X-ray Diffraction Lab

TEM: What is it and How is it related to Diffraction.

Objective: To become familiar with diffraction as applied in a TEM.

Background: Handout, Appendix 2 in Cullity (differences between electron and x-ray diffraction)

This lab will not require a write-up. It is included so you can get some exposure to TEM (transmission electron microscopy) and electron diffraction. There are two main types of electron microscopes, SEM and TEM. In SEM the electrons do not pass through the sample and a reflected beam is used to image a sample. This means that the sample preparation requirements for SEM are less stringent than for TEM. It also means that the resolution of a SEM is less than that of a TEM. SEM's can look at sizes ranging from 10 micron down to about 200Å or so depending on the quality of the instrument. TEM can image extremely small sizes down to about 20 Å.

In TEM the electron beam passes through the sample. Electrons have charge so they pass through only thin samples. (Charge can build up on the sample in non-conducting samples so special sample preparation is necessary to make the samples conductive.) A typical wavelength for an electron is 0.05Å so is much smaller than that of an x-ray.

The electron beam is made from a filament just as it is in an x-ray tube. This beam is accelerated down a large voltage drop (similar to an x-ray tube). The SEM consists of a large evacuated chamber (UHV) where the electron beam can be collimated (with magnets) and can impinge on a thin sample. The beam can be adjusted for imaging (similar to a transmission optical microscope) or diffraction (similar to a pinhole collimated x-ray beam). Generally, the beam is collimated with a fairly diverging beam at first in order to obtain ***Kikuchi*** lines. These lines are formed when secondary electrons (Polychromatic) which result from the initial impact of monochromatic electrons with the sample, diffract from planes in a crystalline sample. This is shown on the first page of the copied journal article which is included.

Diverging, partially collimated electrons diffract forming a pattern similar to a Laue single crystal pattern since only a small region of a sample is irradiated, i.e. we are looking at basically one grain. Similar to a Laue pattern, arcs are formed which correspond to planes of a zone. Theses ***Kikuchi*** lines are not composed of spots (as in a Laue) because of the high polydispersity and divergence of the secondary electron beam. The arcs form continuous lines with bright regions. Similar to a Laue pattern, these arcs can be used as a guide to locate low-index planes at their crossing points. The ***Kikuchi*** lines are used as a road map to the crystalline structure so that one of the crossing points can be centered in the instrument and a diffraction pattern can be taken with a highly collimated beam over a very narrow region of the sample. The crystalline structure can be indexed at these crossing points of the ***Kikuchi*** pattern.

Bright field images in the TEM correspond to images taken using the incident beam and the contrast corresponds to regions of high absorption of electrons (high charge or electron density regions).

After low index planes are found by the ***Kikuchi*** method, an image can be made from the diffracted beam which will show bright spots for oriented planes and dark spots for less oriented regions. In this way nano-scale regions of crystalline orientation can be identified in the Bright field image using these ***Dark Field*** or diffraction images.

By the end of this lab period you should be able to describe what a ***Kikuchi*** line is, what a bright field and dark field image is, generally what the difference between an SEM and a TEM is, how diffraction can be used in conjunction with electron microscopy for crystalline samples, and what some of the differences between x-ray and electron diffraction are. This handout includes copies of a journal article on ***Kikuchi*** lines and a copy of the lab which was written up last year for the TEM for your information.