

## Objective

The purpose of this experiment is to introduce students to the use of X-ray diffraction techniques for investigating various types of crystal structure encountered in metallic materials.

## Abstract

The X-Ray diffraction technique is used to determine the crystal structure and interatomic spacing of crystalline samples through constructive interference of reflected x-ray beams. Bragg's Law and X-Ray diffraction data, in combination with the expressions for interatomic spacing in terms of the lattice parameter and Miller indices for a crystal, can be utilized to identify crystal structures, determine lattice constants, and locate defects within a structure. X-Ray diffraction data has been collected for a FCC brass sample and a BCC steel sample; the data will be analyzed to determine the lattice parameter and preferred orientations for these materials.

## Background

Metallic and ceramic materials utilized in mechanical engineering applications have crystalline microstructures; many material properties are related to the atomic arrangement on various planes within the structures. Three basic crystal structures encountered in metallic materials are the Body-Centered Cubic (BCC), Face-Centered Cubic (FCC) and Hexagonal Close-Packed (HCP) as illustrated in Figure 1.

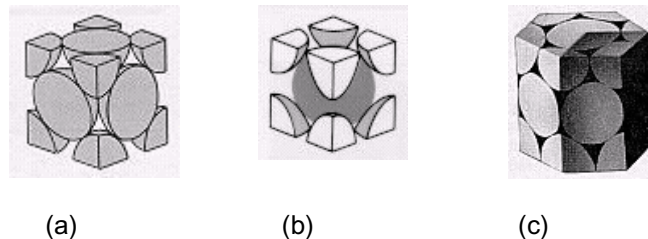


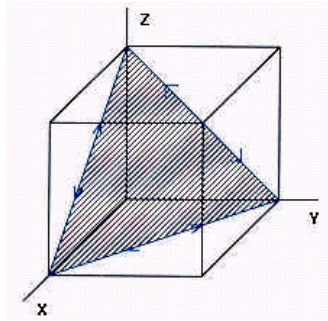
Figure 1 - Atomic arrangement in common metallic crystal structures: a.) Face-Centered Cubic (FCC), b.) Body-Centered Cubic (BCC), c.) Hexagonal Close-Packed (HCP). [Shackelford, 1996]

Engineering metals are commonly composed of substitutional solid solutions. Steels, which often contain carbon interstitially, are alloyed with Manganese, Chromium, Nickel, Molybdenum, etc. substitutionally to enhance performance in both its FCC and BCC structural phases; BCC ferrite is quite important. Aluminum, Titanium, and Copper alloys such as brass, generally contain substitutional atoms in their FCC structures. Titanium alloys have HCP and/or BCC structures. X-ray diffraction techniques can be utilized to identify the structure of various materials; this investigation will focus on the use of X-ray diffraction methods applied to basic metallic materials with BCC, FCC and/or HCP structures.

The atoms are arranged on the densest packed planes on the  $\{1\ 1\ 1\}$  family of planes in FCC structures. There are four sets of non-parallel planar stacks in each FCC crystal or grain, and on each plane there are three non-parallel close packed  $\langle 1\ 1\ 0 \rangle$  directions (two example planes are shown in Figure 2). Slip, which is the main phenomenon in plastic deformation, generally occurs on the most densely packed planes along the most densely packed directions. Since slip can occur along any one slip plane along any one direction on it, there are altogether 12 possible slip directions that can occur in FCC crystals. As a result, there are 12 slip systems in FCC materials such as Austenitic Stainless Steels (FCC), Al-alloys, Ni-alloys, Cu-alloys, etc.

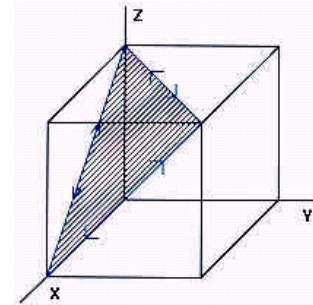
Compared with FCC crystals, the closed packed planes in BCC are not as tightly packed. In BCC, the planes with the highest atomic packing density are those in the  $\{1\ 1\ 0\}$  family of planes. There are 6 sets of non-parallel planes in this family in any one crystal as can be discerned from the BCC unit cell (two example planes are shown in Figure 3). The atoms are in contact with one another along the  $\langle 1\ 1\ 1 \rangle$  family of directions which are the densest packed in BCC crystals; slip occurs along such close-packed directions. There are 2 such directions on any one of these planes thus there are 48 slip systems in BCC materials. However, there are two major differences between slip in FCC and BCC materials: 1.) The BCC slip planes are not close-packed thus more energy is required to cause slip in BCC crystals, and 2.) Slip in BCC crystals can also occur along the same close-packed directions on other less-densely-packed planes after the slip process begins on the  $\{1\ 1\ 0\}$  family of

planes. The second set of planes upon which slip can occur are termed "Secondary Slip Planes"; more energy is required for slip on secondary planes as compared to the primary planes.



(a)

Figure 2a The (111) plane and its  $[1 \bar{1} 0]$ ,  $[1 0 \bar{1}]$  and  $[0 1 \bar{1}]$  slip directions



(b)

Figure 2b The  $(\bar{1} 1 \bar{1})$  plane, and its  $[1 0 \bar{1}]$ ,  $[1 1 0]$  and  $[0 1 1]$  slip directions

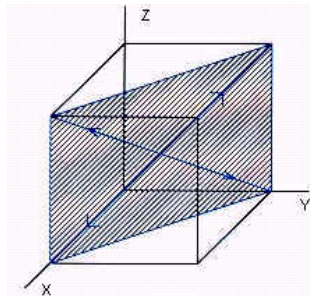
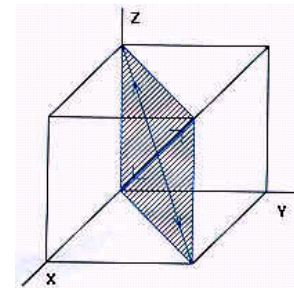


Fig. 3 (a) (110) plane, and its  $[\bar{1} 1 0]$   $[1 \bar{1} 1]$  slip directions

(a)



(b)  $(\bar{1} 1 0)$  plane, and its  $[1 1 1]$   $[1 1 \bar{1}]$  slip directions

(b)

Figure 3

Unlike FCC and BCC crystals, HCP crystals have only one set of slip planes, the  $\{0 0 1\}$  family of densely packed planes; these planes are equivalent in packing arrangement to the  $\{1 1 1\}$  family of slip planes in FCC crystals. Thus there are 3 slip directions on each plane, and hence there are only 3 slip systems in HCP crystals. HCP materials exhibit twinning and have secondary slip systems like BCC crystals and also become brittle at low temperatures; FCC crystals generally remain ductile and tough even at sub-zero temperatures.

### X-Ray Diffraction

"Our present knowledge of crystal structures has been obtained mainly by X-ray diffraction techniques which utilize X-rays about the same wavelength as the distance between crystal lattice planes" [Smith, 1993]. Typical X-rays used for diffraction are electromagnetic waves with wavelengths in the range of 0.05 to 0.25 nm. Diffraction X-rays are produced when a voltage is applied between a cathode and an anode target metal contained within a vacuum; the target metal emits X-rays when struck by electrons. With the wavelengths of the X-rays about equal to the distance between planes of atoms, reinforced diffraction peaks of radiation of varying intensities can be produced when a beam of X-rays strikes a crystalline solid.

If a monochromatic (single-wavelength) beam of X-rays of wavelength ( $\lambda$ ) is incident on a crystal, as illustrated schematically in Figure 4, at an angle such that the wave patterns of the beam leaving the various atomic planes are NOT in phase, then no reinforced beam will be produced (Figure 4a); this is termed Destructive Interference. If the reflected waves are in phase, then reinforcement of the beam occurs (Figure 4b); this is termed Constructive Interference.

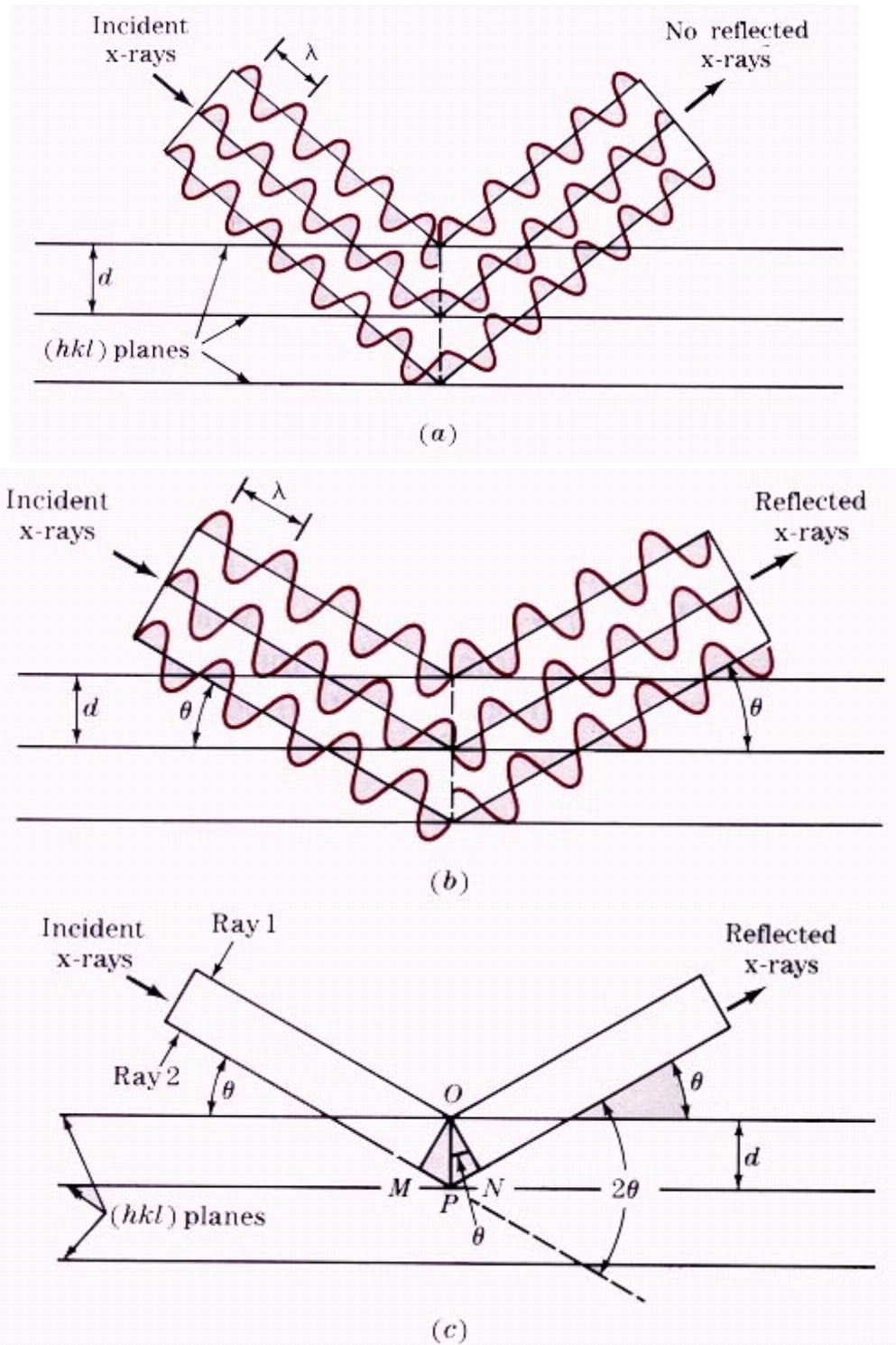


Figure 4 - The reflection of X-ray beam by the  $(hkl)$  planes of a crystal. a.) No reflected beam is produced at an arbitrary angle of incidence. b.) At the Bragg angle  $\theta$ , the reflected rays are in phase and reinforce one another. c.) Schematic representation of the in-phase condition. (Smith, 1993)

Considering incident X-rays 1 and 2 in Figure 4 to be in phase, the extra distance traveled by ray 2 must be equal to MP + PN, which in turn must be an integral number of wavelengths,  $\lambda$ . Thus

$$n\lambda = MP + PN \quad (1)$$

where  $n = 1, 2, 3, \dots$  and is termed the Order of the Diffraction. Since both MP and PN equal  $d_{hkl} \sin \theta$ , where  $d_{hkl}$  is the Interplanar Spacing of the crystal planes of Miller Indices (hkl) and  $\theta$  is the incident angle, the condition for Constructive Interference must be

$$n\lambda = 2d_{hkl} \sin \theta \quad (2)$$

This expression, known as Bragg's Law, gives the relationship among the angular positions of the reinforced diffracted beams in terms of the wavelength of the incident X-ray and the interplanar spacing of the crystal planes.

In most cases the first order of diffraction where  $n = 1$  is used. For cubic crystal structures, such as FCC and BCC, the Interplanar Spacing between two closest parallel planes with the same Miller Indices can be determined as

$$d_{hkl} = \frac{a}{\sqrt{h^2 + l^2 + k^2}} \quad (3)$$

where "a" is the Lattice Parameter (Lattice Constant). Equations (2) and (3) can be combined to represent the wavelength as a function of the lattice parameter; this equation can be used along with X-ray diffraction data to determine if a cubic crystal structure is FCC or BCC.

To analyze X-ray diffraction data, the crystal planes which diffract must be known for each type of crystal structure. For the BCC structure, diffraction occurs only on planes whose Miller indices when added together ( $h + k + l$ ) total to an even number (see Table 1). In the case of the FCC crystal structure, the principal diffracting planes are those whose Miller indices are either all even or all odd (zero is considered even); planes with mixed odd and even indices give rise to zero intensity and their diffraction peaks do not appear in the pattern.

Table 1 - Diffraction Planes in Cubic Crystals. [Shackelford, 1996]

Bravais Lattice	Constructive Interference (Reflections Present)	Destructive Interference (Reflections Absent)
BCC	$(h + k + l) = \text{Even}$	$(h + k + l) = \text{Odd}$
FCC	$(h,k,l)$ All Odd or All Even	$(h,k,l)$ Not All Odd or All Even

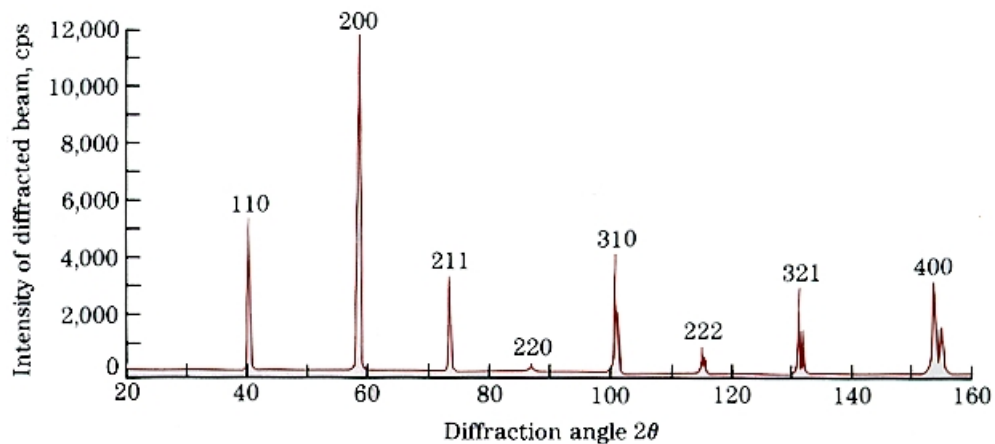


Figure 5- Sample Output (Smith 3.31).

## Lab Requirements

- Briefly explain the differences between  $[1\ 1\ 1]$ ,  $(1\ 1\ 1)$ ,  $\langle 1\ 1\ 1 \rangle$  and  $\{1\ 1\ 1\}$ .
- What is the interplanar spacing for the sets of  $\{1\ 2\ 1\}$  planes in iron? ( $a = 0.2865\text{ nm}$ )
- At what angle will iron diffract Cu-K $\alpha$  X-rays of wavelength  $0.1541\text{ nm}$ ?
- The radius of iron at room temperature is  $0.1241\text{ nm}$  when it has the BCC structure; compute the lattice parameter for BCC iron.
- Draw the  $(1\ 1\ 1)$  plane in a FCC unit cell and identify the three close packed directions.
- Draw the  $(1\ 1\ 0)$  plane in a BCC unit cell and the two diagonals on it; identify these two directions which are the closest packed directions in BCC crystals.
- The X-ray diffraction data collected for two samples (FCC Brass and BCC Annealed Stainless Steel) is provided in ASCII format. Load each file into a spreadsheet and plot the Intensity of the Diffracted Beam (cps) versus the Diffraction Angle  $2\theta$  (Degrees) for each sample. The plots should be similar to that presented in Figure 5. Examine each of the plots noting how the diffraction lines appear.
- Assuming first order diffraction, determine an expression for the diffraction angle ( $\theta$ ) as a function of wavelength ( $\lambda$ ), the lattice parameter ( $a$ ), and the Miller indices ( $h, k, l$ ) based on equations (2) and (3); then:
  - List the Miller indices for the first 5 planes that will diffract in FCC crystals based on this expression and the information in Table 1; begin with the plane which will diffract at the lowest angle, proceed to the next lowest angle, etc. (i.e., arrange the diffracting planes in the order of increasing diffraction angle).
  - Similarly, list the Miller indices for the first 4 planes that will diffract in BCC crystals.
- Analyze the diffraction pattern of the FCC Brass sample based on an input wavelength of  $\lambda = 0.154056\text{ nm}$  and the results determined above in question 8. The first peak in the pattern should match with the first possible diffraction plane and so on. Complete Table 2 for the FCC brass sample.
  - Identify the Miller indices of the planes for each diffraction peak shown.
  - Determine the Lattice Parameter "a" for the brass sample based on the data for each peak then compute the average value of "a" for the brass sample.

Table 2 - Analysis of the diffraction pattern of FCC Brass.

Peak #	Relative Intensity	$2\theta$	$\sin \theta$	Interplanar Spacing (nm)	h k l (nm)	Lattice Parameter (nm)
1						
2						
3						
4						
5						

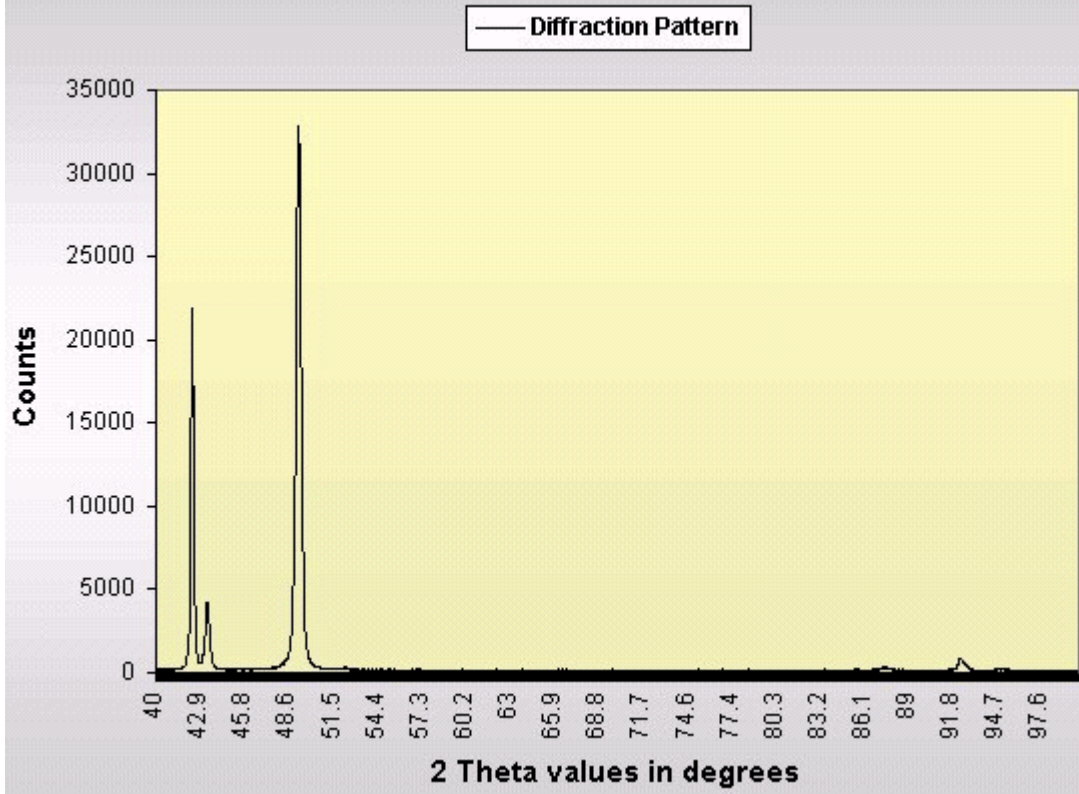
10. Analyze the diffraction pattern of the BCC Annealed Steel sample based on an input wavelength of  $\lambda = 0.154056$  nm and the results determined above in question 8. The first peak in the pattern should match with the first possible diffraction plane and so on. Complete Table 3 for the BCC Annealed Steel sample.
- Identify the Miller indices of the planes for each diffraction peak shown.
  - Determine the Lattice Parameter "a" for the steel sample based on the data for each peak then compute the average value of "a" for the steel sample.

Table 3 - Analysis of the diffraction pattern of BCC Stainless Steel.

Peak #	Relative Intensity	$2\theta$	$\sin \theta$	Interplanar Spacing (nm)	h k l (nm)	Lattice Parameter (nm)
1						
2						
3						
4						

11. Compare the diffraction patterns of the FCC Brass specimen with the diffraction pattern of the BCC Annealed Steel specimen.
- What major differences exist between the patterns?
  - If preferred surface orientations appear then certain planes appear more frequently parallel to the surface which in turn increases the intensity of their peaks. If only one strong peak appears then there is a 100% preferred orientation. What can you infer about the preferred orientation:
    - In the Brass sample?
    - In the Stainless Steel sample?

**X-Ray Diffraction Pattern for Brass**



**X-Ray Diffraction Pattern for Steel**

