

**Sample Preparation
for
Transmission Electron Microscopy**

Why sample preparation for Transmission Electron Microscopy

- Electrons with properties of **particles** and waves
- Strong interaction between electrons of the beam and atoms of the samples → **scattering**
- Sufficient intensity/number of transmitted electrons only for **small thickness** (about 100 nm)
- Essential thickness depends on, e.g., materials properties, acceleration voltage, and requirements of individual investigation method

Demands on sample preparation

- No change of materials properties including:
 - Structure (amorphous, polycrystalline, crystalline)
 - Chemistry (composition of the bulk material, of surfaces, and of interfaces)
- But:
 - Artifacts inherent to any preparation method!**
- Criterion for appropriate preparation technique:
 - Influence on structural and chemical properties as little as possible!**

Type of sample

- Bulk material

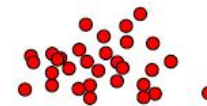


- Particles

not transparent for electrons

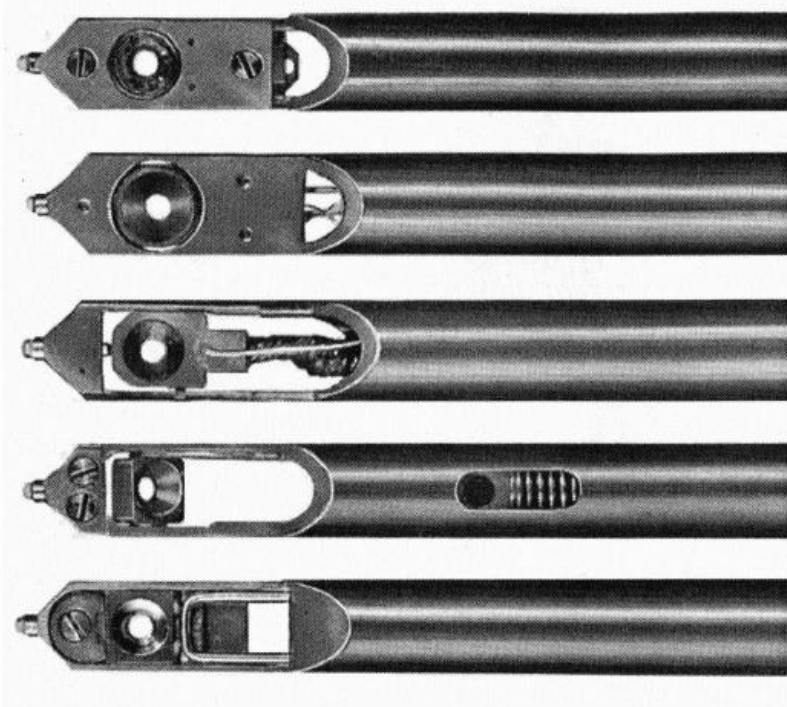


transparent for electrons



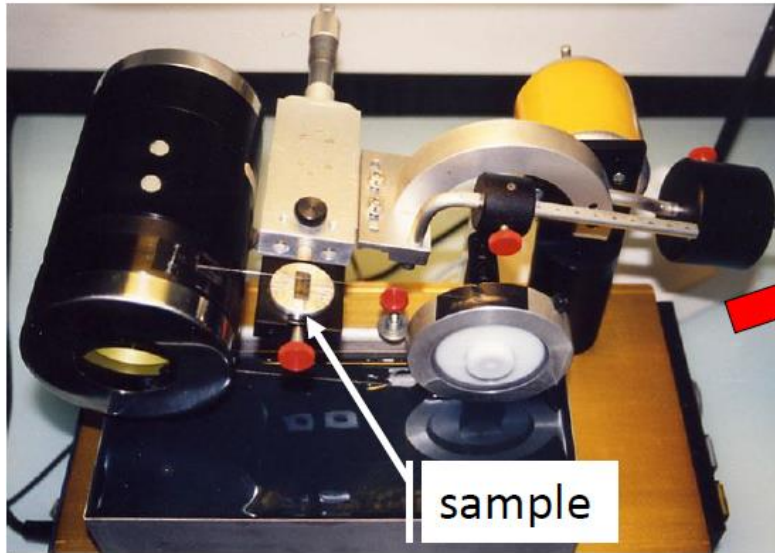
size < 100 nm

Shape of the sample



- Defined by TEM sample holders
- Limits of sample size:
 - Diameter: $\leq 3 \text{ mm}$ due to the furnace of the TEM sample holder
 - Maximum thickness of sample edge: ca. $100 \mu\text{m}$

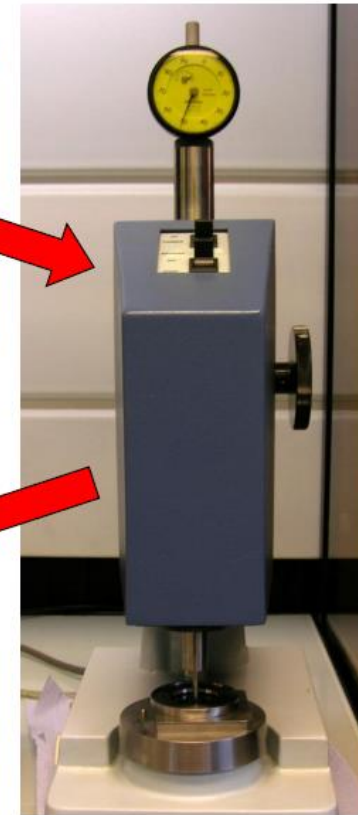
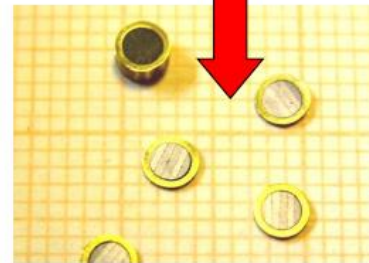
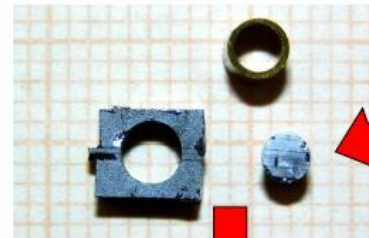
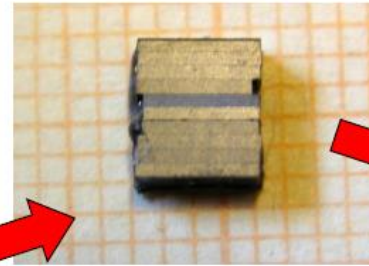
- Bulk material



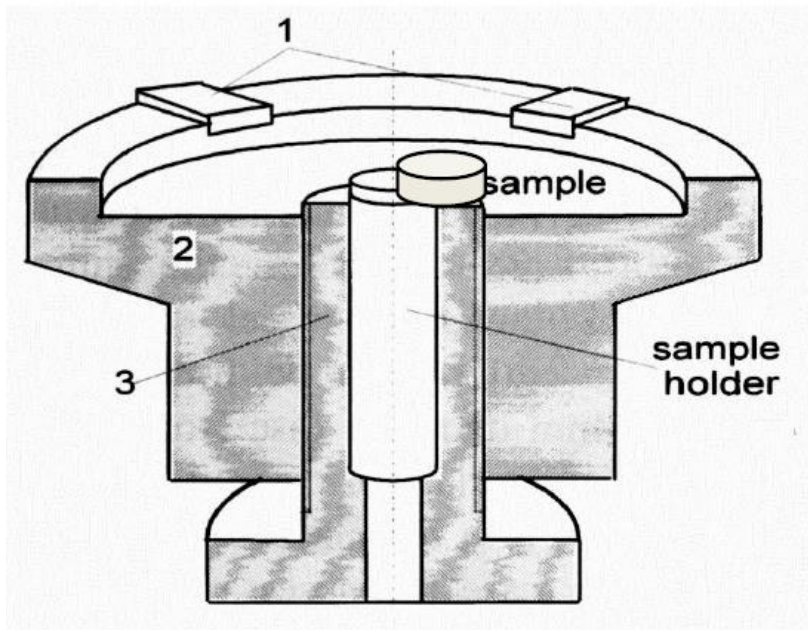
Wire saw with wire of steel covered by diamond particles

Gluing into a cylinder and cutting into discs

Face to face gluing



Ultrasonic cutter

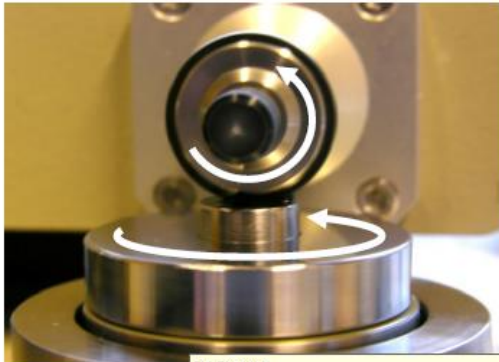


Grinding and polishing tool:
1 - silicon nitride pads,
2 - body part. 3 - screw

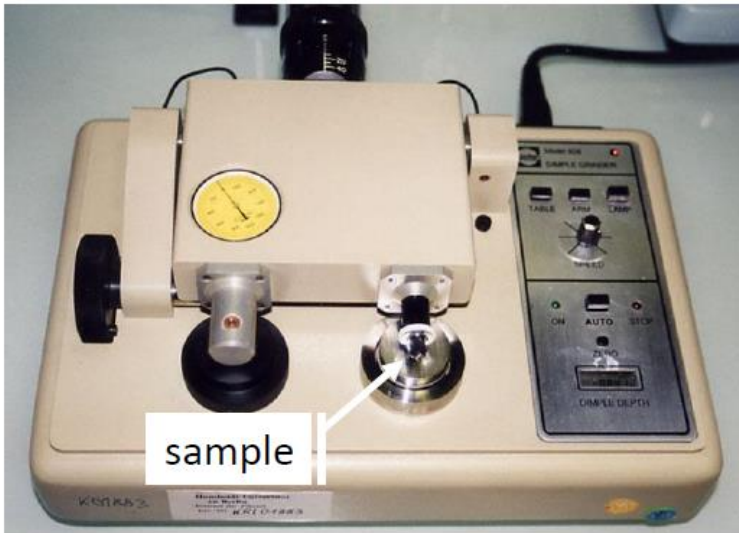


Cross sectional specimen after
mechanical polishing

Dimple grinding

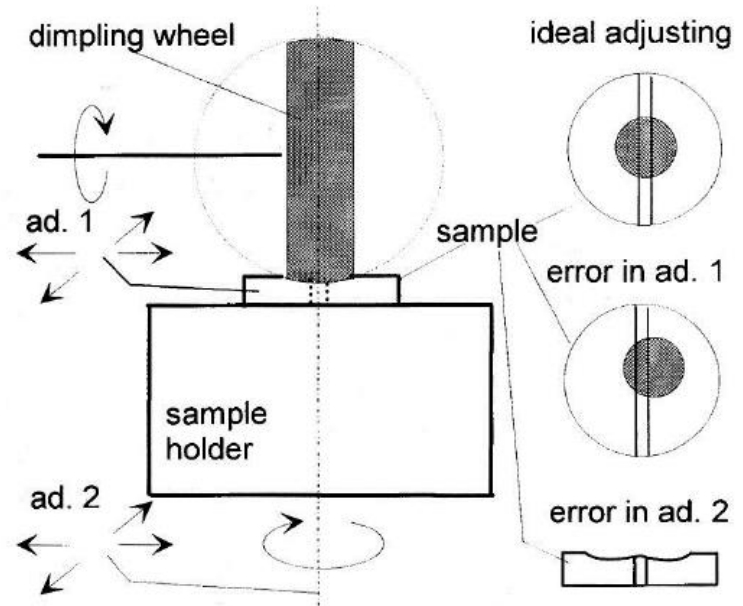


Detail of
D:\Eigene Dateien\geräte\preparation\instruments_pics\DSCN3537a.jpg



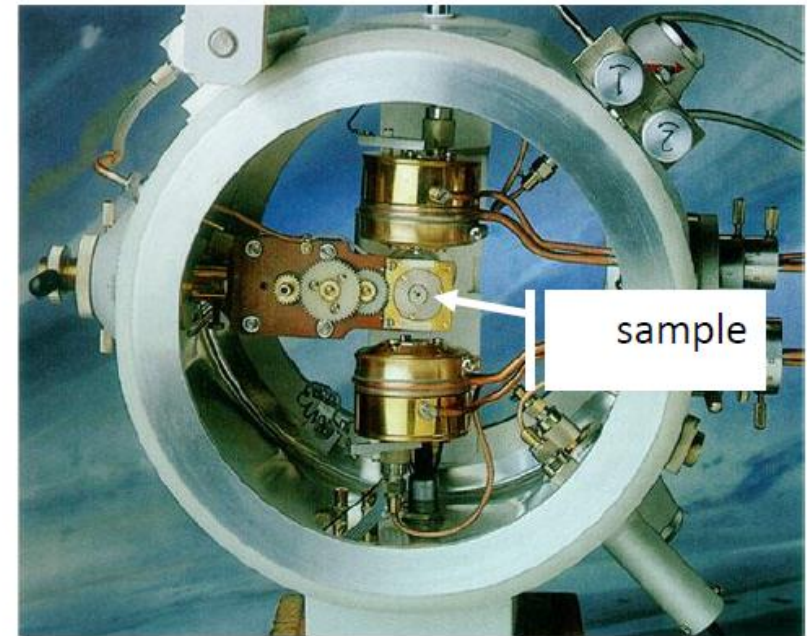
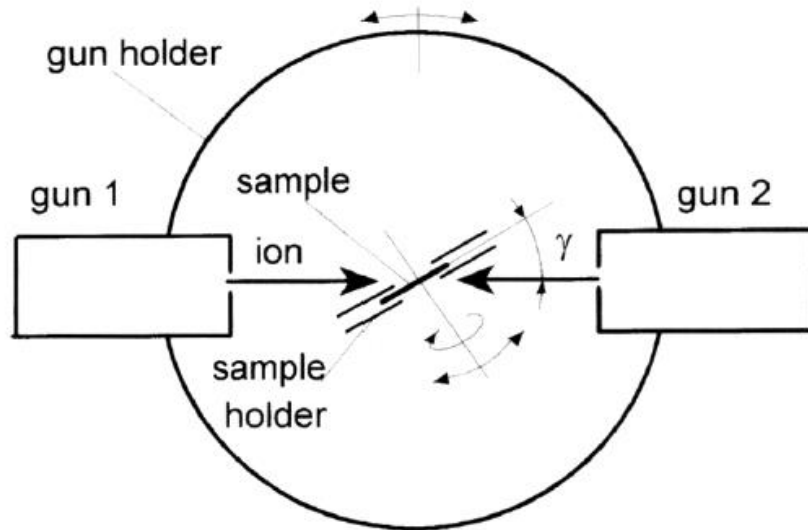
sample

Dimpler grinder of Gatan



Principles of dimpling technique

Ion-beam milling



Layout of a vacuum chamber with two ion guns

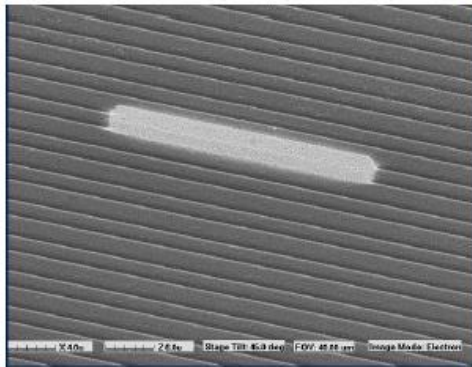
Ion gun arrangement for milling of both sides of the sample;
possible ions: Ar^+ , Xe^+ , I^+ , ...
acceleration voltage: 1...5 kV
usual angle γ : $< 10^\circ$

Precision Ion Polishing System PIPS (Gatan)

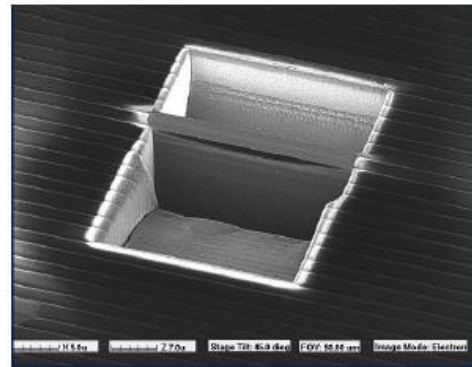


Focussed-ion beam preparation of a device structure

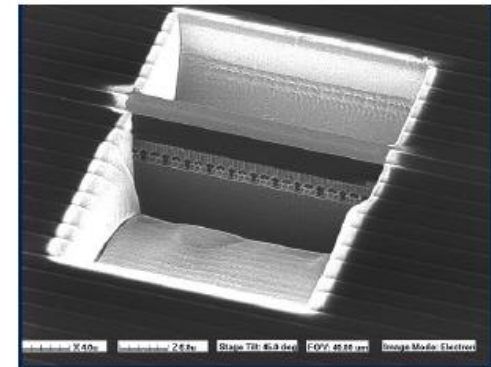
Pt deposition



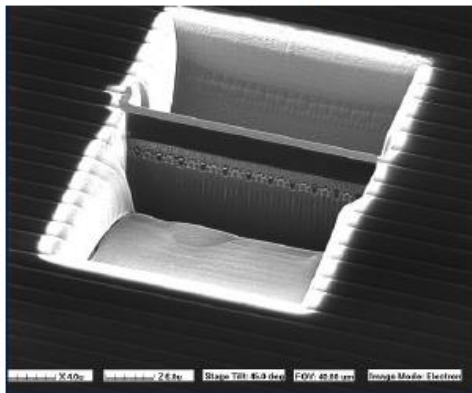
Terrace cut



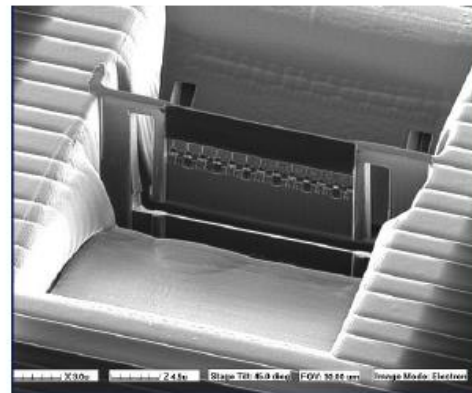
Cleaning I



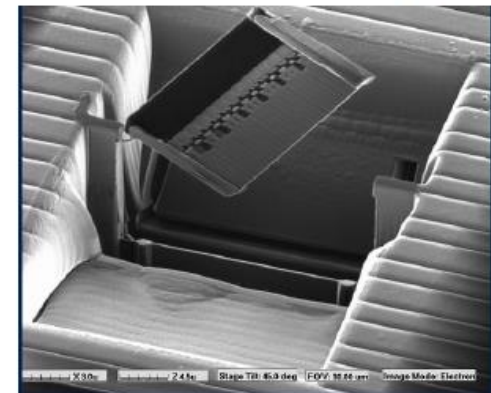
Cleaning II



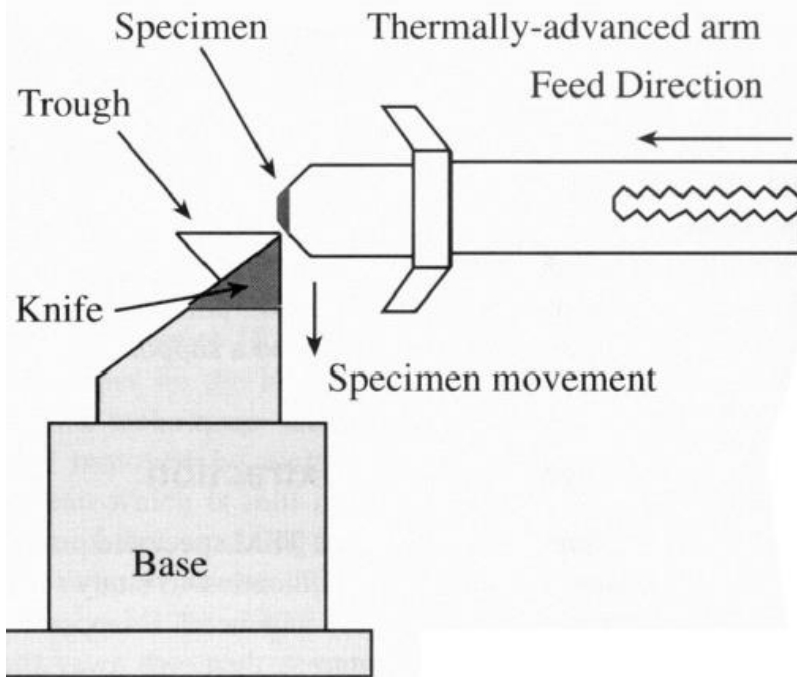
U cut



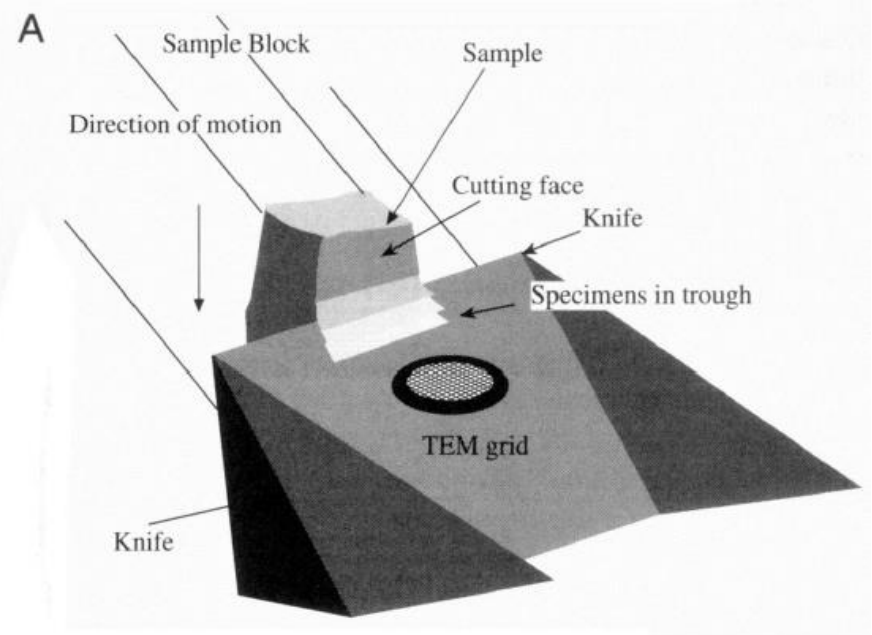
Transfer to TEM grid



Ultramicrotome cutting



Slicing of the specimen embedded in epoxy or other medium

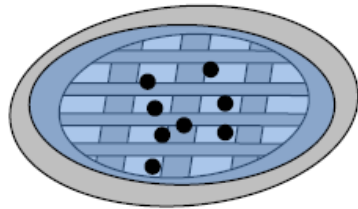


Floating of the slices onto water or an appropriate inert medium and collection on TEM grids

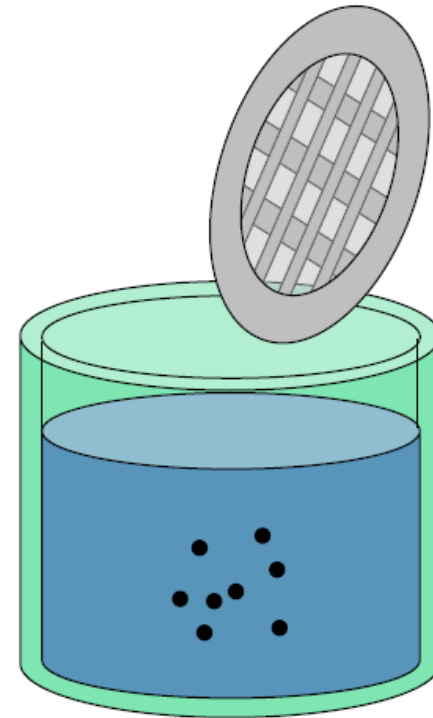
TEM preparation of small particles (1)

e- transparent particles ($t < 100$ nm)

- Dispersion in a non dissolving liquid (e.g.: methanol, water, etc.) in an ultrasonic bath
- Transfer to a carbon film supported by a copper grid

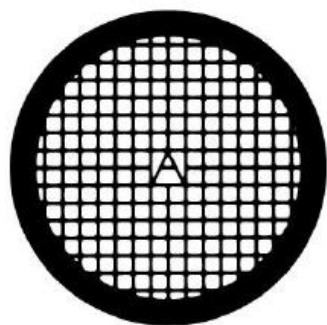


Evaporation of a droplet

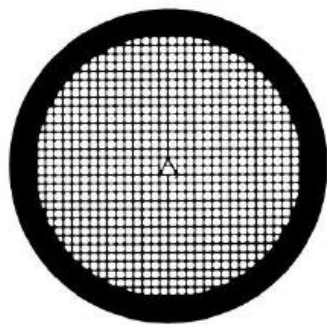


Dipping

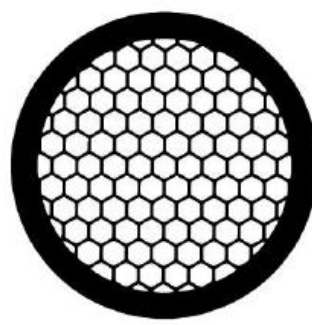
TEM grids



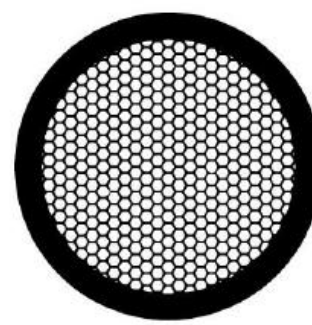
150 mesh



300 mesh



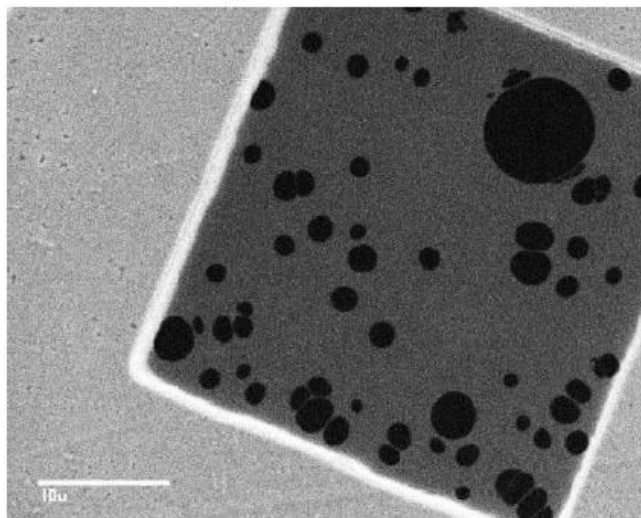
100 mesh



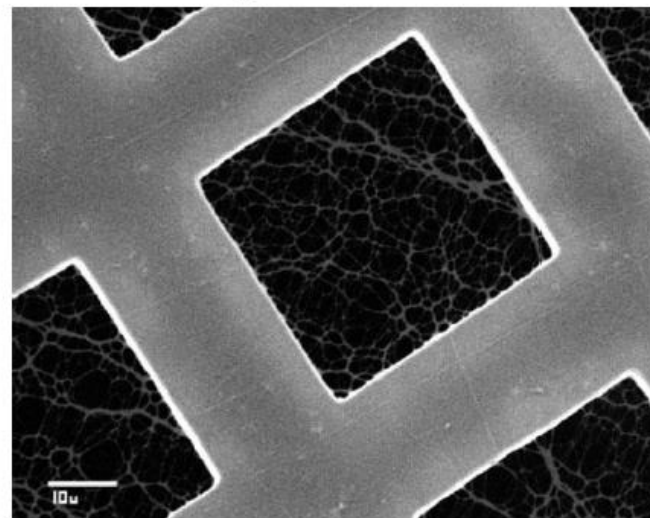
200 mesh

and
many
more

holey carbon film



lacey carbon film



References

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