Microscopes

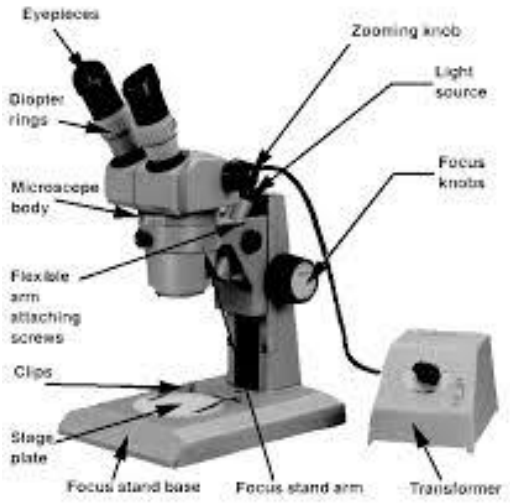
For a material to be a crystal, the material must have some sort of 3-dimensional order. The ***only*** way to determine if a sample has this order is to test if it can produce a diffraction pattern. However, an optical examination, that is required to mount the crystal for study on a diffractometer, will often be *very* suggestive of a material's crystallinity.

In general, most crystals will have *flat faces* and *sharp edges*. Although this is not always the case, most samples with these properties are later shown to be crystalline. Each crystal of the same material will generally have a similar shape or *habit* to other pieces of that material.

No glass has long-range, 3-dimensional order. However, it is possible to cut a piece of glass so that it has very flat faces and sharp edges. Conversely, many materials can have poor surface appearance (rough surfaces, lacking sharp edges) and still diffract X rays quite well.

Suitable samples for examination on a diffractometer are typically 0.1 – 0.5 mm on each edge. Most crystals of these dimensions will diffract quite well on the CCL instrument. The maximum dimension of a crystal analyzed on the CCL instrument should be no greater than 0.6 mm. Because these dimensions are so small, most people cannot visually see if a material looks crystalline without the aid of an ocular device, preferably a microscope.

Parts of a stereo-zoom microscope

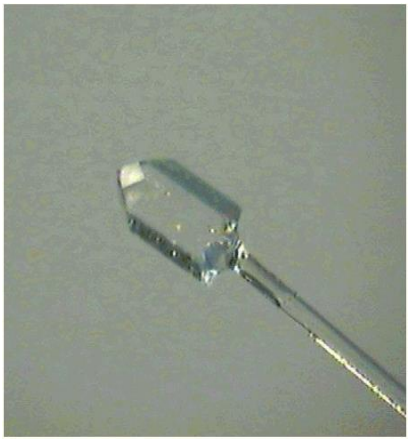


This diagram shows the components of the microscopes in our laboratory.

- The transformer powering the light source with our microscopes have potentiometers (variable resistors) that allow the user to adjust the brightness of the light on the sample.
- The eyepieces may be widened or closed to account for the different distance between the pupils of each person's eyes.
- The focus adjustment moves the microscope, and hence, the focal position, up and down to meet the material being observed.
- The zoom adjustment allows a user to increase or decrease the magnification being applied. Increasing magnification makes the object being observed larger and easier to see, but also decreases the microscope's "depth of magnification" and the overall area of observation.

Crystal Mounting Supports and Adhesives

The point of any mount (sample support) is to hold the sample rigid in the X-ray beam using the least amount of mounting material possible. To keep the diffraction pattern limited to the sample only, the mount support and adhesive should be made of amorphous materials. In the past, thin glass fibers were preferred for supporting the sample. Plastic mounts either as loops made from fishing-line type material or specially made plastic supports are now preferred for supporting the sample on an instrument. Plastic mounts are generally preferred to glass because the Si in a glass contributes more to background scattering than does the C, N, or O in plastic supports.



Glass fiber support

Plastic loop support

Plastic support

The adhesive used to hold the crystal on the support can be any material that will hold the sample rigidly. When the sample will be analyzed at room temperature, then the adhesive should dry to form a solid bond. Often used room-temperature adhesives are 5-minute epoxy, Super-Glue, Duco cement, or Elmer's glue. When the sample will be analyzed at low temperature, then any material that will harden at a low temperature may be used. Thus, inert oils or greases such as mineral oil, Paratone N oil, or high-vacuum grease are used to bond the sample to the support. Note that the *minimum* amount of adhesive necessary to do the task is preferred, because everything in the path of the incident X-ray beam will scatter X rays (which increases the background scattering in the diffraction pattern). Finally, the selected mounting fluid must *wet* the surface of the sample to adhere to the sample.

Best diffraction data will be obtained if the smallest face of the crystal is mounted on the top end of the support. Absorption effects are minimized if the long axis of the crystal is oriented parallel with the support axis. Both of these goals are rather hard to achieve!

Air-sensitive Mounts

Mounting an air-sensitive material requires a dry, anaerobic atmosphere. Such an atmosphere can be achieved in an anaerobic glove box equipped with a microscope, an anaerobic glove bag around a microscope, or a microscope over some container like a dish that is connected to a flow of an inert gas that is heavier than air, such as argon. Because most oils adsorb air, it is best to use a high vacuum grease to mount air-sensitive materials.

When mounting a sample in a dish filled with an inert gas, be sure to wear water-impermeable gloves such as nitrile gloves. Your skin breathes and the water from your skin can react with an air-sensitive sample. If the sample is provided in a Schlenk tube, purge the air from the extra plastic gas tube and the side arm of the Schlenk tube before attaching the gas tube to the Schlenk tube. As noted above, place some stopcock grease on a glass slide and move some of the sample into the grease as quickly as possible. Pick up the selected crystal and mount it on the end of a goniometer-head mount. Often a glass fiber is the quickest type of mount to use. Be more concerned about placing the crystal in the cold-stream of the low-temperature device on the instrument than getting the optimum orientation of the crystal on the mount.

Polarizing Lenses

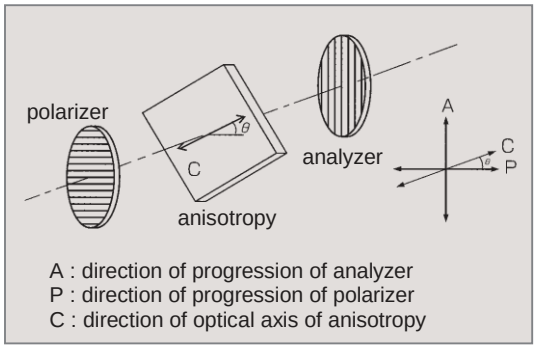


Fig. 2.1 Anisotropy between crossed nicols

When most crystalline materials are placed between crossed-polarizing lenses, special properties of the materials can be observed. Approximate information about the crystal system can be determined. If the sample is not isotropic, then it is sometimes possible to identify bulk twin domains in the crystal.

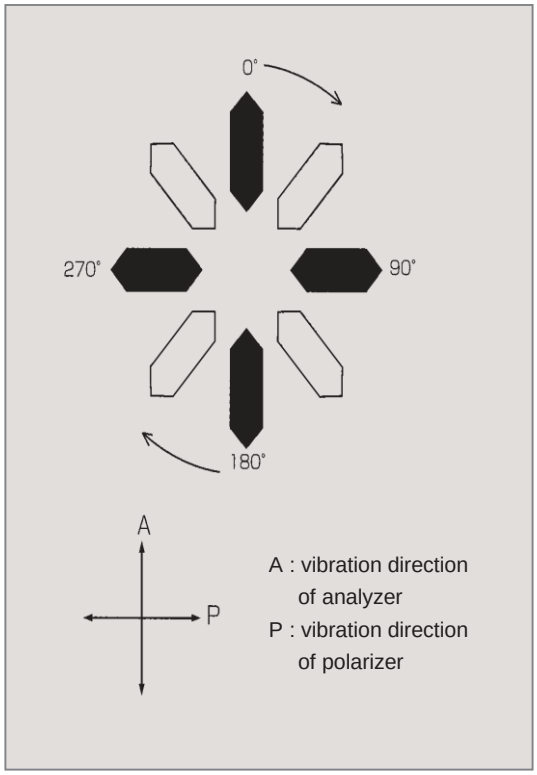
Transparent materials can be divided into three groups of anisotropy. Non-crystalline materials and crystals in the cubic crystal system are called isotropic – they will look the same at any rotation angle when rotated between crossed-polarized lenses.

Crystals with a single axial direction or optically-uniaxial crystals belong to the tetragonal, trigonal, or hexagonal crystal systems. Uniaxial crystals will appear to be isotropic when rotated between crossed polarizers in one particular orientation (viewed along [0 0 1]), but will show anisotropy when viewed in other orientations and rotated between crossed polarizers.

Biaxial crystals, crystals that will exhibit extinction when viewed along any axis and rotated between crossed polarizers, come from the orthorhombic, monoclinic, or triclinic crystal classes.

Extinction similar to that seen below occurs because of the anisotropy of a crystal when that crystal is rotated between crossed polarizer lenses.

If a crystal has bulk twinning, then part of the crystal will show extinction while another part of the crystal will be bright. The line between these regions is the location to cut the crystal to remove the twin.



All drawings shown above were taken from a handout on the "Basics of Polarizing Microscopy" produced by Olympus.
Images of crystals courtesy of Dr. Richard Staples at Michigan State University or Dr. Bruce Noll of Bruker AXS, Inc.