Topic 8a - FIB

- Introduction and History
- Experimental Aspects
- Application to Nanolithography - Nanoprinting Program
- Conclusions

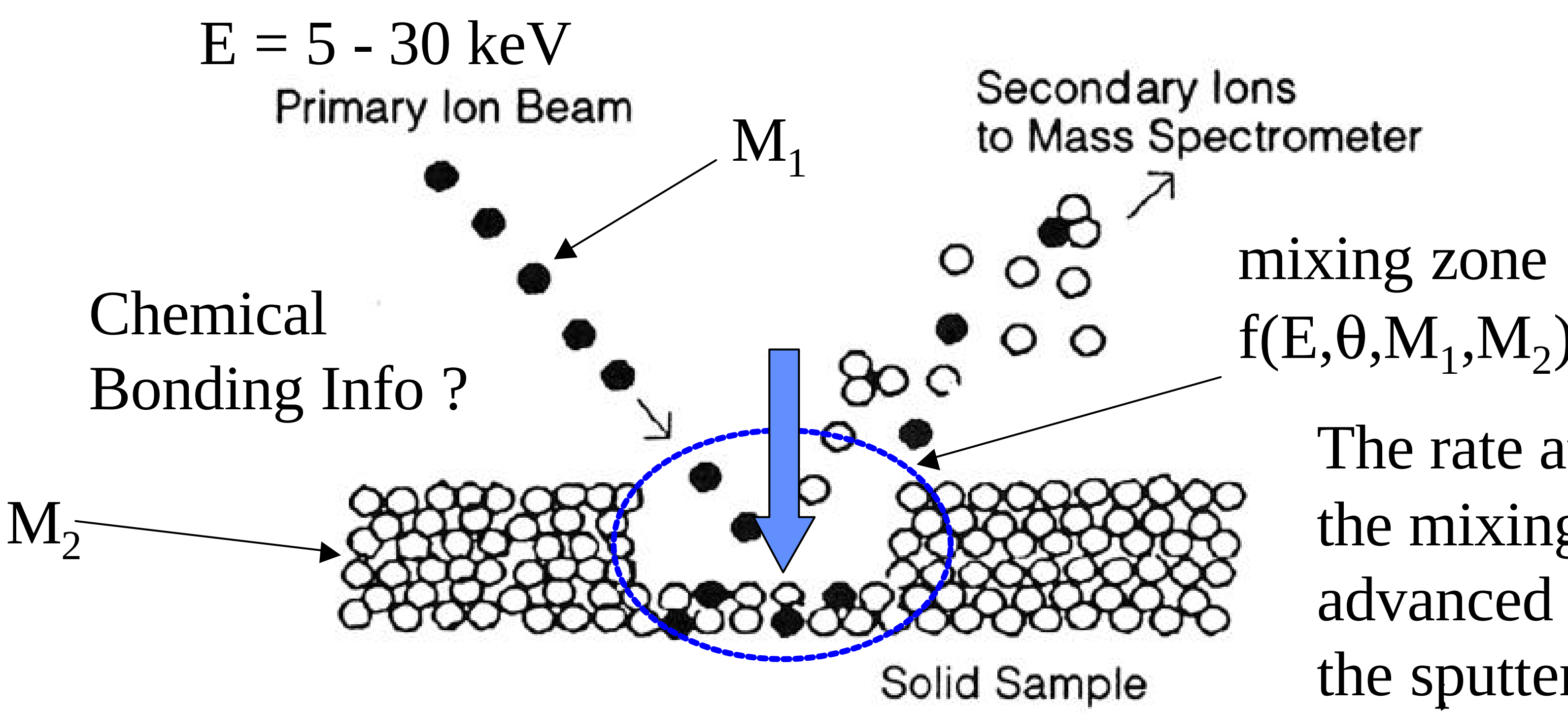
Introduction

***Dynamic* Secondary Ion Mass Spectrometry (Dynamic SIMS)**

• In Secondary Ion Mass Spectrometry (SIMS), a solid specimen, placed in a vacuum, is *continuously* bombarded with a narrow beam of ions, called primary ions, that are sufficiently energetic to cause ejection (sputtering) of atoms and small clusters of atoms from the bombarded region.

• Some of the atoms and atomic clusters are ejected as ions, called secondary ions.

• The secondary ions are subsequently accelerated into a mass spectrometer, where they are separated according to their mass-to-charge ratio and counted. The relative quantities of the measured secondary ions are converted to concentrations, by comparison with standards, to reveal the composition and trace impurity content of the specimen as a function of sputtering time (depth).



The rate at which the mixing zone is advanced is called the sputtering rate.

Introduction

Range of Elements:	**H to U; all isotopes**
Destructive:	**Yes, material removed during sputtering**
Chemical Bonding:	**In rare cases, from molecular clusters, but see**
Quantification Standards:	**Usually needed**
Accuracy:	**2% to factor of 2 for concentrations**
Detection Limits:	**10^{12}-10^{16} atoms/cm^3 (ppb-ppm)**
Depth Probed:	**2 nm-l00 µm (depends on sputter rate and data collection time)**
Depth Profiling:	**Yes, by the sputtering process; resolution 2-30 nm**
Lateral Resolution:	**50 nm-2 µm; 10 nm in special cases**
Imaging/ Mapping:	**Yes**
Sample Requirements:	**Solid conductors and insulators, typically ≤ 2.5 cm in diameter, ≤ 6 mm thick, vacuum compatible**
Main Use:	**Measurement of composition and of trace-level impurities in solid materials as a function of depth, excellent detection limits, good depth resolution**
Instrument Cost:	**$500,000-$1,500,000**
Size:	**10 ft. x 15 ft.**

Introduction

Modes of Analysis

- ❑ Depth profiling mode, by far the most common, is used to measure the concentrations of specific ***pre-selected elements*** as a function of depth (z) from the surface.

- ❑ The bulk analysis mode is used to achieve ***maximum sensitivity*** to trace level components while sacrificing resolution.

- ❑ The mass scan mode is used to survey the entire mass spectrum within the specimen.

- ❑ The imaging mode is used to determine the lateral distribution (X,Y) of specific pre-selected elements, in some cases combined with depth profiling for "tomographic" studies. See posters Allen Kubis et al. on the first floor for full detail.

Experimental Aspects I

- **The focused ion beam (FIB) employs rastering of a Ga+ ion beam for imaging with either secondary electrons or secondary ions.**

- **For milling, high energy (30 keV) Ga+ ions are focused into spots as small as 10 nm to form pixel-by-pixel images.**

- **As the image is created, atoms from the surface are sputtered by the incident Ga+ ions, meaning that we can acquire images from different depths ("slices") within the sample.**

- **These slice images may then be combined in the computer using appropriate interpolation algorithms, to enable a three dimensional reconstruction of the sample to be produced.**

Experimental Aspects II

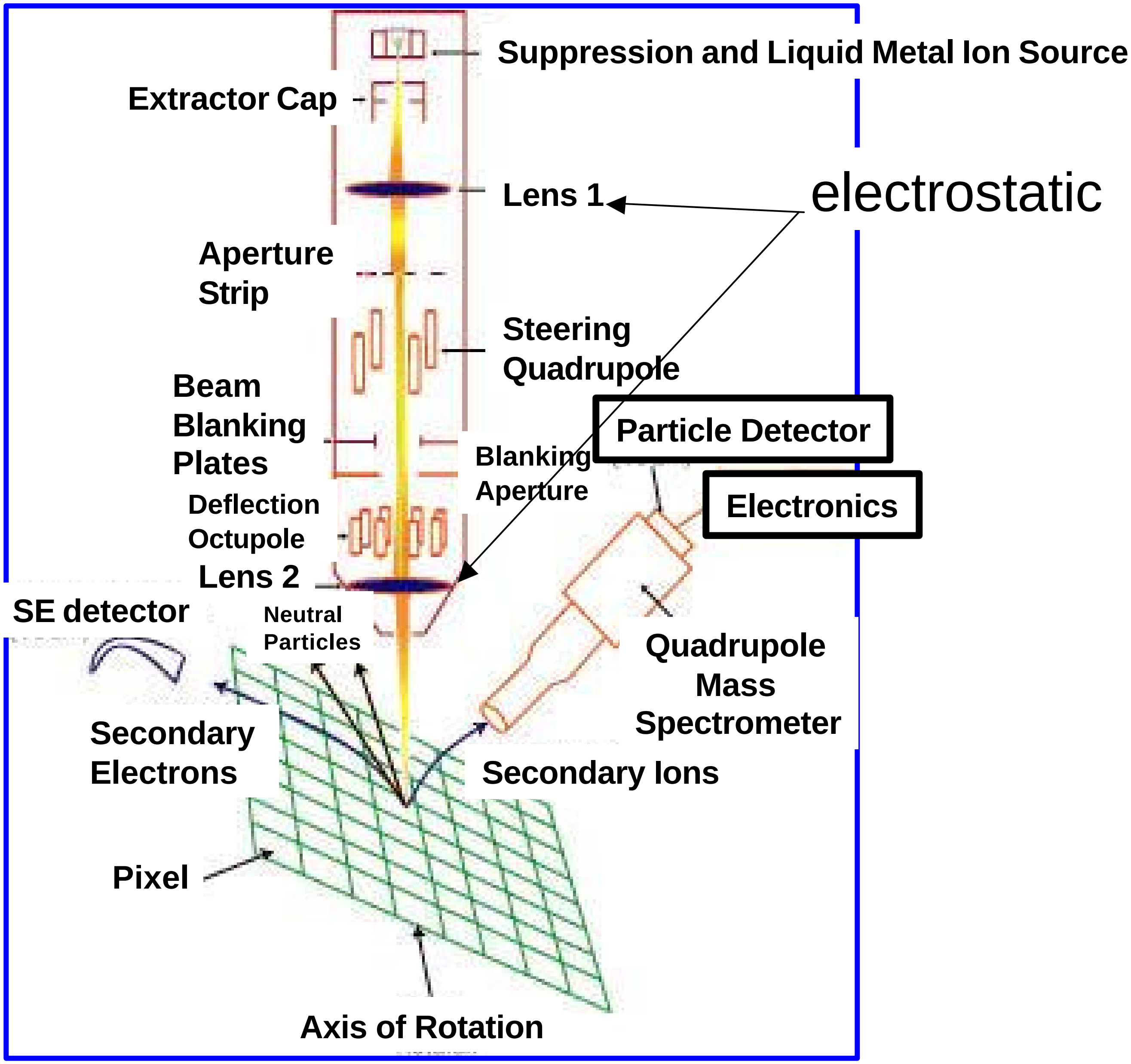
- **Surface chemical information at sub-micrometer (~250 nm) length scale.**

- **Mass resolved *images* of secondary ion species from H+ to fragments organic complexes of high molecular mass are collected virtually simultaneously during image acquisition.**

- **A pulsed Ga+ analysis beam sputters only a few monolayers of material during analysis, thus the technique consumes only a small amount of the specimen.**

- **Additional ion sources (Cs+, O_{2+}, and Ar+) are available for sputtering beneath the surface for both 1 dimensional depth profiling and 3-D ion mapping.**

The Ga^+ Finely Focused Ion Beam (FIB)

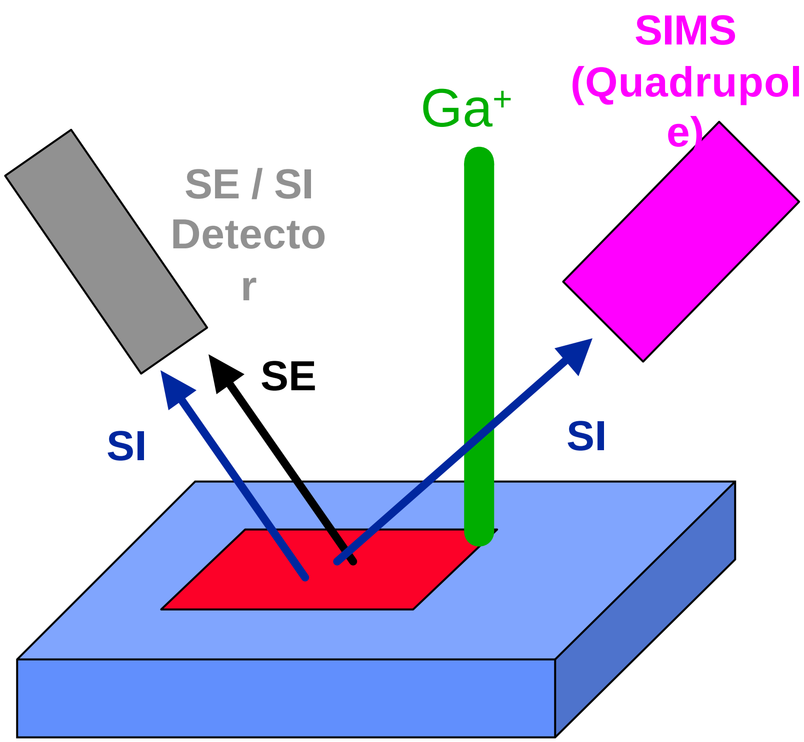


The Ga^+ Finely Focused Ion Beam (FIB)

Figures of Merit: *FEI FIB 200*

- *Minimum spot size < 30 nm*
- *Ion current density > 10 A/cm^{-2}*
- *Ion currents 1 pA - 10 nA*
- *Ion energies 3 keV - 30 keV*
- *Sample translation 2”x2”x1/2”*
- *Pt, SiO_2 deposition sources (organic platinum, Si)*
- *Charge Neutralization (flood gun)*
- *Secondary Electron / Ion Imaging*
- *SIMS Spectrometer*
- *Cooling / Heating (77 – 700 K)*
- *Iodine enhanced etch*
- *Depth of focus ~ 200 mm*

Imaging and Spectroscopy in the FIB



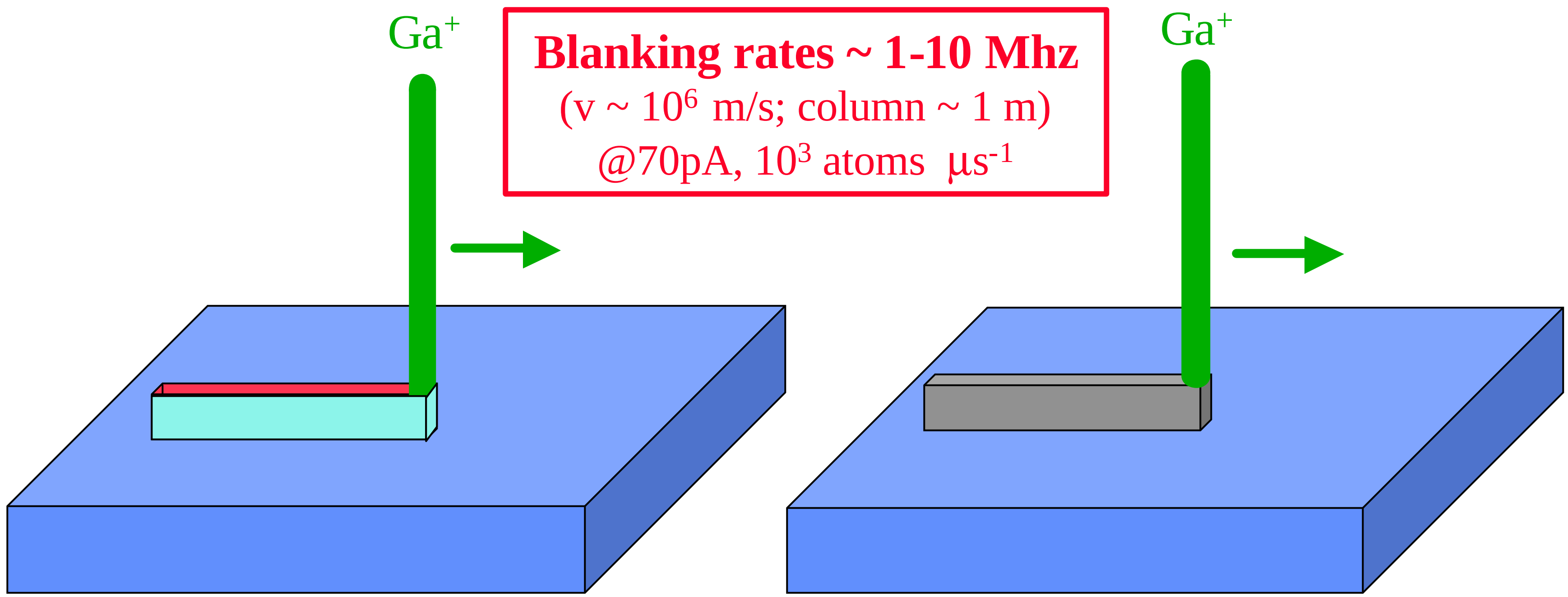
- Pixel by Pixel (1000 x 1000)
- Resolution 20 - 500 nm (1 pA - 10 nA) milling
- Secondary electron yields ~ 1-10 / incident ion
- Secondary ion yields ~ 10^{-5} - 10^{-2} / incident ion
- SE Imaging
- SI Spectroscopy

Imaging and Spectroscopy in the FIB

- For Ion Imaging: A double focusing, electrostatic mass spectrometer achieves mass separation using an electrostatic analyzer and magnet. Secondary ions of different mass are physically separated in the magnetic field, with light elements making a tight arc through the magnet and heavy elements making a broad arc. Some of these spectrometers are capable of stigmatic imaging (ion microscopy), which is used to acquire mass-resolved ion images with a resolution as good as 1 μm.

- For Mass Analysis: Quadrupole-based SIMS Instruments: Mass separation is achieved by passing the secondary ions down a path surrounded by four rods excited with various AC/DC voltages. Different sets of AC/DC voltages are used o direct the flight path of the selected secondary ions into the detector. Primary advantage of this type of spectrometer is the fact that it can rapidly switch from peak to peak and to analyze insulating materials. Quantitative analysis.

Nanoscale Lithographic Patterning



Subtractive Lithography: Sputtering

Sputtering Yields ~ 1 -10 / incident ion: f (material, q, E...)

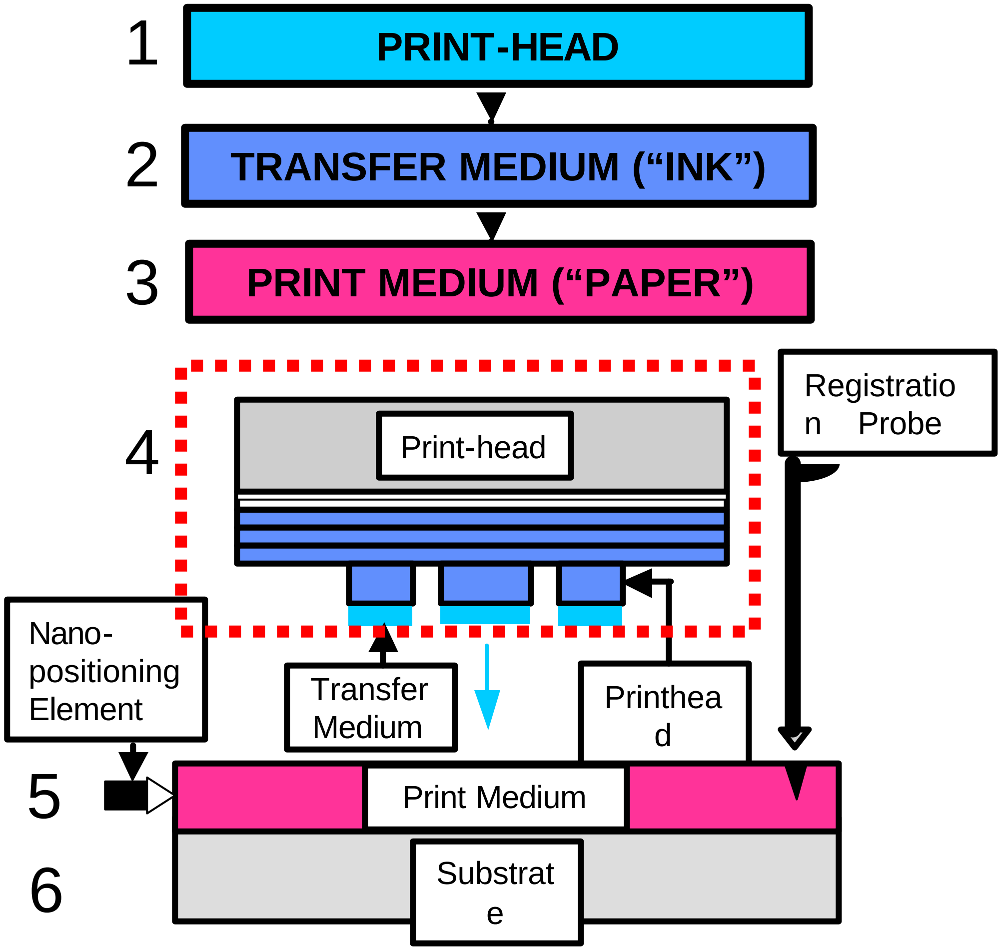
Si ~ 0.5 mm^{3}nA^{-1}s^{-1} *(E = 30 kV, q ~ 0^o)*

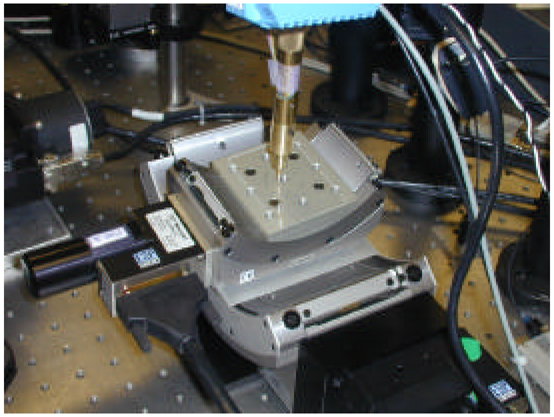
Additive Lithography: Deposition

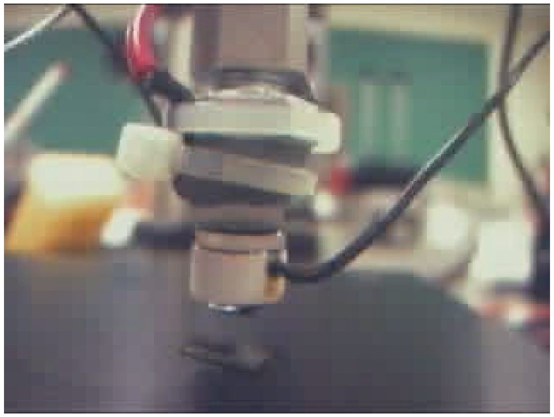
Deposition Yields ~ 1 - 10 / incident ion: f (material, q, E...)

Pt ~ 2 mm^{3}nA^{-1}s^{-1} *(E = 30 kV)*

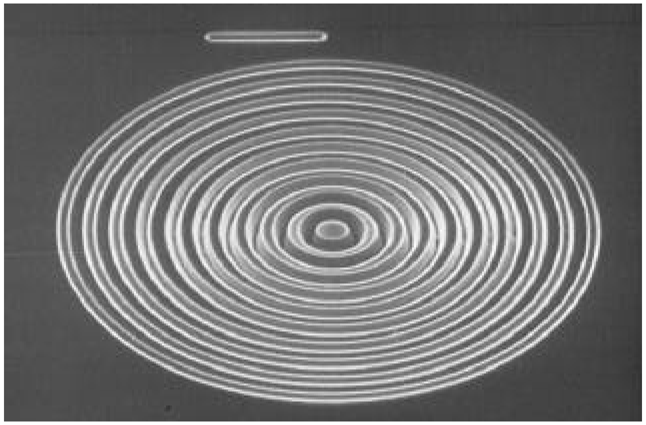
University of Virginia "Nanoprinting Project"





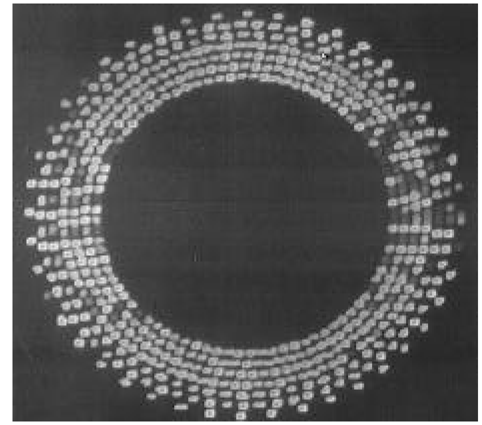


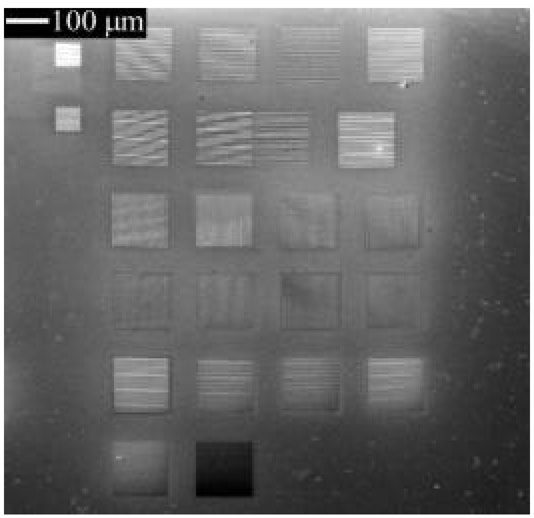
Example: Print-Head Masters by FIB Processing

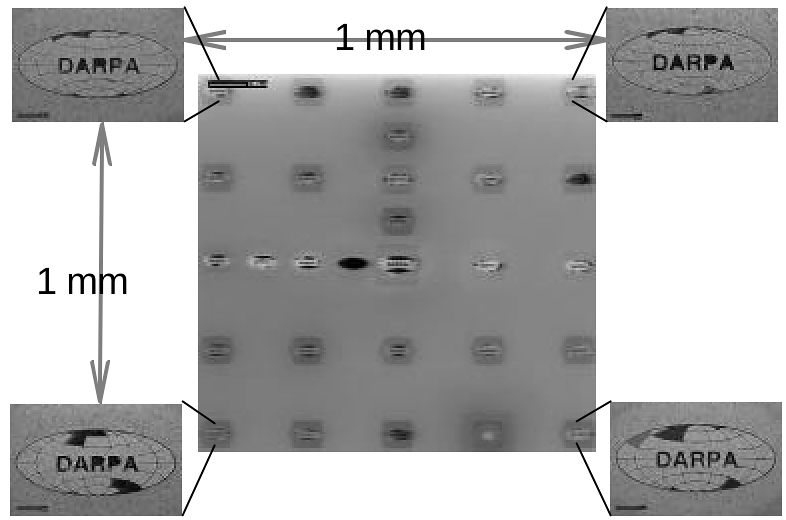


200 nm Lines

50 nm Dots



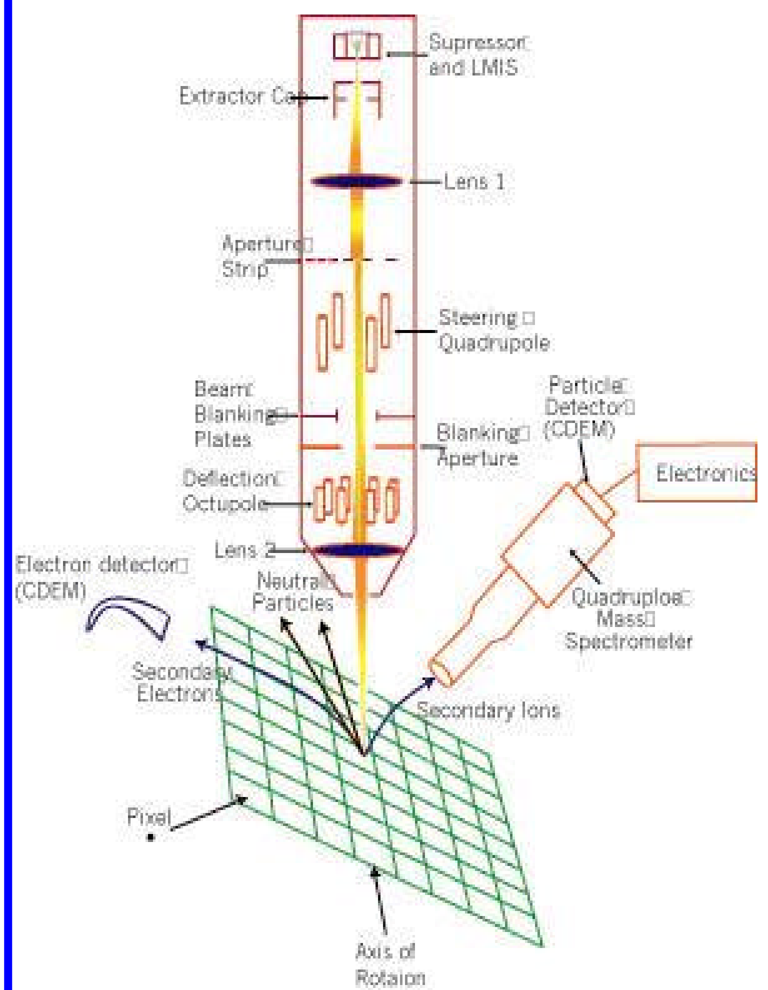




1 mm² Planar (100 nm features)

1 mm² Curved (5 cm ROC)

FIB System Issues and Performance



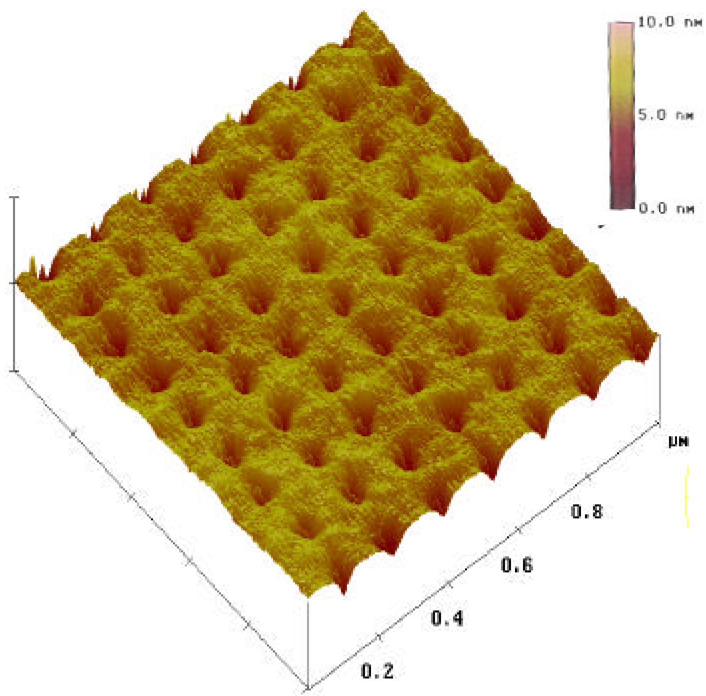
•Why FIB for Mastering?

Rapid prototyping – no mask nor resist

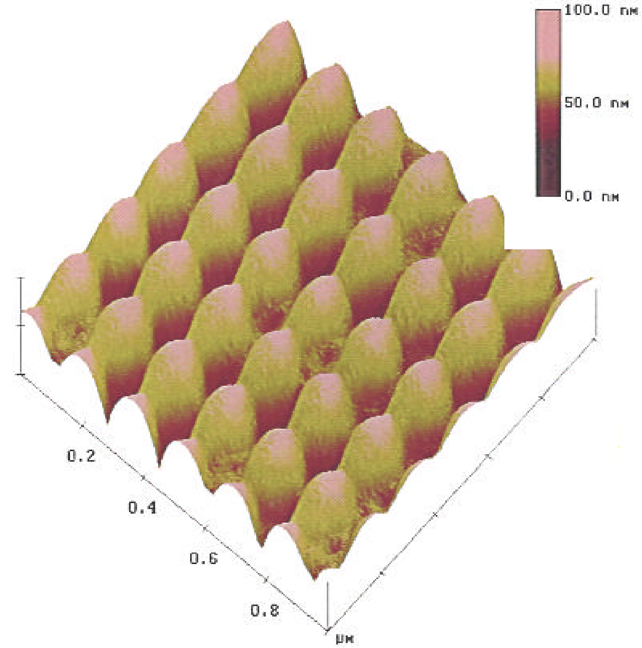
- *Depth of focus*
- *On-the-fly repair*
- *Visual alignment / inspection*

PARAMETER	*MEASUREMENT*
RESOLUTION	< 10 nm beam < 50 nm feature
THROUGHPUT	~ 0.2 – 2.0 $\mu m^3 nA^{-1} s^{-1}$ Deposit or Sputter
FIELD OF VIEW	1 mm^2
STAGE STABILITY	1-2 nm / minute attainable
BEAM REGISTRATION	± 10 nm, multiple scans
DEPTH OF FOCUS	> 100 µm

New Technique for Scaling:
Ultra-Rapid FIB Topographical Writing into PMMA



1 pA, 20 ms / feature



11 pA, 500 ms / feature

Equivalent Sputter Yield 10^3 - 10^4

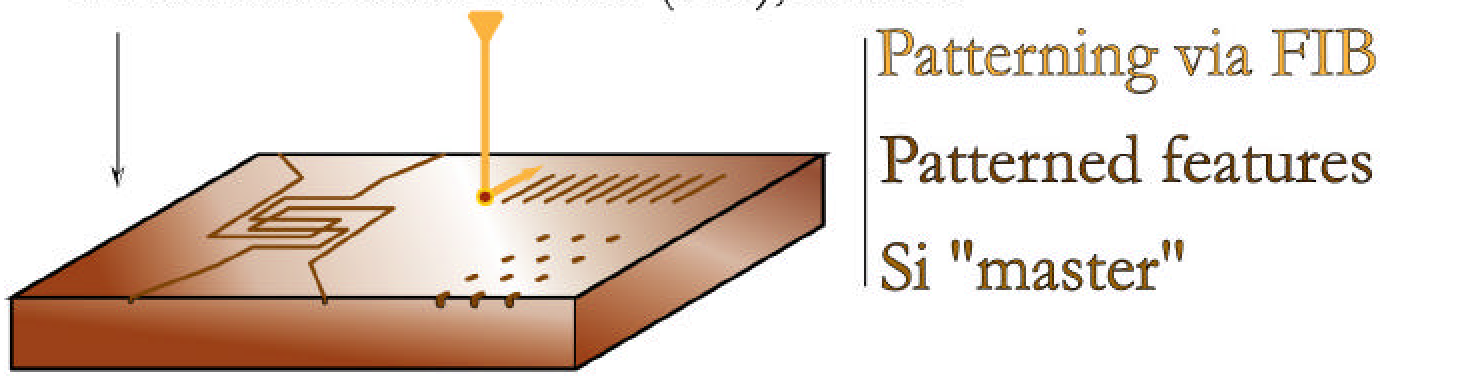
Writing Rate > 10^4 Features / sec : Extend to 1 cm^2 Printheads

vs. a master made out of Si

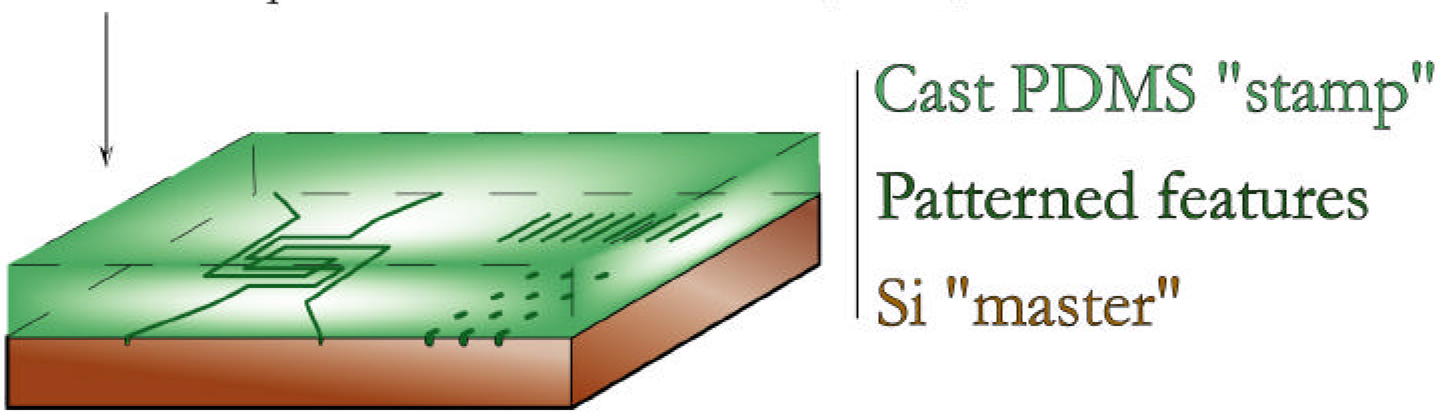
University of Virginia, Dept. of Materials Science and Engineering

Pattern Transfer Mechanisms
Microcontact Printing I

1. Fabricate Master Pattern (FIB), Silanize

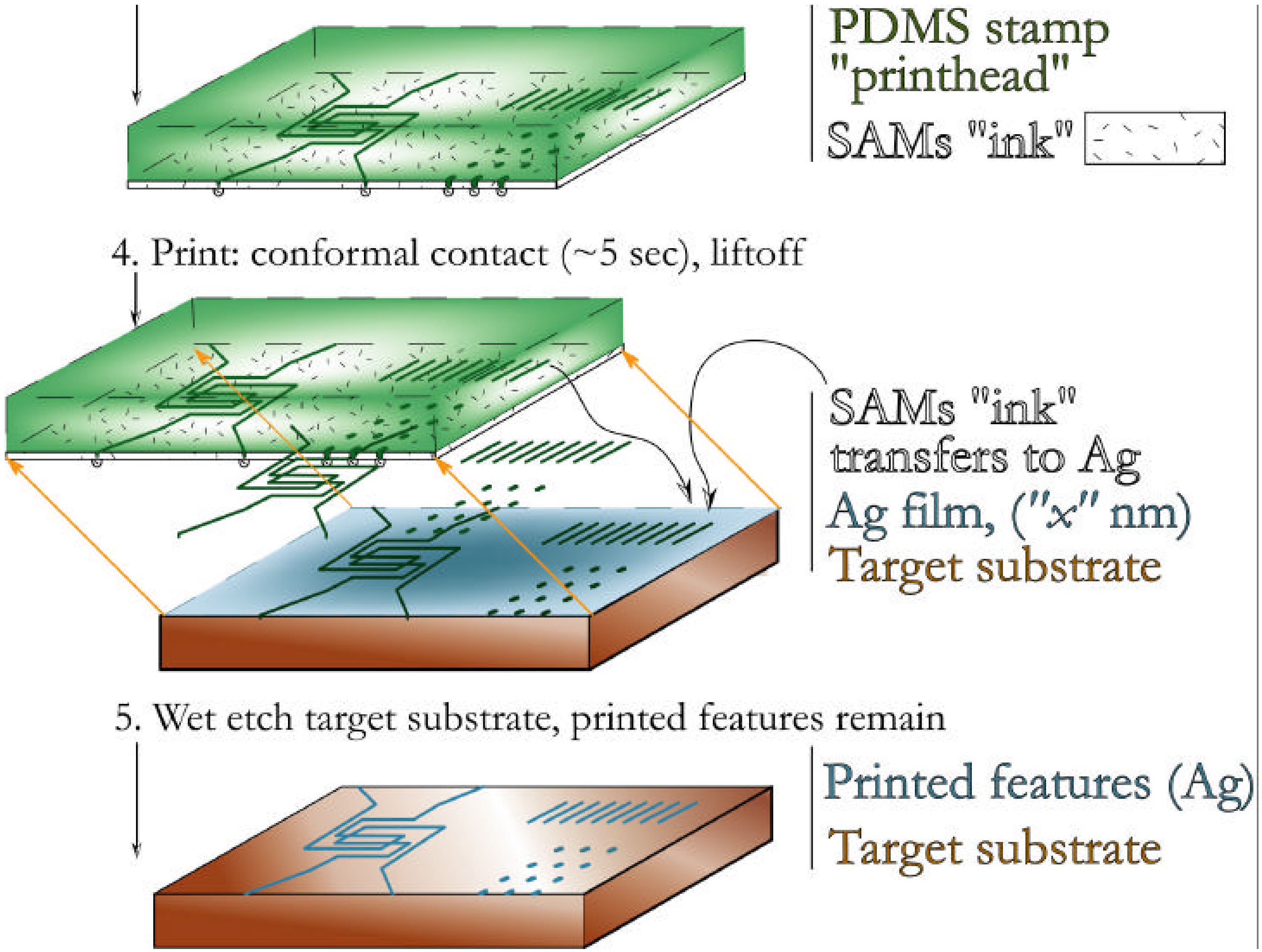


2. Pour liquid PDMS over Si Master, Cure, Release



Pattern Transfer Mechanisms

Microcontact Printing II

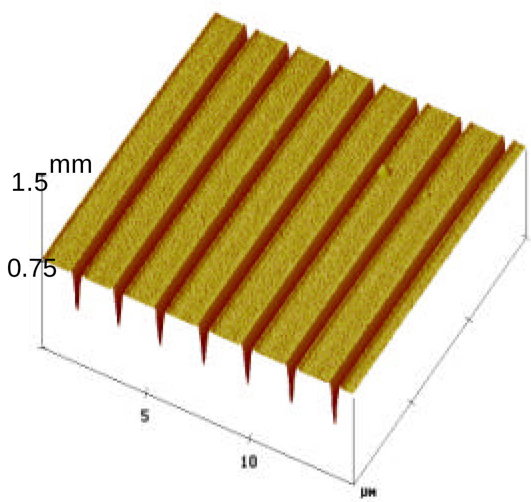


Microcontact Printing Using PDMS Elastomer

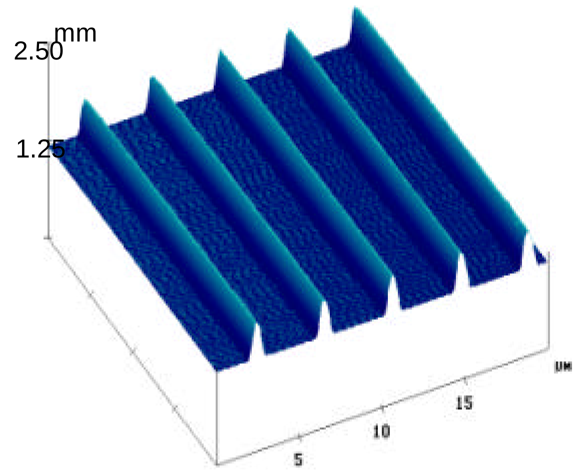
AFM Images (Tapping Mode)

Etched

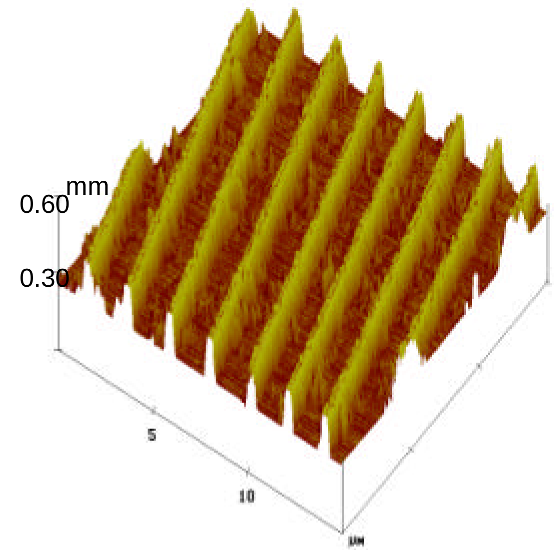
FIB Master → ***PDMS Mold*** → ***mCP Surface***



60 nm lines



150 nm lines

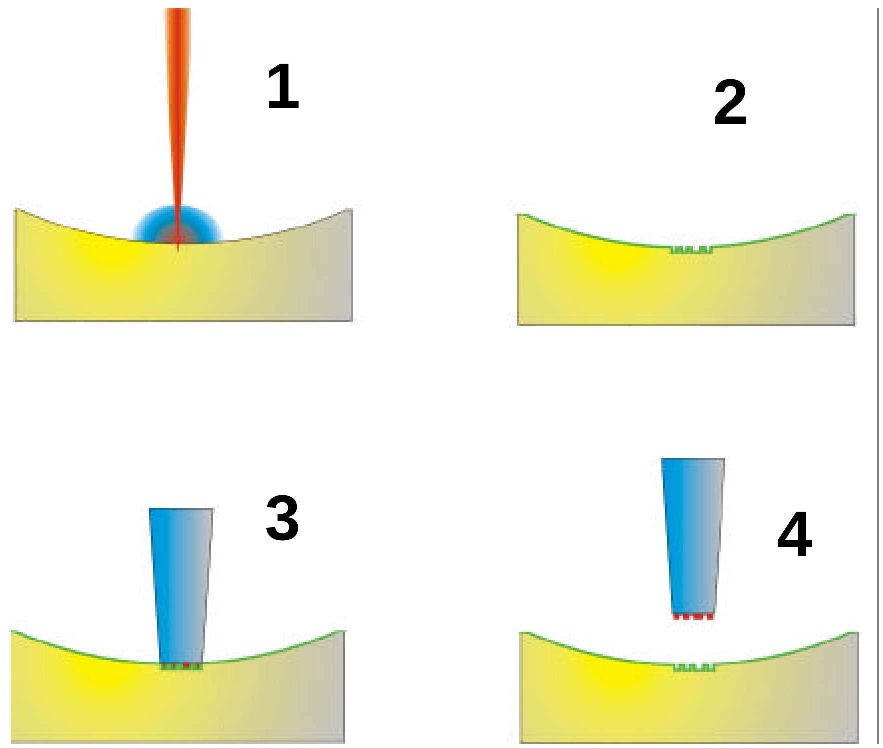


170 nm lines

PDMS is Relatively Low Modulus: Conformable but Low Rigidity Our Attainable Resolution Limit Using PDMS ~ 150 nm

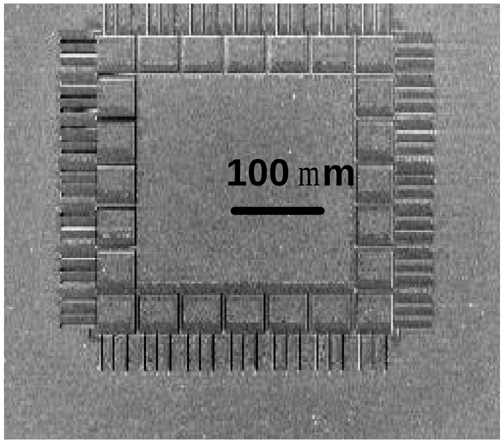
All Polymer Imprinting Process

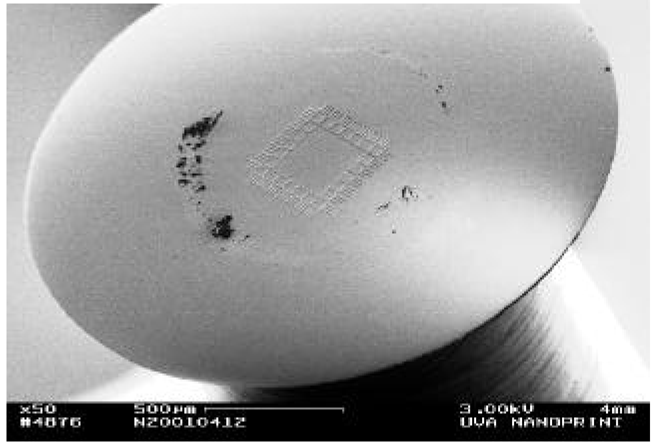
- 1,2. FIB mill a master pattern onto Au-coated lens with appropriate curvature
- 3. Cast mold (epoxy, poly-urethane, SU-8), from master. 4 minutes working time
- 4. Release cast mold from master, Au adheres to mold surface
- 5. SAMs (self assembled monolayers) Inking or Imprinting into Softer Polymers
- Also adaptable to more complex curvature surfaces!



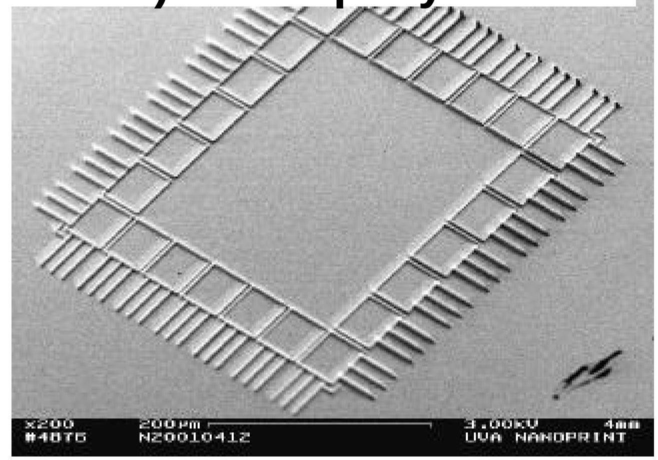
Curved Surfaces

1,2 Curved substrate with a 12.4 mm ROC

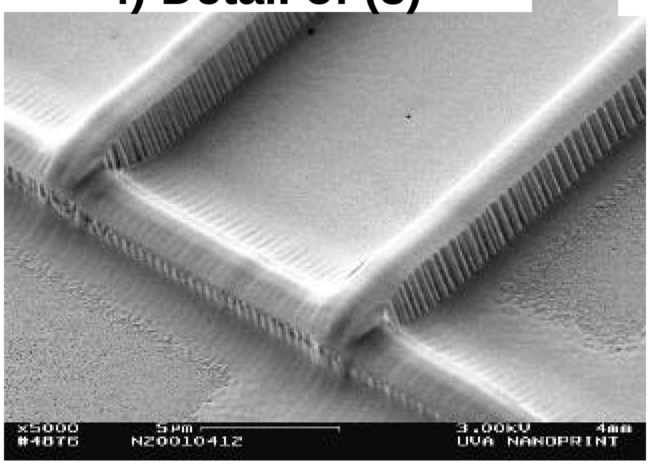




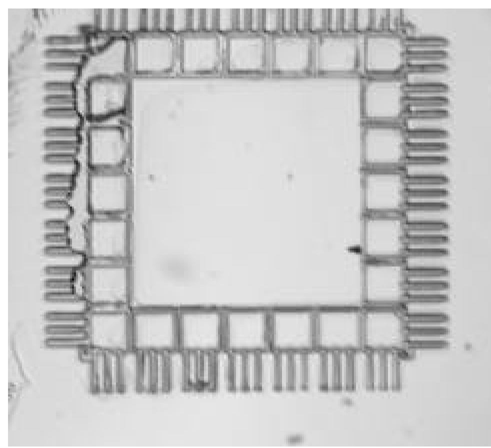
3) Cast Epoxy Mold

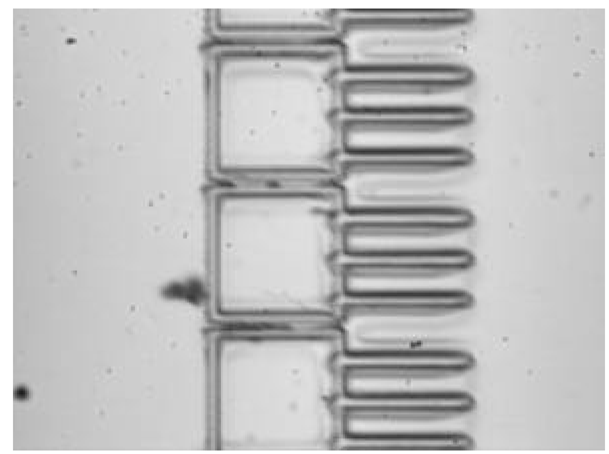


4) Detail of (3)

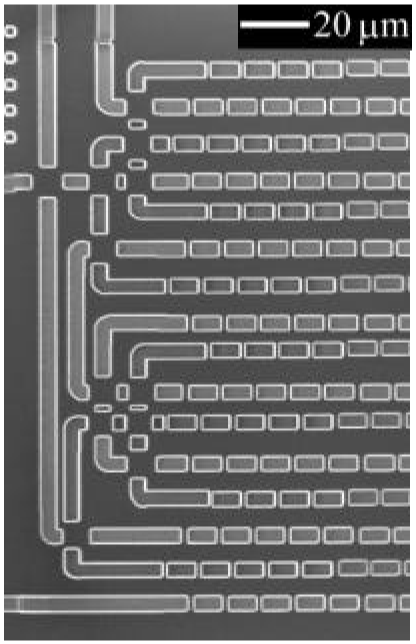


4,5 2 mm Clariant AZ 4210 resist, 11.2 MPa, 5 min, RT

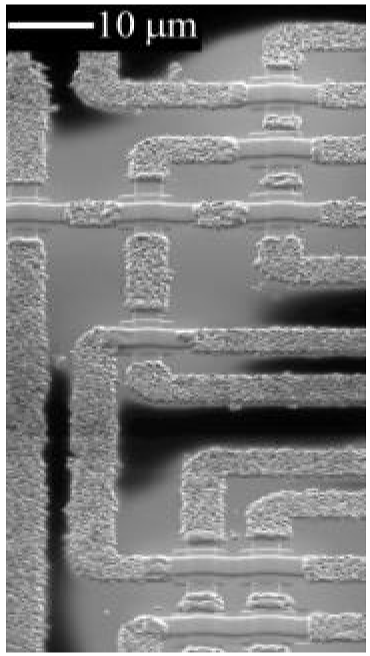




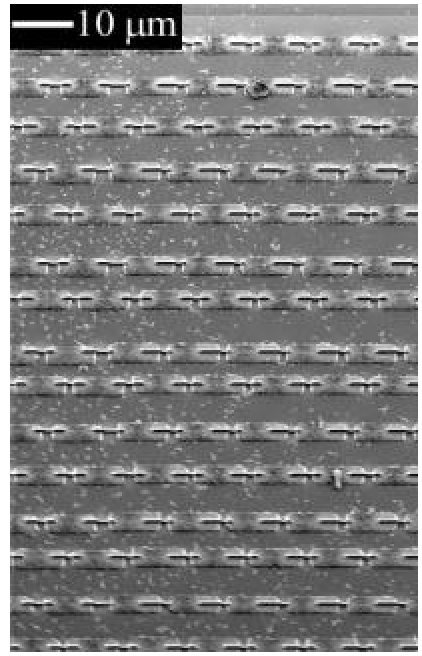
100 Element x Four-Level



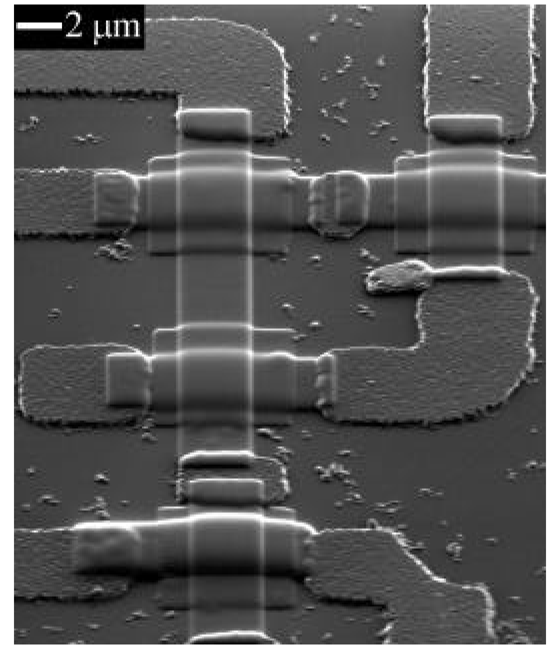
1. FIB Print-Head



2. mCP Pattern in Ag

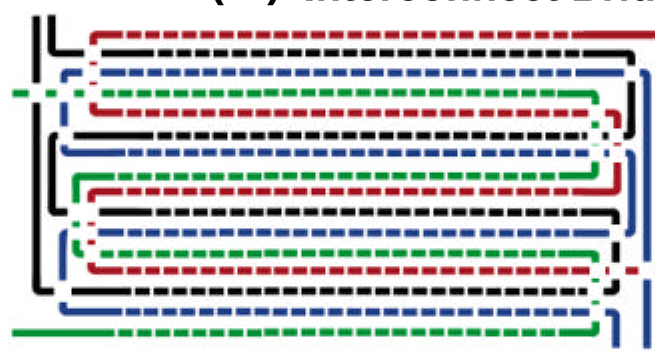


3. FIB Deposited Heating Elements (Pt)



4. FIB Deposited Pt/ SiO_2 Interconnect Bridges

- Combined FIB / uCP Fabrication
- 4 levels, 100 elements each
- 24 bridges total
- 2 mm x 2 mm printhead



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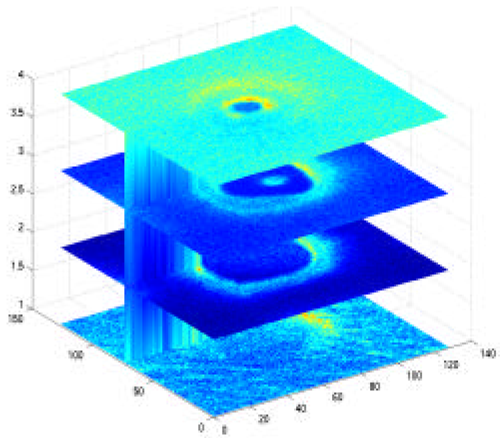
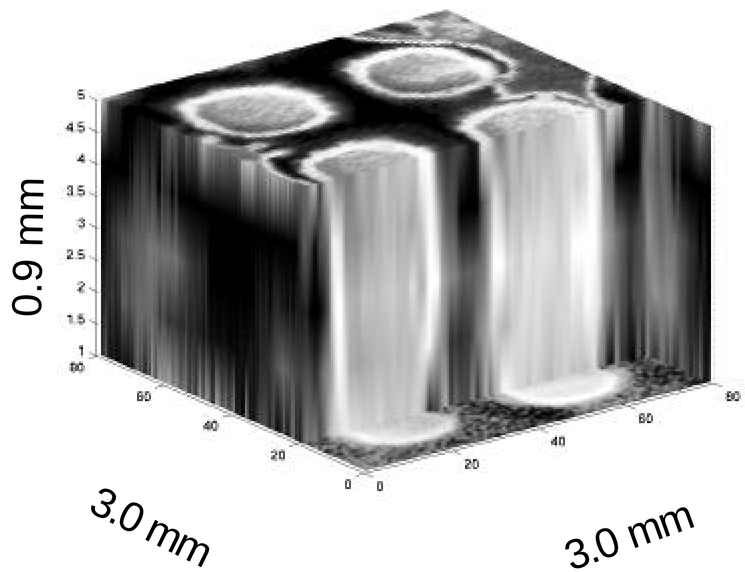
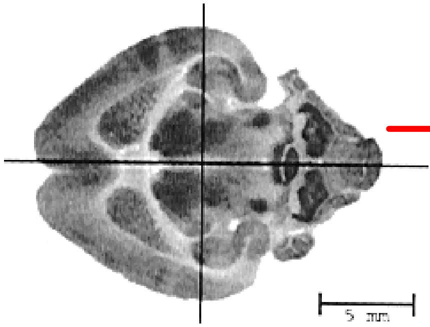
Towards Nanoscale Reconstruction of 3D Structures, Chemistry and Crystallography

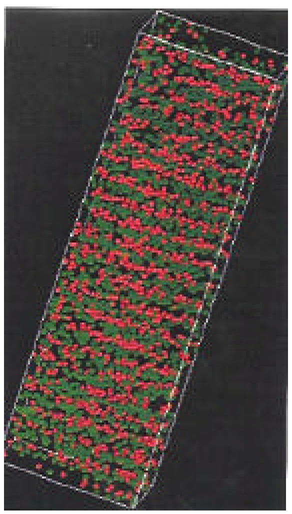


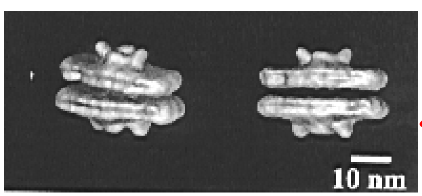


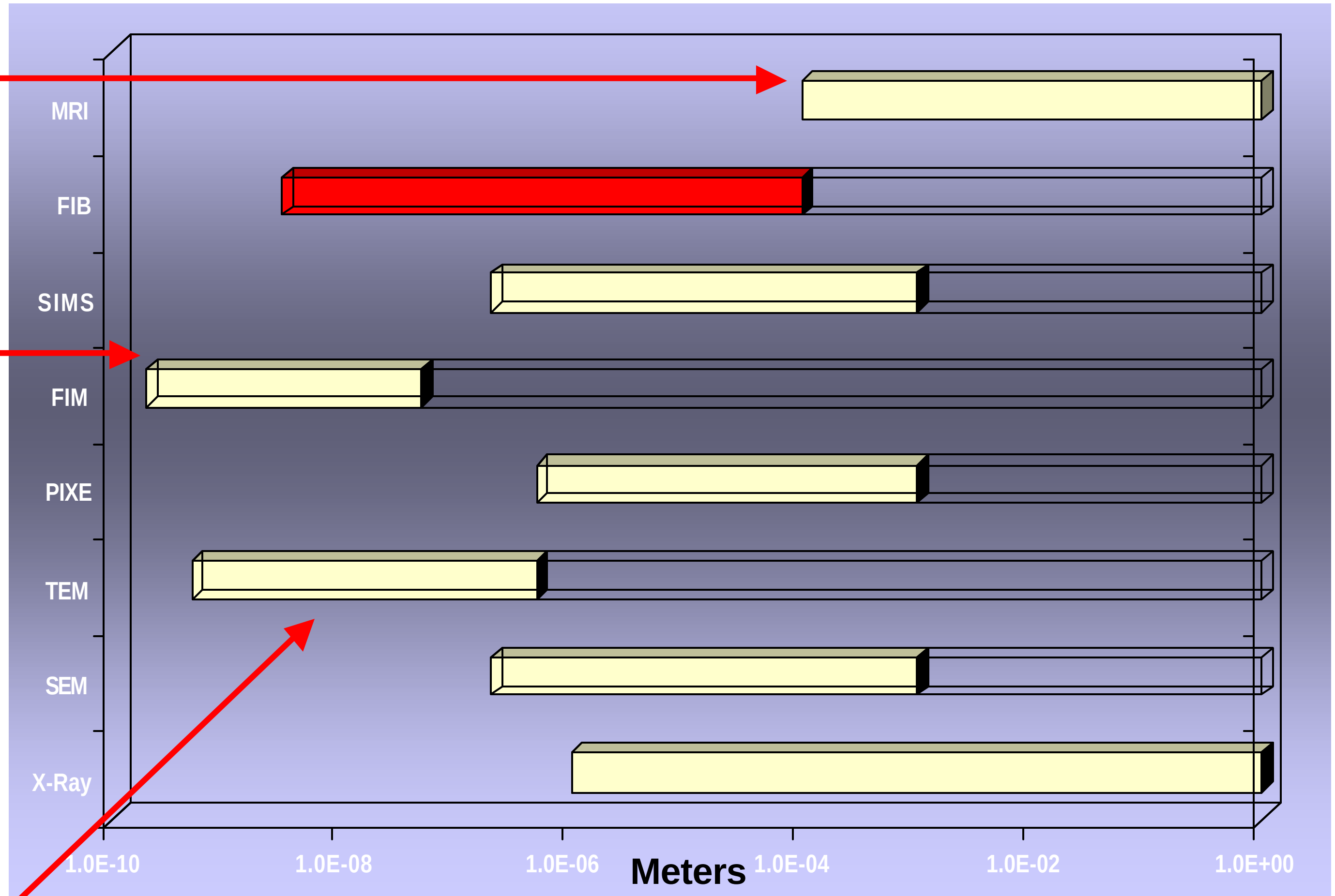
- **Secondary Electrons and Ions**
- **Lateral, Vertical Resolution 20-30 nm**
- **Field of View up to (tens of mm)^3**
- **Reconstructions Contain ~ 10^7 Data Points**

Comparison of Length Scales in *Tomographic* Techniques









FIB – the only technique to span tens of nm to tens of μm

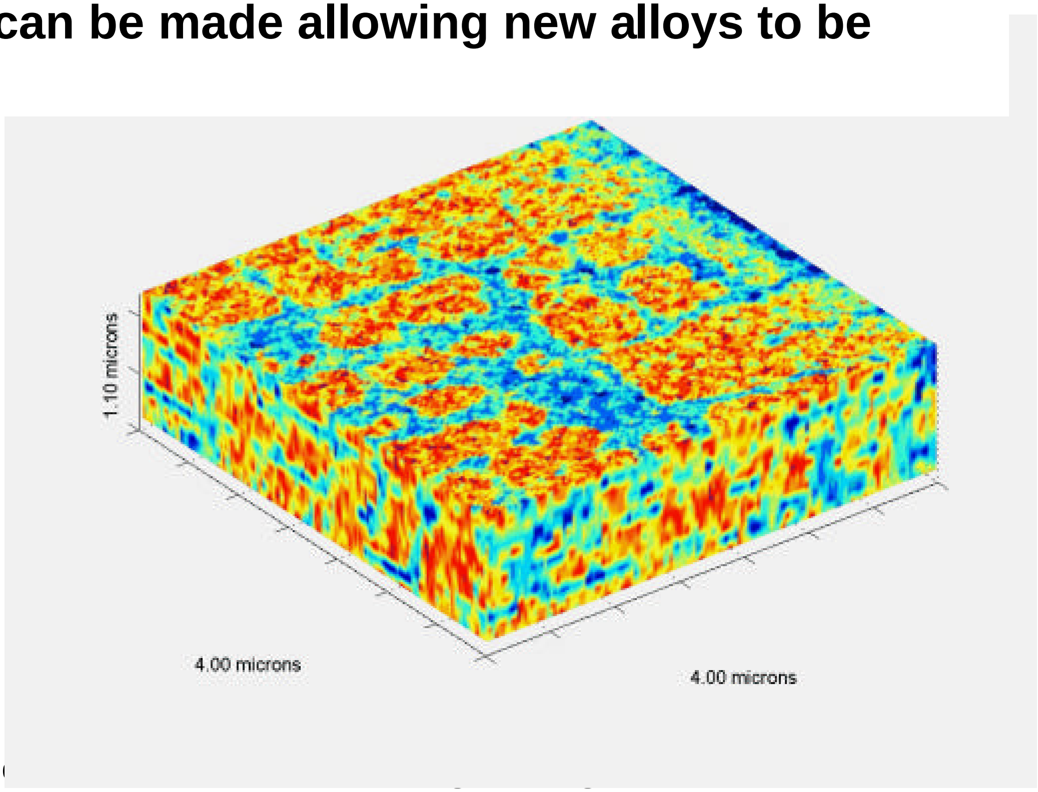
Tomographic Techniques

The accompanying image is a reconstruction of the Al distribution in a Nickel-Based Super Alloy.

In this example the FIB was combined with Secondary Ion Mass Spectrometry (SIMS) allowing elemental mapping of the Al in the alloy.

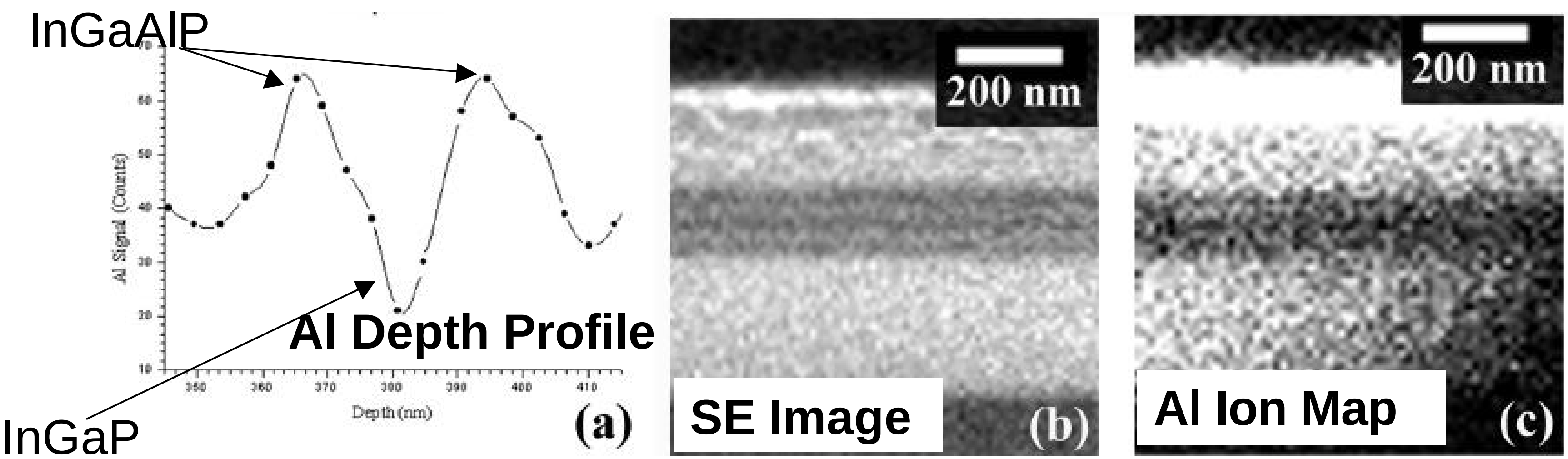
Aluminum is observed preferentially in the cellular precipitates in the structure.

By comparing the structures obtained in the reconstructions to the physical properties of the alloy, correlations can be made allowing new alloys to be formulated for specific applications.



Lateral and Depth Resolution Tests

Test structure: Two 22 nm InGaAlP layers separated by $In_{0.51}Ga_{0.49}$ P



Depth resolution (~ 10 nm) Defined by:

- Secondary electron / ion escape depth (< 1nm)
- Slice thickness required for sufficient signal
- Implant mixing.

Lateral Resolution (~ 20 nm) Defined by Convolution of:

- Lateral Ion Range (~ 7 nm Ga^{+}: Ti, Al).
- Ion Beam Diameter (~ 10 nm)

Conclusions I

- **FIB / Microcontact Printing Techniques Offer:**
  - **Maskless, “Resistless”, Rapid Prototyping**
  - **Direct visual alignment**
  - **High Resolution (< 100 nm)**
  - **Large Range of features sizes (100 nm – 10 μm)**
  - **Large fields of view (1 cm^2, 10^8 features /s attainable)**
  - **Adaptability to wide range of materials**
  - **Adaptability to wide range of surface geometries (high DOF of FIB)**

Conclusions II

- **FIB Tomography Allows 3D Reconstruction of Structure, Chemistry and Crystallography**
  - **20-30 nm Spatial Resolution**
  - **Fields of View up to (Tens of μm)^3**
- **Major Experimental Opportunity: Improve Detected Secondary Ion Yields**
  - **Currently 10^{-7} ± OM**
  - **Existing Improvements (Incidence Angle, Iodine Etch) + 1 OM**