Identification of Crystal Structure and Lattice Parameter

for Metal Powders Using X-ray Diffraction

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Abstract

The objective of the experiment was to identify crystal structure and lattice parameter for a polycrystalline metal powder by performing the diffractometer method and analyzing the x-ray diffraction measurements. X-ray diffraction as a characterization technique was also used to explore the orientation of a single crystal semiconductor, Si. The Si single crustal structure was determined to have a diamond cubic structure with an orientation of 001(crystal plane of 400). With a lattice parameter of 0.54307 nm, the d-spacing of Si crystal was calculated to be 0.135765 nm. Based on the x-ray analysis, the metal powder was found to be composed of two metals, one with BCC and one with FCC crystal structure. The lattice parameters for the BCC and FCC structures were identified to be 0.3310097 and 0.4054096 nm, respectively. It was concluded that the width of the diffraction curve increases as the thickness or grain size of the crystal decreases. The findings implied that a preferred orientation can be determined by running two peak profiles for the same sample, one as single crystal and the other as polycrystalline. If the peak intensity of a particular reflection is much higher than expected for random orientation, then a preferred orientation can be concluded.

Introduction

X-rays are classified as electromagnetic radiation that has high energies and short wavelengths. The wavelengths are on the order of the atomic spacing for solids. "Atoms in crystalline solids are positioned in orderly and repeated patterns that are in contrast to the random and disordered atomic distribution found in noncrystalline or amorphous materials."[1] When a beam of x-rays interjects a solid material, a portion of this beam will be dispersed in all directions by the electrons associated with each atom or ion that lies within the beam's path. In more details, when a parallel and monochromatic x-ray beam with a wavelength λ and angle of incidence θ is diffracted by a set of planes, oriented in specific directions, there are sharp intensity peaks, corresponding to the spacing between the planes, if the conditions of the Bragg's law are satisfied:

$$n\lambda = 2\, d_{hkl}\, \sin\theta_{hkl} \tag{1}$$

When the path difference between two incident and diffracted waves is an integral number of the wavelength, constructive interference occurs.[2] Interplanar spacing is a function of the Miller indices and lattice parameters as well as the crystal structure. Therefore, x-ray diffraction is useful in determining crystal structure, lattice parameters, interplanar spacings and atomic arrangements.[1]

The x-ray technique used in this experiment is the diffractometer method, which determine the angles at which diffraction occurs for powdered specimens. It is advantageous because it provides a quick analysis of both the direction and intensity of the diffracted rays that is easily adapted to a computer.[2] Figure 1 shows schematic diagram of an x-ray diffractometer composed of x-ray source, specimen, detector and the axis around which the specimen and detector rotate and the geometry for the diffractometer method. When the monochromatic x-ray beam is generated, the intensities of diffracted beams are detected with a counter. The specimen, x-ray source, and counter are always coplanar.[1] The counter is mounted on a movable carriage that may also be rotated about the axis. Its angular position in terms of 2θ is marked on a graduated scale. Carriage and specimen are mechanically coupled such that a rotation of the specimen through is accompanied by a rotation of the counter; this assures that the incident and reflection angles are maintained equal to one another. The angular positions of the diffracted lines are determined by the shape and size of the unit cell, while the relative intensities of these lines are determined by the positions of the atoms within the unit cell.[2]
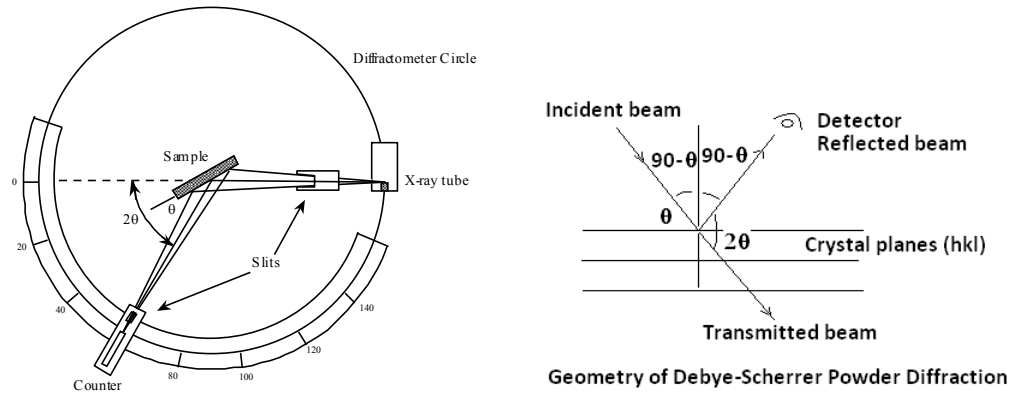
Figure 1. (a) X-ray diffractometer and (b) the geometry for the diffractometer method. [2]

Predicting the diffraction angle for any set of planes in a cubic crystal can be made possible by by combining the Bragg law and the plane spacing equation, which yields the following:

$$\sin^2\theta_{hkl} = \frac{\lambda^2}{4a^2}\left(h^2 + k^2 + l^2\right) = (\lambda^2/4a^2)\,Q^2 \tag{2}$$

where $Q^2 = h^2+k^2+l^2$. This equation predicts for a given cubic crystal of unit cell size a and for a given incident beam of wavelength λ all the possible Bragg angles at which diffraction can occur from the planes (hkl). Table I shows allowed reflections in different cubic crystal structures.

Table 1. The allowed reflections in the different crystal structures. [2]

Crystal Plane (hkl)	Q^2	Space lattices from which reflections are possible			
100	1	SC			
110	2	SC	BCC		
111	3	SC		FCC	DC
200	4	SC	BCC	FCC	
210	5	SC			
211	6	SC	BCC		
–	7				
220	8	SC	BCC	FCC	DC
300, 221	9	SC			
310	10	SC	BCC		
311	11	SC		FCC	DC
222	12	SC	BCC	FCC	
320	13	SC			
321	14	SC	BCC		
–	15				
400	16	SC	BCC	FCC	DC

The objective of the experiment was to identify crystal structure and lattice parameter for a polycrystalline metal powder by performing the diffractometer method and analyzing the obtained x-ray diffraction measurements. X-ray diffraction as a characterization technique was also used to explore the orientation of a single crystal semiconductor.

Experimental Procedure

The model of diffractometer used in this experiment was Rigaku G-Flex 58741. The software used was MDI data Scan. The accelerating voltage and the current for the x-ray tube were 25 volts and 10 mA. The sample being examined was a metal powder, which was prepared by using an amorphous tape and an amorphous glass to prevent any unwanted interruption in the results. The radiation used was Cu K-alpha radiation with $\lambda = 0.15418$ nm. The first step in the experiment was to calibrate the goniometer which measures the θ and 2θ values accurately. The sample used for calibration was a Si (001) crystal. The goniometer was calibrated by iteratively coupling and decoupling θ and 2θ knobs on the goniometer as shown in Table 2.

Table 2. The scans run in the x-ray with the corresponding parameters.

Scan	Purpose	Range	Step Size	Count time (s)	Mode	Coupled
1	Si	$2\theta = (67-71^{\circ})$	0.04°	0.5	Both axis	Yes
2	Calibration	$2\theta = (33-36^{\circ})$	0.01°	0.5	Theta($2\theta = 69.13^{\circ}$)	No
3		$2\theta = (68-70^{\circ})$	0.01°	0.5	Both axis	Yes
4	Powder A	$2\theta = (37-40^{\circ})$	0.04°	0.5	Both axis	Yes
5	Analysis	$2\theta = (35-100^{\circ})$	0.02°	0.5	Both axis	Yes

The Si crystal in the sample stage was placed between the clips so that the surface flushed with the clip. All the doors of the X-ray unit were closed. It is worthwhile to note that when the doors were opened, the beam did not turn on to prevent exposure to radiation. It was taken into consideration that the door was only opened when the beam was turned off or the shutter to the tube was closed so that no X-rays were coming out. The X-ray tube was turned on by slowly increasing the voltage to the allowed value and the current to the allowed value. After running the third scan, the peak position showed the highest counts, which indicates that the goniometer was calibrated. Then the metal powder was mounted. The peak profile was run twice to get better accuracy with a smaller step size as indicated in Table 2. The run of the peak profile was started by turning on the shutter. The software collected the data of intensity versus 2θ. The data were analyzed using an excel file.

Results and Discussion

The data collected gave the 2θ values of the peak positions, which were used to determine the possible hkl values by calculating the ratios of $\sin\theta$ from Bragg's law shown in Equation 1 and how they fit into the allowed reflections shown in Table1. The interplanar

spacings were calculated using the rearrange Bragg's law as mentioned in Equation 2 where λ is 0.15418 nm (Cu Kα x-rays). For the metal powder, the values were sorted out into two sets by trying two different combinations. For FCC, the values of Q^2 are 3, 4, 8, 11, and 12 for and for BCC, the values of Q^2 2, 4, 6, 8, and 10.

Figure 1 shows a diffraction pattern for Si crystal with a peak at 69.13 ° (2θ). Si was found to have a diamond cubic structure with an orientation of 001(crystal plane of 400). With a lattice parameter of 0.54307 nm, the d-spacing was calculated to be 0.135765 nm.
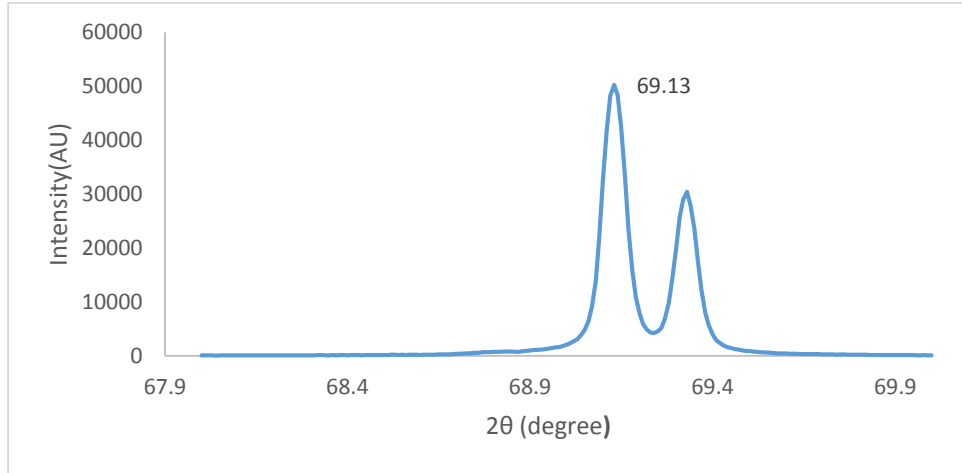

Figure 2. Diffraction pattern for Si Crystal using x-ray diffractometer.

The spectrum for Si crustal exhibited two peaks associated with $K_{α1}$ and $K_{α2}$ x-rays. However, since Si was a single crystal specimen, one peak was expected to be seen due to the fact that single crystal has only one orientation. Single crystals are defined as materials in which the atomic order spreads uninterrupted over the entirety of the specimen. [1] Nevertheless, the smaller peak can be ignored because there was no filter in the instrument. The intensity was high as expected due to the positions of the uniformly oriented atoms within the unit cell. [3]

Figure 3 shows a diffraction pattern for a metal powder using x-ray diffractometer. The powered sample was found to have more peaks than the Si crystal. The peaks were observed to be much thinner than the Si crustal due to the number of grains and have much smaller intensities. This is due to due to its polycrystalline nature. Polycrystalline materials are known to be composed of many small crystals or grains having different crystallographic orientations. Each powder particle can be considered a crystal. [1] Having a large number of them with random orientations ensures that some particles are properly oriented such that every possible set of crystallographic planes will be available for diffraction. [3]
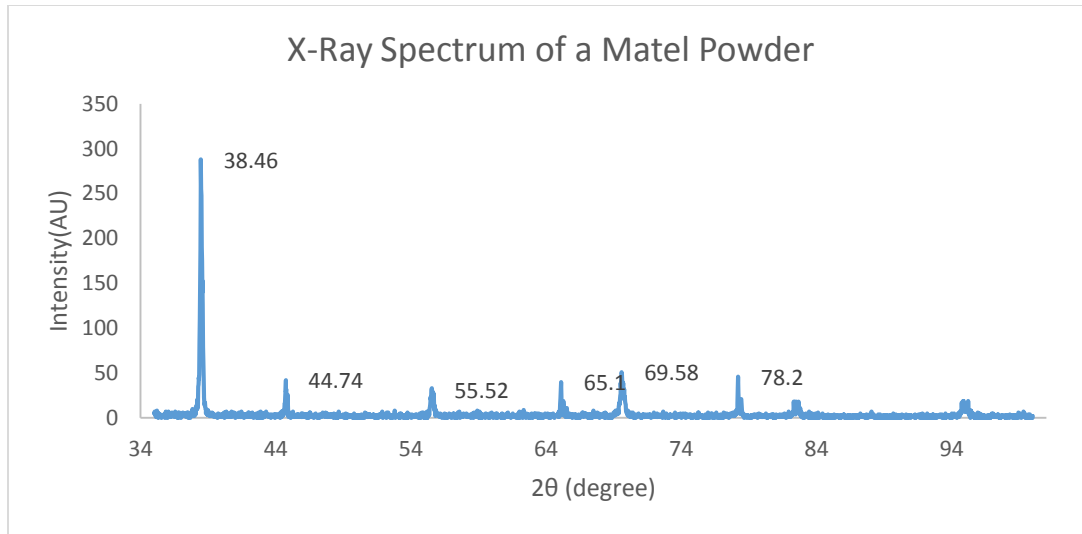
Figure 3. Diffraction pattern for a metal powder using x-ray diffractometer.

Based on the x-ray analysis, the metal powder was found to be composed of two metals, one with BCC and one with FCC crystal structure. The first peak at 2θ of 38.46 ° was a convolution of two peaks. Because the two metals have a peak in approximately the same 2θ, they constructively interfered with each other and appeared as one singular peak. Table 3 shows the 2θ, d-spacings, $\sin^2\theta$, hkl, crystal structure, and lattice parameter determination.

Table 3. 2θ, d-spacings, $\sin^2\theta$, hkl, crystal structure, and lattice parameter determination of the metal powder.

2θ (º)	$\sin^2\theta$	d-spacing (nm)	hkl	crystal structure	lattice parameter (nm)
38.46	0.1084787183	0.2340592	110	BCC	0.3310097
38.46	0.1084787183	0.2340633	111	FCC	0.4054096
44.74	0.1448458807	0.2027048	200	FCC	0.4054096
55.52	0.2169407359	0.1655048	200	BCC	0.3310097
65.10	0.2894820933	0.1433333	220	FCC	0.4054096
69.58	0.3255504004	0.1170296	211	BCC	0.3310097
78.20	0.3977519741	0.1222355	311	FCC	0.4054096

From the two x-ray spectra, it was can be concluded that the width of the diffraction curve increases as the thickness or grain size of the crystal decreases. [2, 3]

Conclusions

A beam of x-rays focused on a crystalline material may experience diffraction (constructive interference) as a result of its interaction with a series of parallel atomic planes

according to Bragg's law. The goal of the experiment was to identify crystal structure and lattice parameter for a polycrystalline metal powder by performing the diffractometer method and analyzing the x-ray diffraction measurements. X-ray diffraction as a characterization technique was also used to explore the orientation of a single crystal semiconductor. X-ray diffractometry is commonly utilized for crystal structure and interplanar spacing determinations.

Overall, the experiment was successful. It identified Si structure to have a diamond cubic structure with an orientation of 001(crystal plane of 400). With a lattice parameter of 0.54307 nm, the d-spacing of Si crystal was calculated to be 0.135765 nm. Based on the x-ray analysis, the metal powder was found to be composed of two metals, one with BCC and one with FCC crystal structure. The lattice parameters for the BCC and FCC structures were identified to be 0.3310097 and 0.4054096 nm, respectively. It is worthwhile to not that the error in the determination of the lattice parameter is smallest for larger angle reflections. In other words, the lattice parameter determination is more accurate from higher angle reflections. [2] The limitations were found in the x-ray spectra exhibiting undesired peaks. These peaks were determined to be due to the unfiltered beam and should be ignored. From the two x-ray spectra, it was can be concluded that the width of the diffraction curve increases as the thickness or grain size of the crystal decreases. A preferred orientation can be determined by running two peak profiles for the same sample, one as single crystal and the other as polycrystalline. If the peak intensity of a particular reflection is much higher than expected for random orientation, then a preferred orientation can be concluded. [2]

References

[1] W.D. Callister Jr., Materials Science and Engineering: An Introduction, Seventh Edition (Wiley, New York, 2007).

[2] L. Reynolds, Characterization of Materials MSE 210 Lab Manual, 2008.

[3] B. D. Cullity, Elements of x-ray diffraction (Addison-Wesley,1978).