X-Ray Diffraction Lab
Experiment 2
Wulff Nets and Pole Figures

A) WULFF NET PROJECTIONS
Objective: To become familiar with Wulff net projections.

Background: Cullity pp. 63-78 (Chapter 2).
Wulff nets are a type of stereographic projection which is typically used for single crystal samples such as silicon wafers in the microelectronics industry. They are also critical to electron diffraction in a transmission electron microscope (TEM) which will be the subject of a latter lab.

Figure 2.27 on page 65 shows the relationship between the plane normal of a crystal, a sphere of projection of this normal, and its depiction on a 2-d Wulff Net. A point on the equator of the sphere of projection is chosen as the point of projection for the Wulff Net with projection lines passing through the plane normals on the sphere of projection to the plane of projection which touches the sphere of projection on the equator. Because of the geometry of the Wulff projection, the Wulff net has the appearance of an equatorial mapping of the earth, i.e. a straight line through the equator passes through the projection at the center and arcs centered on the poles diverge at the top and bottom. The point of projection in relation to the crystal is decided by the geometry of a diffraction experiment. This will be discussed further in later labs.   The purpose here is to manipulate stereographic projections in and of themselves but it should be kept in mind that rotations of these projections correspond to rotations of the sample in a diffraction experiment for identification of crystalline planes in a sample.

Wulff nets can be produced either manually or through the use of a computer program and you will become familiar with both approaches in this lab.   Two programs are available, Desktop microscopist and Crystal 2.29. (The latter is shareware but will only run on a Macintosh with a math coprocessor, i.e. not on a power PC. Desktop Microscopist is limited to Macintosh computers equipped with a special box and will also only run on older macs.) For this reason you will need to run the programs either in 515 Rhodes or in 551 ERC where these older macs are available. This can be done either during lab or at some other time by arrangement.

On a sphere of projection a plane which passes through the center of the sphere intersects the sphere in a circle of maximum diameter on the sphere or a **great circle**. Planes which do not pass through the center of the sphere intersect the sphere in circles of smaller diameter than a great circle, a small circle. Lines of longitude (lines which connect NS) are great circles (meridians). Except at the equator, lines of latitude (EW) are small circles.

The angle between two planes is the same as the angle between the two great circles which are formed by their intersection with the sphere of projection or by the angle between the two normals to the planes, the two poles of the planes. The equatorial stereographic projection (Wulff net projection) of a great circle is an arc and the projection of a small circle is a circle as shown in figure 2-28 on page 66.

Poles, or plane normal projections on the sphere of projection become points on the Wulff Net projection.

Angle between planes:
1) For two poles which lie on the same great circle (NS arc) the angle between the two poles (and their corresponding planes) can be measured directly off the Wulff Net as shown in figure 2-30 pp. 68.

2) For two poles which do not lie on the same great circle the projection (or the Wulff Net) must be rotated so that the two poles do lie on a great circle and then the angle between planes can be determined from the projection. This is equivalent to rotation of the sample in the diffractometer. This procedure is shown in Figure 2-31 on pp. 69. This type of rotation parallels rotation of the sample in a diffraction experiment in a plane normal to the incident beam.

Standard Projections
The plane of projection can be chosen to coincide with a standard low index plane (001) etc. for the crystal system of interest. This is termed a (001) standard projection for instance. For a standard projection the plane of projection will appear at the center of the Wulff net.

Locate a pole (hkl) on a Wulff Net.

1) The two sets of angles in the Wulff net are μ for N(+μ) and S(-μ) angles from the center (μ=0) and for E(+) and W(-) angles from the center (=0). These two angles can be calculated for the cubic system by,

$$\tan = k/l$$
$$\tan \mu = h/(k^2+l^2)^{1/2}$$

2) For a specific standard projection (e.g. 001 of figure 2-36 pp. 76) the north, south, east and west edges of the Wulff Net correspond to orthogonal planes at their respective angles The position of a pole (hkl) can alternatively be determined by calculation of the angles between the plane of projection and the pole of interest using the standard formulas given in the back of Cullity (appendix 3) and the method of determining angles between planes give above.

Determining the great circle trace (intersection of a plane with the sphere of reflection) for a pole.
This procedure is shown in figure 2-32 on page 70.
Rotate Wulff net so pole is on the equator. Then find trace by finding the longitude line (NS) which is 90° from the pole. Angle between traces can be determined as shown in figure 2-33 of Cullity on pp. 71.

Rotation of a pole about an axis.
1) Rotation about an axis normal to the projection can be done by rotation about the center of the Wulff net the required number of degrees.
2) Rotation about an axis lying in the plane of the projection:
     a) Rotate the Wulff Net so the axis falls on the NS line.
     b) Then move the poles along lines of latitude (EW) the required number of degrees.
This is shown in figure 2-34 on pp. 72 of Cullity. W to E is positive rotation angle. Cullity shows what to do if you reach the edge of the projection.
3) Rotation about an axis lying at some angle to the plane of projection (i.e. a pole about another pole):
     a) Bring the pole of the axis of rotation to the center of the projection by compound rotations of the type of 1 and 2 above.
     b) Rotate the required number of degrees using 1 above.
     c) Return the projection back to the original state by undoing a.
This procedure is shown in figure 2-35 on pp. 73 of Cullity.

Part A of this lab will involve performing the problems given on the next pages. These should be done either manually or using the computer programs provided. Everyone should get a chance to become aquatinted with the two softwares which we have for Wulff net construction and manipulation even if you decide to construct the nets manually.

B) POLE FIGURES

Objective: To become familiar with Pole Figures.

Background: Cullity pp. 297 to 321

Wulff nets are one kind of stereographic projection which is useful mostly for single crystal samples or in the TEM where a single crystal in a polycrystalline sample can be isolated through the use of an extremely thin sample and a fine beam. Wulff nets are useful for single crystals used to produce computer chips (silicon or germanium diamond cubic systems) where the electrical properties and chemical properties (used in etching) vary greatly with crystalline lattice direction.

In many industrial circumstances samples are polycrystalline and are formed with a preferred axis of orientation of crystalline planes, i.e. in rolled sheet metal, metal fibers, and in virtually all polymer samples which have been industrially processed, blown films, fibers, extruded samples, cast sheets. In these cases the sample you are interested in has at least one direction related to the processing conditions. This direction is often called the machine direction (MD), or draw direction. If the sample is a fiber the MD is the only important direction since the crystalline orientation is usually considered random about the other two directions, i.e. a fiber is cylindrically symmetric.

For sheet samples there is a sheet normal directions (ND) and a cross direction (CD) which define an orthogonal coordinate system of importance to sample properties. This is true for most polymer applications and many metals applications.

Since such industrial samples differ dramatically from the single crystal samples natural to Wulff net stereographic projections so it is not surprising that a stereographic projection native to these samples has been developed. The samples are polycrystalline so a separate stereographic plot is needed for each set of crystalline planes. Also, the crystals display a distribution of orientations so the plots will show regions of high probability of orientation rather than discrete directions of orientation. Pole figures are very useful to describe the mechanical and electrical properties of a typical industrially processed sample, i.e. the strength of a fiber in the draw direction or the strength and failure properties of a blown film in MD and CD.

Most stereographic projections are produced by computers so there are two features where you are likely to interact with these graphs. First is in setting up the parameters for a triple axis goniometer for creation of a pole figure (this is mostly beyond the scope of this lab but is described in Cullity on page 303. Second in interpretation of a pole figure produced by a computer as an aid in qualitative description of mechanical properties of a sample. This lab focuses on your becoming familiar with pole figures through several exercises and does not involve any measurements unfortunately. We will perform some crude experiments involving pole figures when we use the film camera in a later lab.

Concept of a pole-figure for a polycrystalline sample is fairly simple. A polycrystalline sample with preferred orientation is placed at the center of a sphere so that the MD lies in the equatorial plane of the sphere. Normals to one set of planes are projected on to the surface of the sphere, just as they are in the Wulff net projection. A plane is set in the MD/CD plane along the equator of the sphere and a line is connected from the south pole to the normals on the surface of the sphere. The pole figure looks like a dart board (concentric circles) since it is made by projecting the sphere of projection onto a plane from a polar perspective. Where these lines cross the plane of projection a spot is recorded. Figure 9-7 in the text shows this process for the (100) poles of a cold roll metal with cubic structure where MD is termed the roll-direction (RD) and CD is termed the transverse direction or TD in the figure. In part (a) the crystals are randomly oriented and the metal's mechanical properties are expected to be similar in all directions. In (b) the (100) poles are oriented preferentially with the cubic faces in the RD/TD/ND planes. For this system one would expect to see differences in the mechanical/conductivity properties at 45° to the roll direction for instance.

Since pole figures represent only one plane they must be referred to by the plane which is considered, i.e. the (hkl) pole figure.

For a fiber sample the fiber axis (FA) takes the place of MD. The CD and ND are equivalent so symmetry about the fiber axis is expected in the projection as shown in figure 9-8 on page 299. Using a pole figure it is simple to determine which crystalline planes preferentially align in the fiber axis, for instance, in figure 9.8 the [100] direction aligns in the FA and the (111) pole figure shows arcs which show an average angle from the FA of approximately $= 54.7°$, is the angle of tilt of the (hkl) plane normal from the fiber axis and can be used to determine the predominant crystalline direction which corresponds with the fiber axis. This is termed a **[100] fiber texture**. For a fiber texture only one of the quadrants contains unique information so the pole figures are often drawn as 1/4 of a circle.

The diffraction pattern from an oriented sample, when projected on a plane film or 2-d detector, displays arcs rather than Debye-Scherrer rings. This is shown in figure 9.9 and 9.11. The angle from the fiber axis on the diffraction pattern to the center of the arc is , as shown in figure 9-9. The angle from the main beam to the arc is 2 from the Bragg equation. The relationship between the diffraction angle, arc angle and the angle on the pole figure is given by equation 9-6 on page 301,

$$\cos\ =\cos\ \cos \qquad (9.6)$$

Which plane corresponds to a given arc is determined by 2 using appendix 3. is measured from the diffraction pattern. From a series of these measurements for different planes the fiber axis direction can be determined from known angles between planes for the crystal system (also appendix 3).

For a film or sheet material the preferred orientation is referred to by the preferred family of crystalline directions in the MD of the sample <uvw> and by the family of planes in the MD/CD plane of the sample {hkl}.

This lab is basically aimed at your becoming familiar with pole figures. It involves your conducting several exercises to demonstrate that you are familiar with these stereographic projections.

1) Problem 9.9 on page 323 in the text. Use polar graph paper included in this lab.

2) Polyethylene has an orthorhombic crystalline structure with 2 mer units per unit cell and lattice parameters a=7.40, b=4.93 and c =2.534Å. Why is the c dimension so much smaller and so much more specific in distance when compared with a and b directions?
The diffraction pattern from PE contains three prominent peaks corresponding to the (110), (200) and (020) planes. This lab includes two sets of pole figures for these planes. Determine the ideal orientations which correspond with these pole figures and qualitatively describe the preferred orientation in these two samples. Relate the chain direction to the film directions, (the chain axis is always the c-axis for polymer samples).

Your write-up will diverge somewhat from the standard format but you should include all relevant sections and adhere to the format as much as you think is reasonable.