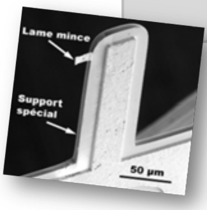
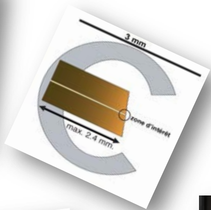
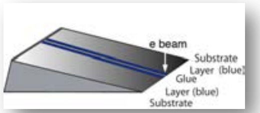
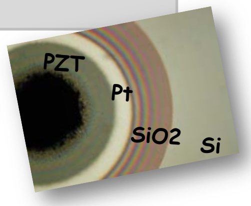
OVERVIEW OF SAMPLES PREPARATION METHODS FOR TRANSMISSION ELECTRON MICROSCOPY (TEM)

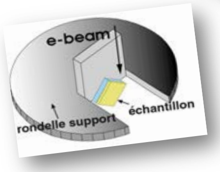
Danièle LAUB, EPFL-SB-CIME, Lausanne CH

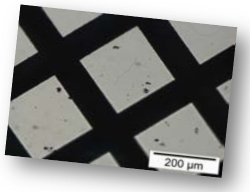








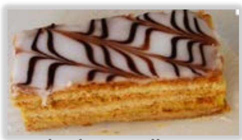




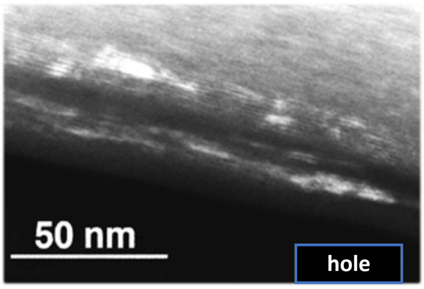
INTRODUCTION

Why is the sample preparation so important ?

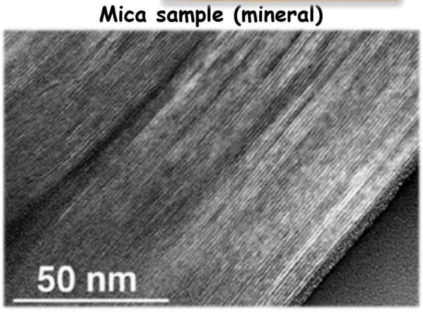
Because no good sample preparation, no good TEM observation !!!



Mica sample (mineral)



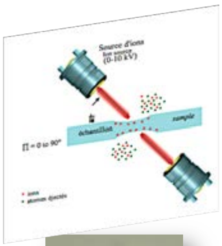
Ion Milling

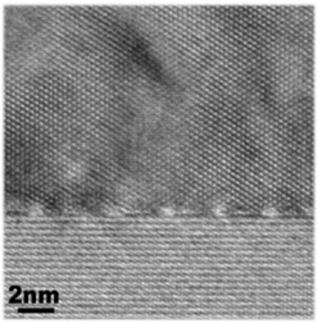


Ultramicrotomy

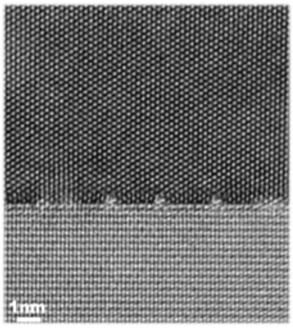
INTRODUCTION

Why is the sample preparation so important ?





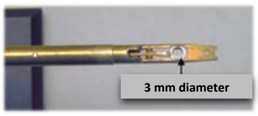
Cross section, zone axis [0001], Ion Milling (>2kV)



Cross section, zone axis [0001] , Ion Milling (down to 100V)

INTRODUCTION

Size and thickness of the sample



Diameter: 3 mm

1) Reduce size of large sample
2) Use 3 mm grid support for small sample

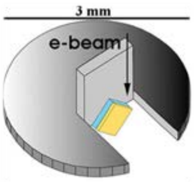
Thickness: between 10 et 200 nm depending on the material and the kind of observation to be done

1) depend on chemical composition
2) high resolution observation, EELS analysis or not

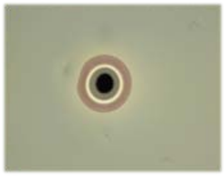
INTRODUCTION

Electrons transparent area, yes, but haw, where ???

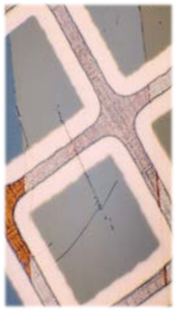
Edge



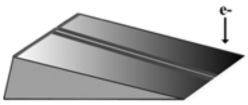
Hole

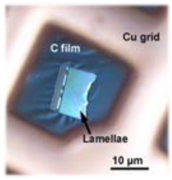


Full thin lamella



Edge





INTRODUCTION

Titan Themis TEM/STEM

300 kV performance: TEM information limit ≤ 0.7 Å;
HR-STEM resolution ≤ 0.7 Å; analytical probe 2 nA in 0.2 nm probe size

INTRODUCTION

Sample as also to be:

- ♣ **electrically conductive**
- ♣ **stable under vacum**
- ♣ **free of hydrocarbures contamination**
- ♣ **should not contain artefacts that could conduct to wrong analyse**

The sample for TEM observation must be representative of the true nature and morphology of the material

It will be impossible to prepare a sample without any artefact, so the good method as to be choosen depending on the type of analyse needed and the type of artefacts induced by one or the other technique

INTRODUCTION

How to choose the preparation technique in relation with the material and the analysis to be done

Material	Geometry	Physical structure	Chemical phases	Physical properties	Electrical properties
Metal Semiconducteor Ceramics Mineral Biological Polymer Composite	Bulk	Compact	Monophased	Hard	Conductive
	Multilayer	Porous	Multiphased	Soft	Insulating
	Small size materials: fibers, nanotubes, …	Liquid phase		Fragile	
				Ductile	
				Resistant	

Analysis

Sample orientation
-Any
-Particular

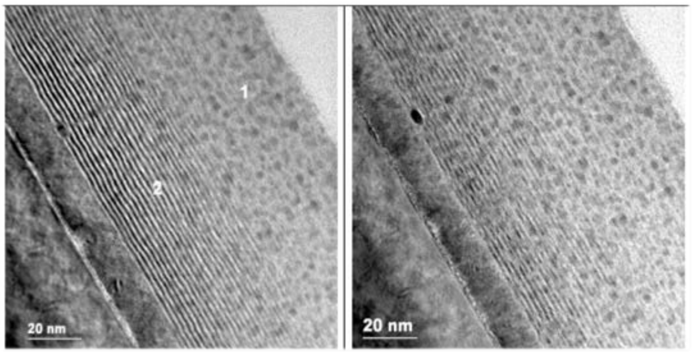
→ Choice of technique depending on analysis to be done and/or induced

→ Technique adaption depending on material

INTRODUCTION

ARTEFACT

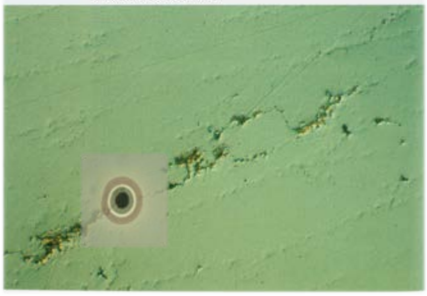
Ru/Zr/$SrTiO_3$
Preparation: tripod planar polish followed by Ion milling



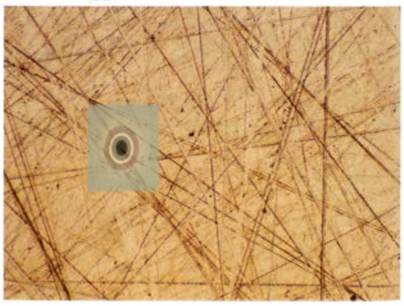
INTRODUCTION

How to observe the true structure of a material ?

Des inclusions arrachées. Nous pouvons apercevoir les rayures provenant des inclusions arrachées.
500x, contraste interférentiel



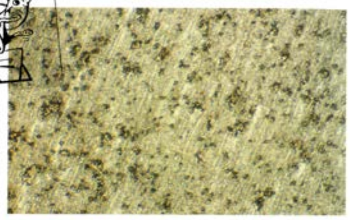
Après le FG, les rayures occasionnées par le PG sont toujours visibles
200x



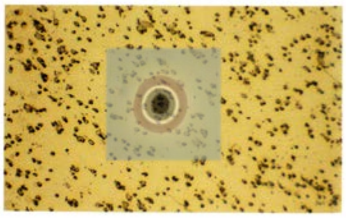
INTRODUCTION

How to observe the true structure of a material ?

Diamond grains used for grinding penetrate into the sample



1. Aluminium, poli aux diamants de 3 µm, sur un disque à faible élasticité. De nombreux diamants se sont incrustés dans l'échantillon 500 x

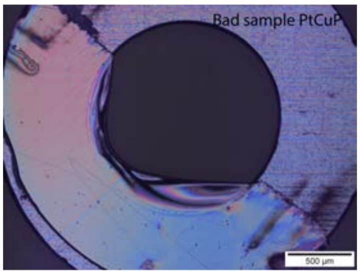


2. Pareil que ci-dessus, après le polissage final. La plupart des diamants sont restés dans l'échantillon 500 x

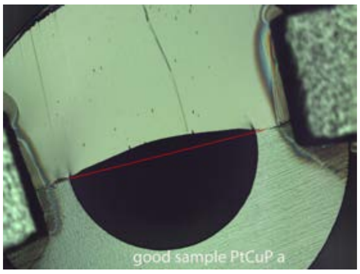
How to observe the true structure of a material ?

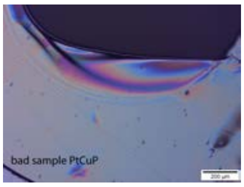
No thermal damages !

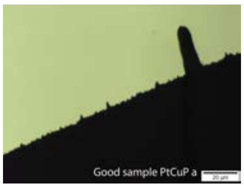
2,5 kV



1kV, -170° C



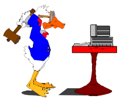




INTRODUCTION

DIFFERENT TYPES OF PREPARATIONS:

Mechanical
- Mechanical polishing down to electron transparency
- Clivage
- Ultramicrotomy
- Crushing
- Nanoparticles dispersion

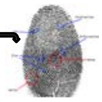


Mechanical + ionic
- Grinding, (dimpling), ion milling

Ionic
- FIB

Chemical
- Electro-chemical polishing
- Chemical polishing or etching

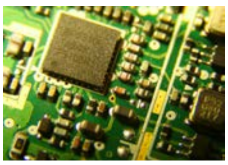
Mechanical-physical
Physical
- Replica (direct or double), extractive
- Thin film deposition
- …

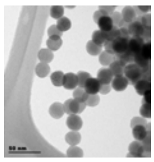


INTRODUCTION

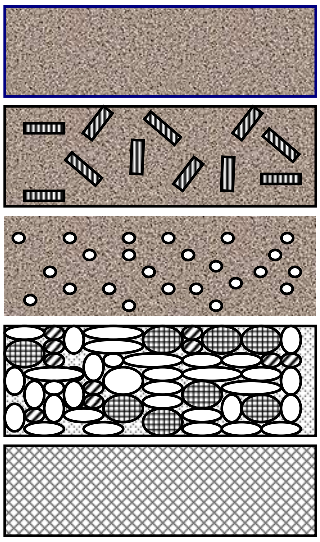
TYPE OF MATERIALS
- Semiconductors
- Metals and alloys
- Polymers
- Minerals
- Cements
- Ceramics
- Wood (paper)
- Etc.

THE OBSERVATION DIRECTIONS
- Planar view
- Transversal view (cross section)
- Anisotropes materials = planar or tranversal view





MATERIAL STRUCTURE AT THE MICROSCOPE SCALE



Amorphous

Slightly organized

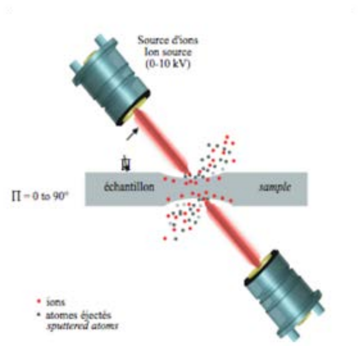
Nano-micro organized

Polycristalline

Mono-cristalline

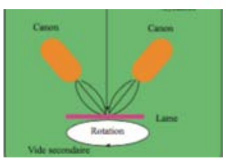
The Ion Milling

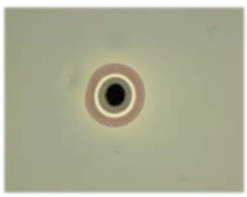
Using electric discharge, Ar^{+} ions of some kV are generated and focused on the sample. The goal is the crystal lattice destruction at the surface followed by ejection of superficial atoms.

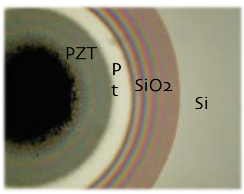


DRAWBACK and ARTEFACT:

- Surface roughness
- Creation of amorphous layer on both surfaces
- Ions implantation
- Creation of dislocations
- Modification of stoichiometry
- Differential thinning rates on different compounds or phases
- Heating







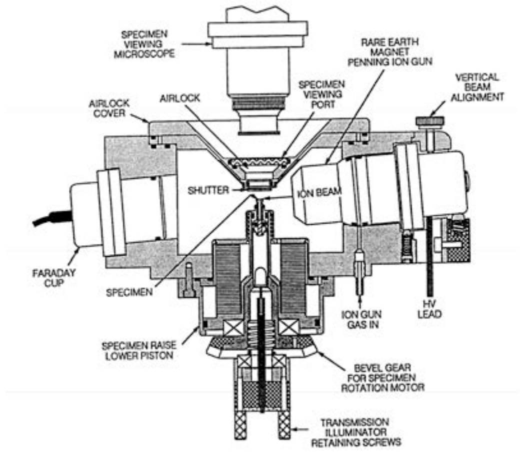


Diagram of an ion milling chamber

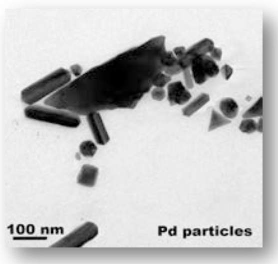
TECHNIQUE: ELECTRON TRANSPARENT NANOPARTICLES/NANOTUBES DISPERSION

*Direction d'observation: **généralement** aléatoire*

But: obtenir des particules bien dispersées, propre, déposées sur le support adéquat.

Observations:

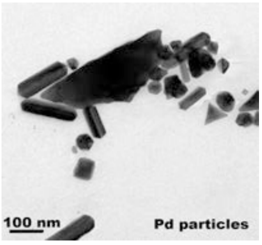
- **Taille et forme des particules**
- **HRTEM**
- **Diffraction**
- **Analyses EDX**
- …



Particles well dispersed

Clean particles, well dispersed

Goal



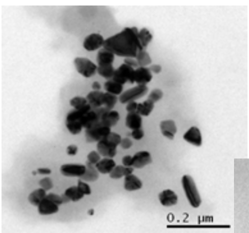
Clean particles, well dispersed on the adequate support

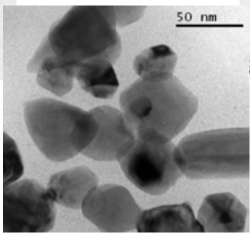
TEM image

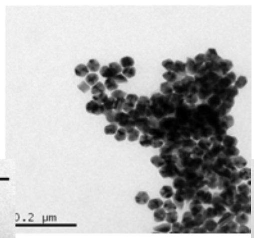
Presence of involatile molecules or inorganic species

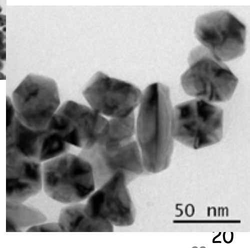
Reduced contrast

Reduced image quality









20

Working with nanoparticles or nanofibers

Safety rule n° 1: preparation under the fume hood !

Absolutely not needed

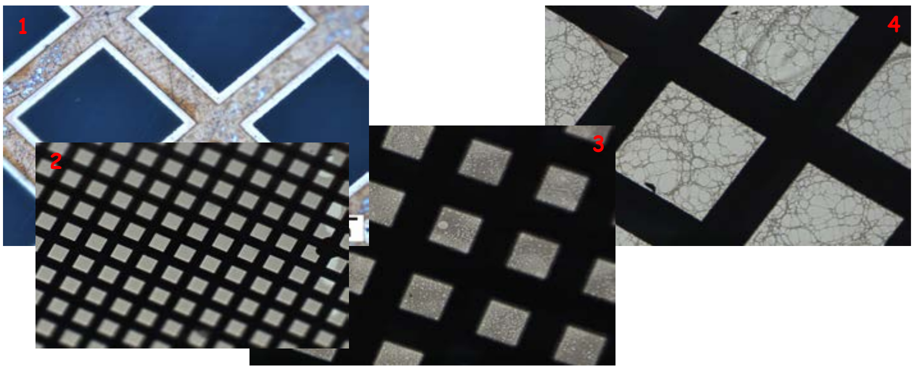
Not needed but up to you !

Absolutely needed

Selection of suitable support grid

Depends on the analysis to be done and to the particles size

- Standard carbon coated grid (C thickness 25-40 nm) 1
- Ultrathin carbon film (4-5 nm) 2
- Holey carbon film with ultrathin carbon windows (5 nm)
- Holey carbon film 3
- Lacey carbon film 4
- …



The use of solvent to disperse particles

Polar, non polar or does not matter ?

Ethanol: polar

Toluene: non polar

Ultrasonic Device to Disperse Nanomaterial

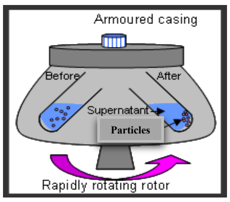
Hielscher Ultrasound

Heating may help

Dispersion time needed: from 1 minute to several hours

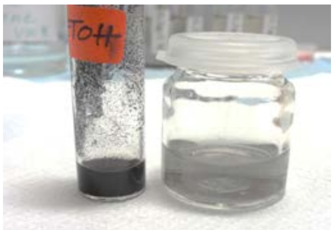
Centrifuging to wash and concentrate the nanomaterials

- To wash the particles if in dirty or inadequate solvent
- To concentrate the particles if too few



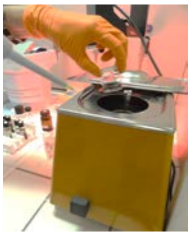
Dispersion

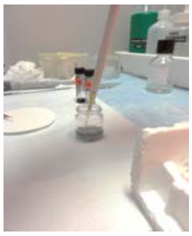
Dilution



Concentration

Ultrasound

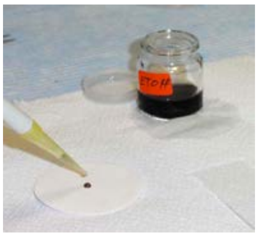


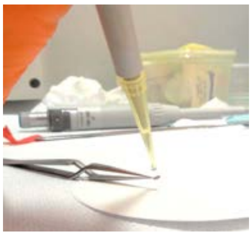


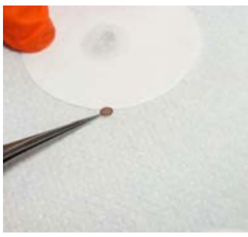
Ready to pick up the droplet

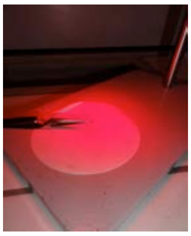
Dispersion

One droplet on the grid: 3 ways







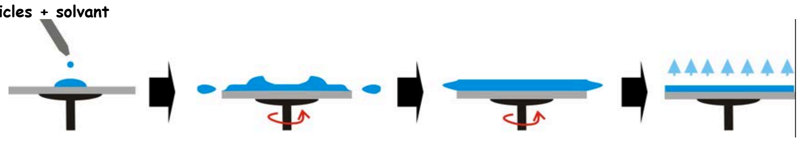


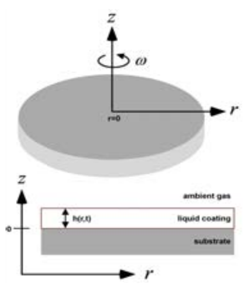
Dry under infra-red or standard desk lamp

With the perfect loop

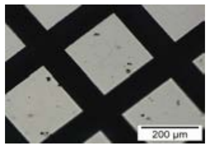
Dispersion via Spin coating

Particles + solvant

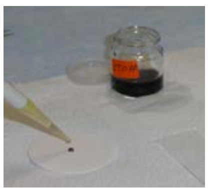


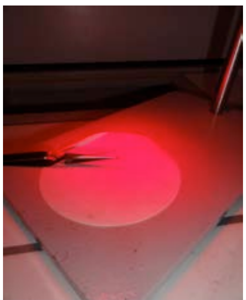


splash
protection



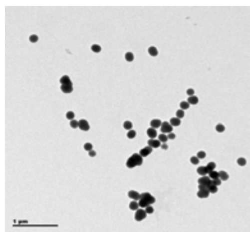
Before TEM observation



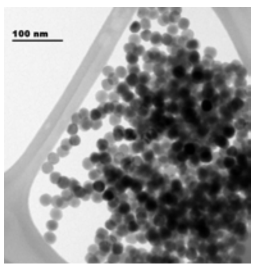


At least 1 hour under infra-red
or standard desk lamp =
removal of some hydrocarbons

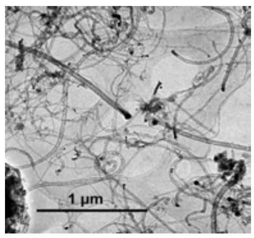
A few examples of dispersion



SiO2_Fe particles



Au particles



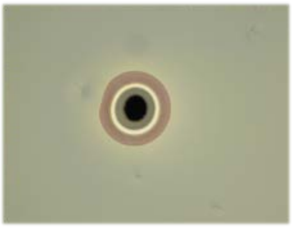
C nanotubes

TECHNIQUE: THE PLANAR VIEW

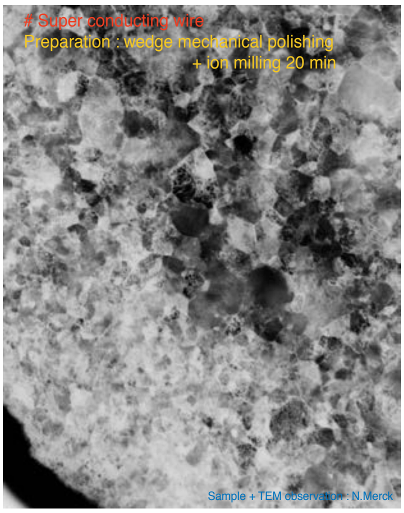
Observation parallel to the growing axis or to the preferential axis of the material

<u>Observations:</u>

- Crystalline defects
- Linear defects (dislocations, ...)
- Planar defects (twins,...)
- Study of structure and granular interfaces
- Précipitation
- ...



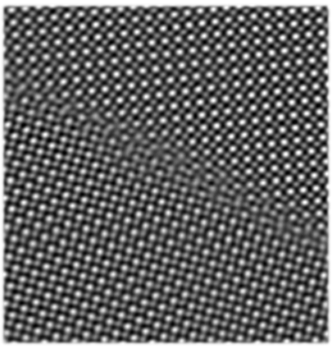
Planar view



Planar view of a supra-conducting wire
TEM image, bright field

<u>Advantage:</u> large thin area

<u>Drawback:</u> no information about different positions along the observation axis



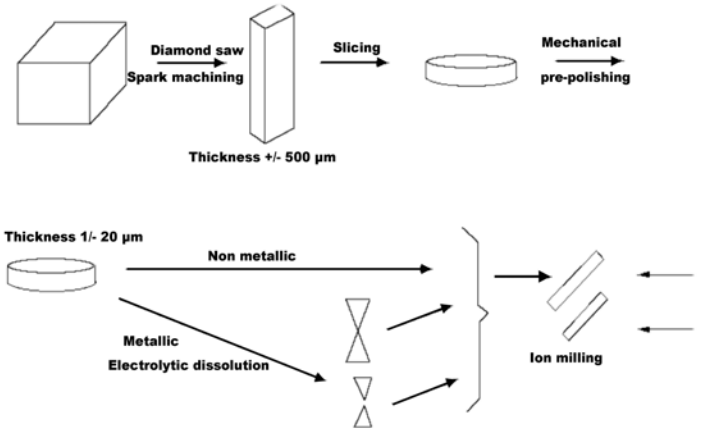
$SrTiO_3$ Grains boundary
(J.Ayache)

Planar view

Materials:
-All kind

Method:

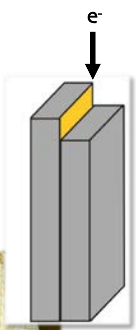
Possible defects
- Dislocations
- Irradiation
- Amorphisation of surface layers
- Modification of chemical composition



TEM Samples Preparations

TECHNIQUE: THE CROSS-SECTION

Observation perpendicular to the growing axis or to the preferential axis of the material

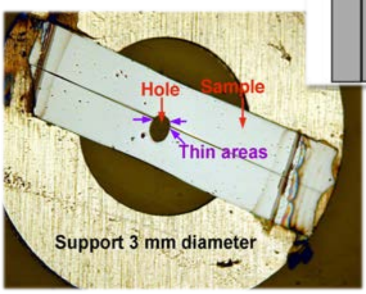


Advantage: observation of anisotropy along the growing axis

Drawback: small thin area

Observations:

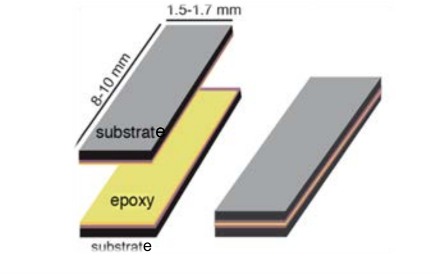
- characterization of multilayer materials
- -layers thickness measurement
- -layers and interfaces structure analysis

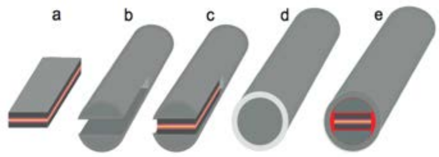


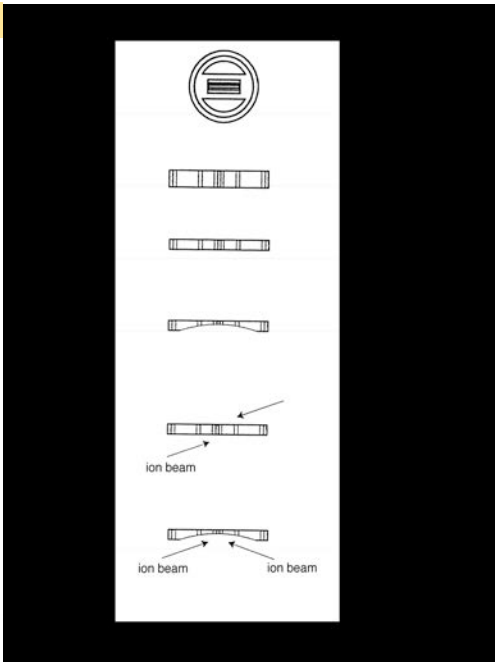
TEM Samples Preparations

Cross-section

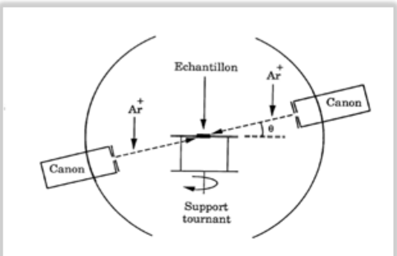
Method



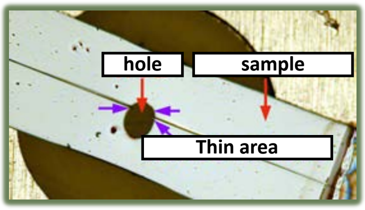


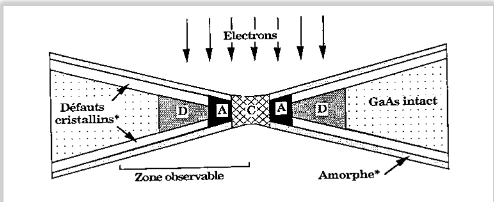


Cross-section



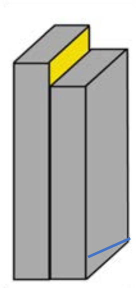
possible disposition of guns and sample in ion bombardment

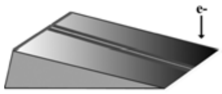


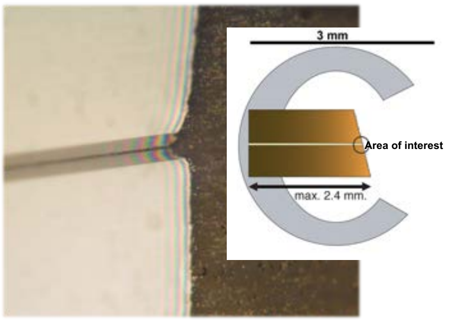


TECHNIQUE: THE TRIPOD

Mechanical thinning, in a wedge configuration, down to electron transparency or to a thickness that requires very short ion milling time



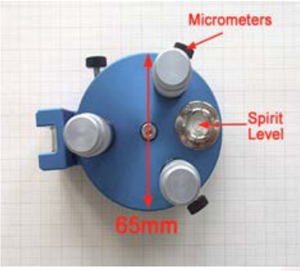


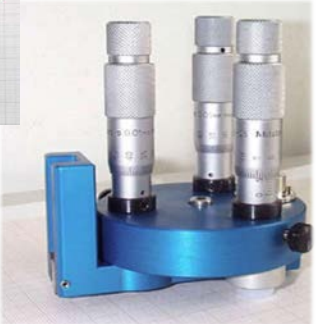


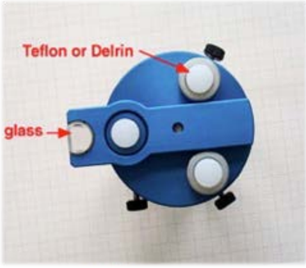
TiO_2 / Silicon,
Optical microscope, reflected light

The Tripod method

Tripod tool

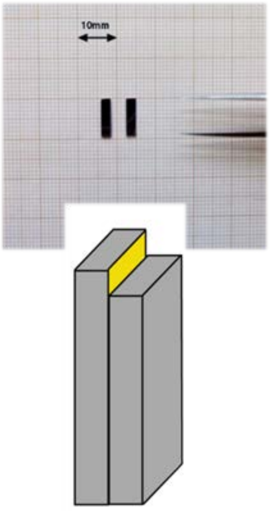


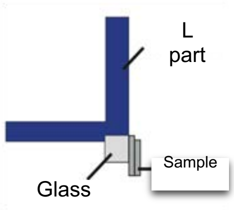


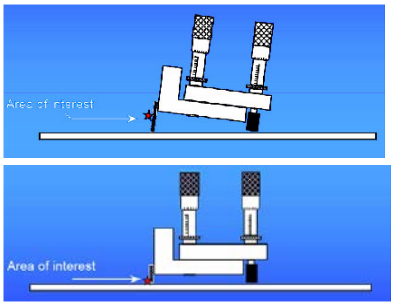


The Tripod method

Preparation for the first side polishing

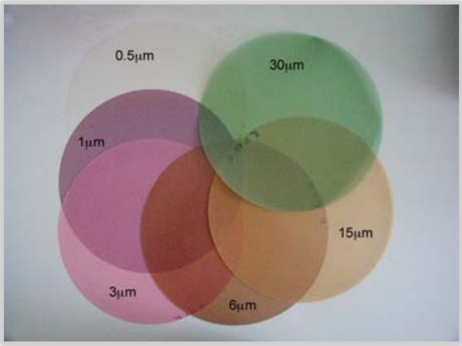


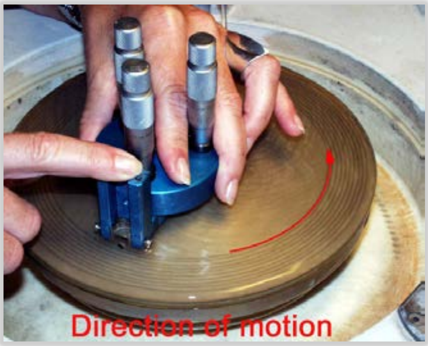




The Tripod method

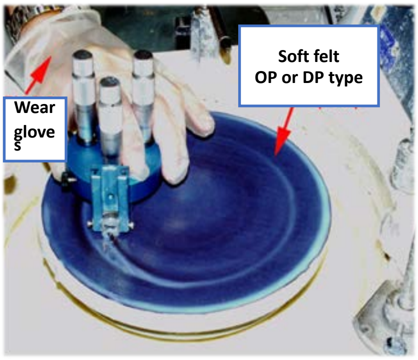
Diamond lapping films





The Tripod method

Final mechanical-chemical polishing with colloidal silica or other medium

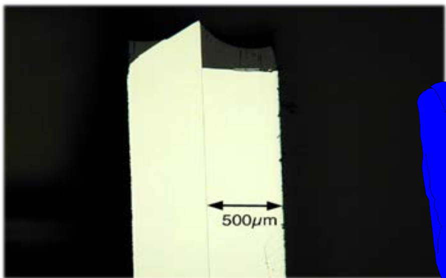


Careful removal of the colloidal silica

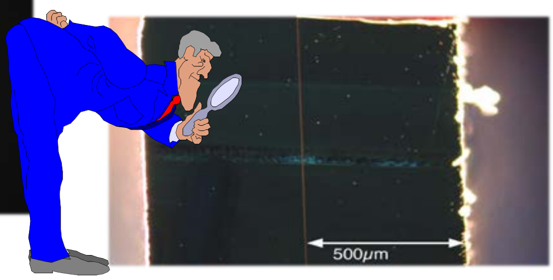
Use hand to remove silica suspension from the pad

The Tripod method

Surface after final polishing



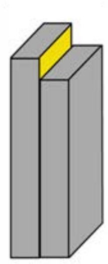
Optical microscope, bright field image

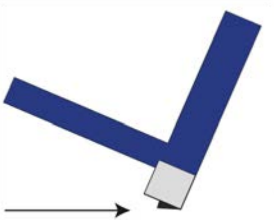


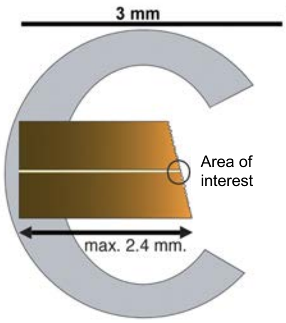
Dark field will show the best information about surface quality

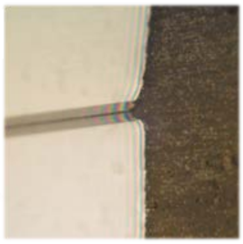
The Tripod method

Second side polishing



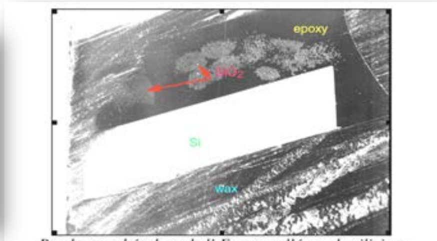






The Tripod method

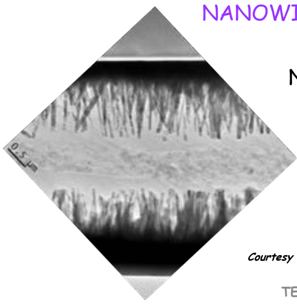
LA PREPARATION D'UNE POUDRE

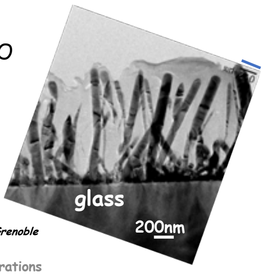


Poudre enrobée dans de l' Epoxy, collé sur du silicium

NANOWIRES PREPARATION

Nanofils ZnO



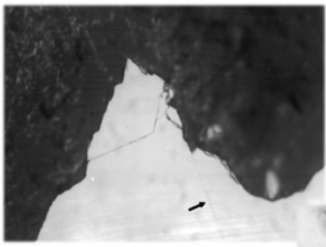


Courtesy L. Rapenne, LMPG, Grenoble

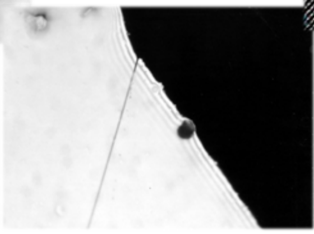
The Tripod method

SOME EXAMPLES

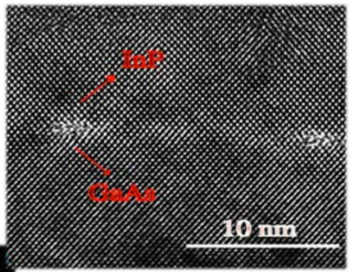
Example 1: InP/GaAs cross -section



After final polishing.
The narrow shows the glue line.



Same sample after ion milling: 1h at 5 keV, 10 min at 2 keV, 16° angle, 2 guns.
Experimental conditions

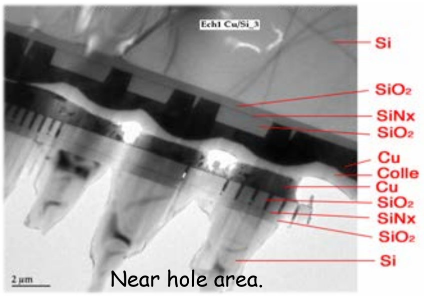


InP/GaAs interface: TEM, bright field
Image L. Sagalowicz, EPFL.

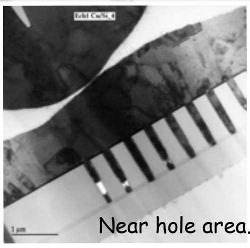
The Tripod method

Cu/SiO2/Si cross-section.

TEM observation

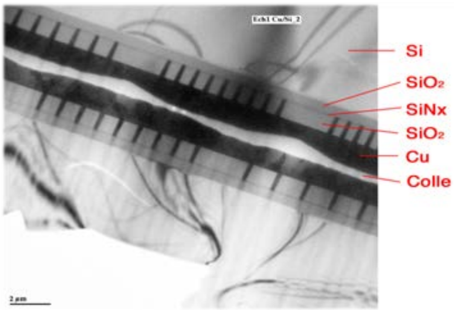


Near hole area.



Near hole area.

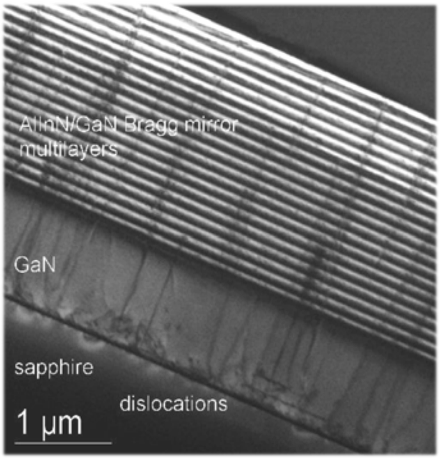
Thicker area



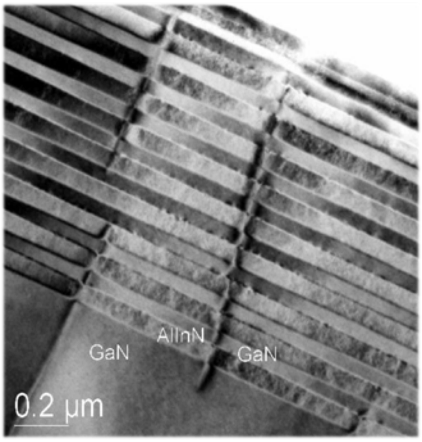
Artefact or not ?

The Tripod method

GaN on sapphire substrat



TEM image, dark field

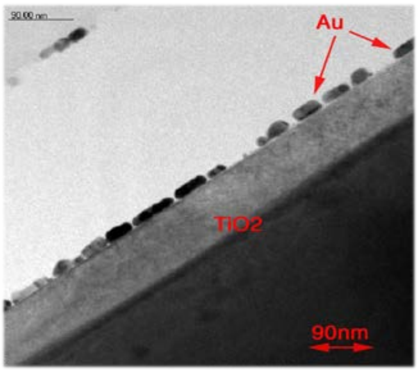


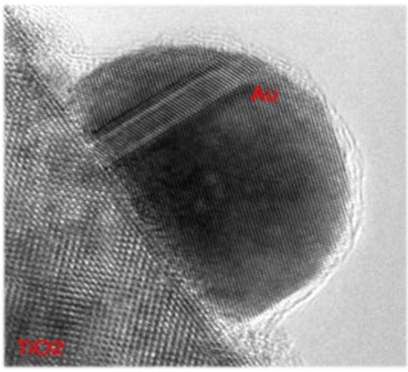
TEM image, bright field

Additional ion milling: 15 minute, 3 and 2kV, 2 guns, sectorial rotation, 5° angle

The Tripod method

Au particles /TiO2, cross-section, planar grinded





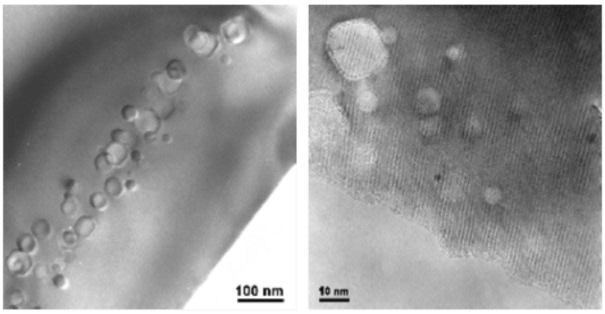
Ion milled at low incidence angle, sectorial rotation for 15 minutes

TEM images F. Cosandey, Rutgers University

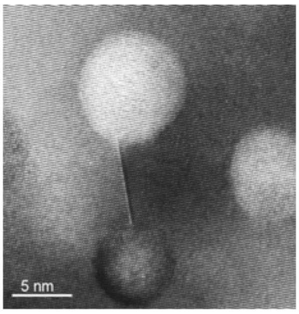
The Tripod method

TEM observation

Si sample doped with He (cavity) TEM image, bright field
No ion milling



Bright field TEM images



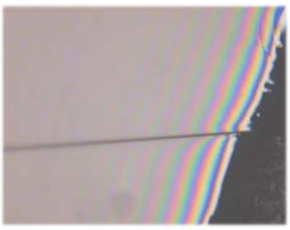
High resolution TEM image

J. Werkmann, IPCMS, Strasbourg

The Tripod method

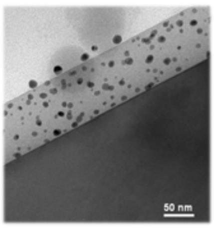
TEM observation

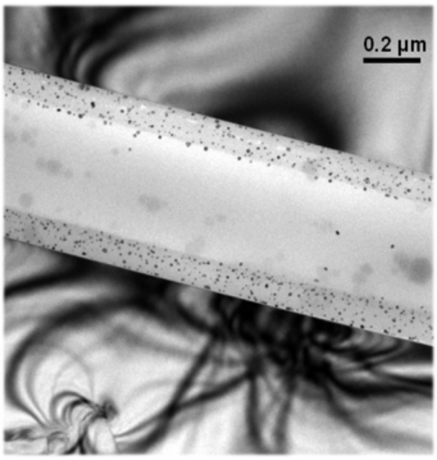
Au/SiO_2 layer on Si Substrate. No ion milling



Optical microscope, reflected light, mag. 1000x

Artefact !!!



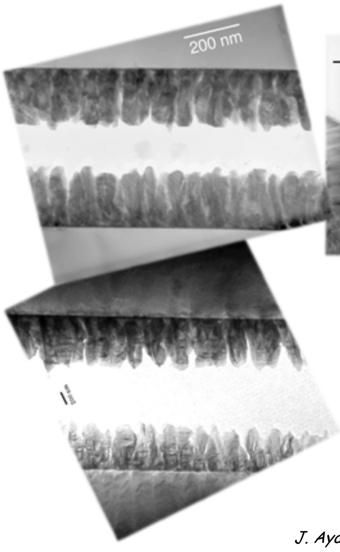


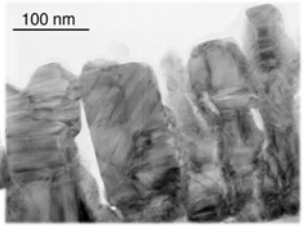
TEM bright field image

A. Schüler, S.de Chambrier, EPFL, Lausanne

The Tripod method

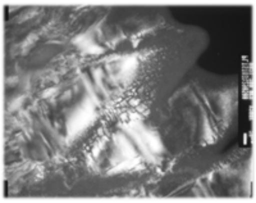
Thin $PbLaTiO_3$ ferroelectric film on $SrTiO_3$ substrate





Tripod no ion milling

Dimpler+ions



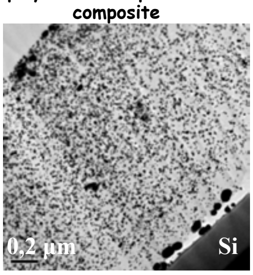
J. Ayache, CSNSM-CNRS-IN2P3, 91405 Orsay

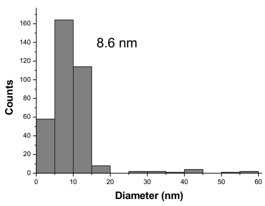
ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

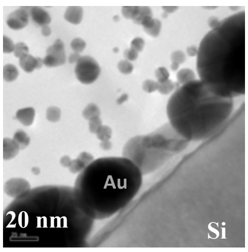
The Tripod method

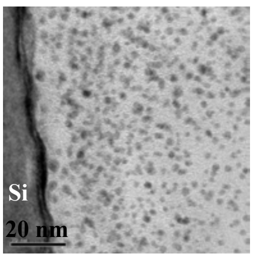
Au particles synthesized in situ in a bio-polymer film for a biological application

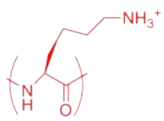
polymère-nanoparticules composite





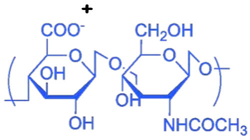






Poly(L-lysine) (PLL)
Amino acid

+



hyaluronic acid (HA)

Tripode technique allows to prepare some kind of polymer

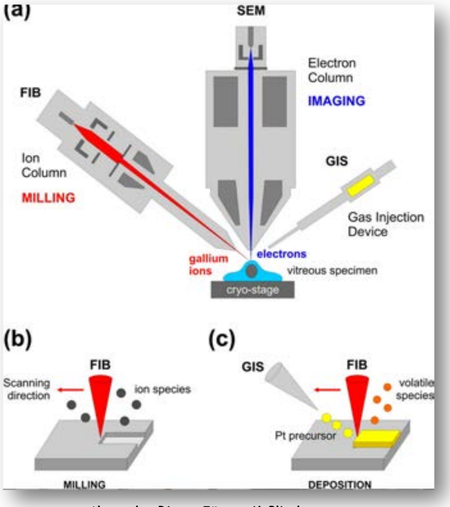
L. Rapenne présentation, Prague 2014

THE FIB (Focused Ion Beam)

Gaz: généralement Galium

Ionisation et émission par effet de champ d'atomes de gallium.

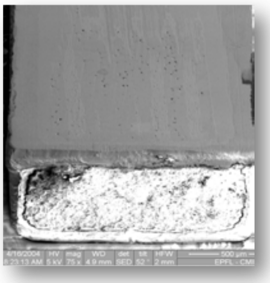
Sonde: +/- 7 nm

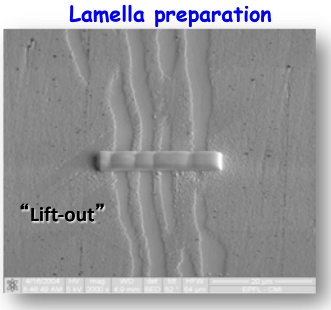


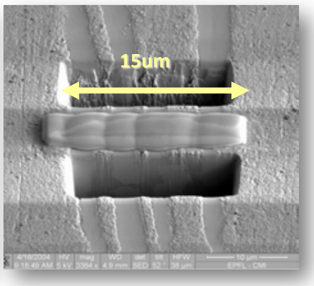
Alexander Rigort/Jürgen M.Plitzko

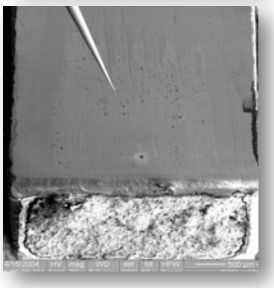
The FIB

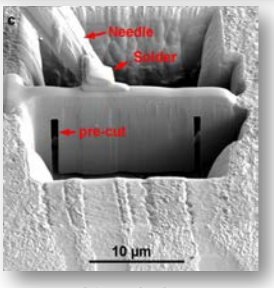
Lamella preparation









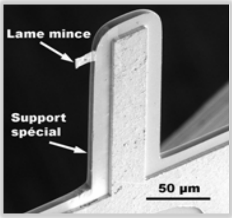


TEM grid, 3mm

58

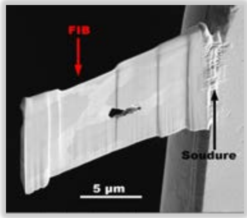
The FIB

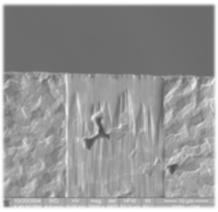
« Lift out »

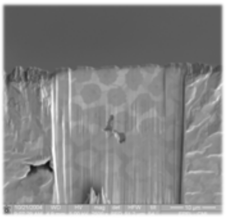


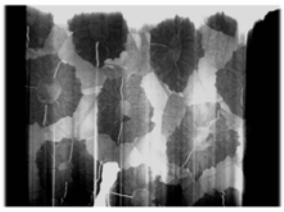
Final preparation of lamella

Nb3Sn multifilaments /bronze matrix





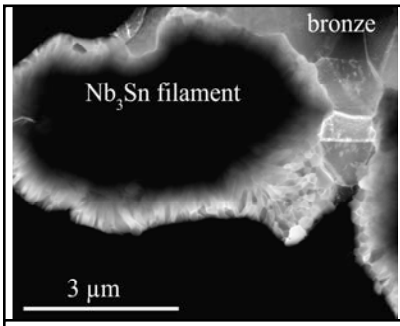


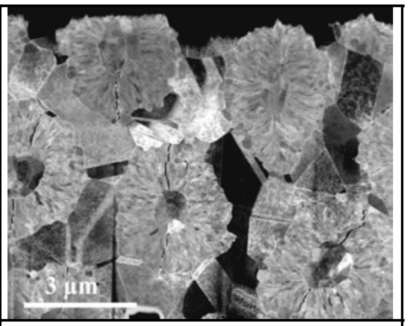


FIB prép.: F.Bobard Images MET: M. Cantoni, CIME-EPFL

The FIB

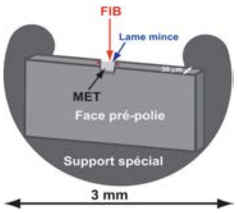
COMPARISON BETWEEN TRIPOD AND FIB TECHNIQUES



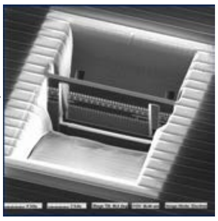


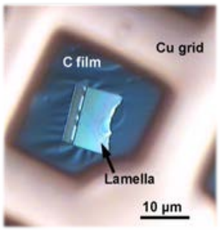
Nb_3Sn multifilament in a bronze matrix. Tripod method + Ion milling low angle (5°). Only the filaments edges and the matrix are electrons transparent M.Cantoni, EPFL-Lausanne	Same sample prepared by FIB.The lamella has a constant thickness and the entire filaments + matrix are electrons transparent M.Cantoni, EPFL-Lausanne

H bar method



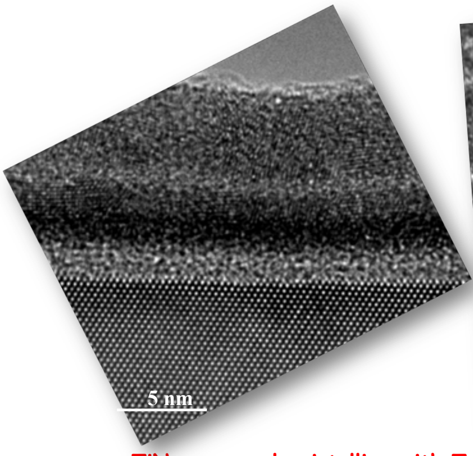
External Lift out



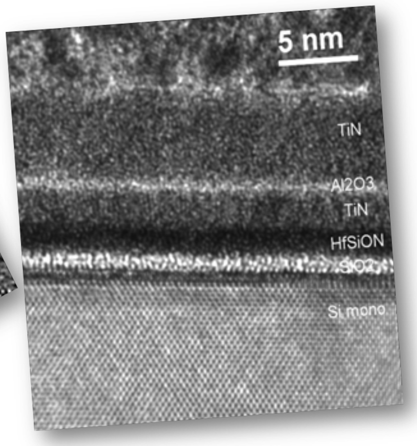


COMPARISON BETWEEN TRIPOD AND FIB TECHNIQUES

Tripod : high quality without ion milling



FIB: artifact from preparation

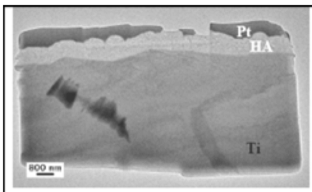
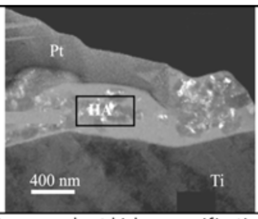


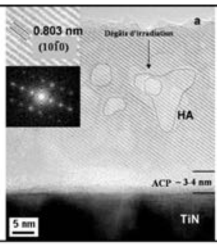
TiN seems polycristalline with Tripod andamorphous with FIB !!!

L. Rapenne présentation, Prague 2014

The FIB

COMPARISON BETWEEN FIB AND TRIPOD TECHNIQUES

	
Low magnification picture of a HA/TiN/Ti.FIB lamella.	Same sample at higher magnification. Dark field image. This FIB lamella is not thin enough to allow HREM

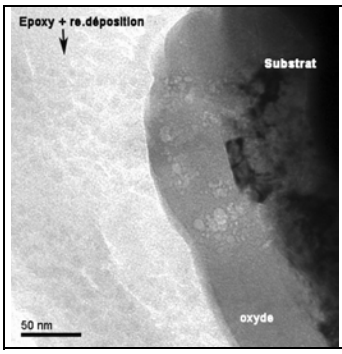
	Same sample prepared with the Tripod technique.. (the Ti substrate is not visible here) A part of the HA layer has been removed during grinding and polishing. Despite that and the iradiation defects, HRTEM observation is possible

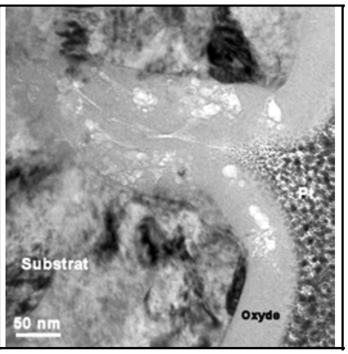
J. Ayache & al.

The FIB

COMPARISON BETWEEN TRIPOD AND FIB TECHNIQUES

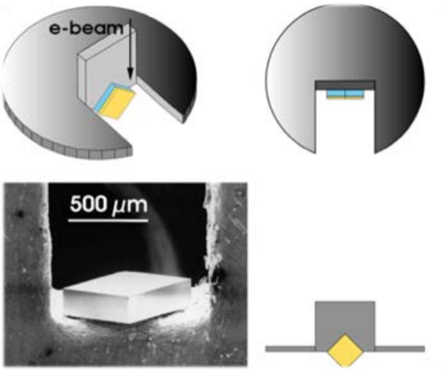
Tripod + Ion milling FIB thinninf



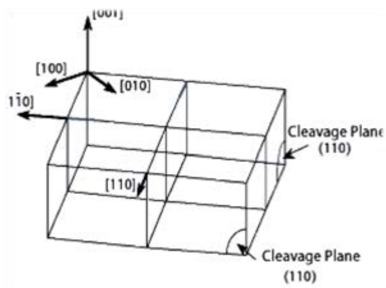


THE CLIVED WEDGE METHOD

The cleaved wedge is a monocrystalline substrate (+ layers),
dimension about 0.6/0.6 mm, obtained by 2 or 3 cleavages along designed atomic planes that give a perfect edge.
Cleavage: make use of the fact that crystals may be split along planes which are weakly bonded



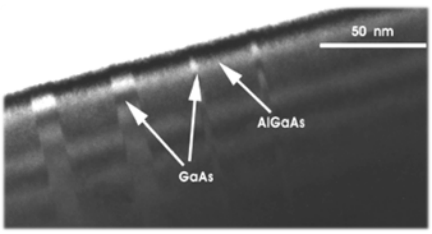
GaAs wafer e.g



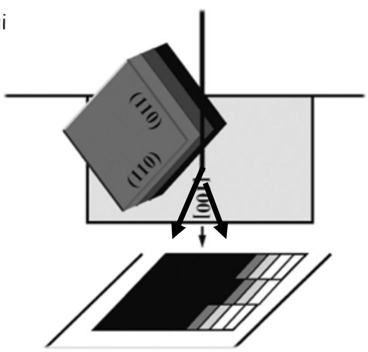
The cleaved wedge

Origin of the contrast:

- The observed contrast is linked to the sample thickness and its chemical composition
- As for a cleaved wedge, the sample thickness is accurately known, the chemical composition can be deduced from the thickness fringes profile
- The electron beam is parallel to the layer interfaces
- The layer interfaces are put forward by a discontinui of the fringes (perpendicularly to the wedge edge)

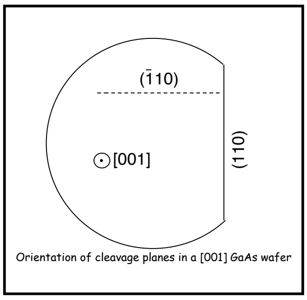


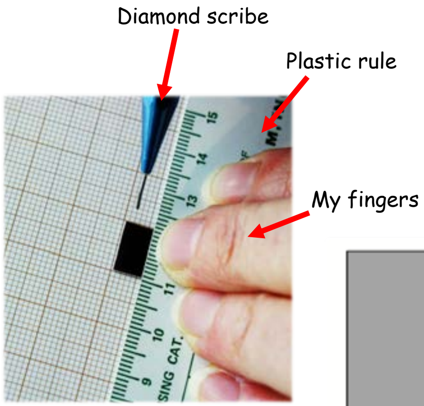
P.A. Buffat, J.D. Ganière, EPFL.



The cleaved wedge

How to cleave ?

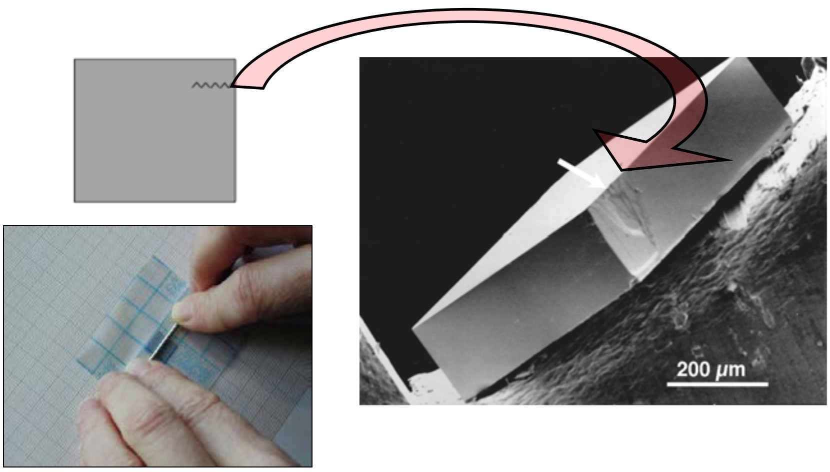




• Partially scribe the sample with a fine diamond scriber **onto layers surface**

The cleaved wedge

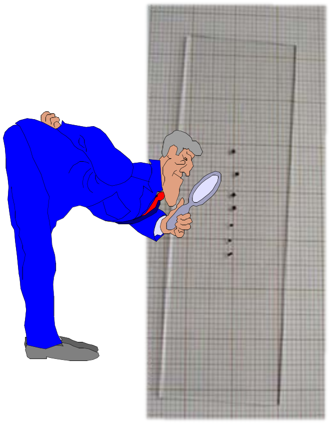
How to cleave ?

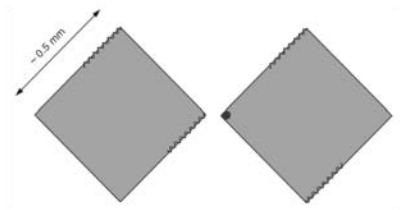


The cleaved wedge

How to cleave ?

- Turn it over, roll on the cylinder on the full sample
- Small samples must be turned over very carefully



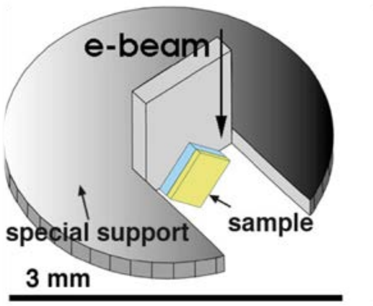


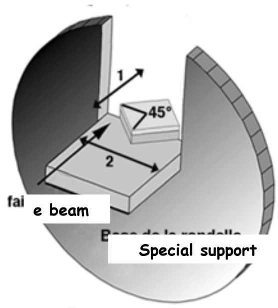
- Select good wedges using optical microscope

The cleaved wedge

Positioning the wedge cleaved sample on the grid support

Take care of:
1) Eucentricity
2) Left and right
3) Sample orientation should be about 45° with respect to the direction of the electron beam

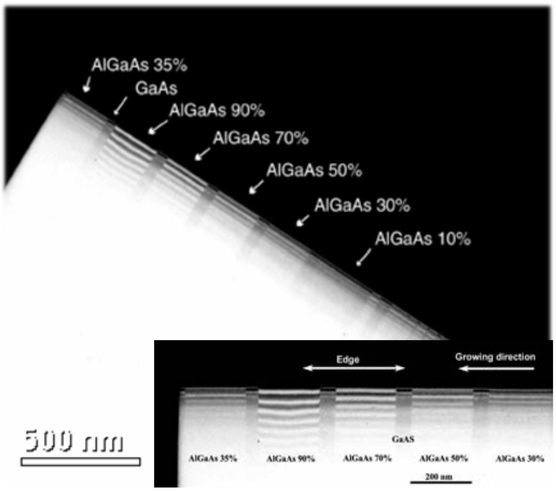




The cleaved wedge

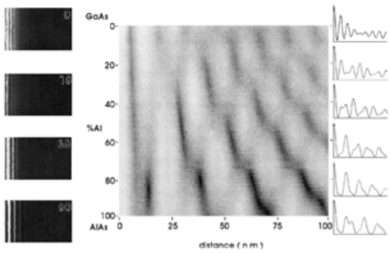
Few examples

AlGaAs/ GaAs



Chemical composition measurements for AlGaAs/GaAs interfaces

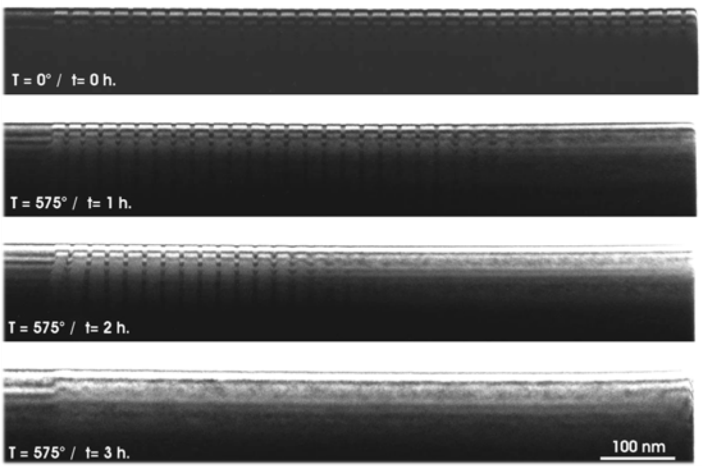
Calculations (JEMS) can be done to interpret the thickness fringes profile in a semi-quantitative way.



The cleaved wedge

Few exemples

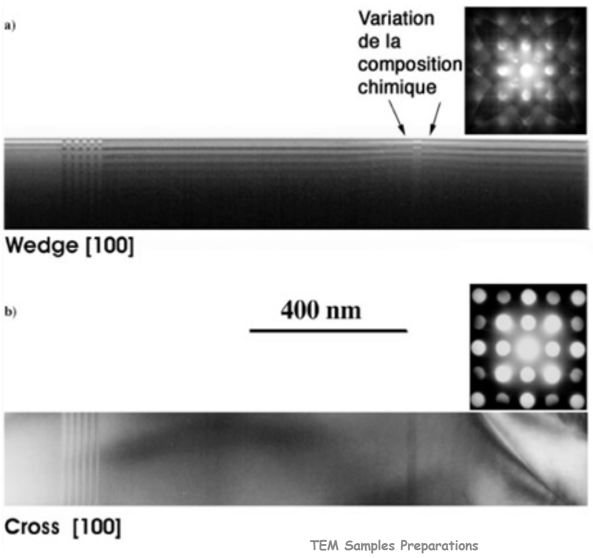
AlGaAs/ GaAs



Quantum wells degradation in AlGaAs by Zinc diffusion from the surface
J.D. Ganière, EPFL

The cleaved wedge

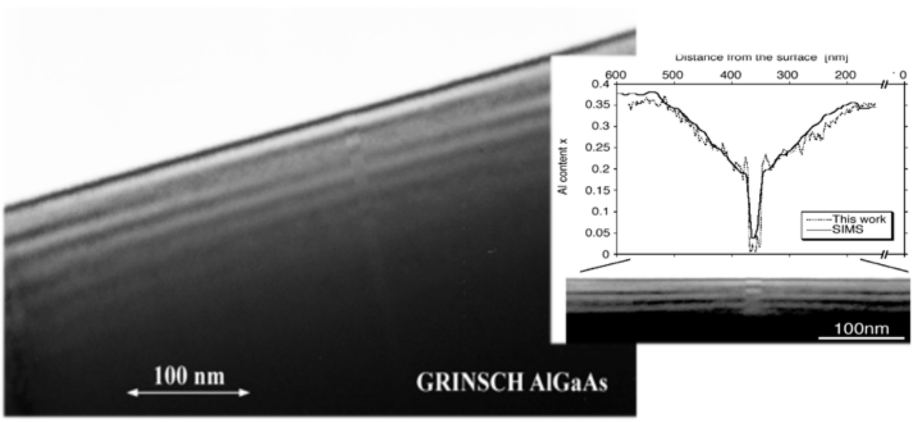
Comparison between a cleaved wedge and an "usual " cross-section of quantum wells AlGaAs/GaAs AlGaAs/GaAs



P.A. Buffat, J.D. Ganière EPFL

The cleaved wedge

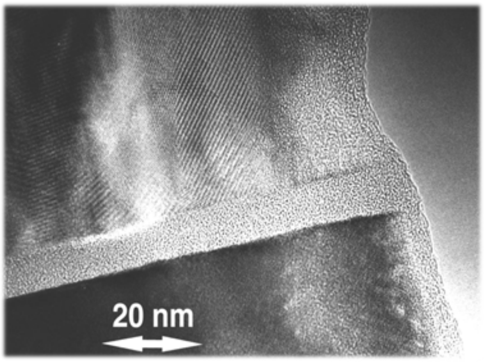
Few examples



P.A. Buffat, EPFL-CIME, Lausanne

The cleaved wedge

Si substrate



poly $Si/SiO_2/Si$ [111]

Cr_Ti 50-50/Si substrate
Supposed to be columnar grown
EELS confirmed Cr-Ti layers

! Cleaving planes are different from the one of GaAs

THE ULTRAMICROTOMY

Slicing of the sample to a constant thickness of 20-200 nm, using a diamond knife, carried out at room temperature

THE CRYO-ULTRAMICROTOMY

Slicing of the sample to a constant thickness of 50-200 nm, using a diamond knife, carried out at low temperature

THE ULTRAMICROTOMY, CRYO-ULTRAMICROTOMY

Observations

- Statistic of particles size
- EDX chemical analysis, EELS chemical analysis (needs thin constant thickness)
- Material microstructure
- Cross-section or plan view of materials that cannot be ion milled, mechanically or electrolytically thinned
- Heterogeneous materials, multilayer
- Small diameter fibres or tips
- Powders (metallic or not)

Materials

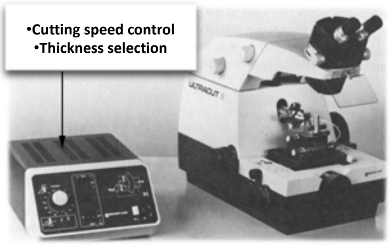
- Polymer /polymers with additional compounds
- Catalyst
- Geological
- Biomaterial
- Wood
- Metal

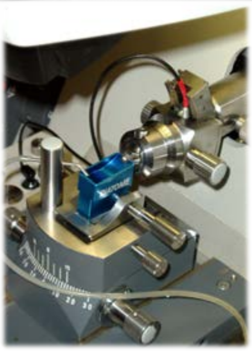
Drawback:

- Deformation of the sample due to compression or/and cracks
- Dislocations
- Shape modification
- …

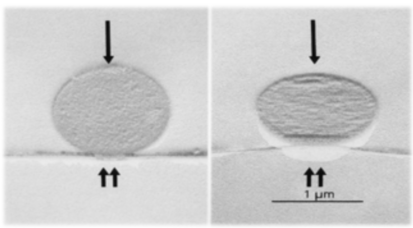
Ultramicrotomy/Cryo-Ultramicrotomy

The ultramicrotome





The knives



PS spheres in Epon cuted with a 15° knife

Cuted with a 45° knife

Courtesy Helmut Gnaegi, Diatome

Ultramicrotomy/Cryo-Ultramicrotomy

Method

- Reduce the sample size if needed
- Embeed the sample if needed

Particles	Multilayer	Bulk

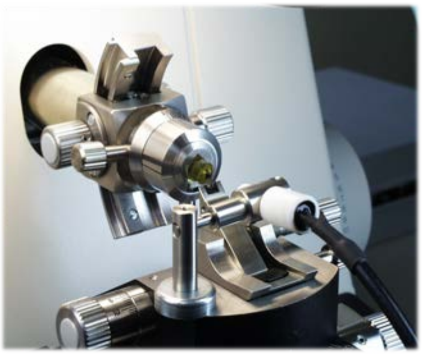
Important: the embedding resin should have the same hardness/ softness as the sample

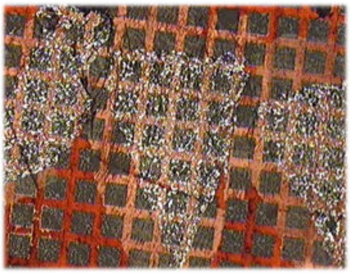
For porous material: embedding under vacuum or infiltration-embedding

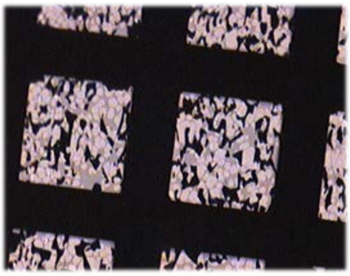
Ultramicrotomy/cryo-Ultramicrotomy

Resin	Knive angle	Compression
Lowicryl K4M	45° 35°	24% 12%
EM-Bed	45° **35°**	20% **14%**
Spurr's (hard grade)	45° 35°	17% 10%
LR White (hard grade)	45° 35°	13% 8%
Epofix	45° 35°	11% 6%

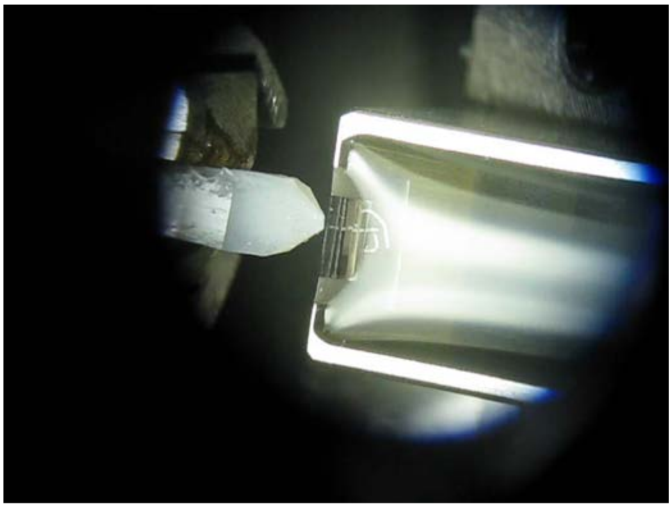
Ultramicrotomy





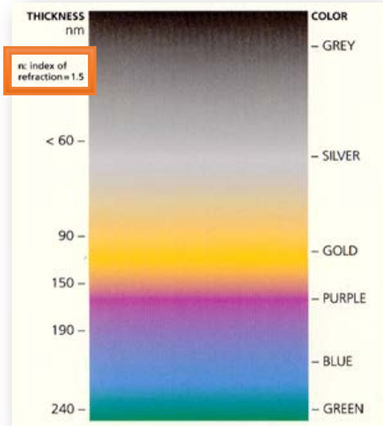


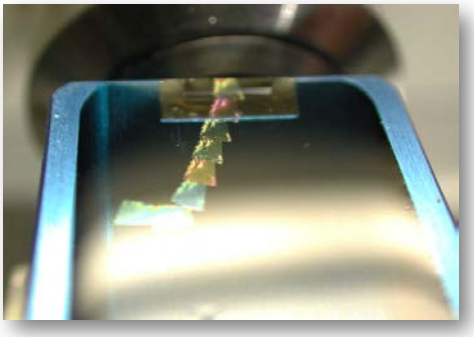
Ultramicrotomy/cryo-Ultramicrotomy



Ultramicrotomy/cryo-Ultramicrotomy

The colour of the resin gives information such as:
- Homogeneity of the section thickness
- Thickness of the section





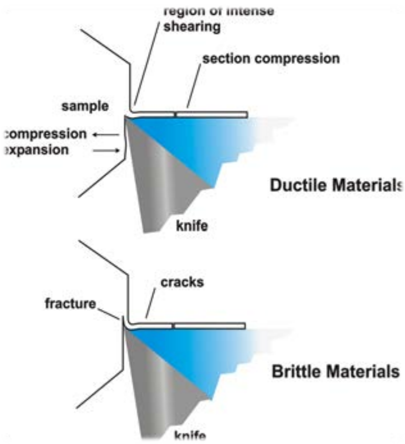
Isabelle Pignot-Paintrand, UPR-CNRS 5301

Ultramicrotomy/cryo-Ultramicrotomy

Cutting the sample to the desired (or possible) thickness

- Section thickness 40 - 50nm
- Sectioning speed 0.2mm/sec

Induced damages

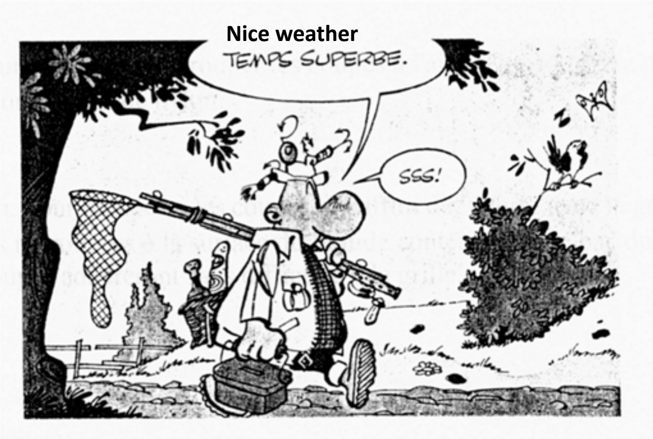


Diatome, Helmut Gnaegi presentation

Ultramicrotomy

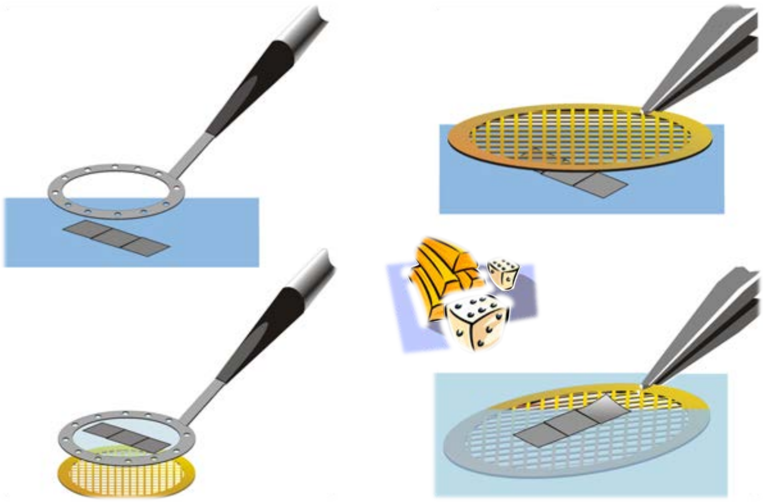
Thin slices are done!

Now we have to fish them !!!



Ultramicrotomy

Sections collection

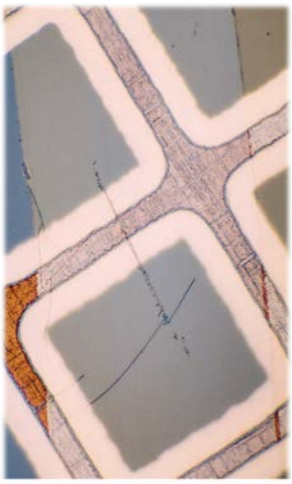


Diatome, Helmut Gnaegi presentation

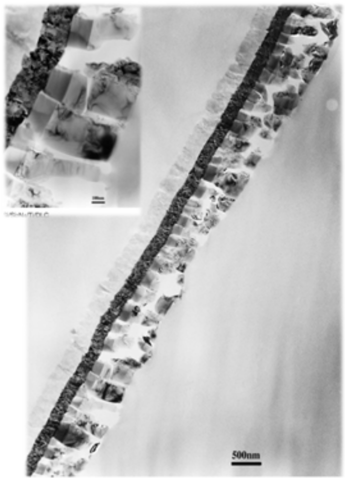
Cryo-Ultramicrotomy

Ultramicrotomy/cryo-Ultramicrotomy

Results



Optical microscope, transmited light



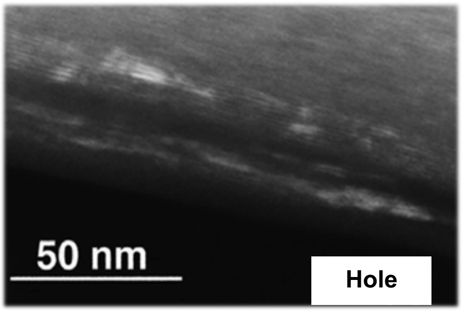
TEM, bright field image

Diatome, Helmut Gnaegi presentation

Ultramicrotomy/cryo-Ultramicrotomy

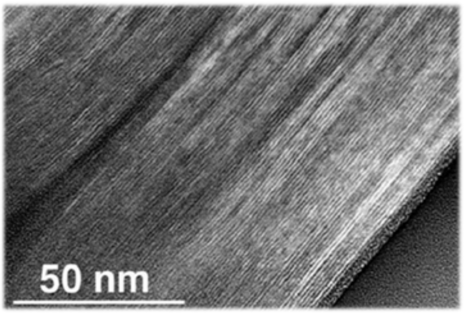
Some examples

Mica sample
Mechanical thinning followed by ion milling did not give a suitable result



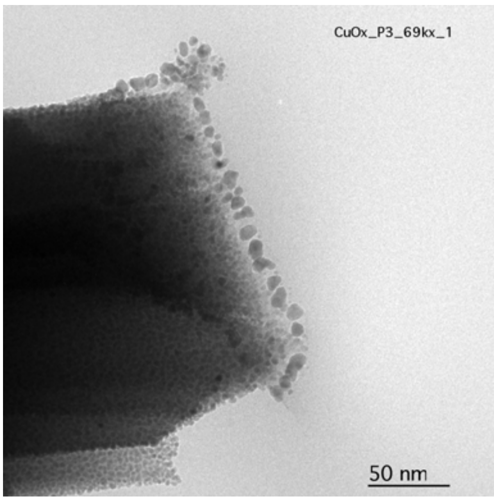
Final thinning by ion milling, optimized for high speed abrasion

Same sample, prepared by ultramicrotomy



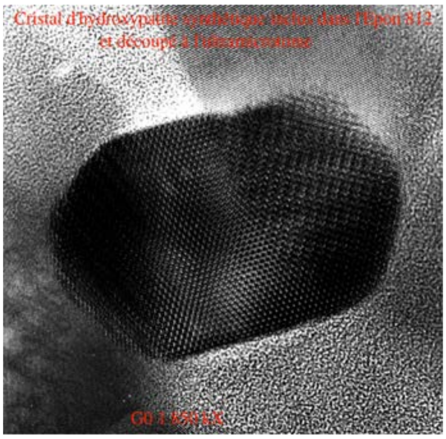
Ultramicrotomy/cryo-Ultramicrotomy

Some examples



Ultramicrotomy/cryo-Ultramicrotomy

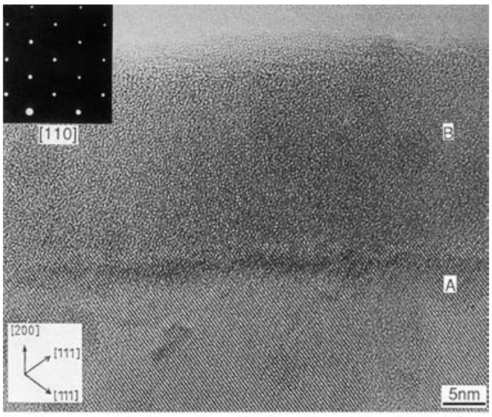
Synthetic hydroxyapatite needle



Sectioned perpendicular to its length (*C* axe)

J.Hemmerlé, INSERM U 424, Strasbourg

Amorphous Si/Si

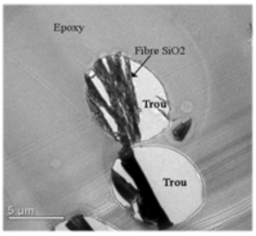


Ion beam deposited amorphous Si+ film (B) on a Si substrate (A)

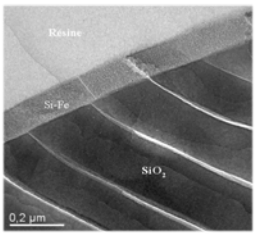
Diatome, Helmut Gnaegi presentation

Ultramicrotomy/cryo-Ultramicrotomy

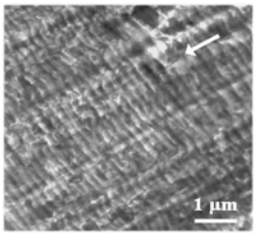
Drawback



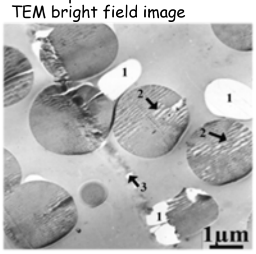
Si-Fe/SiO_2 particles Embedded in epoxy
TEM bright field image



Higher magnification



Tin sample
TEM bright field image



Carbon particles in epoxy resin
TEM, bright field image
J.Ayache, UMR-CNRS-IGR, Villejuif

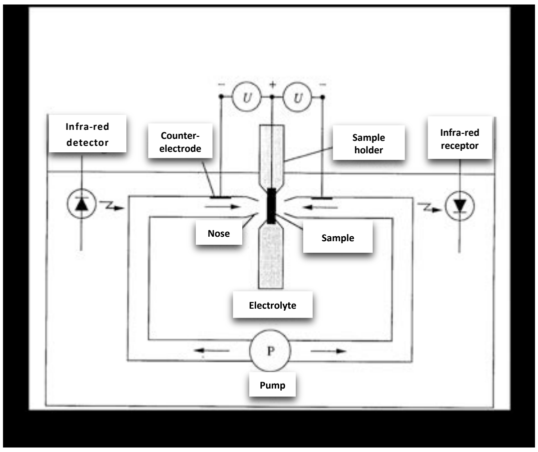
ELECTRO-CHEMICAL POLISHING (JET POLISHING)

Effect of electrolytical polishing is due to anodic dissolution of a pre-polished surface in an electrolyte bath

- A bath for the electrolyte
- A continuous current source
- An anode (the sample)
- A cathode

Observations:

- dislocations (orientation)
- Twins (macles)
- Grain boundaries
- precipitates and phases
- ….

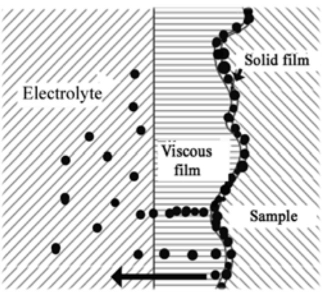


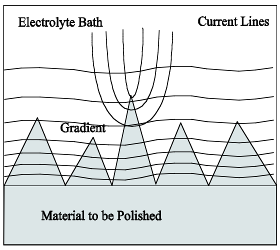
ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

Electro-chemical polishing

Principle

Electrolytic bath: - acid or alcaline solution
- viscous solution
- ionisable liquid





Current density is proportional to the concentration gradient: lower in crevasses, stronger on projections = lavelling of surface roughness.

Electro-chemical polishing

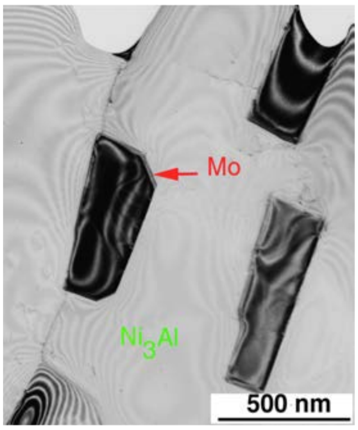
Material must be an electrical conductor

- Metal and alloys, one or more phases
- Carbides
- Graphite
- Some oxides
- Some composite materials with metallic matrix and fine particles

Advantage: non destructive method

Drawback: may cause preferential etching, dissolution of interface or some phases

Possible damages: eventually residual oxidation layer at the sample surface



Ni_3Al matrix with Mo fibres, TEM dark field image

CHEMICAL POLISHING

Same principle as electro-polishing but more difficult to control
The solutions are more reactive and used at higher temperature

- Observations:
- Similar to the planar view or cross section

- Materials:
- Metals
- Semiconductors
- oxides
- glass
- ...

Method:

- Cutting and/or cross section procedure
- Polishing onto soft tissue, specific for chemical addition
- Chemical thinning until hole

Advantage: possible for non conductive materials

Drawback: dislocations, etching (etch pits)

Possible damages: residual oxidation layer at the sample surface

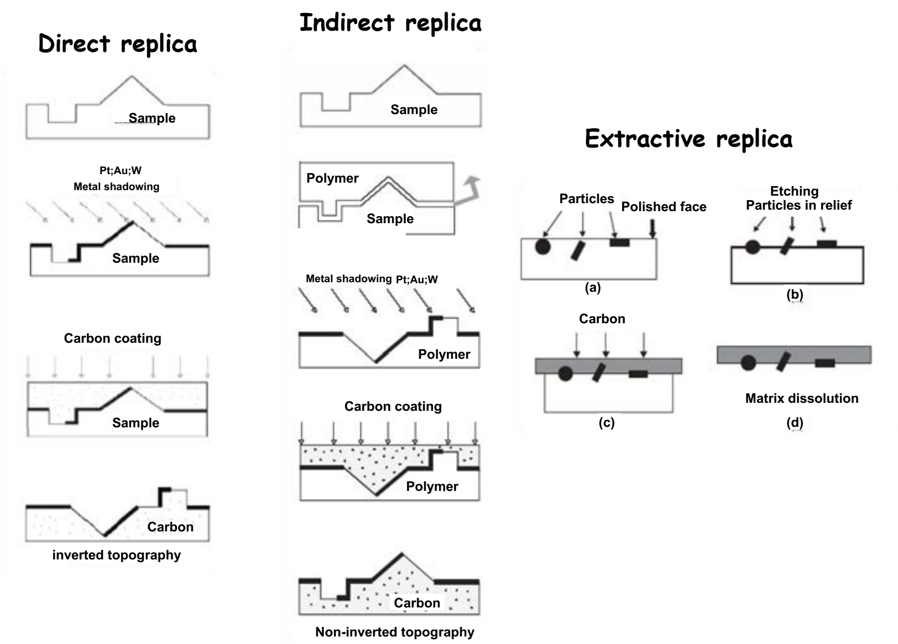
THE REPLICA METHOD

The replica is the reproduction of the sample surface topography.
It is done by polymer, carbone or oxide film deposition onto the surface sample, which is then removed from the sample and observed into TEM.

Observations
•Multiphase materials
•Surface topography
•Second phase particles analysis obtained by the extraction replica method
•Radiation sensitive samples

Method
•Film deposition, either « soft » polymer or in a solvant solution
•Carbon film deposition for non conductive samples

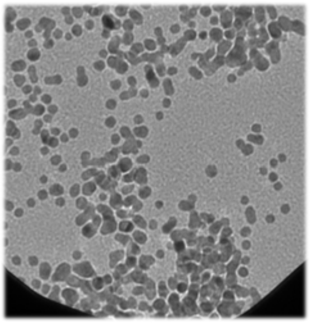
•Pulling away the film from the sample by its immersion into solvant, by pulling out or by chemical etching of the sample.
•Mounting the replica onto a 2.3 mm or 3 mm support grid



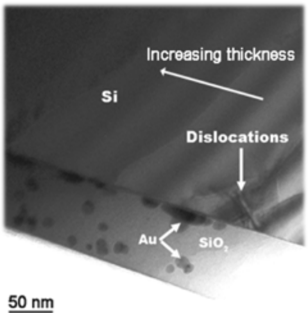
Comparison between techniques:

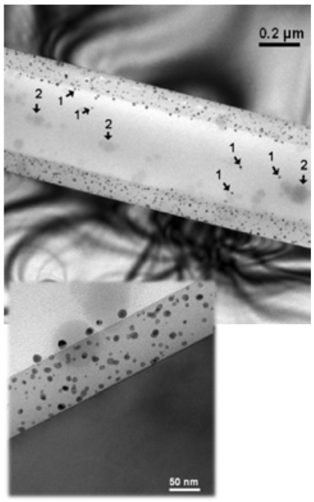
Fine particles dispersion- Cleaved wedge and Tripod method

Au particles



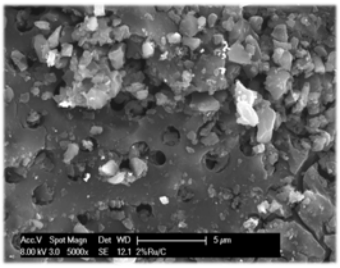
SiO_2/Au particles on Si substrate





Four techniques for one sample !!!!!!

Ru on C particles

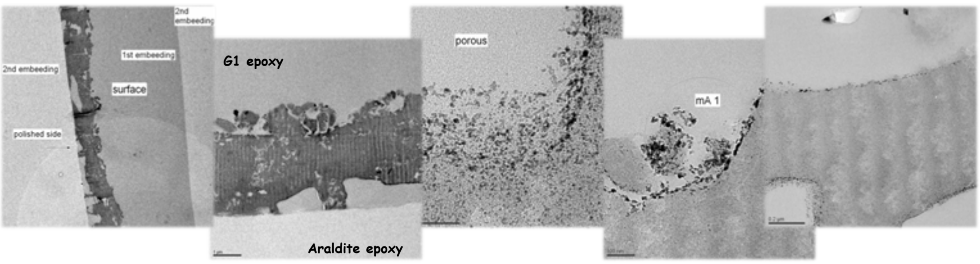


Embedded with G1 (Epotek) resin

Tripod polished (wedge)
Glued on a grid

Embedded in Araldite Epoxy

Sliced with an Ultramicrotome



Don't forget that sample preparation is also...

... like cooking!!!...