Middle East Technical University

Department of Metallurgical and Material Engineering

Mete206 – Materials Processing Laboratory

Experiment 4: Crystallography and X-Ray Diffraction

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ABSTRACT

In the 20th century with the work of Laue, Friedrich, and Knipping the X-Ray diffraction started coming to life and from there on vast advancement was made in terms of material characterization (Epp, 2016). In this experiment with the help of X-Ray diffraction an unknown specimen's crystal structure, lattice parameter and the element itself was able to be found. During the computations Bragg's law as well as interplanar spacing formula for a cubic structure was used.

INTRODUCTION

When it is talked about solid materials, it can be said that they are categorized according to the atoms or ions inside and how these are arranged with respect to another, if for example they are arranged periodically over a wide range than it would be correct to describe that material as crystalline (Callister & Rethwisch, 2016). As materials engineers, the crystallographic structure, chemical composition and its physical properties would like to be known and it can be found by a nondestructive method named as the X-Ray diffraction (F.Sima, 2016). Before giving details about X-Ray diffraction, one must understand the mechanism of diffraction. It occurs when the wave encounters obstacles aligned regularly which may result in the scattering of the wave (Callister & Rethwisch, 2016). In the case that waves are scattered, there are two things that can happen.

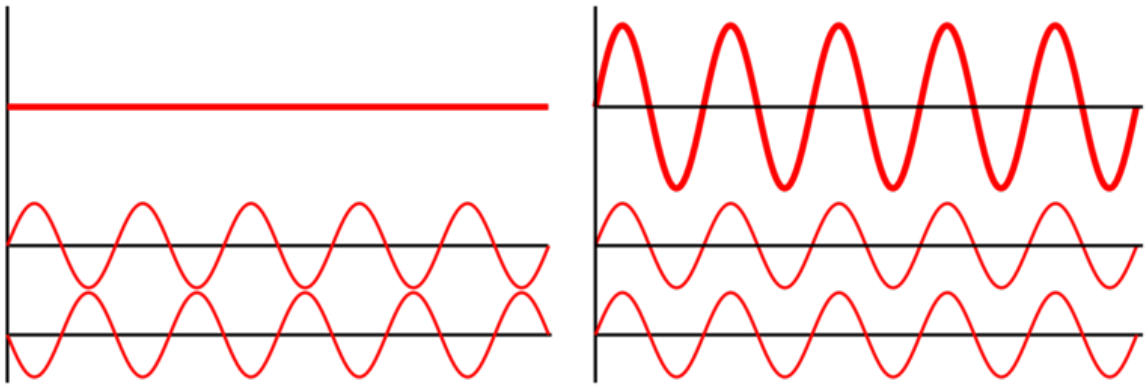

Figure 1. illustration of destructive interference of waves on the left side and mutual reinforcement of waves on the right side (Haade, 2006)

One of them as it can be seen from figure 1 is the destructive interference of the waves and the second one is the mutual reinforcement of the waves as illustrated in the right side of figure 1. When the waves mutually reinforce one another than it can be named as the diffracted beam.

To talk about x-ray diffraction, it means that a wave that has a wavelength very close to that of atoms are sent to the sample and the sample acts like an obstacle which scatters the wave. Braggs law, $n\lambda = 2d_{hkl}sin\vartheta$ shows the relationship of the wavelength, interatomic spacing and the angle of diffraction and it is the condition for diffraction where in the case that it is not obliged then there would be destructive interference among the waves which will lead a low intensity diffracted beam (Callister & Rethwisch, 2016). Figure 2 illustrates the X-Ray diffraction that occurs as well as the diffracted beam.

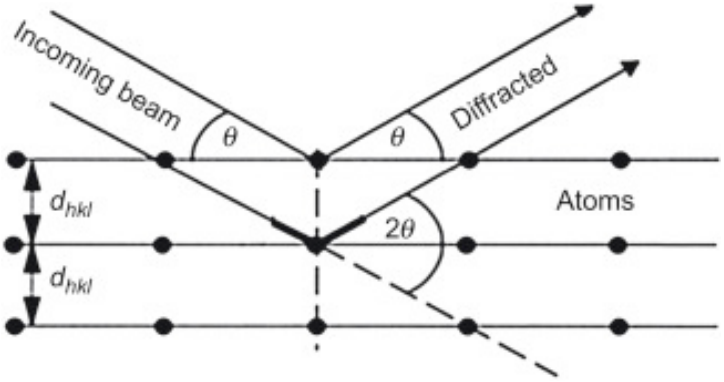

Figure 2. schematic of X-Ray diffraction (Epp, 2016)

The interatomic distance which is showed as d_{hkl} in figure 2 can also be found by doing certain computations. However, for different crystal systems the formula that is used to find the interatomic distance varies.

To find the interatomic separation for a cubic crystal structure the formula;

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

Is used. "a" is the lattice parameter and h, k, l are the miller indices. There are some rules defined as the reflection rule which says that for BCC crystal structure h+k+l must be even and that for FCC crystal structure h, k, l must be all either even or odd (Callister & Rethwisch, 2016).

One can combine the Bragg's law with the formula of interatomic separation to solve for the lattice parameter where the Atomic radius of the material can be found, if it is known that the material is pure. Therefore, if the material has a cubic structure, by combining the two laws;

$$\frac{\sin^2\theta}{h^2 + k^2 + l^2} = \frac{\lambda^2}{4a^2}$$

Equation is obtained. Which can be solved to find the lattice parameter.

X-Ray diffraction tells a lot about a material and a lot of thing can be learned from this test. To give some examples, one could analyze phases, investigate the crystallographic textures and find residual stress which are very useful information to have (Epp, 2016). However, there also exists some challenges in XRD test. For the computer software that does the analyzation of the data, one needs to supply the knowledge of what material that the specimen consists of also there are certain conditions where the XRD test may not be so accurate.

EXPERIMENTAL

Procedure

In the experiment a simulated data of X-Ray diffraction was given to us. From the data the diffraction angles were found and a total of 7 peaks were observed. The knowledge that the specimen is a pure element that has a cubic structure is also given. With the use of this information and also Bragg's law as well as the formula of plane spacing equation, the lattice parameter can be found as well as the crystal structure of the specimen. It is important to note that a wavelength of 1.5406Å was assumed to be used. In the end, the specimen that we did not know what element it is was found.

RESULT & DISCUSSION

To begin with, the specimen that was put through the X-Ray diffraction test was in powder form. The reason for this is that it decreases the probability of error by allowing the observation of all possible peaks.

Table 1. Data obtained from X-Ray diffraction

			$h^2+k^2+l^2$				$\frac{sin^2\theta}{s}$				Lattice Parameter				
Peak #	***2ϑ***	$sin^2\theta$	***DC***	***FCC***	***BCC***	***SC***	***DC***	***FCC***	***BCC***	***SC***	***a(DC)***	***a(FCC)***	***a(BCC)***	***a(SC)***	***hkl***
1	40.60	0.12036435	3	3	2	1	0.04012	0.04012	**0.06018**	0.12036	3.84566628	3.84566628	**3.13997337**	2.22029646	110
2	57.73	0.23304515	8	4	4	2	0.02913	0.05826	**0.05826**	0.11652	4.51320287	3.19131636	**3.19131636**	2.25660144	200
3	73.87	0.36109116	11	8	6	3	0.03283	0.04514	**0.06018**	0.12036	4.2515551	3.62573837	**3.13998154**	2.22030224	211
4	87.87	0.48141652	16	11	8	4	0.03009	0.04377	**0.06018**	0.12035	4.44078137	3.6821014	**3.14010662**	2.22039069	220
5	101.67	0.60113727	19	12	10	5	0.03164	0.05009	**0.06011**	0.12023	4.33061793	3.44162614	**3.14176046**	2.22156012	310
6	116.27	0.72130087	24	16	12	6	0.03005	0.04508	**0.06011**	0.12022	4.44331694	3.62795309	**3.14189954**	2.22165847	222
7	133.13	0.841828	27	19	14	8	0.03118	0.04431	**0.06013**	0.10523	4.36244634	3.65952751	**3.14132039**	2.37461501	321

In table 1 all the necessary calculation and the resulting values can be seen to figure out the lattice parameter of the sample. It is important to note that for $\frac{sin^2\theta}{s}$ value that is constant for every peak is the crystal structure that the specimen has. When it is looked at table 1, BCC and SC crystal structure is continuous up to the 6th peak however, after the 6th peak the SC value can be seen to deviate while the BCC value is still continuous. Therefore, it can be concluded that the specimen has a crystal structure of BCC.
To determine the lattice parameter, the Braggs law and the interatomic separation formula was combined as mentioned before to obtain;

$$\frac{\sin^2\theta}{h^2+k^2+l^2} = \frac{\lambda^2}{4a^2}$$

Where, from here the lattice parameter was calculated for every peak. To obtain the real lattice parameter value the last three peaks that had the highest θ value was chosen and the average

of them was taken to obtain the final lattice parameter which can be seen in table 2. Since it has a BCC structure and a lattice parameter of 3.14 it can be said that the element used in the experiment is Molybdenum (periodictable, 2020).

Table 2. Final results obtained from X-Ray diffraction data

Crystal Structure	*Lattice Parameter(Å)*	*Element*
BCC	3.1417	Molybdenum

Table 2 tabulates the final result of the X-Ray diffraction where the lattice parameter was obtained. Using the lattice parameter from material database the element which had 3.14 Å was found and it is the element molybdenum.

CONCLUSION

X-Ray diffraction is a very important test that gives a lot of information to the user. In this experiment the X-Ray diffraction has been done to find an unknown specimens crystal structure, lattice parameter therefore the element itself. At the beginning, it was only said that the specimen was pure and had a cubic structure. Knowing that the $\frac{\sin^2\theta}{h^2+k^2+l^2}$ must be continuous for every peak it was determined that the crystal structure must be BCC. Lattice parameter was also calculated to eventually find what the element that was tested is. This is one of the examples on how X-Ray diffraction can be used and there are more advanced data that can be obtained from doing this test.

REFERENCES

Callister, W. D., & Rethwisch, D. G. (2016). *Fundamentals of Materials Science and engineering.* Wiley.

F.Sima, C.Ristoscu, L.Duta, O.Gallet, K.Anselme, & I.N.Mihailescu. (2016). Laser thin films deposition and characterization for biomedical applications. *Laser Surface Modification of Biomaterials*, 77-125.

Haade. (2006). *Wikimedia*. Retrieved from Wikimedia: https://commons.wikimedia.org/wiki/User:Haade#/media/File:Interference_of_two_waves.png

Epp, J. (2016). X-ray diffraction (XRD) techniques for materials characterization. *Materials Characterization Using Nondestructive Evaluation (NDE) Methods*, 81-124.

periodictable. (2020). *periodictable*. Retrieved from periodictable: https://periodictable.com/Properties/A/LatticeConstants.html