NE 226 L

Characterization of Materials

Title Page

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1. Introduction

X-ray diffraction techniques are a very useful characterization tool to study, non-destructively, the crystallographic structure, chemical composition and physical properties of materials and thin films. It can also be used to measure various structural properties of these crystalline phases such as strain, grain size, phase composition, and defect structure. XRD is also used to determine the thickness of thin films, as well as the atomic arrangements in amorphous materials such as polymers.

Powder diffraction is a technique used to characterize the crystallographic structure, crystallite size (grain size), and preferred orientation in polycrystalline or powdered solid samples. Powder diffraction is commonly used to identify unknown substances, by comparing diffraction data against a database maintained by the International Centre for Diffraction Data (ICDD). As such, powder diffraction is mainly used for *finger-printing* solid materials. It may also be used to characterize heterogeneous solid mixtures to determine relative abundance of crystalline compounds which can provide structural information on unknown materials.

Figure 1 shows the basic features of an X-ray diffractometer, in which the diffraction angle 2θ is the angle between the incident and diffracted X-rays.

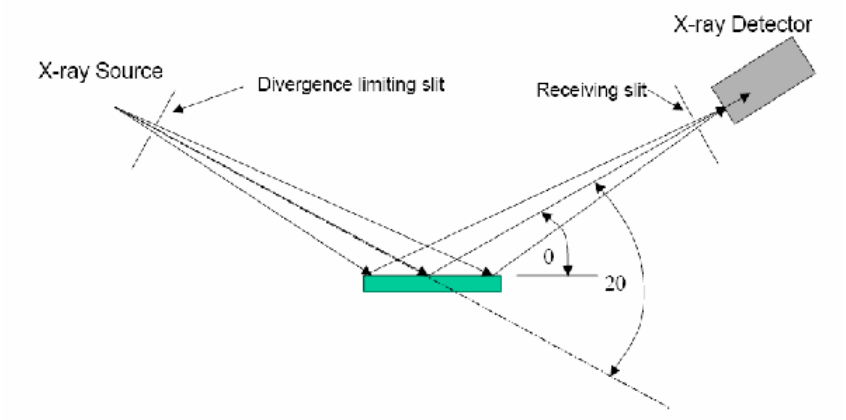

Figure 1 Basic features of an X-ray diffractometer

A typical diffraction spectrum consists of a plot of reflected intensities versus the detected angle 2θ. The 2θ values of the peak depend on the wavelength of the anode material of the X-ray tube. By choosing the right anode and energy of accelerated electrons, a known wavelength and therefore a known energy of X-rays will be generated. Copper X-ray tubes are most commonly used for X-ray diffraction of inorganic materials. For practical applications of X-ray diffraction, we typically want to use x-rays of a single wavelength, i.e. monochromatic radiation to improve experimental results. In general, Kα radiation is used for analytical work while all other radiation (Kβ, etc.) are removed by means of a nickel filter.

The incident X-rays interact with the sample to create secondary "diffracted" beams. These diffracted beams are related to the interplanar spacings between the numerous crystalline planes in the crystalline powder. The equation governing this diffraction angle is known as Bragg's Law:

$$n\lambda = 2d\sin(\theta)$$

where n is any integer,
λ is the wavelength of the incident X-rays
d is the interplanar spacing, and
θ is the diffraction angle

The objective of this experiment is to determine the lattice constants and other structural details of a crystalline sample ($CaCO_3$) by means of Bragg's Law, and to identify unknown materials with the aid of an XRD database.

2. Materials and Methods

The known materials to be tested are $CaCO_3$ nano powders. The instrument that is used in this experiment is a X-ray powder diffractometer: X'Pert Pro manufactured by PANalytical.

The experimental procedure to operate X'Pert Pro is outlined in the lab manual of *Characterization of Materials* [1], under experiment number 4 X-Ray Powder Diffraction. The sample is prepared by grinding them to a fine powder, loaded into the sample holder, the necessary measurement parameters set up.

3. Results

Figure 2 summarizes the 2θ versus intensity output obtained from the X-ray diffractometer.

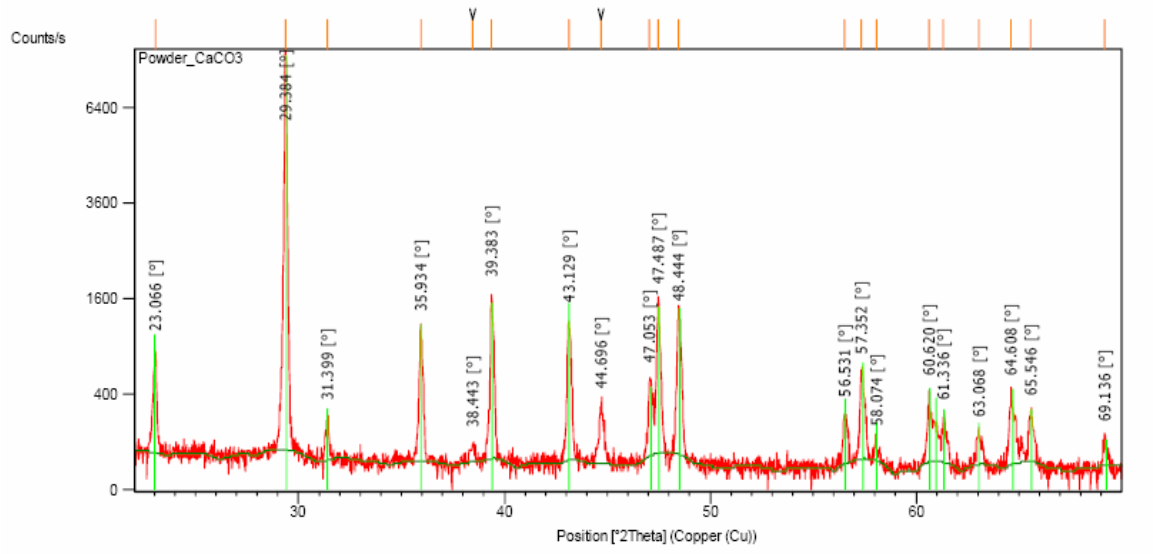

Figure 2 Diffractometer output for $CaCO_3$

Table 1 outlines the d-spacings of the five strongest peaks using Bragg's Law for the provided $CaCO_3$ demo sample.

Peak #	θ (°)	Peak Intensity (counts/s)	d (Å)
1	29.384	1685	1.57
2	39.383	310	1.21
3	43.129	265	1.13
4	47.487	285	1.05
5	48.444	324	1.03

Table 1 Interplanar spacings of the top five strongest peaks

4. Discussion

Calcium carbonate is an inorganic compound that has some excellent structural properties. It belongs to the rhombohedral crystal system and has lattice parameters corresponding to a, b, c, α, β, γ values of 4.99 Å, 4.99 Å, 17.08 Å, 90°, 90°, and 120°. It has a calculated density of 2.70 g/cm^3 and a unit-cell volume of 368.93 10^6 pm^3.

Because the maximum peak intensity occurs at an angle of 29.38°, we infer that the [hkl] parameters that is best predicted is the [1 0 10] parameter system.

X-ray powder diffraction in general can be used in many different applications. Some of these applications are:

- Measurement of structural properties of crystalline phases
- Strain characterization within crystallite domains

- Grain size determination
- Phase composition description
- Defect structure characterization

Relative to other methods of analysis, X-Ray powder diffraction allows for rapid, non-destructive analysis of mixtures with possibly multiple components. One drawback of this method is the focus needed on sample preparation. In today's modern day of automated data collection and analysis, sample preparation is the most critical factor that influences the quality of the analytical data obtained. Therefore sample preparation can have a significant impact on the results of diffraction experiment.

X-Ray powder diffraction allows scientists to quickly analyze unknown materials and perform materials characterization in numerous fields. Identification is performed by comparison of the obtained diffraction pattern to a known standard or to a database such as the International Centre for Diffraction Data's (IDCC's) Powder Diffraction File (PDF) or the Cambridge Structural Database (CSD).

5. Conclusions

In conclusion, X-Ray Diffraction (XRD) is a very powerful technique used to uniquely identify the various crystalline phases present in materials.

In this experiment, we studied the crystal structure of an inorganic compound: calcium carbonate. We determined certain key lattice constants associated with it and also analyzed other structural details with the help of Bragg's Law.

In particular, we determined that the interplanar spacing associated with the strongest peak intensity was 1.57Å. This occurred at an incident angle of 29.38°. Because the maximum peak intensity occurred at an angle of 29.38°, we inferred that the [hkl] parameters that is best predicted is the [1 0 10] parameter system.

6. References

[1] Q. Xie, F. McCourt, *Nanotechnology Engineering NE 226 Lab Manual*, University of Waterloo, Waterloo, pp 43-56 (2007).