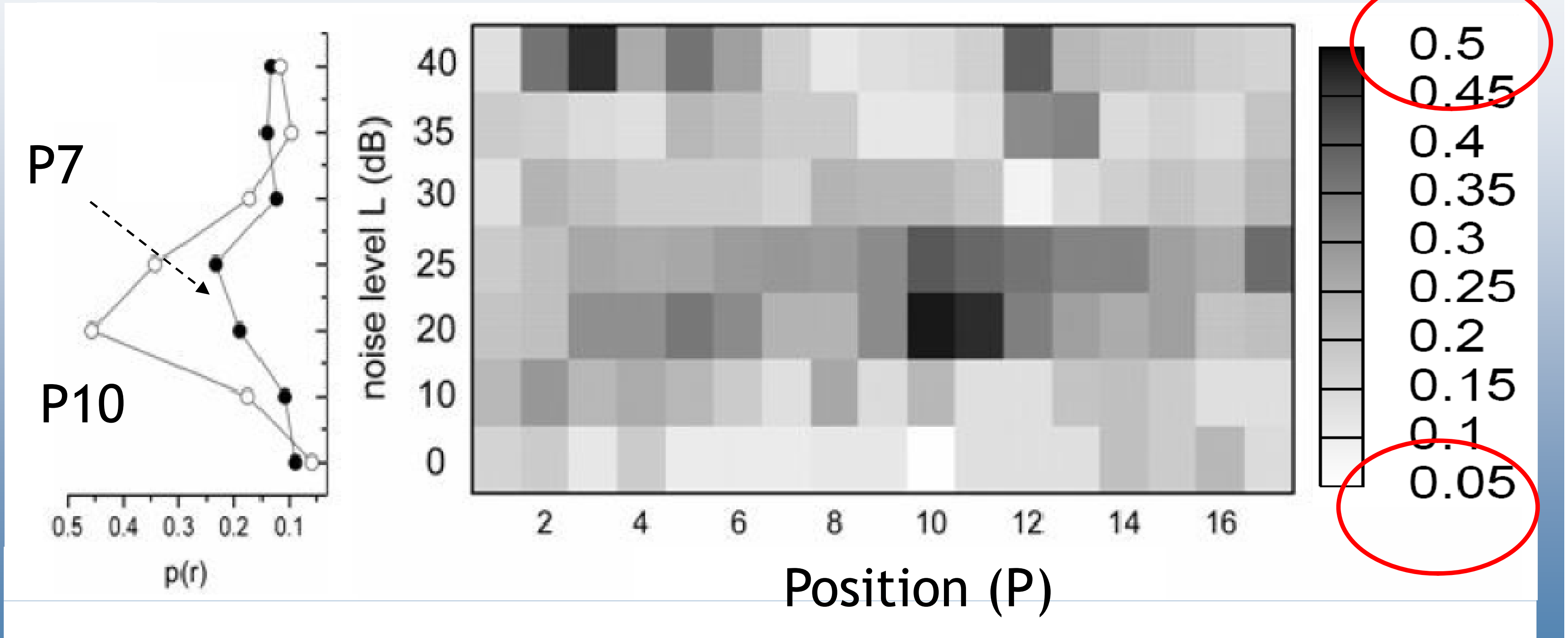
SEM images of size 65 μm x 40 μm (resolution: 3072 x 2304 pixels)





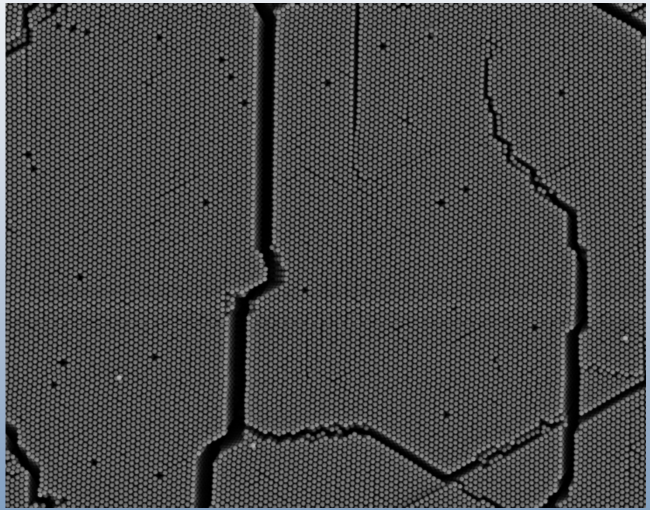
D = 368 nm

r = 5.5 μm ≈ 15D, and ε = 43 nm ≈ 0.12D



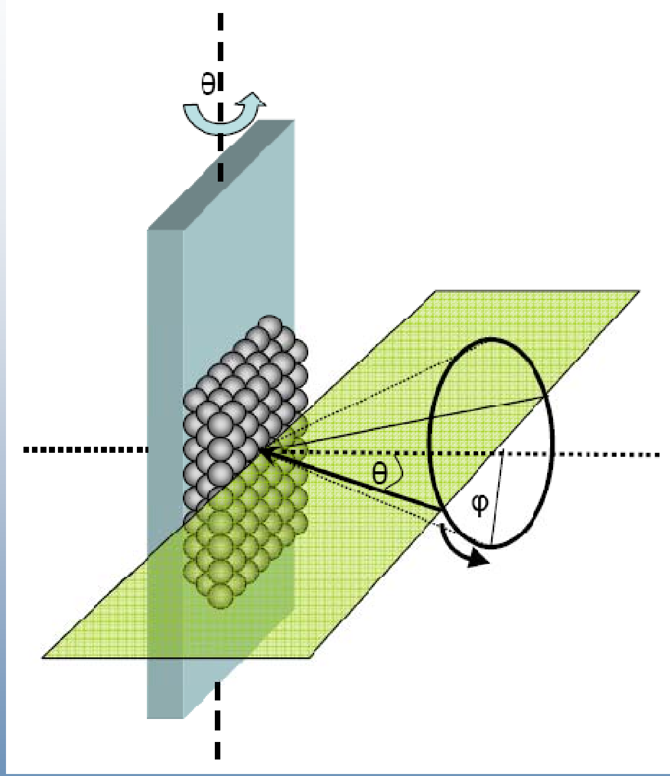
3 D ordering - Experimental approach

2D analysis



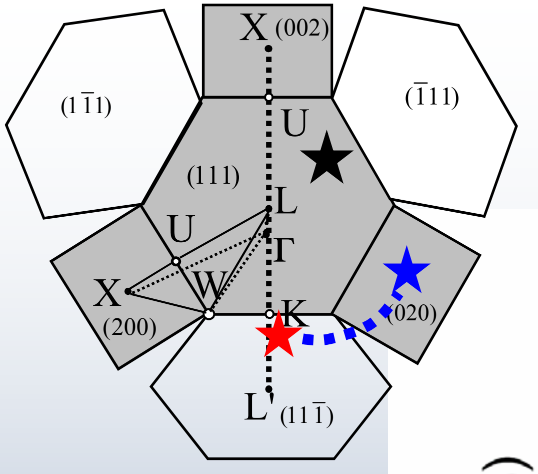
SEM images

3D analysis



θ – incident angle
φ – azimuth angle

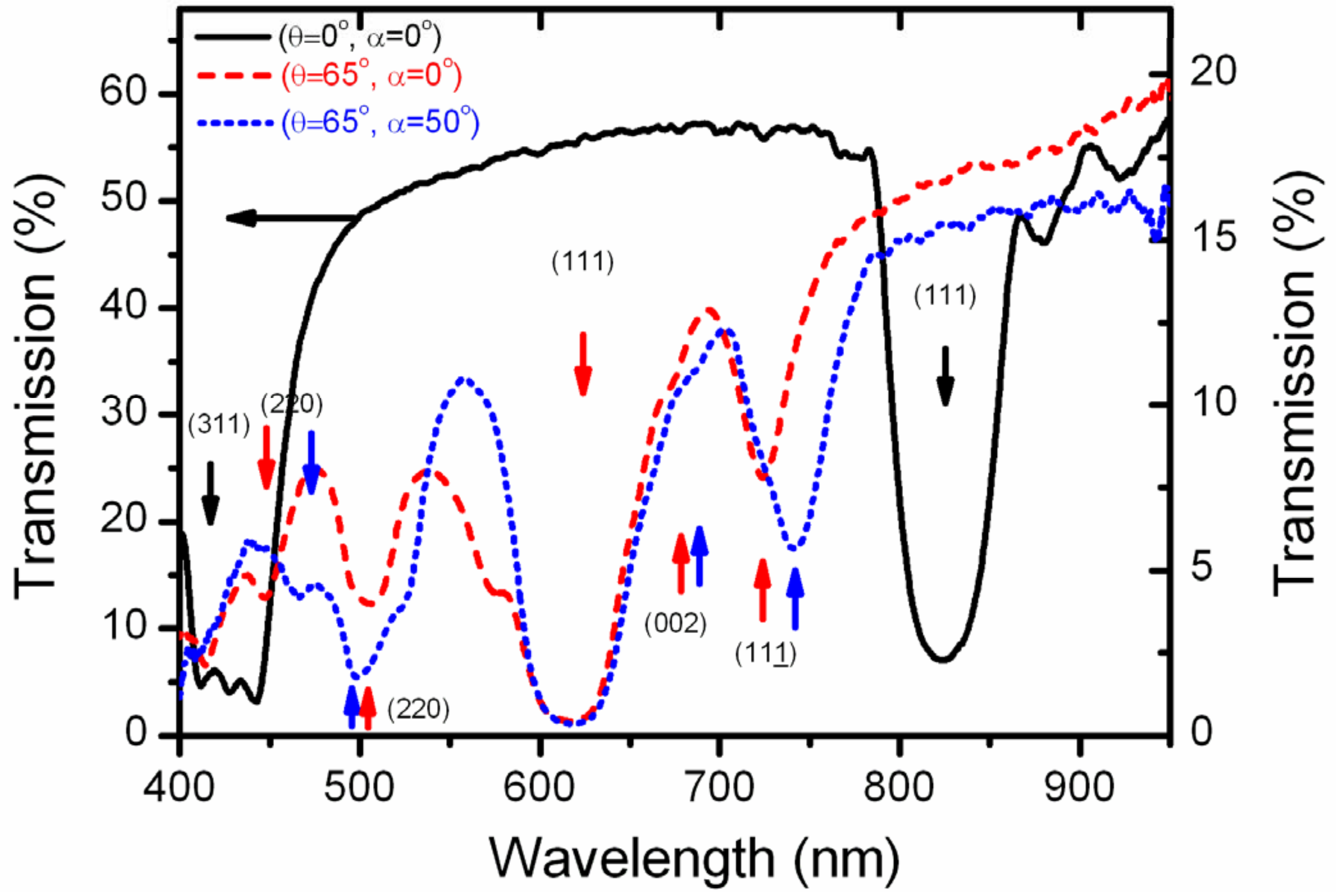
Transmission spectra



W.Khunsin, Adv. Funct. Mater. 18 (2008)

Bragg's Law

$$\lambda = 2 * n_{eff} d_{hkl} \sqrt{(1 - \sin^2(\alpha_{hkl}))}$$

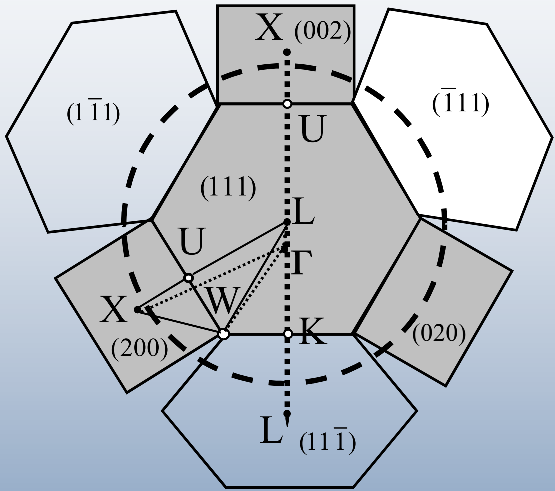


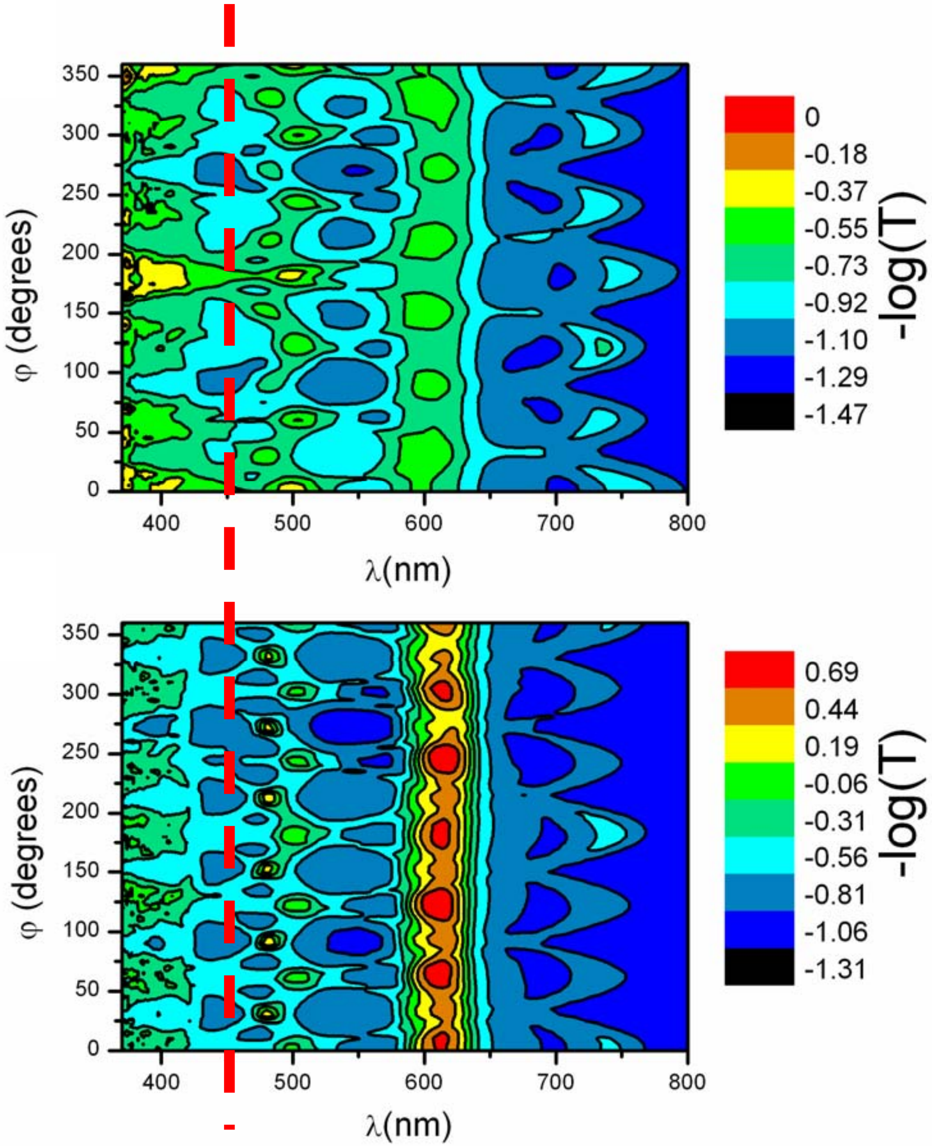
Rotational symmetry of T(φ)

ICN Institut Català de Nanotecnologia

Without Noise

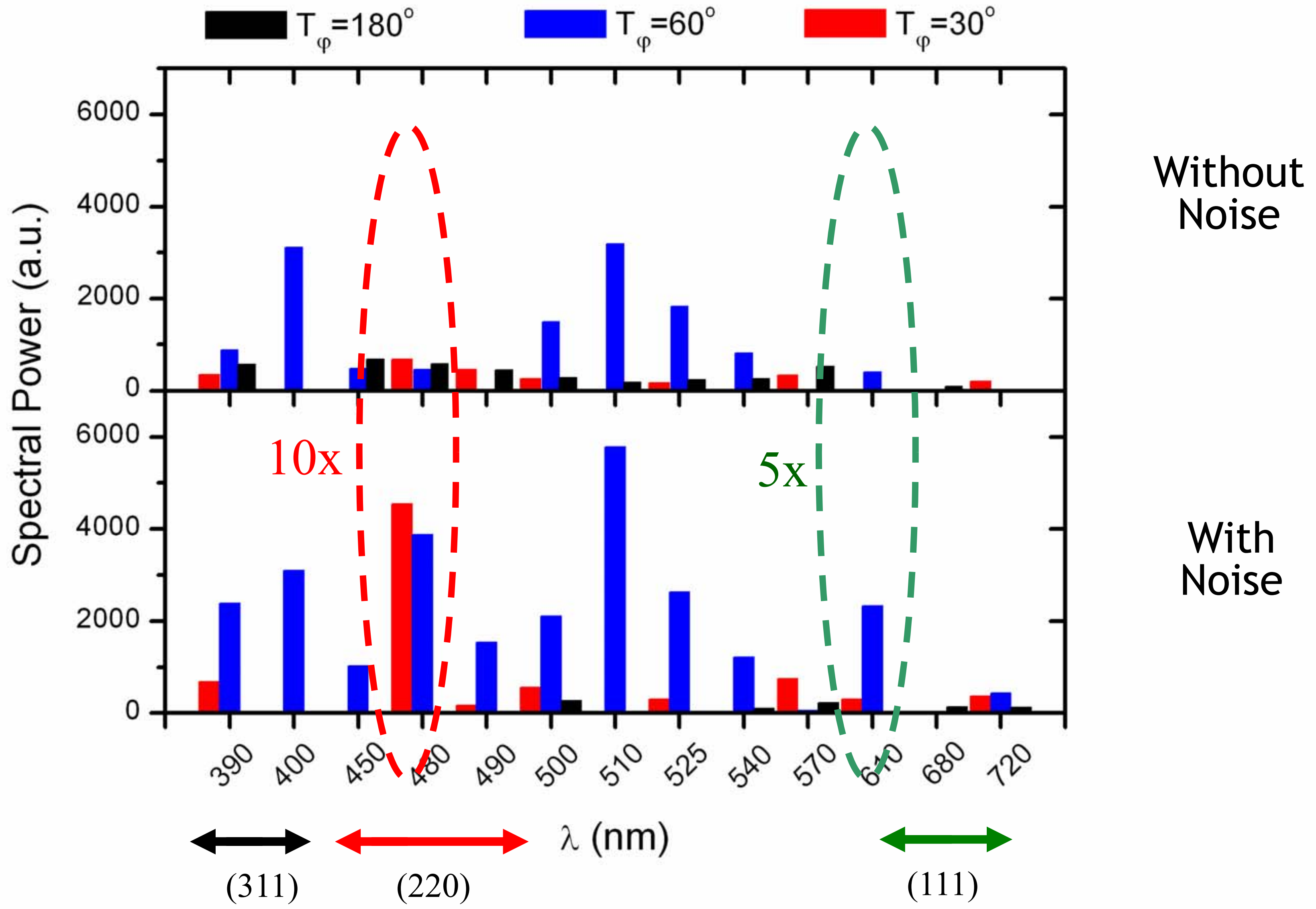
Noise





W Khunsin et al, J. Nonlinear Optical. Physics & Materials 17 97 (2008)

Noise Susceptibility: lattice planes dependency



Conclusions

- New methods presented to characterise nanostructures fabricated by Nanoimprint Lithography (NIL) and self-assembly.
- Sub-wavelength diffraction found sensitive to defects, line-width and profile
  - This is potentially in-line metrology method.
- Photoacoustic metrology demonstrated suitable for dimensional and physical measurement of printed structures
- We propose a robust and generic approach to analyse quantitatively two-dimensional lattice ordering.
  - Opposite partners
  - Rotational diffraction symmetry