FIB artifacts and how to overcome them

Tutorial
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Outline

1. Basics
2. Curtaining
3. Rippling
4. Crystal damage
5. Implantation
6. Mixing and demixing

Acknowledgements:

Samples, Material and slides provided by:

Livie Carter-Dorwling, Dr. Robin Schäublin, Dina Klimentyeva, Dr. Karsten Kunze, Yuan Xiao, Dr. Jeff Wheeler, Dr. Tomáš Hrnčíř, Marek Šikula, Dr. Anna Evans, Dr. Brandon Bürgler, Dr. Henning Galinski, Tamara Popovic, Anna Hambitzer
Different types of FIBs and applications

1. Basics

Liquid Metal FIB
- Typically Ga source
- Alloy sources: Au, Bi, Si, …
- Current range: 1 pA – 50 nA

Plasma FIB
- Typically Xe
- Current range: 100 pA – 1 uA

Applications
- Cross section → flat cut
- TEM lamella → thin & defect free
- Pillar → steep sidewalls, no overetch for mechanical testing
- Nanostructuring → no redeposition, smooth surfaces
Scan strategy

Parameters we control
- Dwell time
- Spacing
- Order
- Dose

Layer by layer
- Used for imaging
- Parallel floor
- Delayering
- Low redeposition

Line by line
- Used for cutting (cross sections /polishing)
- Fast removal of large volumes
- Tilted floor: shallow to deep
- Redeposition on floor and sides

Rings /following outlines
- Used for holes /pillars /tips
- Circle by circle: polishing
- Ring over ring: parallel
## Sputter yield and sputter rate

**Sputter yield** $Y$ = atoms sputtered per incoming ion  
**Sputter rate** $S$ = volume removed per dose

### Basics

![Diagram of sputtering process](image)

<table>
<thead>
<tr>
<th>Element</th>
<th>Sputter yield* ($Y$ [–])</th>
<th>Sputter rate** ($S$ [um$^3$/nC])</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>1.69</td>
<td>0.18</td>
</tr>
<tr>
<td>Al</td>
<td>3.47</td>
<td>0.29</td>
</tr>
<tr>
<td>Si</td>
<td>2.78</td>
<td>0.24</td>
</tr>
<tr>
<td>Ti</td>
<td>2.28</td>
<td>0.46</td>
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<tr>
<td>Cr</td>
<td>5.15</td>
<td>0.1</td>
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<tr>
<td>Au</td>
<td>15.75</td>
<td>1.5</td>
</tr>
<tr>
<td>W</td>
<td>7.59</td>
<td>0.12</td>
</tr>
</tbody>
</table>

Sputtering by 30 kV Ga+ normal incidence

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* SRIM data from: “Introduction to focused ion beams” Lucille A. Giannuzzi and Fred A. Stevie (Eds.) Springer, 2005  
** “Nanofabrication using focused ion and electron beams” Ivo Utke, Stanislav Moshkalev, Phillip Russell, Oxford University Press, 2012
Collision cascade model

Interaction of high energy ion with a solid: ion incidence is normal to solid surface.

Ion solid interaction

- Surface atoms can leave (sputtering / milling)
- Liquid state after 1 ps *
- Relaxation and remaining defects after 10 ps
- Implanted ions
- Amorphization
- Electrical damage zone
- 30 kV Ga+ on Si: ca. 30 nm

* “Introduction to focused ion beams” Lucille A. Giannuzzi and Fred A. Stevie (Eds.) Springer, 2005
Milling at an edge

“Edge effect”

- Increased sputter yield (ca. 8x)
- Scan strategy matters because former pixel creates a side wall → line by line milling is efficient
- Long dwell time → strong side walls → fast milling
Reminder on everyday tricks and tips

General tricks

• Small currents for high precision
• Large currents for speed
• Defocus on high currents *
• Tilt sample into beam for perpendicular cut
• Short dwell time for high control of shape /depth
• Long dwell time for hard materials /speed
• Several passes for nanopatterning to avoid redeposition
• Work with a well tuned beam
• Observe and reflect
• Be open to new effects, they might be useful
• Literature

What is curtaining?

Curtaining (aka waterfall effect)
- Milled face shows lines (more / less material was removed)
- Irregular non flat surface: bad for quantitative image analysis, may obscure features

Porous ceramic on Ni: strong curtaining due to rough surface
Porous cermet: mild curtaining due to pores

Samples by Dr. Anna Evans, Dr. Henning Galinski, NMW, ETH Zürich
Curtaining is created by spatial variation of the sputter rate of the sample and the modulation of the current density by forward scattering of the ions:

- Porous materials
- Rough surfaces
- Height steps (e.g. gates in semiconductors)
- Composites of hard and soft materials

Sample topography leads to deflection of ions, thereby modulating the dose locally and thus the milling speed

* “Nanofabrication using focused ion and electron beams” Ivo Utke, Stanislav Moshkalev, Phillipp Russell, Oxford University Press, 2012
How to reduce /avoid curtaining? #1

Tricks

• Hide it:
  - BSE signal
  - Post processing

• Thick, uniform and dense protection cap to smoothen surface, small currents may help

• Backside milling
  - Works at an edge *
  - For TEM lamellas **, ***

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* “Nanofabrication using focused ion and electron beams” Ivo Utke, Stanislav Moshkalev, Phillipp Russell, Oxford University Press, 2012


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Scope

Samples from Dr. Henning Galinski and Dr. Brandon Bürgler, NMW, ETH Zürich
How to reduce /avoid curtaining? #2

Tricks

• Stage rocking
• Rocking stage

Stage rocking:
From (a) rotate stage 90° to get to (b) then tilt back 10° to get to (c), then blindly mill until curtaining changed direction, get back to (a) and polish again, stop before curtaining reappears

Rocking stage from Tescan:
Piezo motors allow for additional tilt axis → can monitor progress of removal of curtaining

How to reduce /avoid curtaining? #3

### Tricks

- Infiltrate the sample:
  - low viscosity resin
  - vacuum – air cycling

Porous cermet infiltrated with epoxy resin under vacuum and cured under isostatic pressure.

Vacuum chamber for vacuum infiltration and two embedding resins: Epon and Körapox.

Cylinders with embedded samples for metallography (mechanical polishing).
Rippling vs Curtaining

Ion rippling

- Self organization
- Instability of process
- Material dependent (PI, rocks, are bad; Al and Si are fine)
- Ion dependent (size of ion vs atoms in solid)
- Current density dependent
- Mostly see it in Xe PFIB

Typical ion rippling of Xe PFIB cut cross section on silicate rock*: small plateaus and then curtaining at each end.

The geometry of the rippling artifact makes that stage rocking won’t help.

* Sample from: Dina Klimentyeva, Institute of Geochemistry and Petrology, ETH Zürich and Marja Lehtonen, Hugh O’Brien - Geological Survey of Finland (GTK)
How to get rid of rippling?

Tricks

• Thick and very dense deposition
  → Pt deposited at 10 kV in PFIB

• Masking technique
  → cover ROI with a thick piece of Si wafer

(a) Pick a Si mask using manipulator and SEM glue
(b) Place mask onto ROI
(c) Mill using a high current

Cross section polished using Si mask

Dense and homogeneous platinum deposition created using 10 kV Xe beam.

Sample from: Dina Klimentyeva, Institute of Geochemistry and Petrology, ETH Zürich and Marja Lehtonen, Hugh O’Brien - Geological Survey of Finland (GTK)
Masking is fantastic!

Massive ion rippling when milling SiC sample, microstructure completely obscured.

Cross section polished using Si mask, microstructure clearly visible.

For more details see poster “Plasma FIB artefact-free milling using TRUE X-sectioning technique” presented by Miloš Hrabovský.

Tomáš Hrnčíř, Marek Šikula, Jozef Oboňa & Pascal Gounet: “How to achieve artefact-free FIB milling on polyimide packages”, ISTFA 2016 Conference proceedings, pg. 642-646
4. Crystal damage

Types of damage to crystal structure

Relevant parameters
- Energy and type of ion
- Incidence angle
- Type of solid

Amorphization
- Local melting and quenching of solid
- Irregular atom positions obscure HRTEM

Point defects
- Vacancies and interstitials are created
- Electronic structure is disturbed
  → affects TEM Holography
- Accumulation of point defects leads to dislocation loops
  → will show in diffraction contrast TEM

Damage zones in a crystalline solid C
A = amorphized
P = point defects
4. Crystal damage

**Amorphization**

To reduce thickness of amorphous layer (*, **, ***)

- Low kV milling
- Larger ion
- Lower angles

(a) 160 nm total incl. 60 nm amorphous
(b) 90 nm total incl. 60 nm amorphous
(c) 40 nm total incl. 10 nm amorphous

To reduce thickness of amorphous layer (*, **, ***)

- Low kV milling
- Larger ion
- Lower angles

Overview of amorphization layer thickness in Si, from *


** Schaffer, M, et. al., "Sample preparation for atomic-resolution STEM at low voltages by FIB", Ultramicroscopy 114 (2012) 62-71

*** Süess, M, et. al., "Minimization of amorphous layer in Ar+ ion milling for UHR-TEM" Ultramicroscopy 111 (2011) 1224-1232
Tungsten – flash polishing

• FIB creates damage to W crystal that in turn spoils TEM image
• Flash polishing in NaOH removes damaged layer
→ TEM reveals the oxide particles embedded in the W matrix.

‘Focused ion beam application on the investigation of tungsten-based materials for fusion application’
L. Veleva, R. Schäublin, A. Ramar, Z. Oksiuta, N. Baluc
European Microscopy Congress 2008, Volume 2: Materials Science, Eds. S. Richter and A. Schwedt,
Micropillars for mechanical testing

Pillar morphology

- Presence of Ga in Al is visible by SEM
- XeF$_2$ while Xe milling improves quality of pillar

Displacement controlled microcompression setup by Alemnis

Jeff Wheeler, Yuan Xiao, LNM, ETH Zürich
Comparison between Ga and Xe prep’d

Ga in Al
• Ga accumulates at grain boundaries → weakening of grain boundaries
• Deformation mode is changed → grains pop out!

Liquid Metal Embrittlement (LME)
• Ga is one of the worst, it affects:
  Al, Cd, Cu, Fe, Ag, Sn, Zn

Empirical rules for LME
1. Metals form alloy / intermetallic → no embrittlement.
2. Metals are *immiscible* → embrittlement possible!

Comparison of tested micropillars
Microcompression testing: ultra-fine grained aluminum cylinders prepared by Ga FIB and Xe FIB


Mixing of layers

Procedure

• Si wafer with Si$_3$N$_4$ passivation
• Evaporation of 3-layer stack either
  “A” Ti-Au-Ti (10-5-10 nm)
  “B” Cr-Au-Cr (10-5-10 nm)
• FIB patterning at 10 pA, several circles from out to inside
• TEM lamella across patterned area
• STEM imaging and EDX mapping

→ Mixing is much stronger for Ti than Cr!

<table>
<thead>
<tr>
<th>Material</th>
<th>Sputter rate (um$^3$/nC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al$_2$O$_3$</td>
<td>0.08</td>
</tr>
<tr>
<td>Cr</td>
<td>0.1</td>
</tr>
<tr>
<td>Ti</td>
<td>0.46</td>
</tr>
<tr>
<td>Au</td>
<td>1.5</td>
</tr>
</tbody>
</table>

Sample from Livie Dowling-Carter (Biomedical Engineering), TEM
STEM by Christian Zaubitser (ScopeM)
Bleeding /preferential milling

Explanation *
- P sputters faster than In
- Accumulation of In at surface
- Heat during sputtering allows droplet formation

Tricks hinder droplet formation
- Apply XeF₂ etching gas**
- Cooling to low temperature*
- Small milling currents*

Bright spots covering
FIB polished InP

HEMT sample from Tamara Popovic and Anna Hambitzer, MM-Wave Electronics Group, ETH Zürich


** "Nanofabrication using focused ion and electron beams" Ivo Utke, Stanislav Moshkalev, Philipp Russell, Oxford University Press, 2012
Questions