

Focused Ion Beam

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Outline



- Principle of Focused Ion Beam
 - Comparison between SEM and FIB
 - Physical effects of incident ions
- FIB at CEN
- Basic Applications
- •TEM sample preparation
 - Lift out / H-Bar/ Ex-Situ lift out
 - tricks and tips (others)
- FIB for 3D microstructure characterization
 - 3D slice and View
 - 3D EBSD
- Damage
- •Other Ions sources

Principle of Focused Ion Beam

Focused Ion Beam (FIB) was developed in the late 1970s and the early 1980s

Ion column structure similar to that of SEM

Major difference :

Source: Liquid Metal Ion Source (LMIS)

Electromagnetic lenses are not of sufficient strength to focus the heavy ion beam and so <u>electrostatic lenses</u> are used.

Principle:

A strong electromagnetic field causes the emission of positively charged ions from the liquid metal cone which is formed on the tip of a tungsten needle.

Liquid metal is usually Ga

ONS



Principle of Focused Ion Beam



Figure 2. Schematic diagram of a FIB ion column.

Principle of Focused Ion Beam





Schematic diagram of a FIB ion column Source: IBM Almaden Research Center



Ref.: J. Orloff, Scientific American, Oct. 1991

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Electrons	Ions
are very small	Big
Inner shell reactions	-> Outer shell reactions (no x-rays)
High penetration depth	Less penetration depth
Low mass -> higher speed for given energy	High mass -> slow speed but high Momentum milling !!!
Electrons are negative	Ions are positive
Magnetic lens (Lorentz force)	Electrostatic lenses

=> One Gallium-Ion ist approx. 127.000x heavier than an electron!

Ref.: Tim Armbruster

Why Gallium ?

Ga is metallic, low melting point, in the middle of the periodic table, no overlap with other elements in EDX

Ref.: Marco Cantoni

Comparison between SEM and FIB



		FIB		FIB	SEM		Ratio
Particle	type		Ga+ ion		electron		
	elementary charge		+1		-1		
	particle size			0.2 nm	0.00001 nm		20'000
	mass		1.2	.10 - 25 kg	9.1.10-31 kg		130'000
	velocity at 30 kV		2.8	8.105 m/s	1.0 108 m/s		0.0028
	velocity at 2 kV		7.3	3.104 m/s	2.6.107 m/s		0.0028
	momentum at 30 kV		3.4.1	0 - 20 kgm/s	9.1.10-23 kgm/s		370
	momentum at 2 kV	8	3.8.1	0-21 kgm/s	2.4.10-23 kgm/s		370
Beam	size		nm range		nm range		
	energy	up to 30 kV		to 30 kV	up to 30 kV		
	current		pA t	to nA range	pA to uA range		
Penetration depth	In polymer at 30 kV		60 nm		12000 nm		
	In polymer at 2 kV			12 nm	100 nm		
	In iron at 30 kV			20 nm	1800 nm		
	In iron at 2 kV			4 nm	25 nm		
							1
Average electrons	secondary electrons		1	00 - 200	50 - 75		
signal per 100	back scattered	0		0	30 - 50		
particles at 20 kV	electron						
	substrate atom	500		500	0		
	secondary ion	30		30	0		
	x-ray		0		0.7		

Ref.: Marco Cantoni

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Physical effects of incident ions

The most important physical effects of incident ions on the substrate are:

- ✓ Sputtering of neutral and ionized substrate atoms
- ✓ Secondary electron emission
- ✓ Displacement of atoms in →
 the solid
- ✓ Emission of phonons
- ✓ Chemical interaction
 including the breaking of
 chemical bonds



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Basic operating modes



1. Image mode



2. Milling mode



3. Deposition mode



Basic operating modes - Image

During FIB imaging the finely focused ion beam is raster scanned over the substrate and secondary particles are generated in the sample (Emission of atoms, secondary ions and electrons)



Imaging with FIB induces some damage:

- Ga⁺ Ions implantation (depth is related to ion energy, and angle of incidence)
- Some milling always occurs

Ref.: S. Reyntjens and R. Puers 2001 J. Micromech. Microeng. **11** 287 Use of low ion current

FIB imaging



- A. Crystallographic orientation (channeling) contrast
- B. Material contrast
- C. Topographic contrast
 - A. Orientation contrast arises from <u>channeling of the incident ions</u> <u>between lattice planes</u> of the specimen. Depth varies with the angle between the ion beam and the lattice plane and the interlunar spacing of the lattice.

Secondary electron yield is greater than that of secondary ions and less sensitive to changes in chemistry

FIB imaging – Crystallographic orientation

Pure Aluminium



Each line represent the change in intensity of a discrete grain

Deviation from the normal incidence as the speciment is tilted relative to the ion beam

Fig. 2. FIB secondary electron mode orientation contrast of a fully annealed, nominally pure polycrystalline aluminum specimen changes the intensity (grey level) of each grain as a function of deviation from normal incidence as the specimen is tilted with respect to the gallium beam. The same region is imaged (inset) at angles of 0, 15, and 30° tilt (foreshortening due to tilt angle is evident). The change in intensity (in arbitrary 'grey level' units) is plotted as a function of tilt angle in 1° increments. Dark grains (low intensity) represent significant channeling of the primary ions. The angular width of channeling 'troughs' and the angular distance between troughs can be used to calculate the relative orientation of different grains.

Ref.:M.W. Phaneuf / Micron 30 (1999) 277–288

FIB imaging – Crystallographic orientation





SEM image



0.0	HV	curr	mode	WD	HFW	tilt	10 μm
0	30.00 kV	93 pA	SE	16.0 mm	32.0 µm	16 °	Helios 600

FIB image : Ion induced secondary electron image

Ref.:Ni electrodeposit (*Hossein Alimadadi*)

FIB imaging – Crystallographic orientation



FIB image : Ion induced secondary electron image



Ref.:Ni electrodeposit (Hossein Alimadadi)

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FIB imaging – Material contrast

B. Material contrast arises from difference in the yield of secondary particles as a function of specimen chemistry

[>]This effect is most readily observed in FIB secondary ion images

Secondary ion yields from galium beam can be increased by up to 3 orders of magnitude for metalic species in the presence of oxygen.

Secondary electron



Secondary ion



The presence of electronegative oxygen increases the probability that the sputtered atom or molecule can release an electron, thus becoming a positive secondary ion which can be observed.

Corroded crack in a steel pressure vessel

Ref.:M.W. Phaneuf / Micron 30 (1999) 277-288

FIB imaging - Topographic contrast





Fig. 1. FIB secondary electron mode image of a nickel foam 'strut' which forms a component of nickel-hydroxide-based batteries intended for automotive applications. One third of the tip of the strut has been sectioned in the FIB (inset) to expose internal detail. Both the sectioned and original surfaces have been imaged so as to illustrate the grain orientation contrast achievable in FIB secondary electron images, and the ability to reveal sub-micron voids and other fragile specimen features without the stress and potential damage involved in mechanical sectioning.

Topographic contrast is frequently overwhelmed by channeling contrast in certain material systems and geometries. In general, topographic contrast in FIB has been explained in a similar manner to topographic contrast in SEM.

 \rightarrow <u>Differences in signal as a function of the</u> angle of incidence of the primary beam relative to specimen surface normal as the local inclination of the specimen surface varies.

Further study of topographic contrast is required to resolve differences in topographic contrast between secondary-ion and secondary-electron imaging in the FIB, and differences between secondary electron imaging in the FIB and in the SEM.

Basic operating modes - Milling



Use of high ion current

By scanning the beam over the substrate, an arbitrary shape can be etched.

Etch rate depend on:

- Material
- Scanning type
- Redeposition
- Angle of incidence



Rectangle ...

Material	Sputterrate			
	[µm³/nC]			
Si	0.27			
Thermal Oxide	0.24			
TEOS	0.24			
Âİ	0.3			
AI2O3	0.08			
GaAs	0.61			
InP	1.2			
Au	1.5			
TiN	0.15			
Si3N4	0.2			
C	0.18			
Ti	0.37			
Cr	0.1			
Fe	0.29			
Ni	0.14			
Cu	0.25			
Мо	0.12			
Та	0.32			
W	0.12			
MgO	0.15			
TiO	0.15			
Fe2O3	0.25			
Pt	0.23			
PMMA	0.4			



Basic operating modes - Milling

- Ion sputtering yield (Y) depende on incidence angle (θ)
- A larger Y is achieved by increasing the θ from normal incidence.
- The maximum sputtering yield is typically achieved at θ ≈ 80°, and Y dramatically approaches zero from 80° to 90°



Schematic diagram of the effective region of ion track at different incident angles



Ref.: Jing Fu in J. Micromech. Microeng. 18 (2008)

Basic operating modes -Deposition



Chemical interaction:

- Deposition
 - Principal is of CVD (chemical vapour deposition) with better resolution and lower deposition rate.
 - Commercially available:
 - Pt, W, SiO₂ , TMCTS (tetramethyl-cyclotetrasiloxane), O₂ or H₂O
- The precursor gases are sprayed on the surface by a fine needle, where they absorb →the incending ion beam decomposes the adsorbed precursor gases than the volatile reaction products desorb from the surface while the desired reaction products remain fixed on the surfaces as a thin film.
- The smallest feature that can be deposited are of the order of 100nm (lateral) and the minimal thinckness is about 10nm. Aspect ration between 5 and 10 are obtained.

Ref.: S. Reyntjens and R. Puers 2001 J. Micromech. Microeng. **11** 287

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E-beam deposition: ^Deposition by lower KV and higher beam current

The ion beam deposition is much faster than deposition with the electron beam. This is primarily due to the ion beam producing many more secondary electrons near the surface of the sample compared to the electron beam.

for standard accelerating voltages (30 kV for the ion beam and 5 kV for the electron beam) at a given beam current, the electron beam deposition is \sim 10 times slower than the ion beam when depositing platinum for example.

W deposition is in general able to fulfill the same use cases at Pt deposition, but for electrical applications where a deposit with good conductivity is required, W deposition is preferred over Pt deposition. However, the W deposition rate is slower . The W deposition is harder than Pt so it is useful for making mechanical structures such as probing tips and for using as the protective layer on very heterogeneous materials before cross sectioning to reduce the curtaining effect. W deposition also has a lower proportion of carbon in the final deposit when compared with Pt deposition.

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Basic operating modes -Deposition

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The deposition rate of a material depends on a range of parameters.

- Distance to the GIS needle
- Sample orientation
- Ion beam current,
- Pattern area,
- Pattern speed (dwell time and overlap)
- Refresh time (time allowed for each point to replenish with adsorbed gas)



1 μm thickness in 300 seconds



fixed

Ref.: http://www.fei.com/uploadedFiles/Documents/Content/AN-GIS_Beam_Chemistries-AN-web-2010.pdf

Basic operating modes – Enhanced Etching



Chemical interaction:

- Enhanced etching is used
 - •To speed up milling
 - to increase selectivity toward different materials
- Etching gas is introduced to chemically facilitate the removal of reaction products.

	Aluminium	Tungsten	Silicon	SiO ₂ , Si ₃ N ₄	Photoresist, polyimide
Cl ₂	10-20	_	10	_	_
Br ₂	10-20	_	6-10	_	_
ICI	8-10	2-6	4-5	_	_
XeF ₂	_	10	10-100	6-10	3–5

Table 2. Typical GAE gases and their etch rate enhancement factors on various materials.

Enhanced etching



- Delineation Etch is used for the etching of integrated circuit (IC) cross sections \rightarrow Good for Oxide layers, Insulators and Nitrides
- Insulator Enhanced Etch (IEE) is used to rapidly etch films of many types of insulating material with the assistance of xenon difluoride (XeF2), a halogen compound. IEE removes insulating materials preferentially and leaves the conductor. (speeds up maching of glass, nitrides and other isulators.)
- Selective Carbon Mill (SCM) uses water vapor to increase the removal rate of carbon-containing materials such as polyimide, PMMA (polymethyl methacrylate), and diamond. In addition, SCM decreases the removal rate of other materials (e.g., Si and AI). This effectively increases the etch selectivity of polymers over these other materials. Table 1 provides sputter rates and typical enhancements for various materials when using a 30 kV beam voltage. Actual values



Use of IEE in SiO_2

vary depending upon the conditions.

Table 1 Etch Rates and Enhancements for Various Materials

Material	Typical Sputter Rate (µm ³ /nC)	Typical Etch Rate Enhancement with IEE
Silicon (Si)	0.2	7-12
Aluminum (Al)	0.5	2.13
Gallium Arsenide (GaAs)	0.7	_
Indium Phosphide (InP)	1.2	_
Gold (Au)	0.6-1.6	1
Tetraethyl orthosilicate (TEOS)	0.3	7.2
Thermal Oxide	0.3	7.2
Titanium Nitride (TiN)	0.26	7.5
Silicon Nitride (Si ₃ N ₄)	0.16	7

Others available GISes



- Enhanced Etch etches metals and to some extent silicon and some nitrides faster. Helps to prevent re-deposition and enables higher aspect ration holes. Uses halogens.
- Insulator deposition used to produce insulating coating
- Carbon deposition
- Gold deposition

FIB Ion Milling Yield of Various Material with and without Water Vapor

Materia	<u>l</u>	Yield (µm ³ /nC)				
	0.0	0.5	1.0	1.5	• • 7.5	8
Polyimide				-		
PMMA				-		
Diamond						
Gold						
Si Oxide						Con.
Si Nitride						
Silicon			yield with	n water		
Aluminum						

T.J. Stark, et. al. J. Vac. Sci. Technol. B13, 2565 (1995)

Hardware configuration CEN – Quanta 3D

Scanning electron microscope (W- filament SEM)

observation microstructure

FIB = focused ion beam

- 30KV, 15KV, 5KV beam energies
- Second electron detector and backscattered electron detector
- STEM detector
- High and low vacuum mode
- CDEM- Charge neutralizer 2 Gas Injection System:
- deposition of W and Pt films from organic precursor gasses

In-situ manipulation of specimens using Omniprobe manipulator







Hardware configuration CEN – Helios 600

Scanning electron microscope (FEGSEM)

observation microstructure (high resolution)

FIB = focused ion beam

- 30KV -500V beam energies
- Second electron detector and backscattered electron detector
- Thru-the-Lens detector
- CDEM- Charge neutralizer 5 Gas Injection System:
 - deposition of W and Pt films from organic precursor gasses
 - use reactive gases XeF2, water vapor and TFA

Quantitative images with EBSD and EDX

 quantitative characterisation of microstructure

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In-situ manipulation of specimens using Omniprobe manipulator







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Basic applications

- Direct device fabrication or lithography instruments
- Sectioning for failure analysis
- Mask repair
- Micromachining
- Nanofabricated structures
- TEM sample preparation
- Atom Probe sample preparation
- Manufacture of AFM tips

• 3D microstructure characterization





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TWIPUnverformt.001.tif Overview thin foil from FIB 14:29 06/01/07 TEM Mode: Imaging

2 microns HV=200kV Direct Mag: 4400x X: 141.102 Y: -1.007 T:0

TWIPUnverformt.002.tif one grain with individual dislocations and a twin 14:37 06/01/07 TEM Mode: Imaging

2 microns HV=200kV Direct Mag: 7800x X: 139.214 Y: -.5 T:0



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TEM sample preparation – H-bar

 \checkmark specimen must first be mechanically polished as thin as possible before the sample is placed in the FIB for milling.



Figure 1. (a) Schematic illustration of the H-bar focused ion beam (FIB) technique. Material on opposite sides of a region of interest is FIB-milled until it is electron-transparent.

(b) Scanning electron microscopy (SEM) image showing the topdown view of an H-bar FIB specimen in progress. The metal sample was mechanically thinned to ~40 mm and glued to a transmission electron microscope (TEM) half-grid. (Figure courtesy of Richard Young, FEI Co.













Date

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Bath containing 0.04g/l of saccharin Bath interface Middle direction Substrate interface Growth WD |mag ⊞| tilt 16 5 mm WD mag 🎛 🛛 tilt 30 µm Bath 1 750 x Halia .00 kV 73.1 µm 16.5 mm Middle Substrate interface

HV mag 🖽 WD HFW

mag III

WD

det



Samples preparation: Steve Reyntjens

TEM sample preparation – Ex-situ lift out

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- Freestanding site-specific region is FIB milled to electron transparency,
- The thin lamella is removed from its trench with a micromanipulator under an optical microscope.
- The specimen attaches to the micromanipulator tip via electrostatic forces and can be removed easily from its trench.
- Specimens can be transferred to carbon-coated TEM grids, form varcoated grids, holey carbon grids, or directly to the surface of small mesh grids.

TEM sample preparation – plan-view specimen





Figure 3. Method to prepare a plan-view specimen from a specific site based on the microsampling technique (see text for details).

Block- Lift out





30.00 kV 93 pA SE 16.1 mm 213 µm 0 ° Helios 600









FIB-SEM: Slice and view









- No stage movement
- no charge neutralization
- Use of gas to increase milling rate: <u>Selective Carbon Mill</u> (water vapor) to increase the removal rate of carboncontaining materials

FIB-SEM: Slice and view











"Rotation" set-up: Helios





"Rotation" set-up: Helios





Movements:

- rotation 180°
- x and y movements
- z movement 4 to 8 mm WD
- tilt (in case the sample is not perfect align with sample holder)

Milling strategy (rotation set-up)





Damage



In semiconductor materials, the thickness of the amorphous layer formed on the FIB-prepared sample surfaces is nearly proportional to:

- the range of Ga ion implantation, which in turn is roughly proportional to the primary energy of the Ga ions.
- The reduction of amorphous layer formation in a sidewall of Si as a function of Ga energy
- For ion energies of 30 keV, 5 keV, and 2 keV, the observed sidewall damage is \sim 22 nm, 2.5 nm, and 0.5–1.5 nm, respectively.

Alternatively, low-energy ion milling with a broad Ar ion beam can be used to remove damage layers created by the FIB during lamellae formation and also to further reduce the specimen thickness. Special ion polishers are in development that will enable milling to a controlled final specimen thickness.

Parameters to be Optimized

- ▶ Ion energy \downarrow sputtering rate \downarrow and bombardment induced damage \downarrow .
- Milling time.
- Angle of incident ion beam (θ) ↑ sputtering rate ↑, bombardment induced damage ↑ and shadowing ↓.
- **Oscillation angle (\varphi)** \uparrow curtaining effects \downarrow .
- Type of protection layers: Pt/C/SiO₂ protect the area of interest / shadowing / conducting or non conducting.
- > Type of supporting grid minimize redeposition.
- > Position of the FIB lamella on the supporting grid minimize redeposition.





Column Alignment





EFUG2005

SII NanoTechnology Inc.

Anisotropic sputtering & curtaining

DTU

Anisotropic sputtering on Fe 3% Si





Experiments and calculations on anisotropic sputtering of Cu *B.W. Kempshall et al., J.Vac.Sci.Tech. B19 (2001), 749*



- Additional to surface amorphization here have been several reports in the literature that FIB milling of fine-grained fcc metals can change the orientatic and size of the surface grains and form Ga intermetallic compounds.2
- The extensive use of FIB milling for sample preparation and the use of ion channeling contrast to characterize and measure grain sizes require that these ion beam-induced modifications be properly understood.





Cu grain structure modification to a depth of 200 nm by an ion dose of $2.5 \setminus 1017$ Ga/cm2, despite the fact that calculated ranges for 30 kV Ga ions are close to 50 nm (Figure 5b).

EBSD orientation mapping of the sample shows that the surface grains are reoriented so that the <110> direction, the strong channeling direction in fcc crystal structures, is oriented parallel to the incident ion beam

Similar effects were observed in other fcc metals (Au, Ni), and bcc metals were observed to reorient at the surface with a <111> crystallographic direction normal to the exposed surface

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• 30 kV Ga+ ion exposure can cause extensive microstructural modification of metal samples, even to depths beyond the expected, nonchanneling-orientation, ion range.

•Until these effects are understood and/or catalogued, caution should be used when FIB is employed to prepare samples for microstructural investigation, as even short exposures can result in unwanted changes to the sample.

•One must also be careful that the implantation of Ga into the sample does not result in changes induced by the formation of new Ga-containing phases.

•The addition of Ga to many metals can result in low-melting temperature phases.

•Cu₃Ga has been observed at the bottom of FIB-milled trenches in Cu.

• Thin TEM samples prepared from AI may show Ga enrichment at the grain boundaries.

• In extreme cases, because of its low melting point, Ga can form phases with other metals that have melting points at or below room temperature.

• For example, Ga addition during ion milling to In results in eutectic formation, with a melting temperature of 15.3 °C. Thus, the potential exists to have a liquid phase present for the FIB milled samples of In.



• The addition of 2.5 at.% Ga to Ge will also result in liquidphase formation. Other metals, like AI, Zn, and Pb, have similar problems.

• It is highly recommended the phase diagram be reviewed before FIB-milling new materials with Ga to avoid problem materials or prepare them in a different manner.

FIB-beam-induced material changes



Amorphisation (see e.g. *Kato et al., J.Vac.Sci.Tech. A17(1999), 1201)*



Fe₃Al matrix: excellent diffraction patterns

Laves phase inclusion: complete amorphisation

Reaction of Ga and Al



damage due to Ga-Al interaction at grain boundaries under a nano-indentation in Al

Beam-induced α-γ phase transformation in Fe-Ni





transformation of metastable austenite into martensite during milling

Comparison of Ion Sources for FIB

Type of ion source	Ion Species	Virtual source size (nm)	Energy spread,∆E (eV)	Unnormalized brightness, B (A/cm ² sr)	Angular brightness (µA/sr)
Liquid metal	Ga^+	50	>4	3x10 ⁶	50
Gas field ion (supertip) (ref. 11)	$H^{+}, H_{2}^{+}, H_{2}^{+}, He^{+}, Ne^{+}$	0.5	~1	5x10 ⁹	35
Multicusp Plasma (ref.9)	Kr^+	17	1 - 3	0.55x10 ³	40
Penning (pulsed) (ref. 12)	Ar ⁺		4.5	10 ³	

Ref.: John Melngailis

FIB imaging – Material contrast





Secondary Electron



FIB Ion Image







Iron



Oxygen



Chromium



Nickel

Silicon 100 µm



Manganese

Ref.:M.W. Phaneuf / Micron 30 (1999) 277–288

Basic operating modes - Milling

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θ, deg

