Parkland College

A with Honors Projects

Honors Program

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X-Ray Diffraction

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For this project I had originally planned to analyze a compound used frequently in my old job at

Euclid. I was going to find out what this Powder "x" was by doing chemical test that I had learned to

perform in chemistry 102 and 101 here at Parkland. My professor thought this was an excellent and

unique idea and was thoroughly supportive of this. Since she herself has a background in geological

chemistry, she proposed that since this was a dry powder there was a technique called x-ray diffraction

that allowed one to be able to quickly and accurately identify a chemical compound. Due to time

constraints with University of Illinois geology faculty, I wasn't able to get to try this for the honors

project, but this whole new process had me interested. That is why my honors project is a review into

what is, who developed, and why use x-ray diffraction.

X-ray diffraction (XRD) is the go-to technique used when one has a sample of an unknown solid

compound (Barthelmy). Due to the availability and speed of XRD machines many geological scientist use

this testing method first. The basic technique takes a dry ground up sample, which may be mixture, and

bombards it with electron emissions from a cathode ray tube at a fixed wavelength. Then by using

Bragg's law ($n\lambda=2d\sin\theta$) with the measured angle of reflection the inner atomic spacing is found. (SERC)

This all began thanks to a man named Max von Laue. In 1912, he came up with the hypothesis

that crystalline substances act as 3D diffraction grating for x-rays (SERC). Following this W.H Bragg and

his son W.L. Bragg made the law that is now used. They performed three diffractions on sodium chloride

(NaCl), zinc sulfide (ZnS), and diamond (C). These test and creation of the law led them to receive a

noble prize for their work in 1915 (Moeck).

Bragg's law is one of the most important aspects of XRD. The equation is $n\lambda=2d\sin\theta$. Where n is an integer, λ is the wavelength in nm, d is the inner spacing, and theta (θ) is the angles of the diffractions. This equation is used to create a graph that plots the angles to find intensity. On this graph three prominent spikes are use to identify what the sample is. **Figure #1 shows a sample graph.**

Figure #1: Sample XRD Graph.
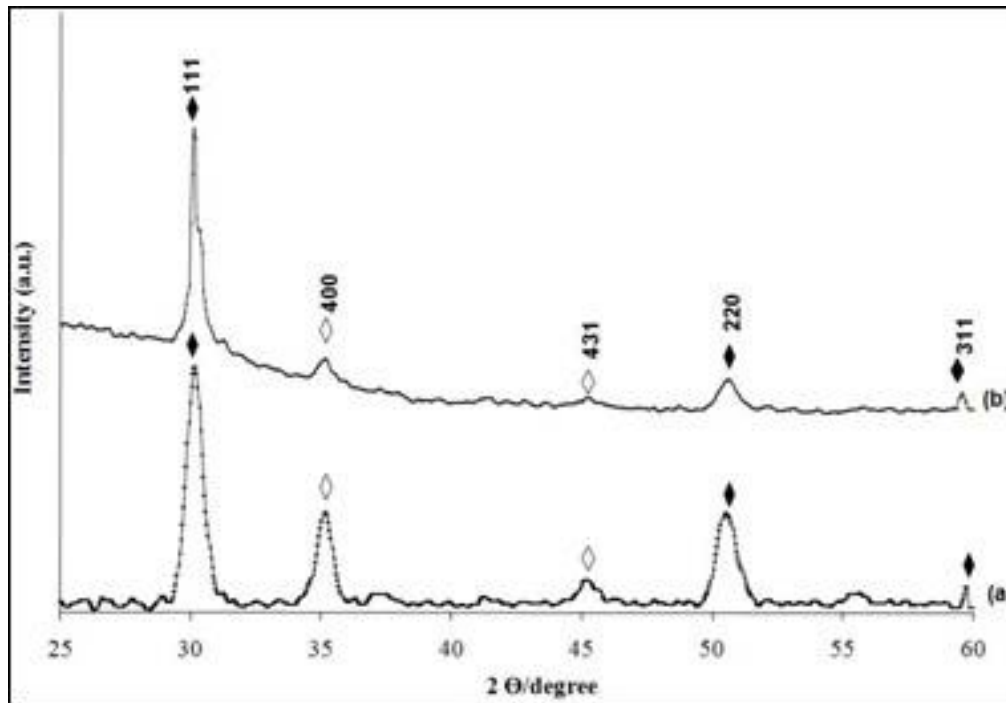
Today XRD allows for any crystalline substance to be identified. To classify a mineral, the observer takes a look at the resulting graph of the d-spacings found with Bragg's law and then uses the three most prominent spikes from its angles that produce the most diffraction. It has also been found there is a possible of seven crystal class a mineral's unit cell can be a part of, cubic, tetragonal, hexagonal, trigonal, orthorhombic, monoclinic, or triclinic. **Figure #2 shows these seven shapes**. These classes can help lead to prediction of the mineral such as strength, boiling point, and melting point

(Moeck). Every mineral has a unique id, which is made up of its specific angles, which when found is entered into a global or national database (SERC).

Figure #2: The Seven Crystal Systems. Every mineral can only be one of the seven classes.

Source: http://www.alientravelguide.com/science/chemistr/crystals/seven.htm
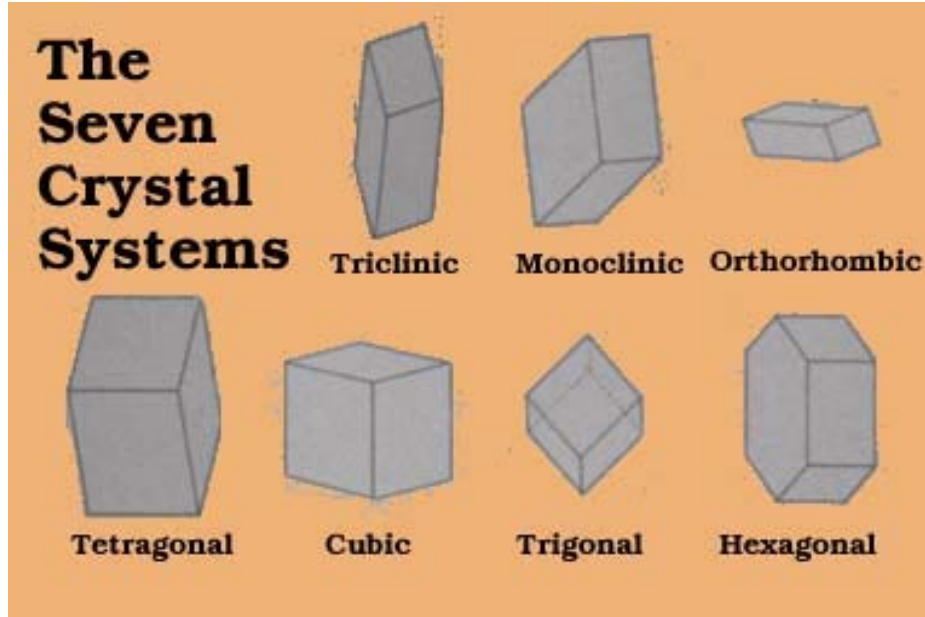
XRD began by using a cathode ray to shoot electrons at a crystalline substance, then the user using Bragg's law to calculate inner atomic spacing (d) and making a graph. Now most labs have a machine that is fully automatic. After inserting a sample the machine can perform multiple tests and create any number of graphs and average them all for very fine precision. Most of these machines range anywhere from $560,000 to $1,600,000 (Moeck).

XRD has three primary uses in today's research. First and foremost, it is used to identify individual mineral samples and their corresponding characteristics. Secondly, it allows identification of specific mineral components within mixed clay or soil samples. Thirdly, it provides an insight of unit cell dimensions, which is the distance between the inner protons and neutrons (SERC).

XRD has many strengths. Firstly, this process is very rapid usually taking under twenty minutes. The results are unambiguous, with very little error. The preparation is very minimal, just sample

collection and cleaning of the machine. The XRD machines are common and widely available around the world. The final interpretation is very straightforward allowing for easily replicated results (SERC).

XRD does present its share of drawbacks as well. To begin with XRD works best when one has a homogenous sample, though it can identify mixtures there must be enough of each. On the average a sample must contain at least 2% of a specific substance for that compound to be identified correctly. Another problem is that all the samples must be ground up and this is sometimes hard depending on what the sample is. When doing an XRD to identify one must also have access to a baseline/standards list to interpret the results. Finally non-isometric crystals pose problems due to the angles not being uniform like the equation deals with (SERC).

In conclusion, I look forward to maybe one day doing an x-ray diffraction. I'm glad my professor introduced me to this whole new idea. This whole concept amazes me and I'm glad I can take this new knowledge going ahead. It provides me a new look on what some companies that supplied us at my old job did when they made us raw ingredients for our cements and paints. Even though I originally wanted to do something more hands-on, I can't describe how interested this topic had gotten me when my professor first brought it up.

<div align="center">Works Cited</div>

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Moeck, Peter. "X-ray Diffraction (XRD)" XRD. Portland State University, 31 Mar. 2004. Web. 20 Apr. 2014.