Sample preparation for Transmissdion Electron Microscopy

Corrado Bongiorno: *IMM-CNR, Catania*

- *Electrons and matter*
- *The right thickness*
- *How to obtain a good sample*
- *Mechanical preparation*
- *Drop casting*
- *Ultramicrotome*
- *New developments*

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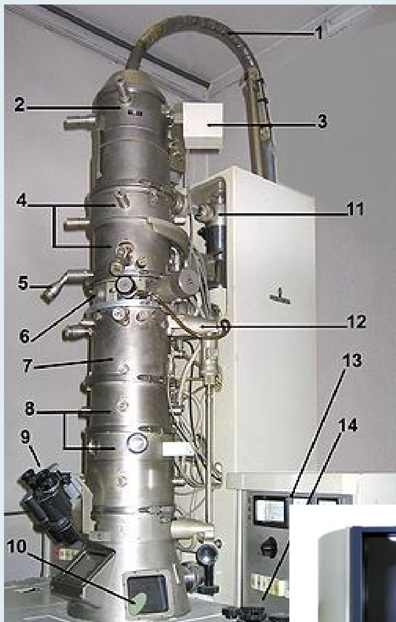
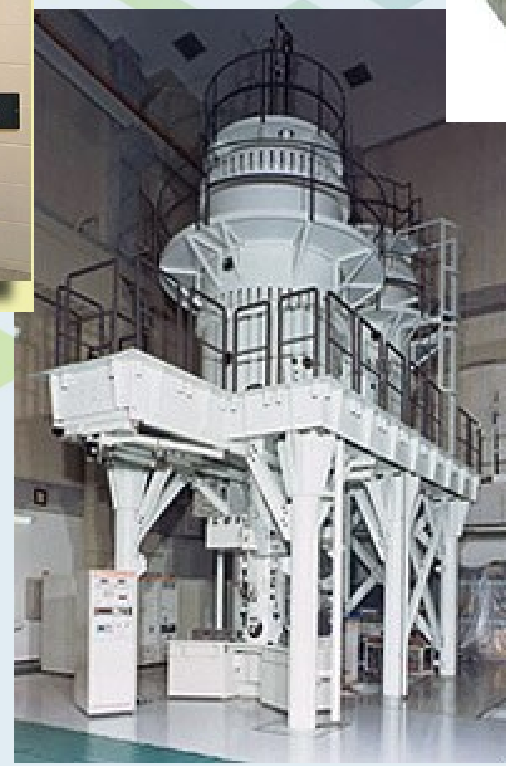
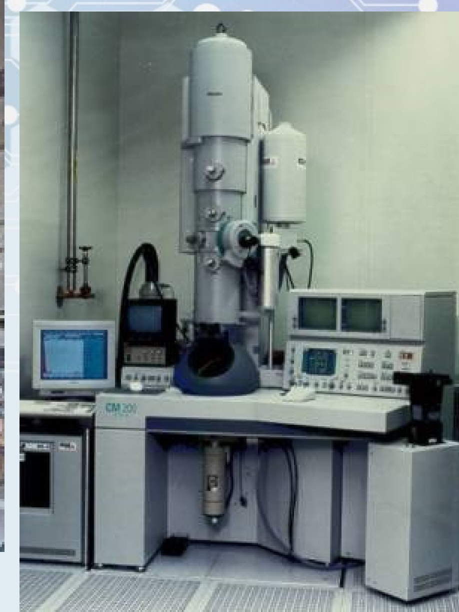
Transmission Electron Microscopy

Very powerfull technique

Main drowback is the sample preparation







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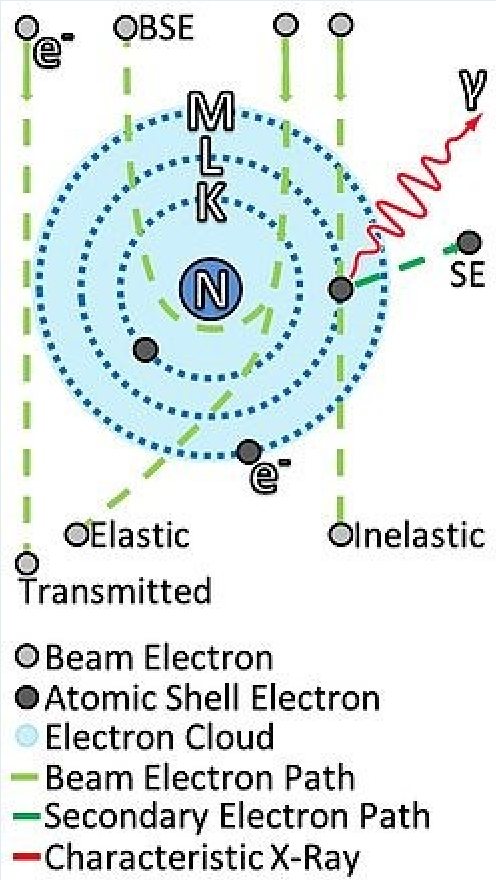
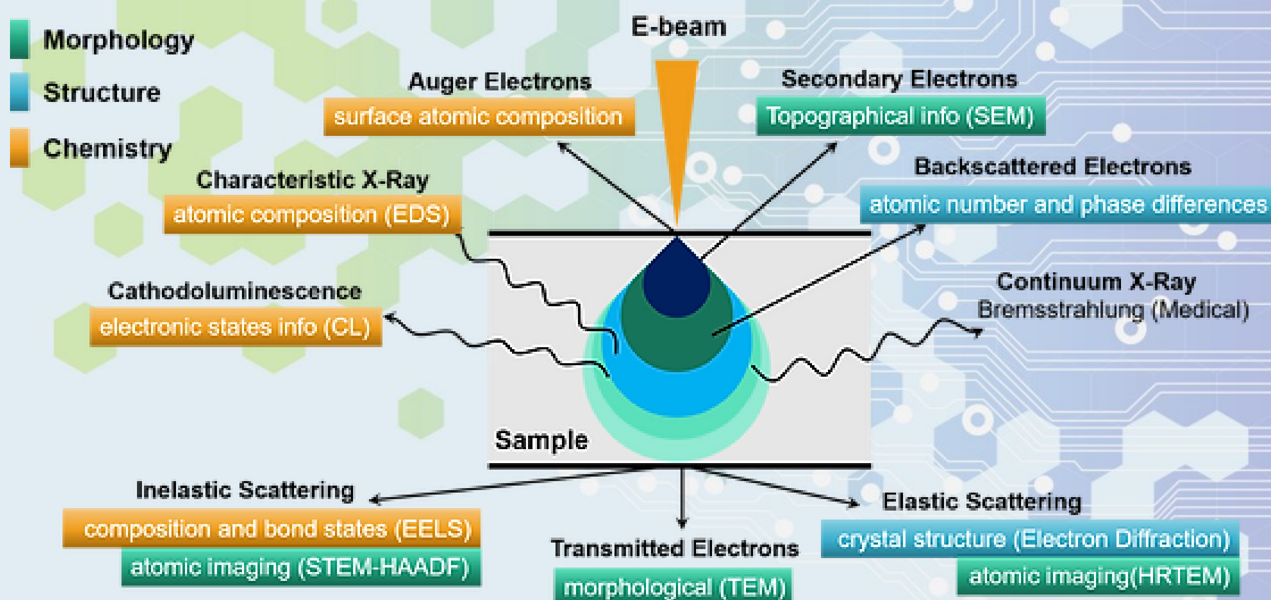
Electron/matter interaction



Every interaction has different cross-section.

Cross-section change with incident energy and materials



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Electrons are strongly absorbed

The right thickness

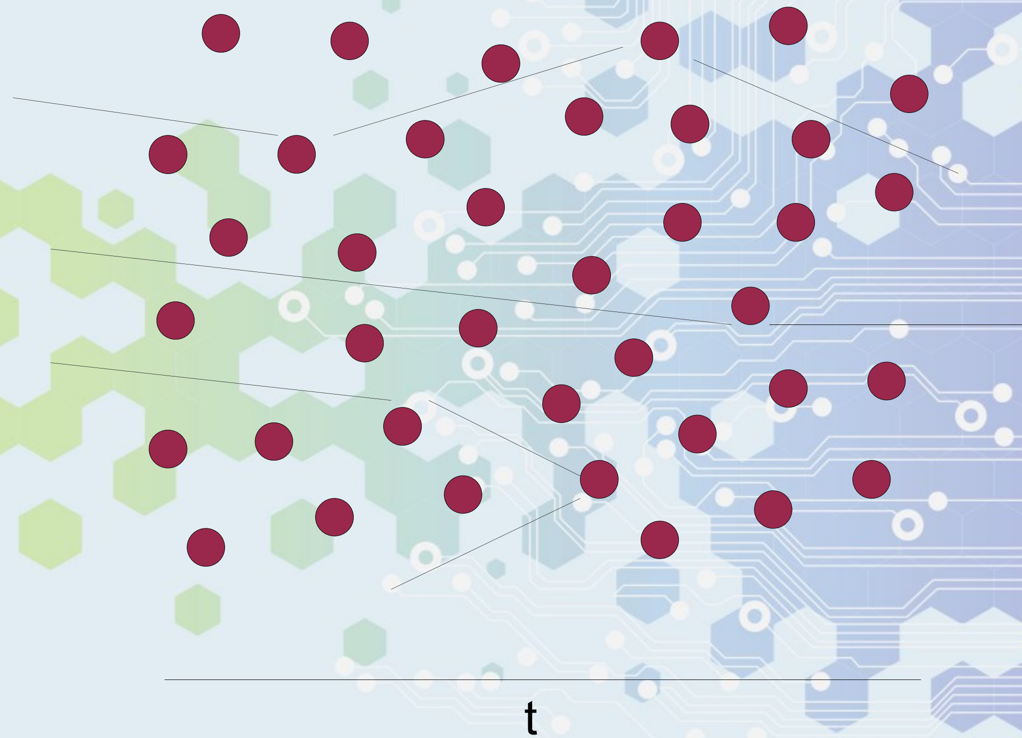
Average distance between two scattering events

λ = mean free path

Increasing thickness increase noise/signal ratio

In TEM I(t) should be very similar to I_0

$t \sim \lambda$



$$I(t) = I_0 \exp(-t / \lambda(E))$$

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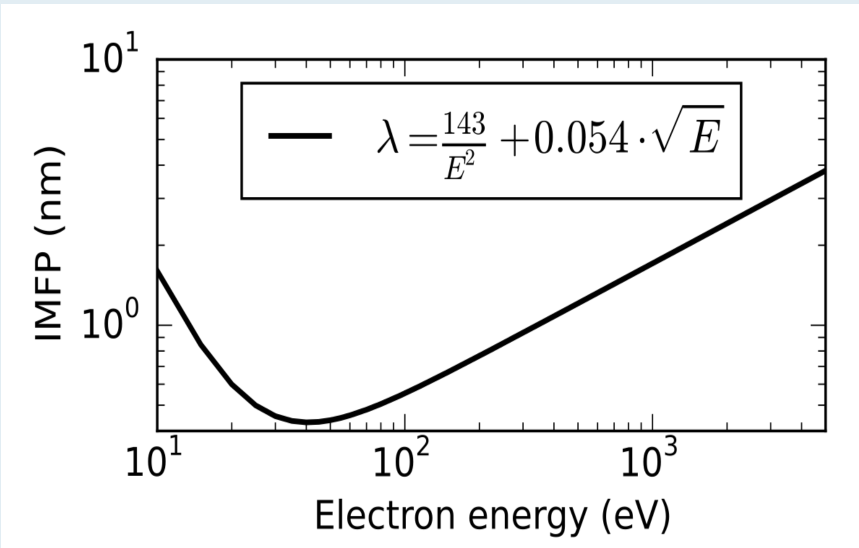
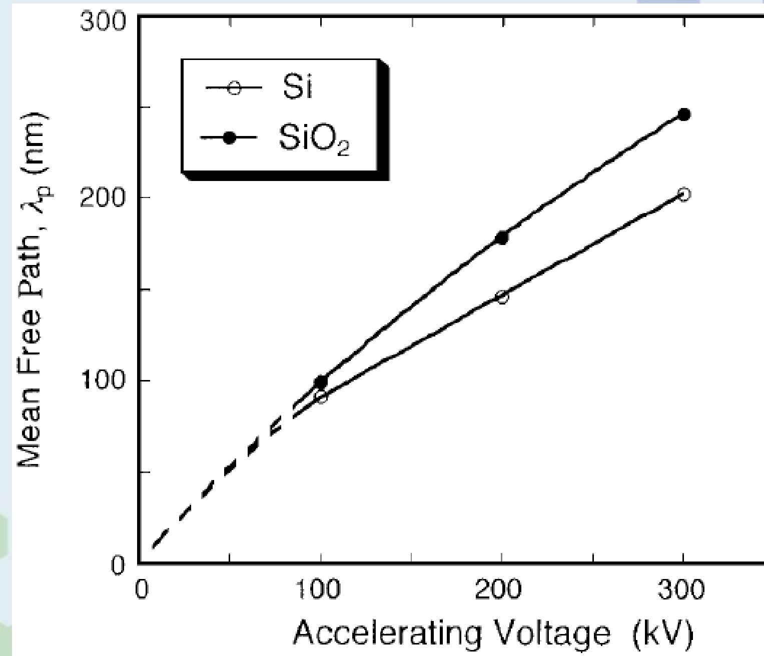
λ change with incident electron energy
Change with materials

Larger is λ more «transparent» is the material.

The right tickness is λ dependent





t/λ is the parameter used for different materials comparison.

t/λ=1 is usually a good sample.
t/λ=1 in silicon at 200keV means t=150nm
t/λ=1 in gold at 200keV means t=40nm

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- HR-TEM Image: Multiscattered elastic electrons

(t/λ =0.3 - 1.5)

- HR-STEM image: High angles scattered electrons

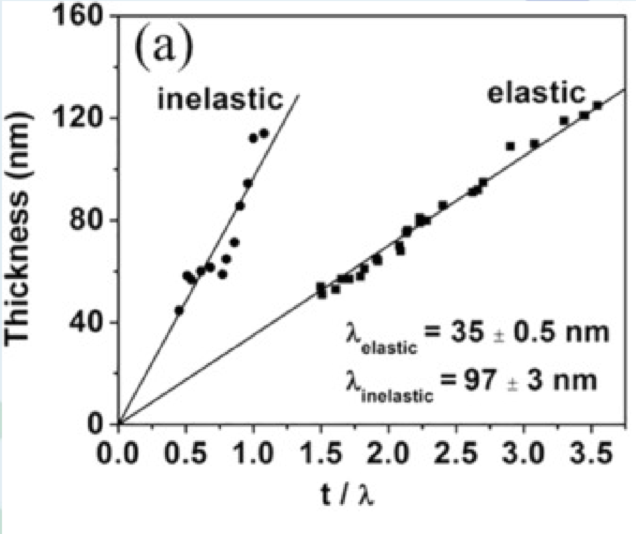
(t/λ =0.3 - 2)

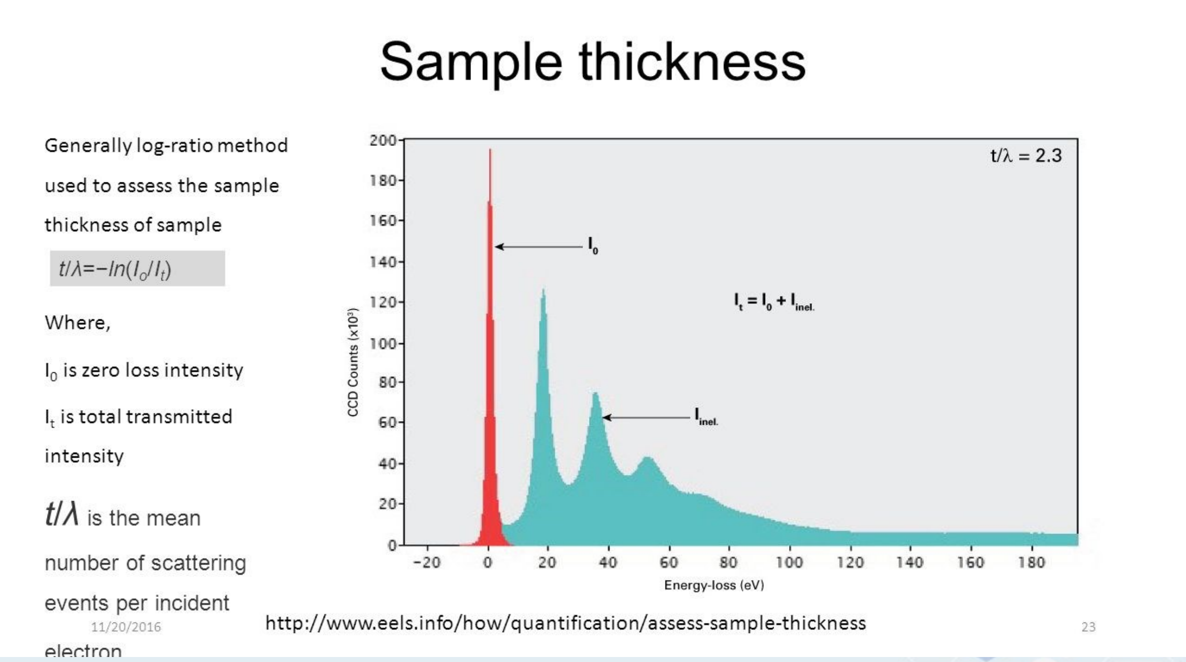
- EDX: secondary emissions due to inelastic events

(t/λ =1- 3)

- EELS: Inelastic transmitted single scattered electrons

(t/λ =0.3 - 1)





t/λ EELS calculation

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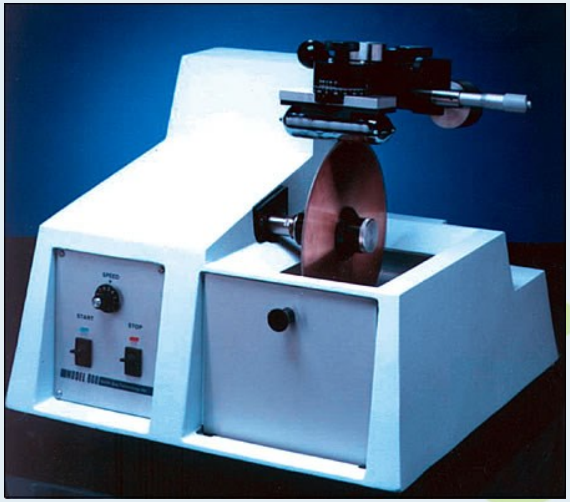
How to obtain a good sample



Polishing/grinding
Systems

Focused Ion Beam

Ultramicrotome

Cryo-EM

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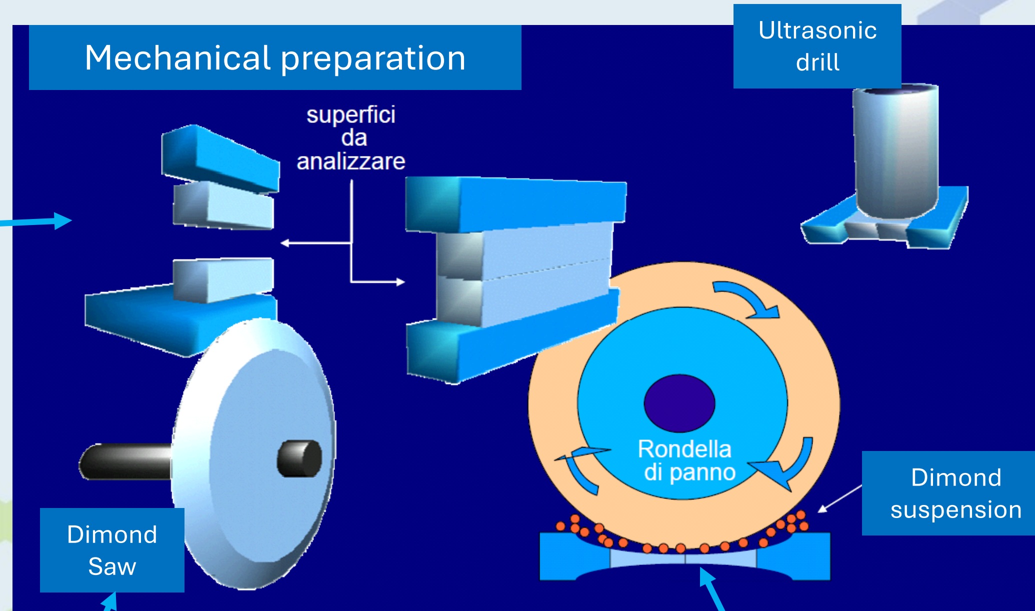
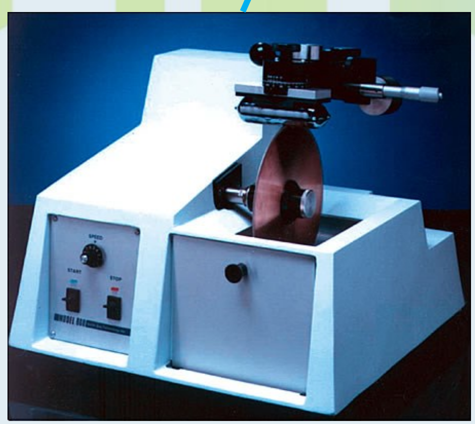
Grinder





Dimond saw

Dimple grinder

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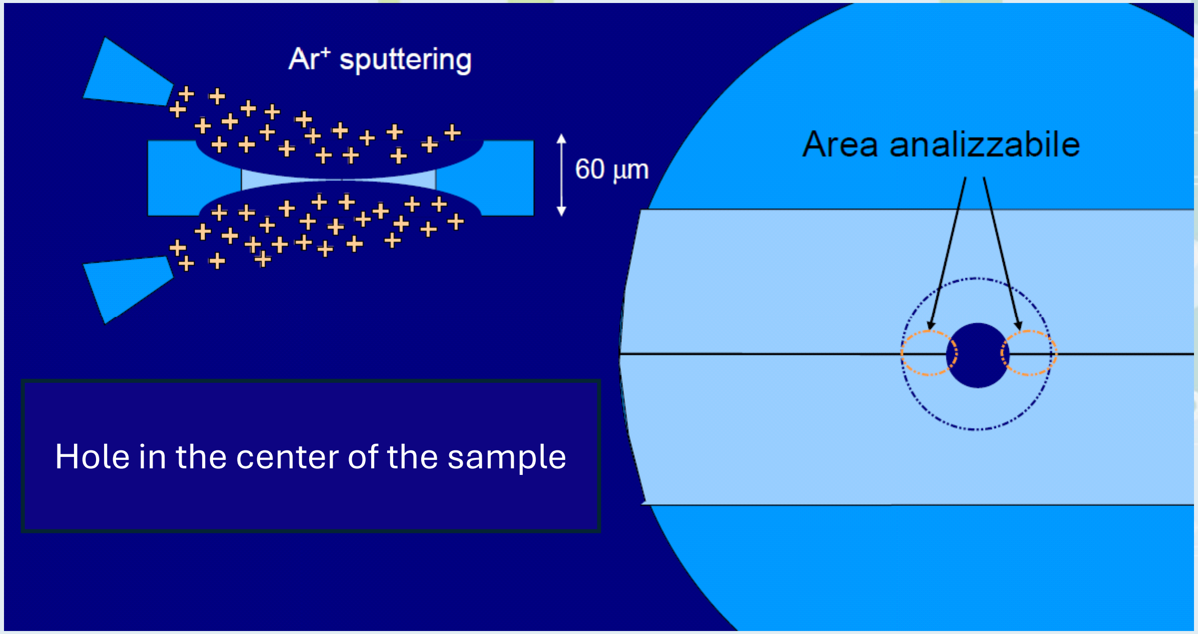
Final Ion milling process

From 5 to 0.1 keV Ar^+ ion beam

Less then 1mm wide beam

Local high temperature (300°C)

Precision Ion Polishing Sistem



Preparation time:
3h – 2 days
Suitable Area:
20-60um, high depth

Only for planar and large area distribution sample

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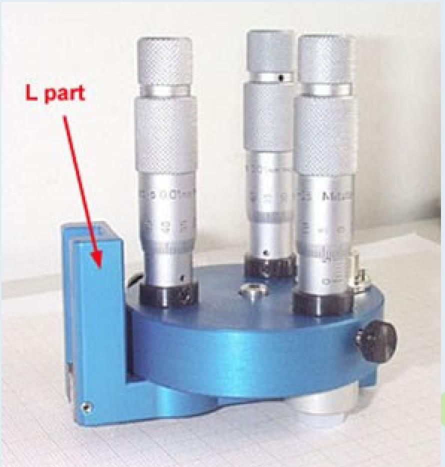
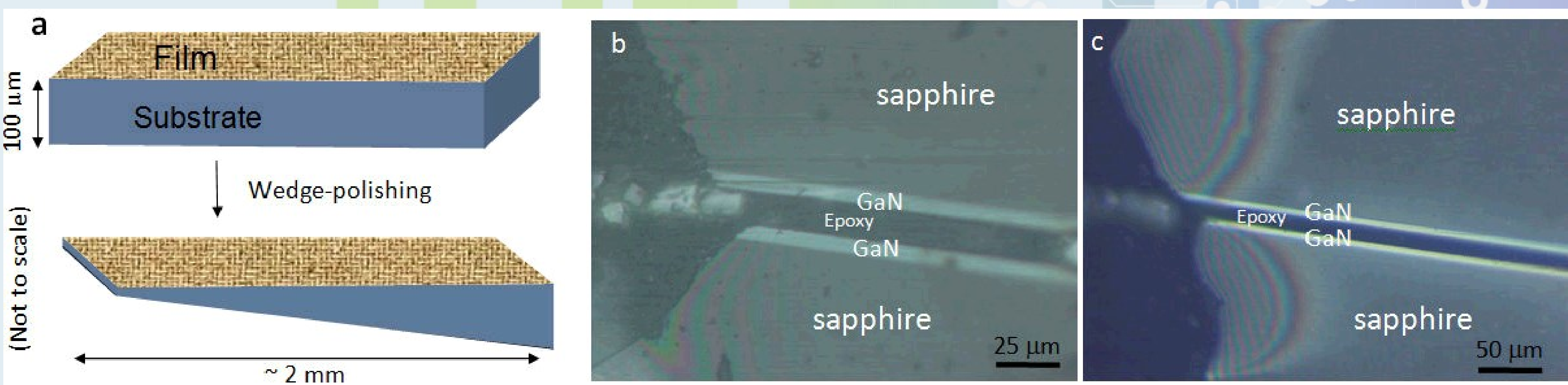
Wedge mechanical preparation



Tripod

Dimond films



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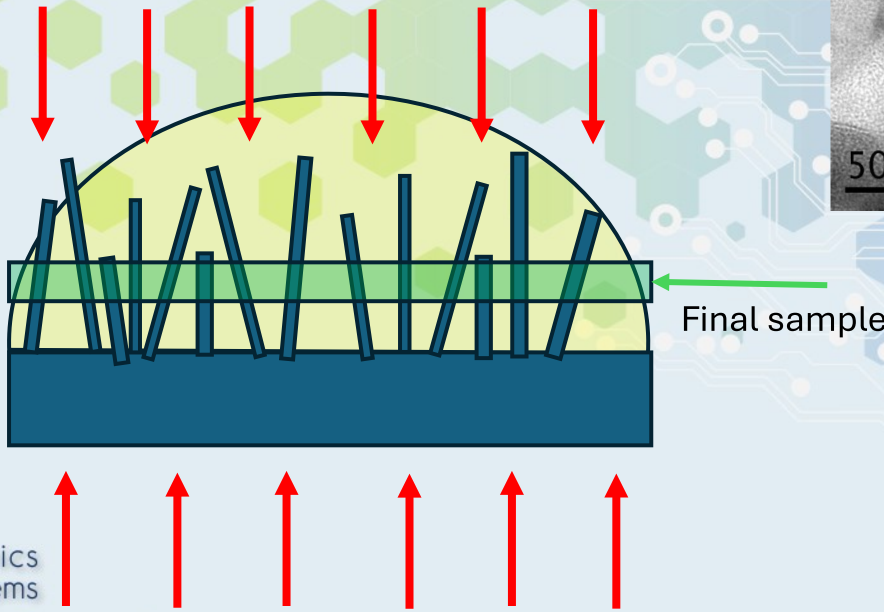
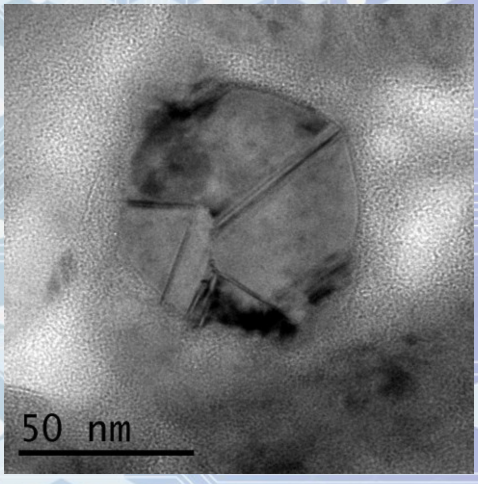
Metallic cilinder for wax embedding

- Wax envelope of large particles
- Ion milling surface remover.
- Chemical etching for junction delineation
- Deposition on pre-prepared samples





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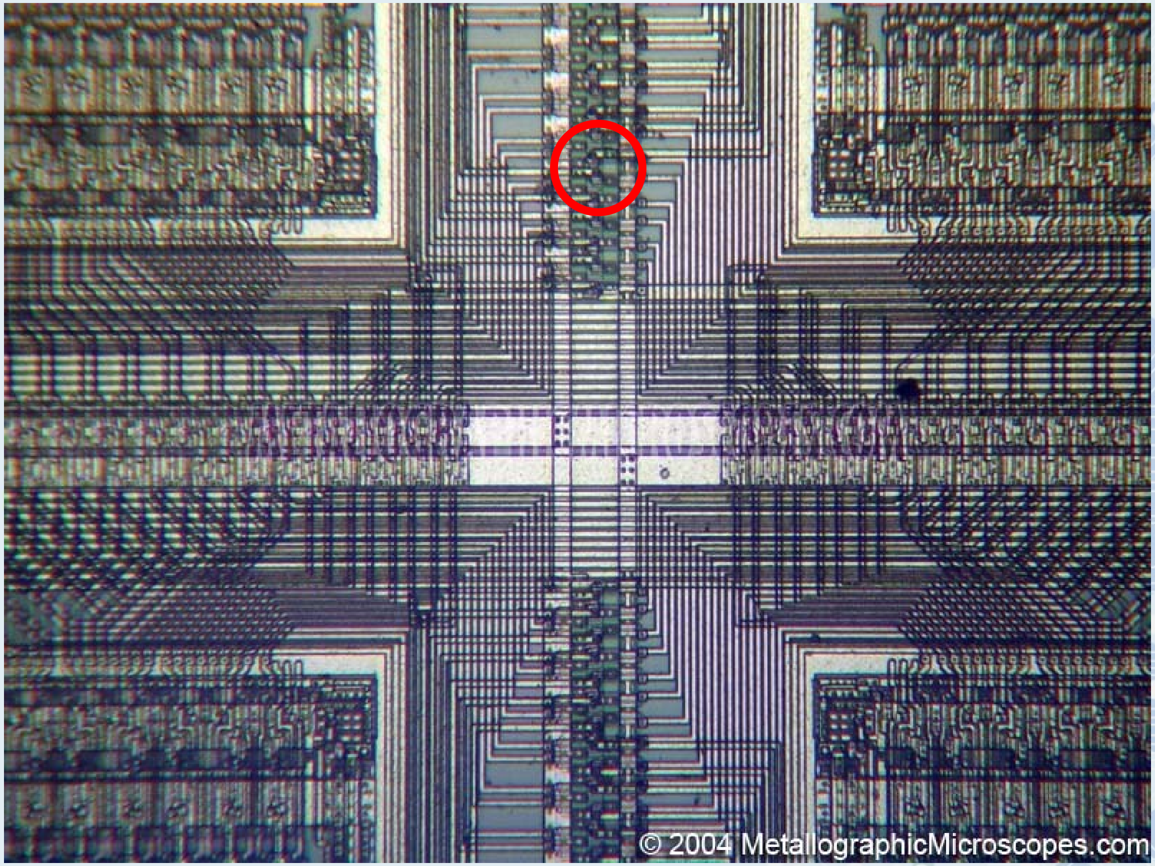

If I want to look there?

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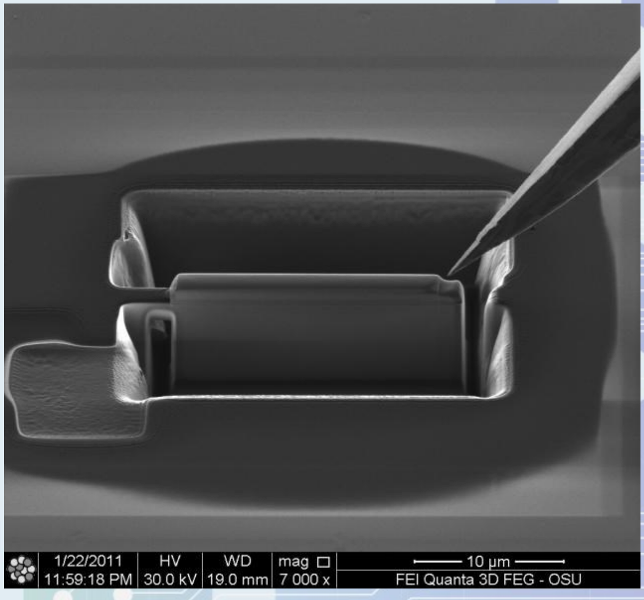
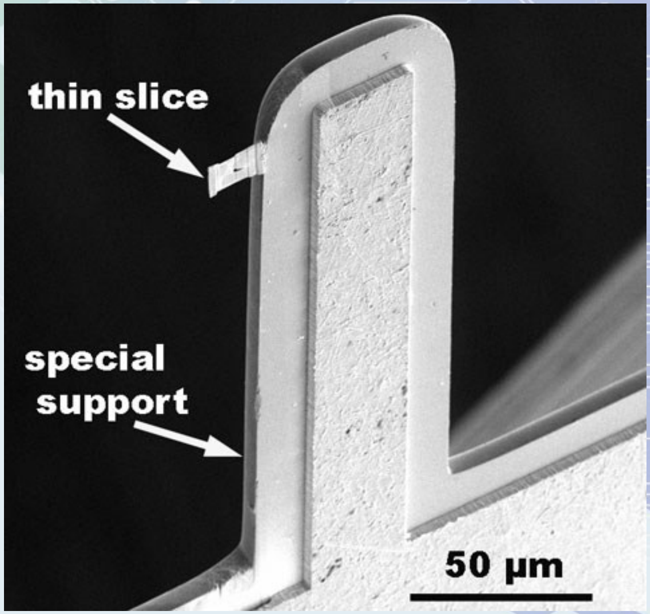
Focused Ion Beam

- Precise position
- Uniform thickness
- 10x10μm wide area





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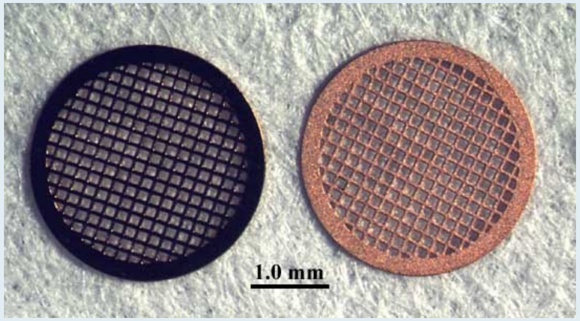
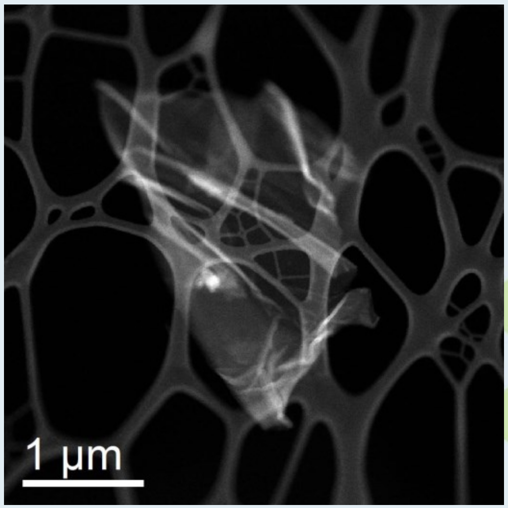
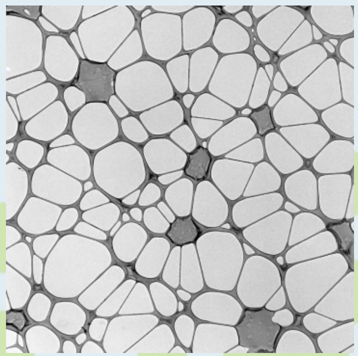
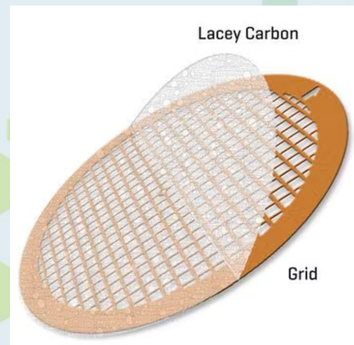


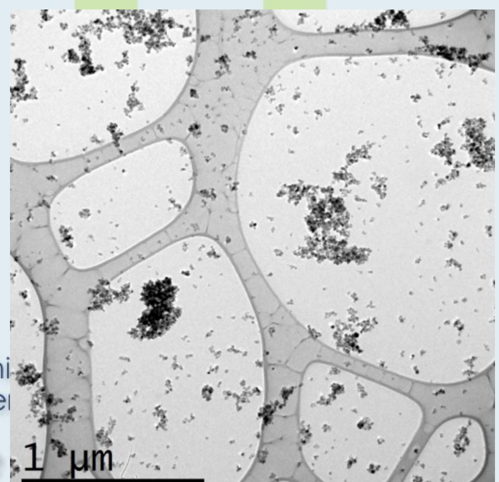
Drop Casting

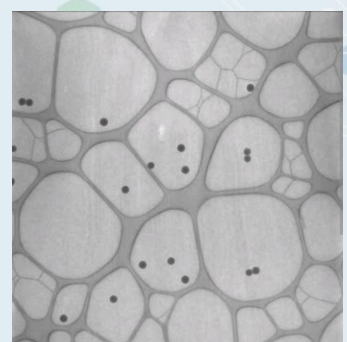
- Continuous carbon layer (50nm)
- Lacey carbon film
- Ultra-thin carbon layer on lacey.











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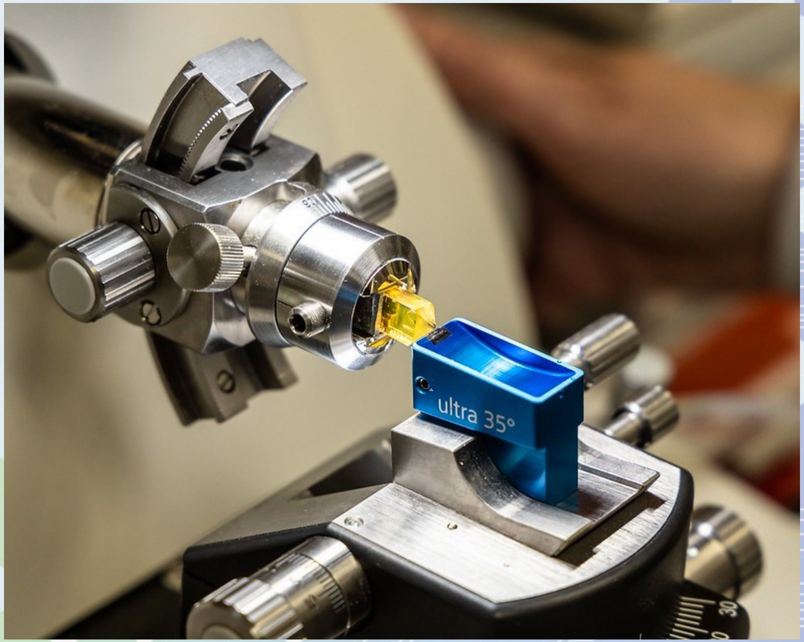
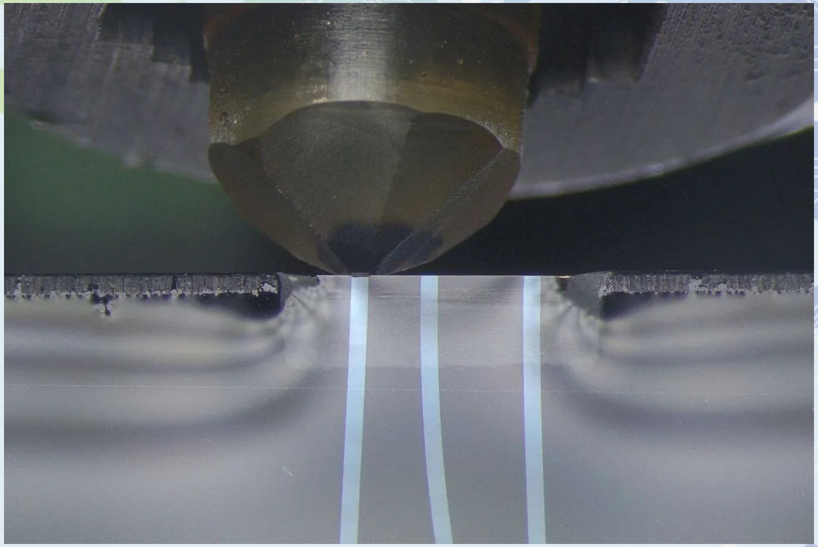
Ultramicrotome

Ultramicrotome



Dimond blade



Trains of lamellae

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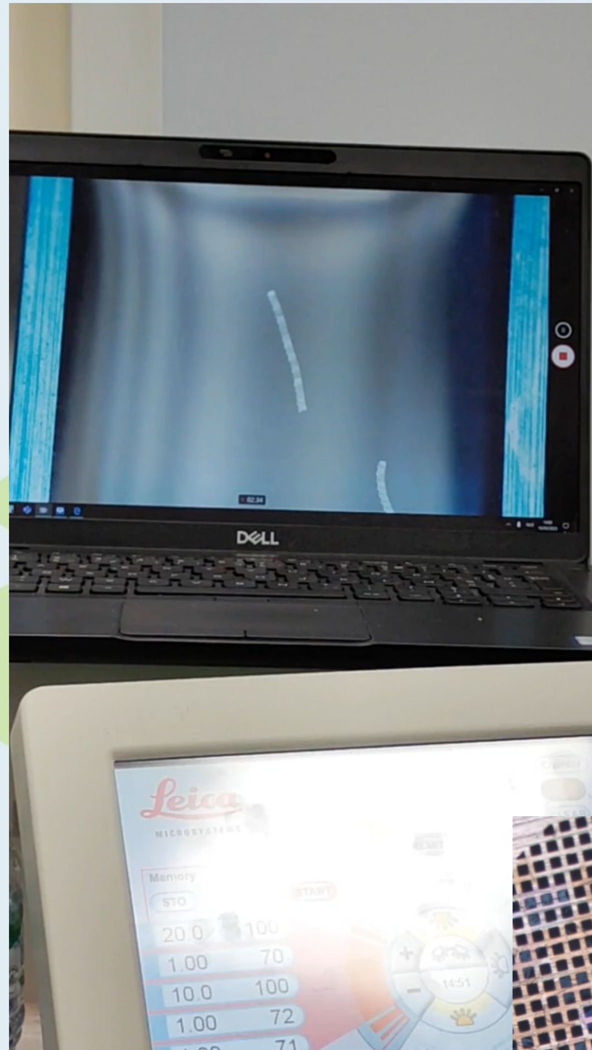

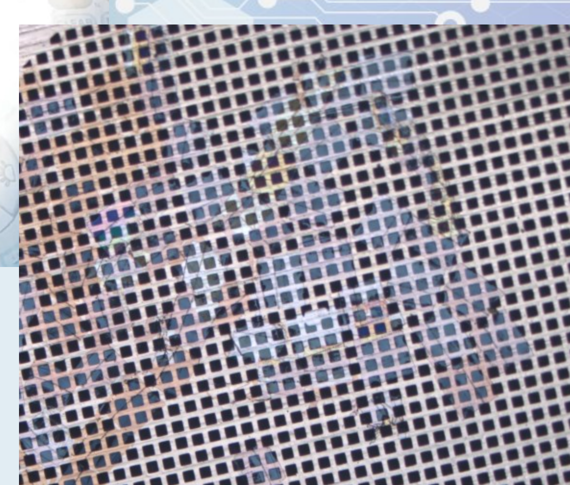

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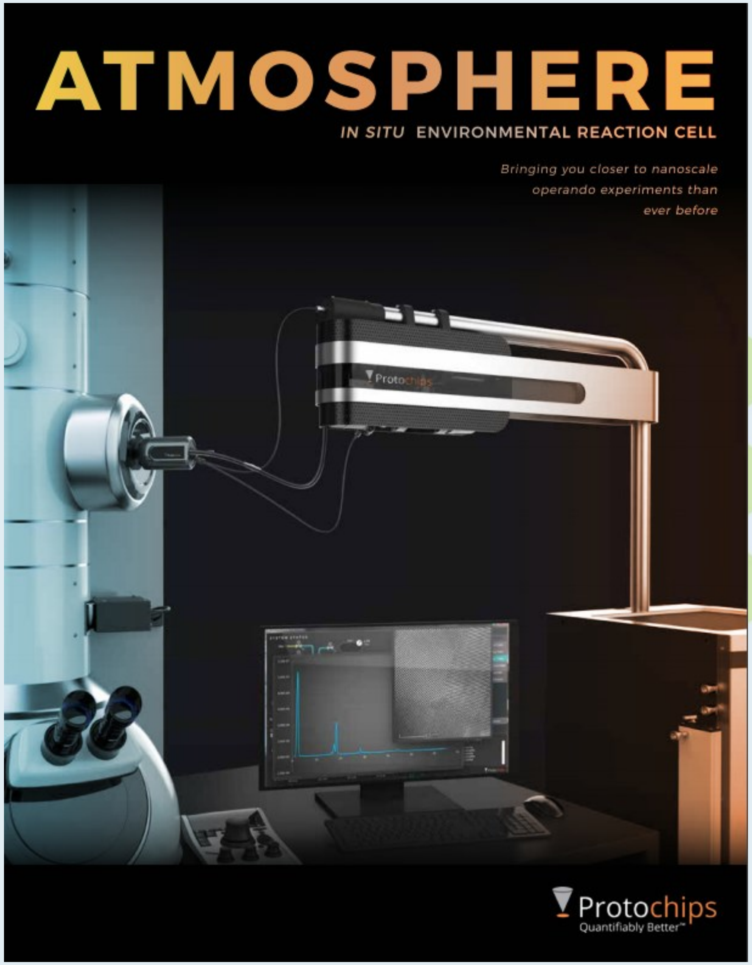
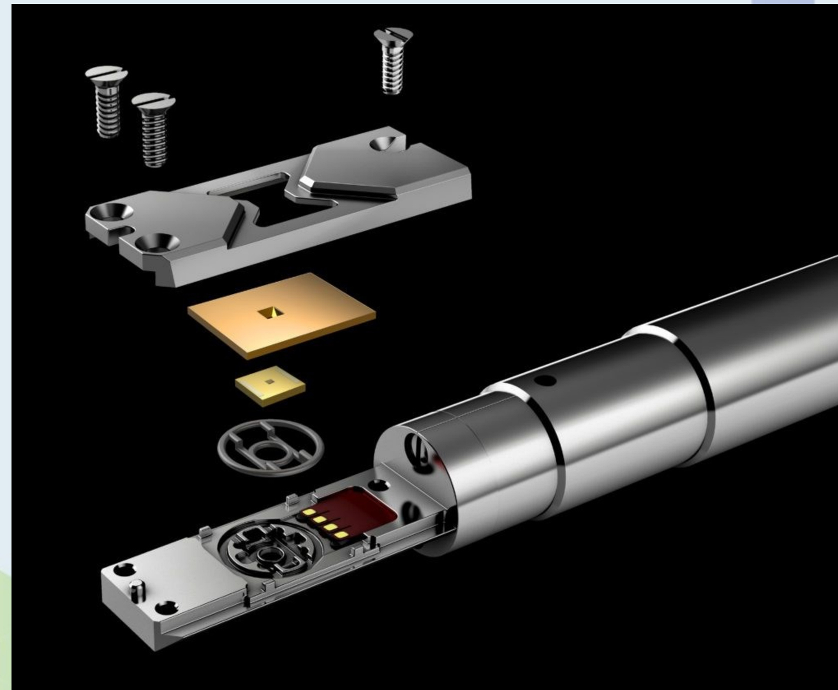
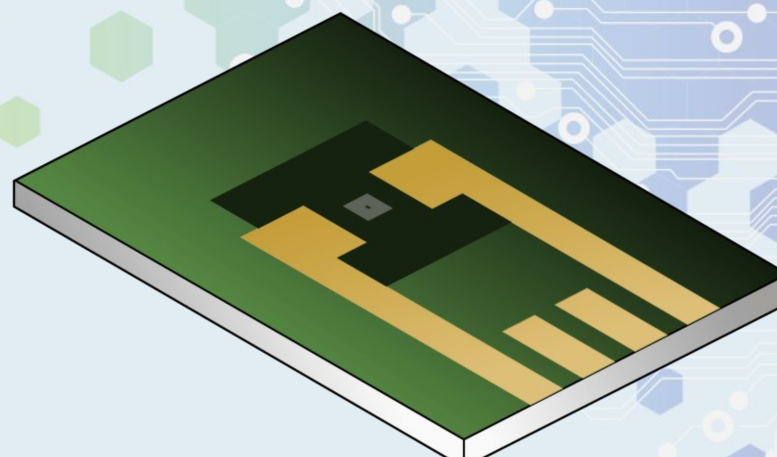
In-situ gas and liquid sample holder







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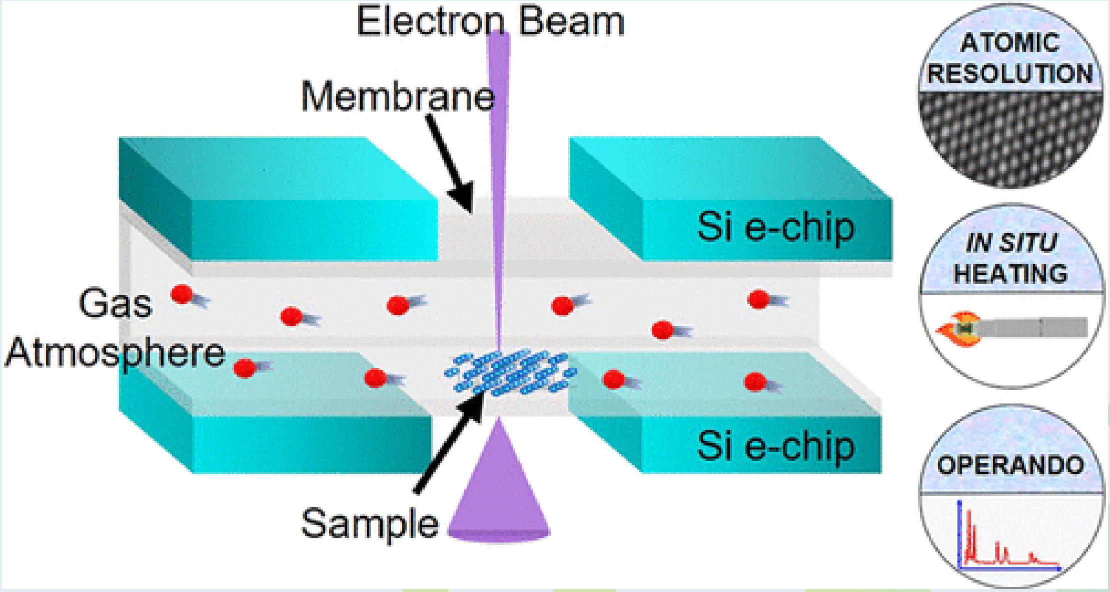

- Precise deposition
- Thin particle size
- Right reactions

Gold sputtered precisely in the viewing area and on the electrodes on a Fusion electrothermal E-chip

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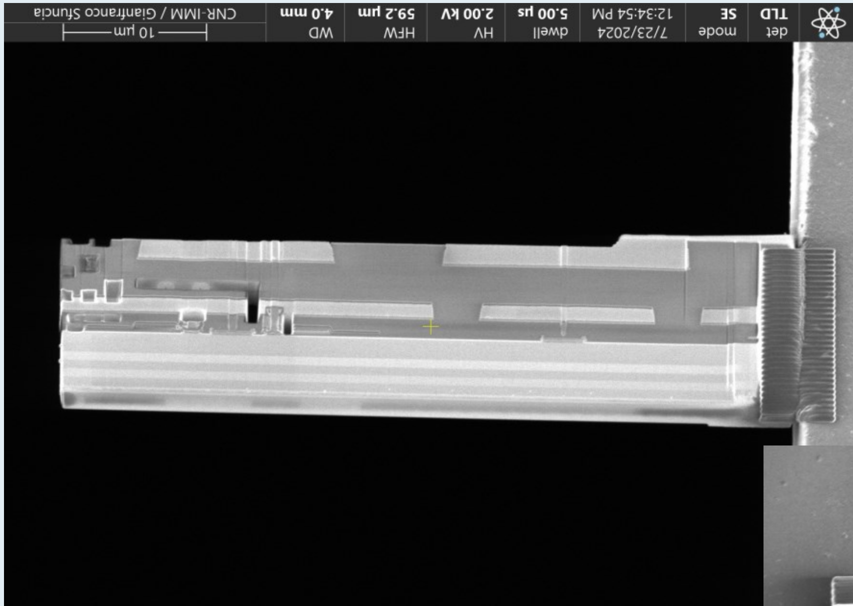
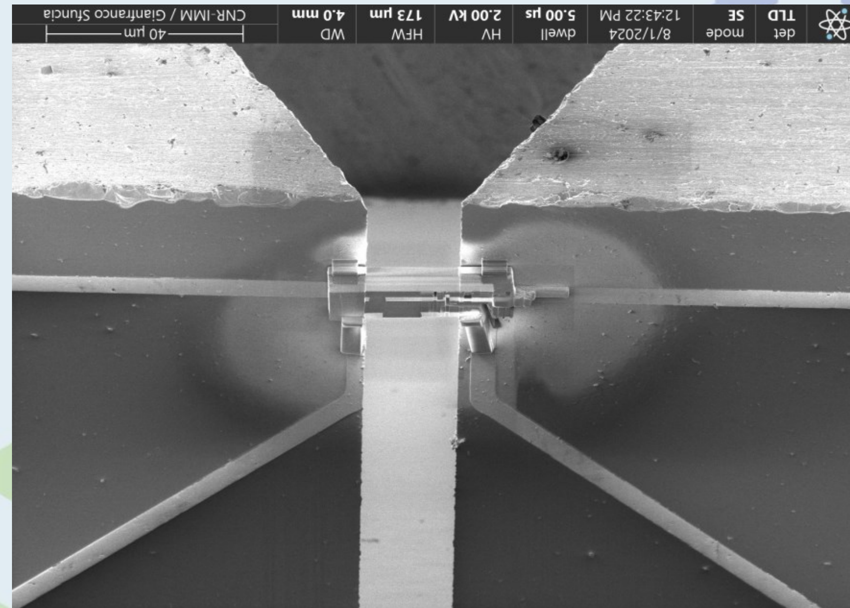
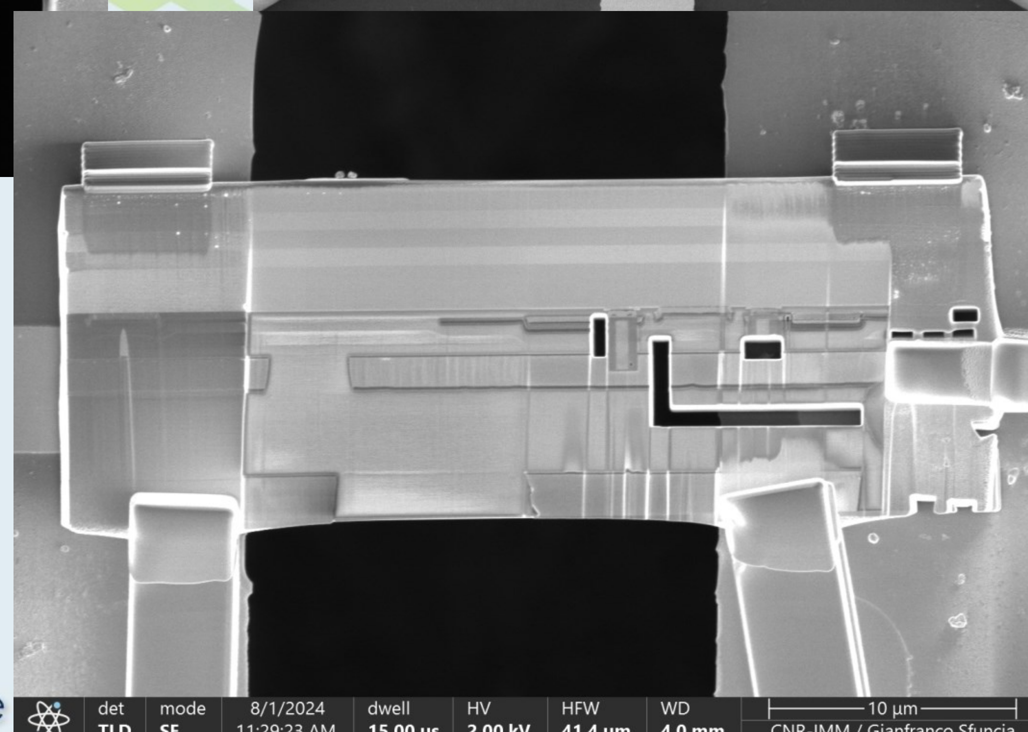
In situ electrical measurements







FIB lamella bonded in a four contact TEM sample holder

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