
بسم الله الرحمن الرحیم

K. N. Toosi University of Technology
Faculty of Materials Science and Engineering

# Materials Characterization Methods

## Ninth Session
## (Transmission Electron Microscope)

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# Introduction

Transmission Electron Microscopy (TEM)

In 1936, the first transmission electron microscope was built by the Metropolitan-Vickers company in England. The TEM has the same function as the light microscope, with the difference that instead of light with a wavelength of 5000 angstroms, electrons with a wavelength of 0.05 angstroms are used to illuminate the sample.

# Introduction

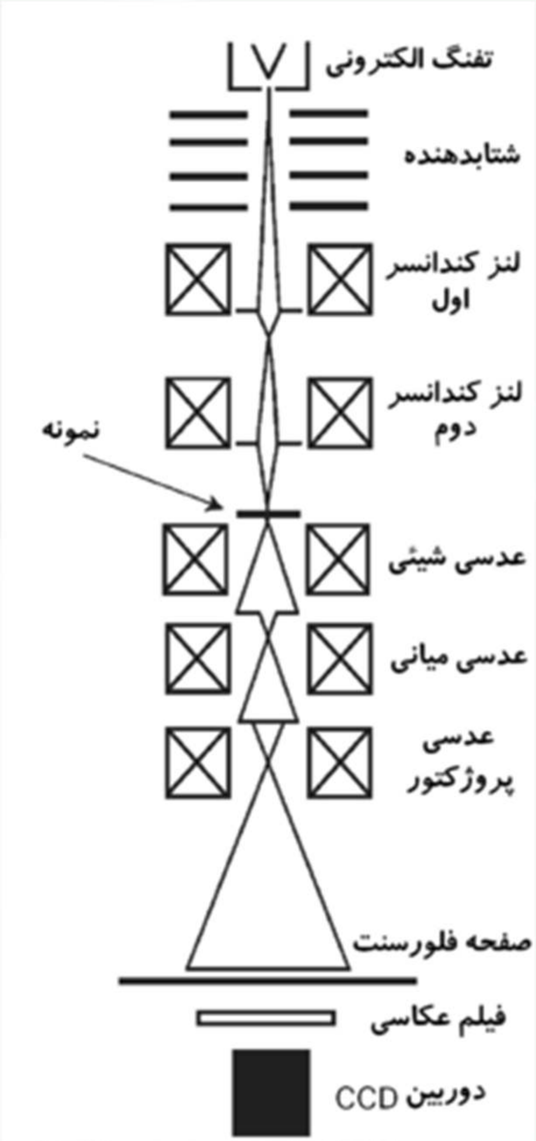
- Theoretically, the resolution limit of a TEM microscope is about 100000 times higher than that of an optical microscope.

- Of course, due to limitations such as lens errors, the resolution of the TEM microscope is about 2 Angstroms, which is about 1000 times better than the resolution limit of the optical microscope.

# Principles of Image Formation in TEM

- ✓ In the TEM, by combining the main components of electron microscopes (electron gun, lenses, apertures, vacuum pumps, etc.) and using transmitted rays, images of the structure of the sample are created.
- ✓ Collecting lenses and apertures focus the electron beams from the electron gun and reduce the diameter of the beam.
- ✓ After the electron beams collide and pass through the sample, they are re-converged by the objective lens and aperture.

# Principles of Image Formation in TEM




- ✓ Intermediate lenses are usually used to change the image magnification and determine the working mode (diffraction pattern or image formation). Finally, the final image is created on the screen with the help of the image lens.
- ✓ The screen in TEM is made of fluorescence, which produces visible light due to the impact of electron beams. Therefore, the formed image is real.

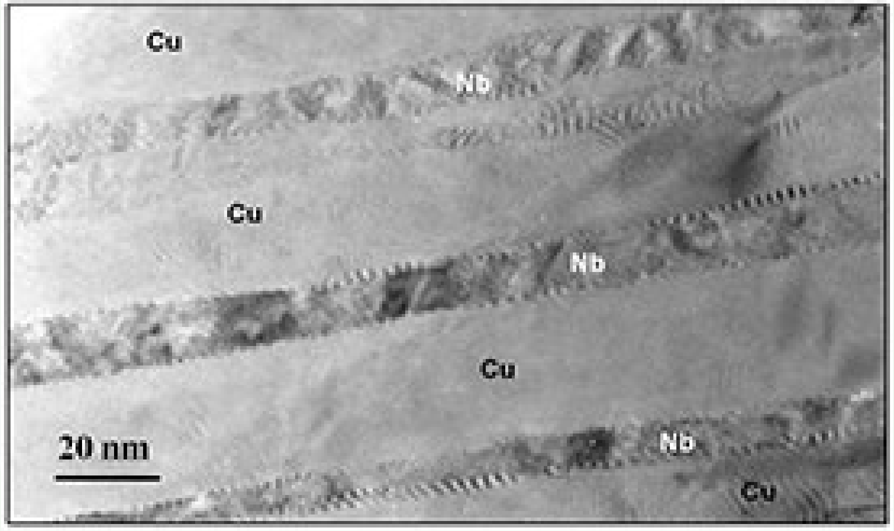
# Working Modes in TEM

## Bright Field

When the image is created only by the transmitted beam, we will have this mode, which is used to observe structural defects and dislocations. In this case, the deflected beam is removed by a suitable aperture.

Transmission electron micrograph of dislocations in steel, which are faults in the structure of the crystal lattice at the atomic scale.

# Working Modes in TEM




Bright field image showing nano-scaled Nb filament in a copper wire

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# Working Modes in TEM

## Dark Field

- ✓ When the image is formed by the deflected beam.
- ✓ This mode is created by using a suitable aperture or by tilting the source.
- ✓ This image is in the form of bright points on a dark background and is suitable for detecting structural phases.

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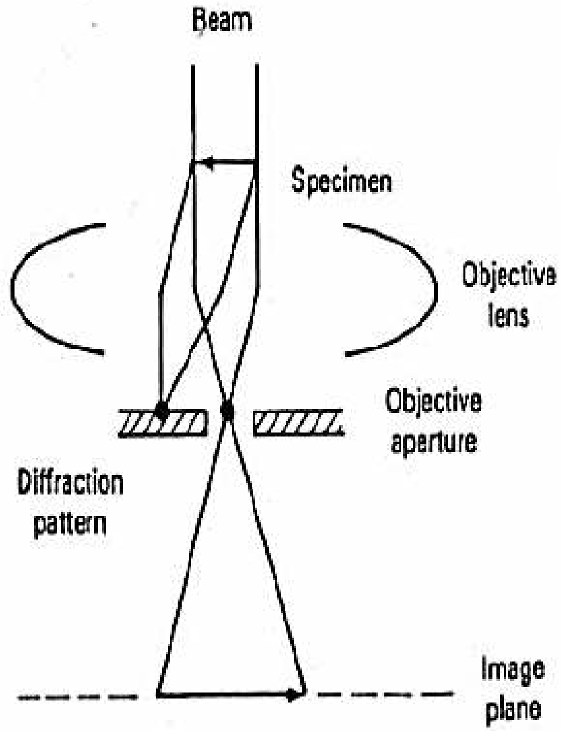
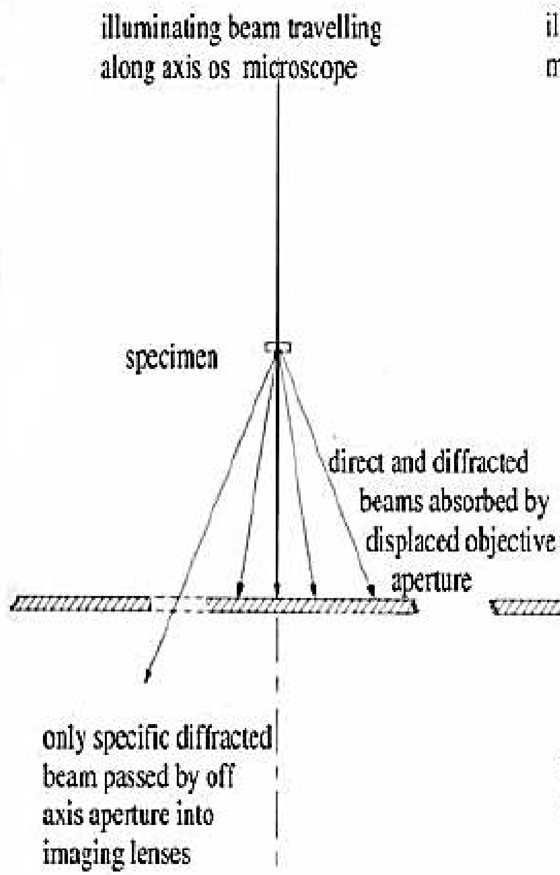
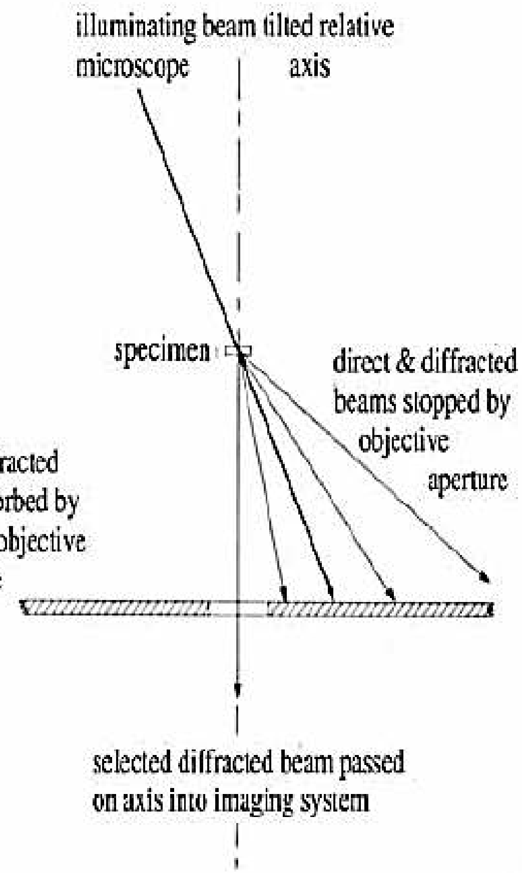
# Working Modes in TEM




Bright field Microscopy

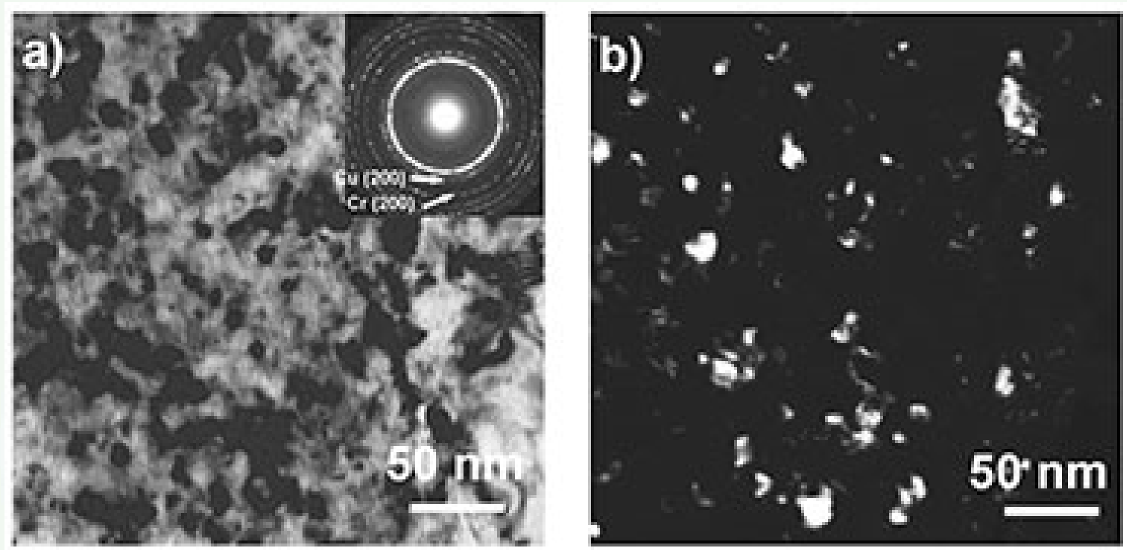



**a) Dark field microscopy by displaced objective aperture**




**b) Tilted beam dark field microscopy (precision dark field)**

# Working Modes in TEM




Bright field (a) and dark field (b) images of a nanoscaled CuCr composite

# Working Modes in TEM

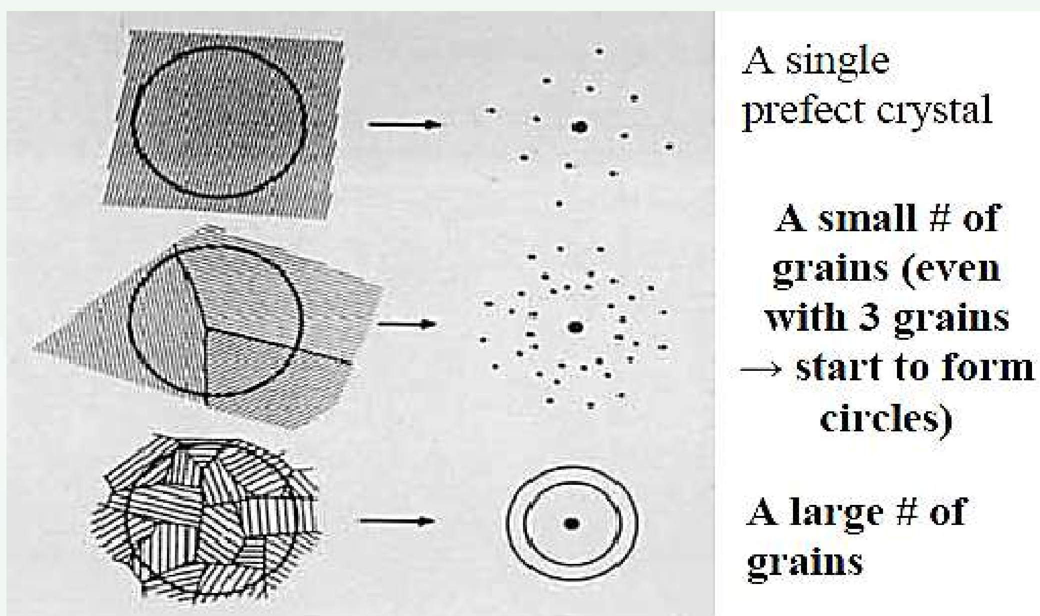
## Weak Beam Dark Field

In this case, a weak deflected beam is used to form the image. The transmitted ray and the scattered beam are removed by suitable aperture and tilting of the source.

# Working Modes in TEM

## Diffraction Pattern

- ✓ Rays diffracted by Bragg's law form an image at the focal plane of the objective lens.
- ✓ The image formed in a single crystal is in the form of a set of points and in fine-grained polycrystals in the form of a set of rings.



# Working Modes in TEM

- ✓ The diffraction pattern is different depending on whether it is single crystal or polycrystalline.
- ✓ Spot diffraction is for single crystals consisting of regular rows of spots, which occurs when they are oriented in such a way that several sets of planes are parallel to the electron beam.
- ✓ If the sample contains several crystals with different orientations, the diffraction pattern is created from the sum of the individual diffraction patterns of each of the crystals.

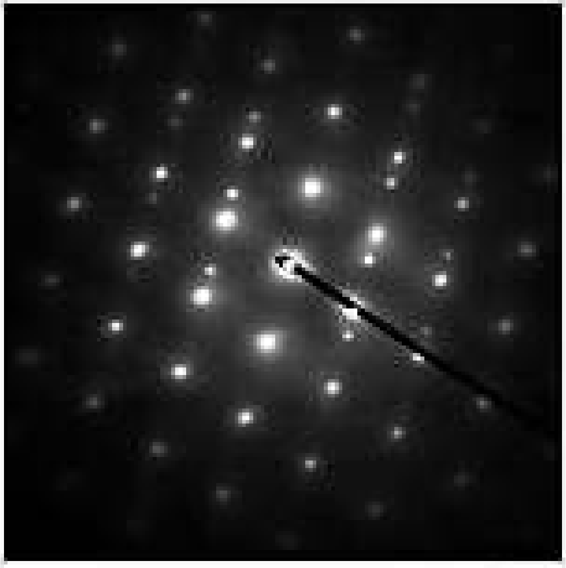
# Working Modes in TEM

- ✓ Because only certain planes can cause diffraction, that is, the number of possible distances d and consequently the distances r on the diffraction pattern are limited and the points are not randomly distributed, so they form circles, each of which has a constant r.
- ✓ In the small number of grains, the diffraction pattern is point and the points are not connected but have a circular shape. But with a large number of random grains, they form connected points and circles.

# Working Modes in TEM

## Selected Area Diffraction Pattern (SAD)

In this case, by using the appropriate aperture, the diffraction pattern is obtained from only a small part of the sample.



SADP of a single austenite crystal in a piece of steel

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# Working Modes in TEM

## Lattice Image (LI)

- ✓ In this case, TEM is used to create an image of the crystal lattice.
- ✓ The said image consists of the interference and combination of information related to different electron beams (transmitted and deflected, at least two beams).
- ✓ Device parameters must be carefully adjusted to create an LI image.

# Sample Preparation for TEM

- ✓ Sample preparation for TEM is very important. The sample should be very thin (in the range of one micron), no fundamental changes will be made in it due to preparation, and it should be sufficient strength to be moved and examined in a microscope. Sample preparation methods are divided into two categories:

1. Cutting the sample in such a way as to produce a very thin sample or a very thin section of a sample.
2. Removal of unwanted material by electrochemical or chemical or mechanical methods until a thin sample remains

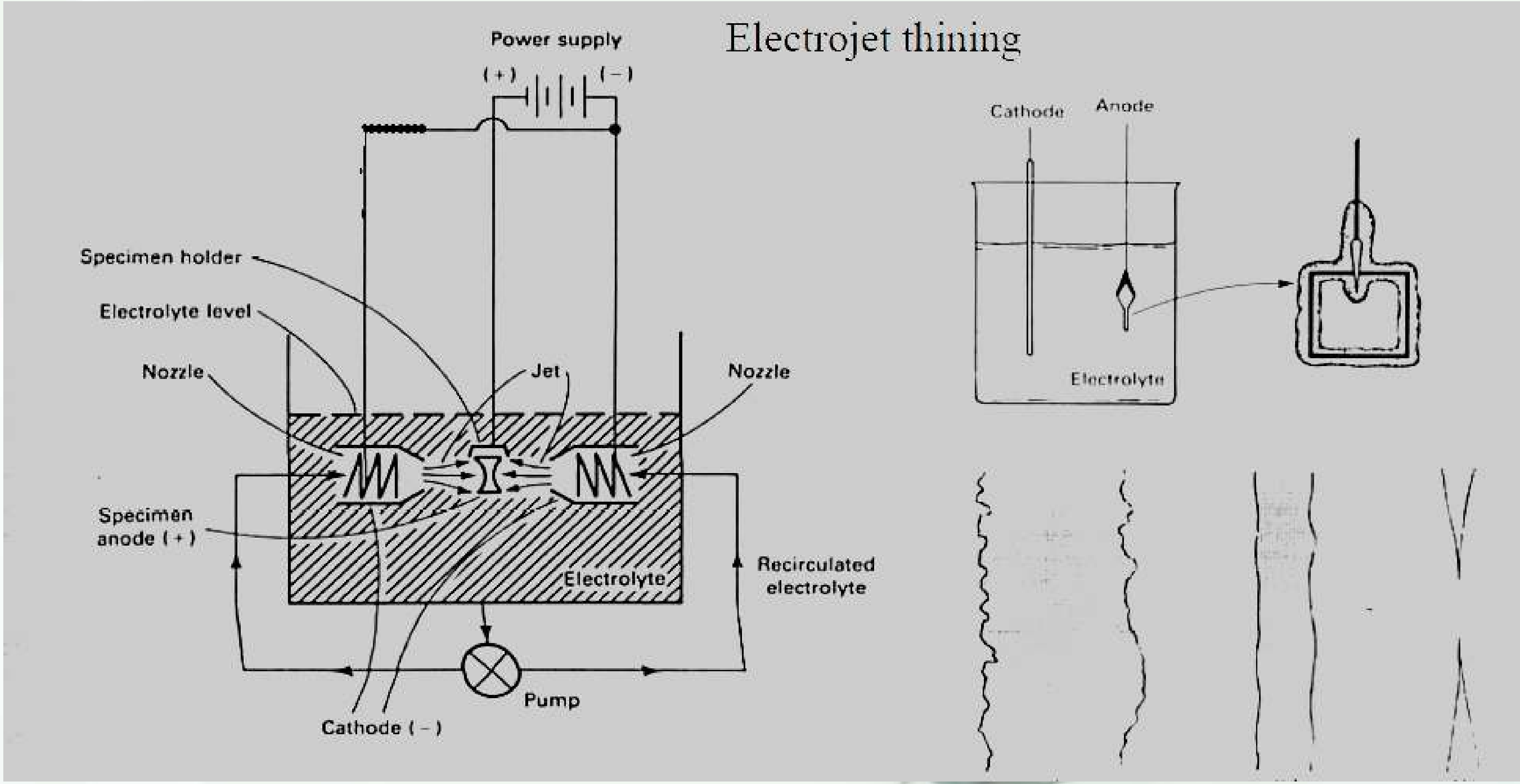
# Sample Preparation for TEM

## Electropolishing, Chemical Polishing and Spark Methods

- ✓ A common method for thinning electrically conductive materials (metals and alloys) is electropolishing.
- ✓ The basis of the method is to place the sample as anode in the electrolyte cell.
- ✓ As the current passes, the metal is separated from the anode (sample) and deposited on the cathode.
- ✓ After the sample reaches the appropriate thickness, the sample is ready to be checked.
- ✓ In some cases, the perforated sample and its surrounding areas, which are very thin, are used for examination.

# Sample Preparation for TEM

## Electropolishing, Chemical Polishing and Spark Methods



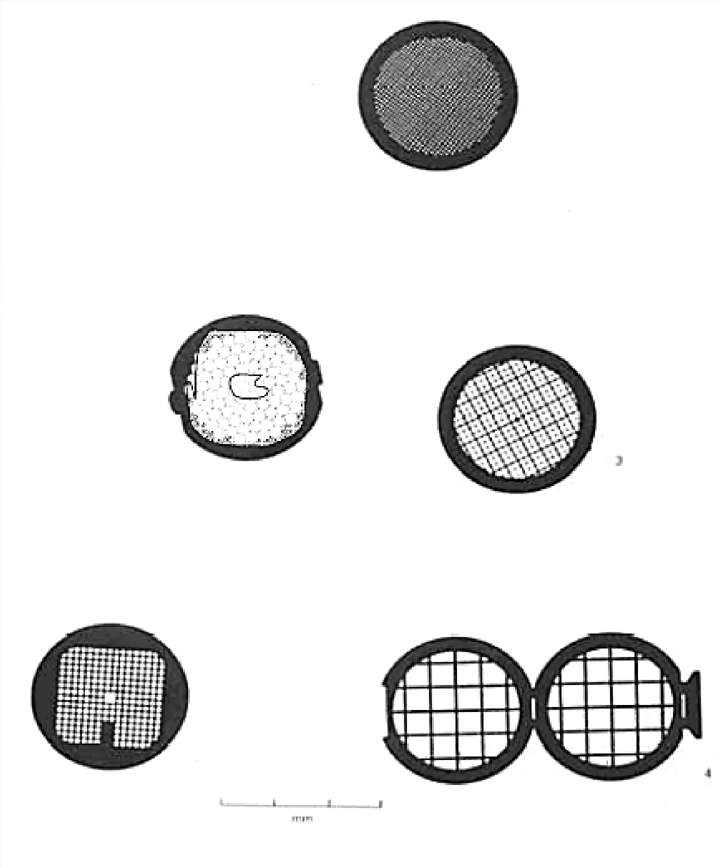
# Sample Preparation for TEM

## Electropolishing, Chemical Polishing and Spark Methods

- ✓ In some cases, disk samples with a diameter of 3 mm with thicker edges compared to thinner central samples are prepared by automatic electropolishing devices and placed directly in the sample holder of the microscope.
- ✓ In some cases, thin samples prepared nonautomatically are reinforced with 3 mm mesh discs made of copper or other materials that do not interfere with analysis.

## Sample Holder in TEM

Specimen Support Grid



# Sample Preparation for TEM

## Electropolishing, Chemical Polishing and Spark Methods

- ✓ Electropolishing cannot thin non-conductive materials. Therefore, for these materials, chemical thinning is used using a mixture of acids without applying electric current.
- ✓ Chemical polishing method is used for ceramics, glasses and semiconductors.

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# Sample Preparation for TEM

## Electropolishing, Chemical Polishing and Spark Methods

- ✓ The disk sample (3 mm) is mounted on a neutral holder using glue and then this set is immersed in the etching material or a thin stream of this material is sprayed on the sample until a small hole appears on it. Then the sample is separated from the holder by dissolving the glue in a solvent and after washing several times the sample will be ready.

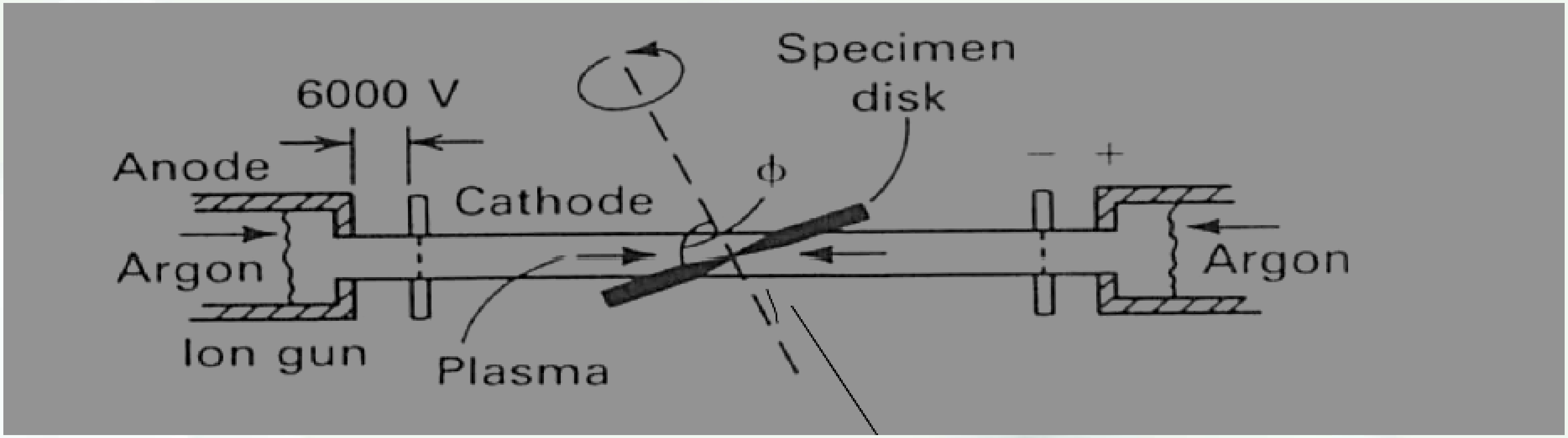
# Sample Preparation for TEM

## Ionic and Atomic Wear Method

- ✓ If beams of ions or high-energy atoms are irradiated to a sample, there is a possibility of ejecting the sample atoms, which is considered a method of thinning the sample.
- ✓ There are two types of guns that use argon gas and liquid gallium for this purpose. In these guns, there are different sources of atoms or ions, and an electric field is used to create acceleration in them.

# Sample Preparation for TEM

## Ionic and Atomic Wear Method

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# Sample Preparation for TEM

## Mechanical Polishing Method

- ✓ Using rough and then soft sandpapers for wearing and polishing samples mechanically is a conventional method in materials engineering.
- ✓ Before TEM examination, samples should generally be subjected to ion wear.

# Sample Preparation for TEM

## Replica Method

- ✓ In this method, a thin layer of carbon (or some other materials) is placed in a vacuum environment by creating an electric arc between two carbon rods on the surface of the sample in the device chamber.
- ✓ Then the resulting sample is floated in the liquid until the resulting thin layer is separated from the surface of the sample and placed on the microscope holder.

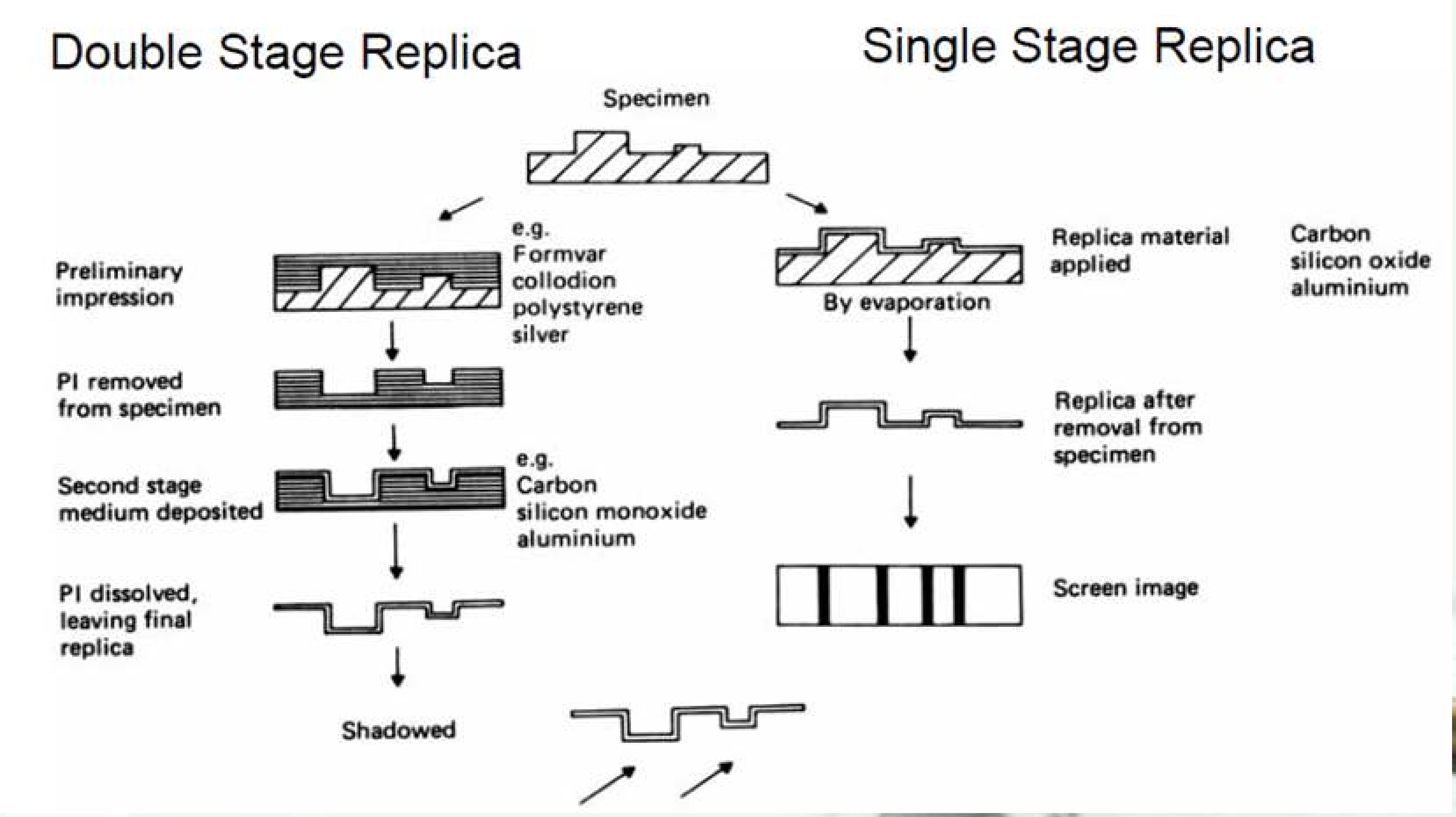
# Sample Preparation for TEM

## Replica Method

- ✓ The surface structure of the sample can be revealed and studied by varying the thickness of the carbon deposited on the surfaces or re-layering in a heavy metal (such as Pt) at an angle to the surface.

# Sample Preparation for TEM

## Replica Method

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# Applications of TEM

- ✓ Study of structural defects and atomic planes
- ✓ Determining the dislocation Burgers vector and stacking fault energy
- ✓ Structural studies (microstructure with resolution limit up to atomic dimensions)
- ✓ Phase transformations
- ✓ Recovery and recrystallization
- ✓ Phase investigations of materials using the preparation of diffraction patterns
- ✓ Study of failure levels
- ✓ Fatigue
- ✓ Oxidation

# Applications of TEM

- ✓ TEM can provide detailed information about metals such as the distribution and movement of dislocations, size, number and distribution of deposits and inclusions, nucleation and growth mechanism, crack movement, etc.

# Limitations of TEM

- ✓ Sample preparation is tedious and time consuming.
- ✓ The resolution limit of the image is about 0.2 nm.
- ✓ The minimum size of the analyzed area is 30 nm in diameter and the sensitivity limit of the device is 0.5-1 wt.% for elemental analysis.
- ✓ The accuracy of quantitative analysis is relatively about 5-15%.