CHAPTER 2 – CHARACTERIZATION TECHNIQUES

2.1 If a Bragg angle of 41.31° is observed for the first order diffraction from the {110} planes, of body-centered cubic niobium using copper $K\alpha_1$ radiation ($\lambda = 0.1541\ nm$), what is the interplanar spacing of the {110} planes?

Solution:

Using the interplanar spacing equation to determine d_{110} gives:

$$d_{110} = \frac{a}{\sqrt{1^2 + 1^2 + 0}} = \frac{0.3301}{\sqrt{2}} = 0.2334\ nm$$

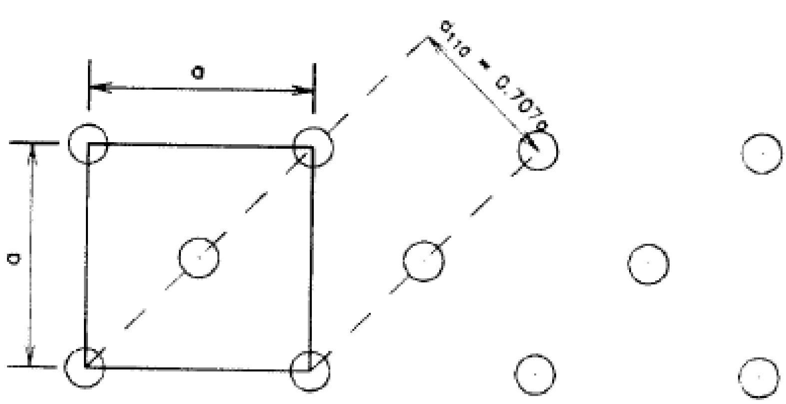
However, by the Bragg equation, assuming a first order reflection, we have $\lambda = 2d_{110}\sin\theta$. Solving this equation for d_{110} , one obtains:

$$d_{110} = \frac{2\lambda}{2\ \sin(\theta)} = \frac{2\ \times 0.1541}{2\ (0.660)} = 0.2334\ nm$$

Note by the Bragg law determination, the spacing between {110} planes equals that obtained by the interplanar spacing equation. In the body-center cubic lattice, a set of {110} planes also passes through the center atoms of the unit cells. The first computation above accordingly corresponds to the spacing between the {110} planes of a simple cubic lattice. The second gives the spacing in a body-centered cubic crystal.

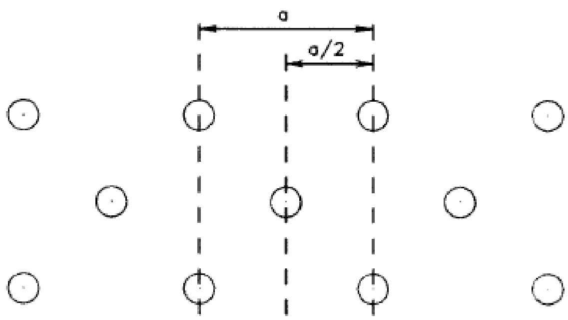
2.2 With the aid of a sketch similar to that for a simple cubic lattice in Fig. 2.10, demonstrate that the interplanar spacing of the {110} planes in the body-centered cubic lattice also equals $a/\sqrt{2}$.

Solution:



2.3 Using a geometrical argument similar to that in Prob. 2.2, show that the {100} interplanar spacing in the bcc lattice is $a/2$ and not *a,* so that the planes are actually {200} planes.

Solution:



2.4 Consider the Bragg equation, with respect to first-order reflections, from {100} planes of a bcc metal. By how much of a wavelength do the reflected path-lengths differ for two adjacent parallel (100) planes, if the interplanar spacing, *d*, is taken as *a* instead of *a*/2? Does this explain why the {100} plane is not listed as a reflecting plane in Appendix C? Explain.

Solution:

If it is assumed that constructive interference occurs at an angle of θ, which satisfies the Bragg equation for a first order reflection from {100} planes with a separation equal to *a,* then the path length after reflection will equal one wavelength or λ. The angle of reflection will be given by:

$$\theta = \sin^{-1}\left(\frac{\lambda}{2a}\right)$$

For reflection from planes with a separation of *a*/2 at this angle, the path-length $\lambda_{a/2}$ will be:

$$\lambda_{a/2} = 2(a/2)\sin\theta = \lambda/2$$

This signifies both a destructive interference, and that a reflection will not occur when $\theta = \sin^{-1}\left(\frac{\lambda}{2a}\right)$. It also accounts for the fact that the {100} plane is not listed as a reflecting plane in Appendix C.

2.5 With the aid of Eq. 2.2, calculate d_{hkl} for all the reflecting planes listed in Appendix C, using copper with *a* = 0.3615 nm.

Solution:

$$d_{111} = \frac{0.3615}{\sqrt{1+1+1}} = 0.2087\ nm \qquad d_{222} = \frac{0.3615}{\sqrt{12}} = 0.1090\ nm$$

$$d_{200} = \frac{0.3615}{\sqrt{4}} = 0.1808\ nm \qquad d_{400} = \frac{0.3615}{\sqrt{16}} = 0.0904\ nm$$

$$d_{220} = \frac{0.3615}{\sqrt{8}} = 0.1278\ nm \qquad d_{331} = \frac{0.3615}{\sqrt{19}} = 0.0829\ nm$$

$$d_{311} = \frac{0.3615}{\sqrt{11}} = 0.1090\ nm \qquad d_{420} = \frac{0.3615}{\sqrt{20}} = 0.0808\ nm$$

2.6 Given a Laue back-reflection camera (see Figs. 2.5 and 2.7), with a 5 cm file-to-camera distance, a 10 cm in diameter circular film, and the (100) plane of a copper crystal specimen normal to the X-ray beam:

(a) What is the maximum angel that some other plane of the copper crystal can make with the (100) plane and still reflect a spot onto the film of the camera?

(b) With the aid of the data listed in Appendix A, now determine which of the planes whose poles are plotted with the 100 standard projection shown in Fig. 1.33, will produce spots on the camera film.

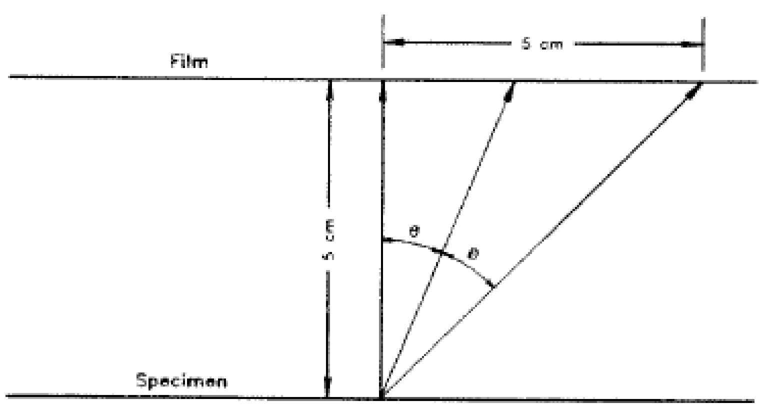


Fig. for Prb. 2.6

Solution:

(a) From the above figure: $\tan(2\theta) = 5\ cm/5\ cm = 1$, so that $2\theta = 45°$ and $\theta = 22.5°$.
(b) The planes whose poles are given in Fig. 1.33 that would produce spots are:
$(310), (3\bar{1}0), (301), (30\bar{1}), (410), (4\bar{1}0), (401), (40\bar{1})$.

2.7 Determine the powder pattern, *S*, values for the first four reflecting planes, listed in Appendix C, if the specimen is a gold powder. Assume that $Cu\ K_{\alpha 1}$, radiation (λ = 0.1541 nm) is used, and that the lattice parameter of the gold crystal is 0.4078 nm.

Solution:

The pertinent reflecting planes are {111}, {200}, {220}, and {311}. Now with regard to the Bragg equation, that is $n\lambda = 2d_{hkl}\sin\theta$, the order of reflection number *n* is included in the Miller indices. Consequently, solving the Bragg equation for the angle of reflection, θ results in the following relation:

$$\theta = \sin^{-1}\frac{\lambda}{2 \times d_{hkl}}$$

The arc distance along the file, *S*, can now be obtained from the relation S = 2Rθ. The values of d_{hkl} will now be determined.

$$d_{111} = \frac{0.4078}{\sqrt{1^2 + 1^2 + 1^2}} = 0.2354\ nm$$

$$d_{200} = \frac{0.4078}{\sqrt{4}} = 0.2039\ nm$$

$$d_{220} = \frac{0.4078}{\sqrt{8}} = 0.1442\ nm$$

$$d_{311} = \frac{0.4078}{\sqrt{11}} = 0.1230\ nm$$

The corresponding values of θ are as follows:

$$\theta_{111} = \sin^{-1}\left(\frac{0.1541}{2 \times 0.2354}\right) = \sin^{-1}(0.2373) = 19.11° = 0.334\ rad$$

$$\theta_{200} = \sin^{-1}\left(\frac{0.1541}{2 \times 0.2039}\right) = 22.30° = 0.388\ rad$$

$$\theta_{220} = \sin^{-1}\left(\frac{0.1541}{2 \times 0.1442}\right) = 32.30° = 0.564\ rad$$

$$\theta_{311} = \sin^{-1}\left(\frac{0.1541}{2 \times 0.1230}\right) = 38.79° = 0.677\, rad$$

With the aid of the above values of θ, it is possible to determine the *S* values. Thus:

$$S_{111} = 2 \times R \times \theta(0.334) = 3.34\, cm = 33.4\, mm$$

$$S_{200} = 10(0.388) = 3.88\, cm = 38.8\, mm$$

$$S_{220} = 10(0.564) = 5.64\, cm = 56.4\, mm$$

$$S_{311} = 10(0.677) = 6.77\, cm = 67.7\, mm$$

2.8 Assume that the electrons in a Transmission Electron Microscope are accelerated through a potential of 80,000 volts. Determine:

(a) The electron velocity given by this potential, by assuming that the energy the electrons gain falling through the potential equals the gain in their kinetic energy.

(b) The effective wavelength of the electrons.

(c) The Bragg angle, if the electrons undergo a first order reflection from a {100} plane of a vanadium crystal. Take the lattice parameter of vanadium as 0.3039 nm.

Note: See Appendix D for values of the constants needed in the solution of this equation.

Solution:

The information needed from Appendix D is:

1 electron volt = 1eV = 1.6 × $10^{-19}\, J$

The mass of the electron = *m* = $0.911 \times 10^{-30} kg$

Planck's constant = $6.63 \times 10^{-34}\, J/Hz$

(a) The energy that an electron received in falling through 80,000 V is:

$$80{,}000\ (1.6 \times 10^{-19}) = 1.24 \times 10^{-14} J$$

This is now equated to the kinetic energy $1/2\ mv^2$,where *v* is the velocity of the electron
$1/2(0.911 \times 10^{-30})v^2 = 1.24 \times 10^{-14}$

Solving this latter equation for the electron velocity yeilds:

$$v = \left(\frac{2(1.24 \times 10^{-14})}{0.911 \times 10^{-30}}\right)^{1/2} = 1.68 \times 10^{8} ms^{-1}$$

(b) The wavelength of the electron may now be determined as follows:

$$\lambda = \frac{h}{mv} = \frac{6.63 \times 10^{-34}}{0.911 \times 10^{-30}(1.68 \times 10^{8})} = 4.33 \times 10^{-3} nm$$

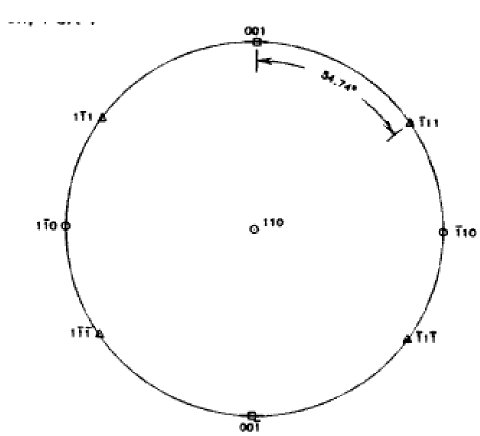
(c) The Bragg angle equation in the case of the electron microscope reduces to, $\theta = \lambda/2d$, where *d* is the interplanar spacing. This latter depends on the lattice constant of the crystal and the Miller indices of the reflecting plane. In the case where the reflecting plane is {200} in a bcc metal, the interplanar spacing is *a*/2 (see Prb. 2.2). This in vanadium $d_{200} = 0.3039/2 = 0.1520\ nm$. Substituting into the Bragg angle equation gives the results:

$$\theta = \frac{\lambda}{2d_{200}} = \frac{4.33 \times 10^{-3}}{2(0.1520)} = 0.0142\, rad = 0.816$$

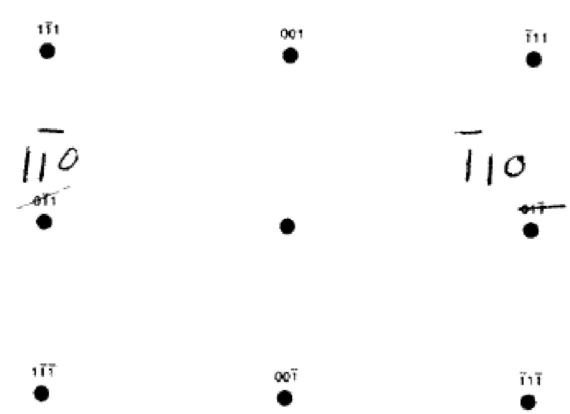
2.9 Rotate the 100 cubic standard projection of Fig. 1.32 about its north-south axis to obtain a 110 standard projection, and draw a figure similar to Fig. 2.19 showing the poles of the major planes of the zone whose axis is [110]. On the assumption that the electron beam of an electron microscope is parallel to [110], make a sketch, drawn to scale, of the electron diffraction pattern for this case that is similar to the one in Fig. 2.20 where the bean was paralell to [100].

Solution:

<u>Part I</u>



<u>Part II</u>



2.10 If the effective area of the raster of an electron microscope specimen, supplying information to the CRT of an electron microscope, has a 5nm diameter and the semicone angle, *α*, of the electron beam is 0.01 rad.:

(a) What would be the maximum usable magnification of the microscope if a digital spot on the screen of the CRT has a 100 μm diameter?

(b) What would be the depth of field at this magnification?

(c) What would be the depth of field if this microscope were to be operated at a magnification of 2000X?

Soltuion:

(a) The usable magnification, M, is a function of the picture element size, PES, and the size of a digital spot on the CRT screen. The relation is:

$$PES = \frac{spot\ size}{M}$$

Solving for M and substituting the values of the given parameters yields:

$$M = \frac{100 \times 10^{-6}}{5 \times 10^{-9}} = 20{,}000X$$

(b) By Fig. 2.28, the depth of field, D, is given by:

$$D = \frac{(PES)}{\tan \alpha} = \frac{5}{0.01} = 0.5\ \mu m$$

(c) For a magnification of 2000X the PES will equal 50 nm so that D will equal 5 μm.