

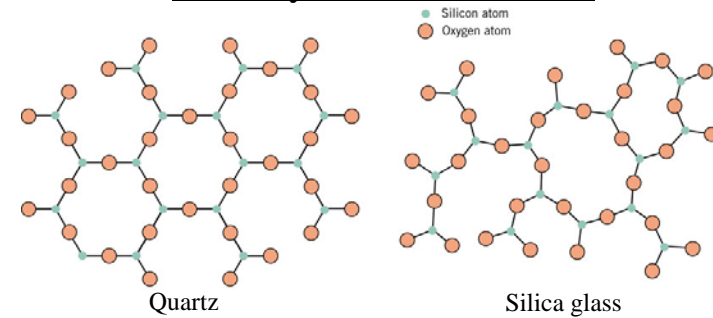


Outline:

- Crystallization
- Grain boundaries
- Grain size determination
- Types of microscopes
 - LOM
 - SEM
 - TEM
 - SPM



Non-crystalline Materials

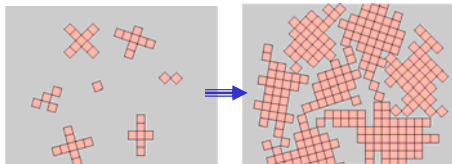


- Non-crystalline materials are ones which show no long-range order in their structure and are *amorphous*
- structure is similar to that of liquids-*supercooled liquids*
- silica (SiO_2) can either be crystalline (*Quartz*) or amorphous (*silica glass*)
- *slight* change in bond angles causes long-range order to be lost



Polycrystalline Materials

Most materials are polycrystalline and are made of many single crystals



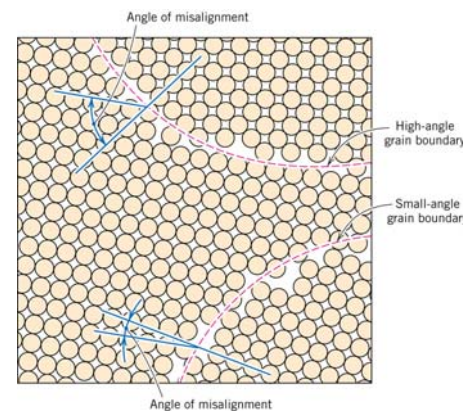
- during *solidification* the crystal nucleate and grow from the liquid in a *random orientation*



- the *grains* impinge on each other when the solidification is complete
- junction of grains are *grain boundaries*



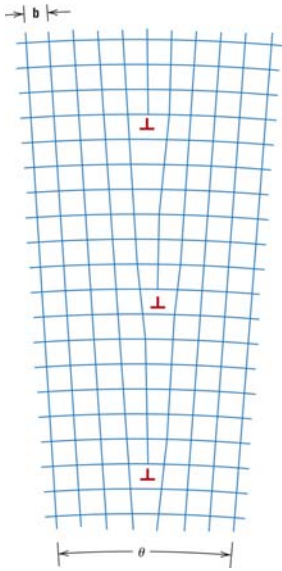
Grain Boundaries



- Occur due to the crystallographic mismatch when two grains meet
- when mis-orientation is large → *high angle grain boundary*
- when mis-orientation is small, → *low angle grain boundary*
- atoms are *less bonded* and the atomic packing is lower than in the grain (*lower coordination*)
- the result is an energy difference → *interfacial surface energy or grain boundary energy*



Grain Boundaries



- grain boundaries are more chemically reactive
- segregation of impurities due to higher energy
- total grain boundary area smaller in *coarse grained* than *fine grained* material
- low angle grain boundary is described as an array of dislocations
 - tilt boundary (edge)
 - twist boundary (screw)



Observation of Grain Structure

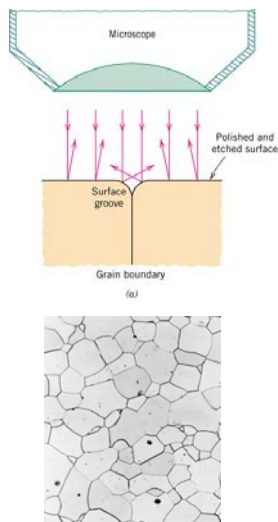


FIGURE 4.10 High-purity polycrystalline lead ingot in which the individual grains may be discerned.

- *Macrostructure* can be observed with naked eye
- coarse grains can be revealed this way (e.g. Al streetlight posts e.g. zinc galvanized garbage cans)
- *microstructure* is when the grains can only be observed with a microscope
- imaged using a camera for archiving
→ *photomicrograph*



Sample Preparation for Microscopy



- Preparation requires meticulous *grinding* and *polishing* of the surface
- the microstructure is revealed by attack using *etchants* (chemical reagents)
 - preferential attack of grain boundaries
- effect is that these features scatter the incident light and create **optical contrast**



Grain Size Determination

- properties are affected by *grain size*
- measurement of *grain volume, diameter and area*
- average grain diameter can be determined using the *linear intercept method*
 - lines of same length placed on micrograph
 - measure number of grains intercepting each line
→ *average grain diameter*
- *ASTM grain size* (n) based on number of grains/square inch (N)
- expression relating the two parameters:

$$N = 2^{n-1}$$
- use comparison charts to determine size of microstructure of interest at x100 magnification → simple to implement



Three-dimensional (volumetric) defects

- Inclusions
- precipitates
- Porosities or voids
- Cracks

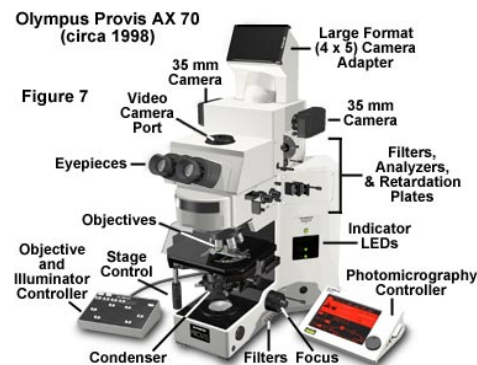
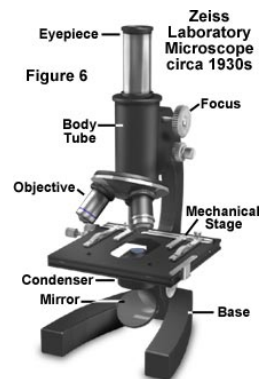


Types of Microscopy

- *Optical microscopy is limited to x2000 magnification*
- *electron microscopy uses electron waves (very small 0.003nm) rather than light*
- *can reveal microstructural features down to atomic scale (x1,000,000)*
- scanning electron microscope (SEM)
 - sample preparation similar to optical microscopy
 - can use to observe fracture surface (fractography)
- transmission electron microscope (TEM)
 - samples are very small
 - requires very thin (electron transparent) samples



Optical Microscopy



Scanning Electron Microscope (SEM)



The SEM is designed for direct studying of the surfaces of solid objects

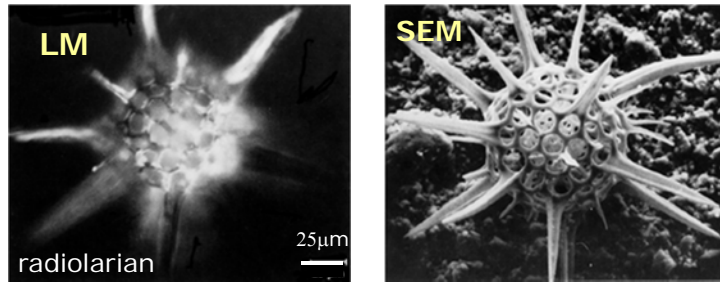
Scanning electron microscope (SEM) is a microscope that uses electrons rather than light to form an image. There are many advantages to using the SEM instead of a LM.



Advantages of Using SEM over LM

- The SEM has a large depth of field,

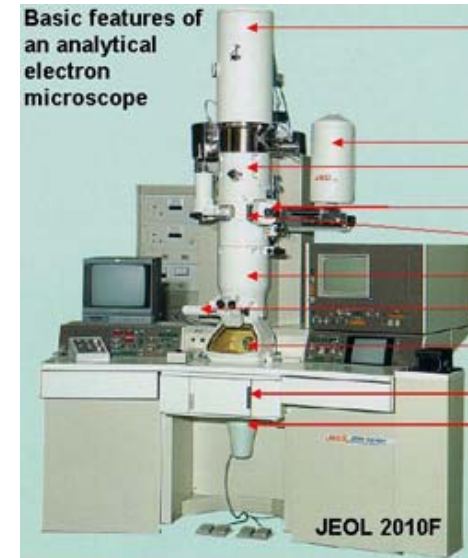
This allows a large amount of the sample to be in focus at one time and produces an image that is a good representation of the three-dimensional sample.



- The SEM also produces images of high resolution, *closely features can be examined at a high magnification.*
- *The combination of higher magnification, larger depth of field, greater resolution makes the SEM one of the most heavily used instruments in research areas and industries, especially in semiconductor industry.*



Transition Electron Microscope (TEM)



Electron Gun

EDS Detector
Condenser Lens

Specimen Holder
Objective Lens

Magnifying Lenses

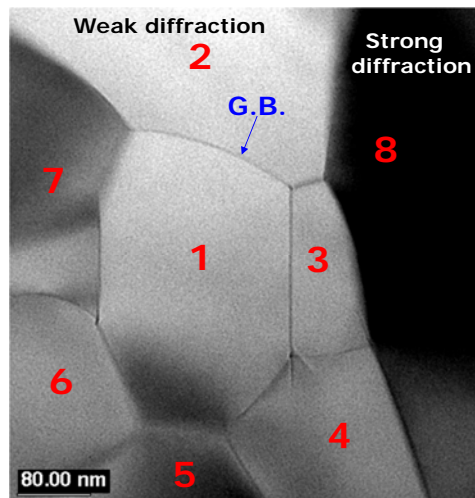
HAADF Detector
Viewing Chamber

Camera Chamber

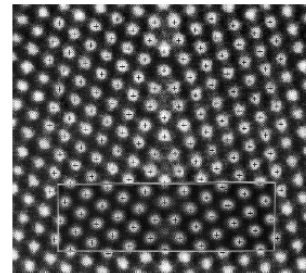
STEM Detector or
EELS



Transition Electron Microscope (TEM)



8 grains are in different orientations

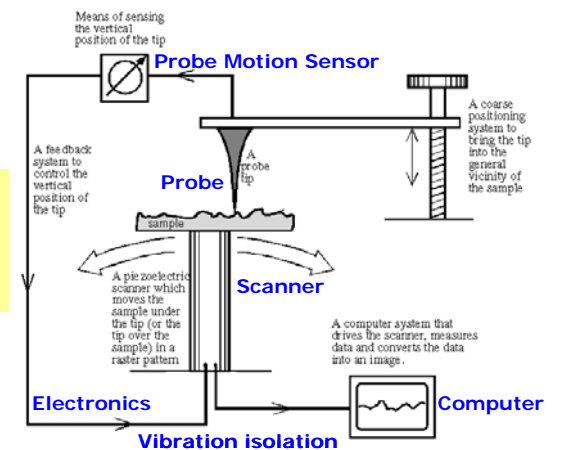


High-resolution TEM image of a tilt grain boundary in Al, Sandia National Lab.



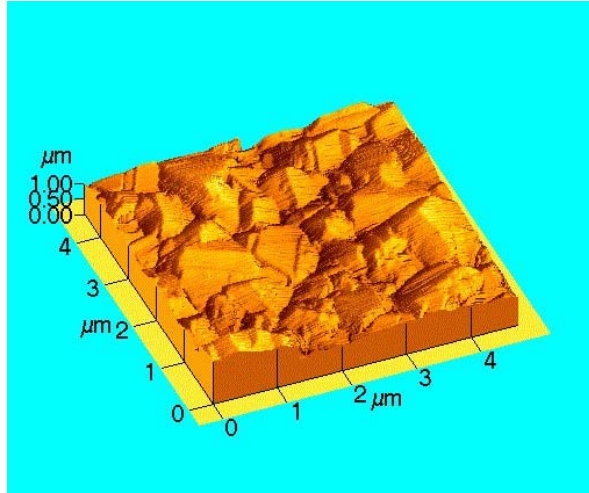
Scanning Probe Microscope (SPM)

SPMs are used for studying surface topography and properties of materials from the atomic to the micron level.





SPM



Question:

Why were commercial TEMs developed from about 1938 and SEMs from about 1965, whereas SPMs were not around before 1980's?



General Resolution of Microscopes

Type of Microscope	Approx. Resolution
Human Eye Å
Optical Light (OLM)	3000 Å
Scanning Electron (SEM)	10-50 Å
Transmission Electron (TEM)	2-5 Å, near atomic
Scanning Probe (SPM)	... Å, atomic



Next Time:
Diffusion