

August 16, 2006

Dr. Christopher Ibeh
Pittsburg State University
Pittsburg, Kansas

Dear Dr. Ibeh,

I have completed my work under Dr. Tomas. The following paper is a review of the work that I have accomplished. It is my hope that the information within will provide the necessary information in regard to the possibilities of using x-ray diffraction.

If you have any questions or comments in regard to the report please feel free to contact me.

Sincerely,

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X-ray Diffraction

Camren Ray

Graduate Student

Supervisor: Dr. Tomas

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Summary (abstract)

Polyurethanes with different amounts of nano clay were provided by BTI ranging from 0% to 40% constriction. The clay was analyzed using the methods of X-Ray diffraction. The point of this experiment was to see if any noticeable changes would occur. Both wide and small angle diffraction is need but only the wide angle patterns where completed. The other part of the work was helping Dr. Tomas in finding possible methods of small angle diffraction with the current running equipment that is available.

Introduction

In 1901 Wilhelm Röntgen was awarded the Nobel Prize for physics with his discovery X-rays which he discovered in 1895. By around 1912 it was becoming evident to scientists that the unknown rays where just another form of electromagnetic radiation, i.e. "light". The difficulties in diffraction or refraction of X-rays in comparison to visible light suggested that the wavelengths of X-rays where much shorter than the visible range.

It was Max Von Laue that made the excellent suggestion to use the atoms in crystals to diffract X-rays¹. Using the atomic spacing in crystals, Laue was able to successfully diffract the x-ray and demonstrate the lattice structure, thereby proving the wave property of x-rays².

Depending on the pattern obtained, X-rays can provide different information about the structure of different material. There are two main methods of X-ray diffraction, small angle and wide angle. The small angle tells about the relationship between molecule to molecule and wide angle tells about the relationship between atom to atom or the internal structure of the material.

In terms of understanding the data, the small angle covers information over just a few degrees, where as the wide angle will cover tens of degrees. In short, one is a picture of a small area and the other is a general overview of the material.

The samples provided by the Business and Technology Institute (BTI) had differing amounts of nano-clays within the polyurethane, ranging from 0% to 40%. BTI wanted both the wide and small angel X-ray diffraction patterns, to see if any internal changes had occurred. Due to the small

¹ Thornton, p 152

angle apparatus being out of commission, only the wide-angle patterns were completed.

Literature Review

Cullity's book *Elements of X-Ray Diffraction* is a general discussion of the principles of the art methodology of X-ray diffraction. One of the possible problems with this book is the age, but according to Dr Tomas there has been no significant change in the practice of X-ray diffraction. Given that information presented in the book is still valid.

Guinier's book *Small Angle Scattering of X-Rays* is a detailed study of the theory and methodology of small angle diffraction. The difficulty with this book is the same as Cullity's, but the book is still of value because the same ideas and theories expressed within still apply.

Vietti's masters thesis *The Construction and Characteristics of an X-Ray Diffraction system with both low and high angle capabilities* looks at the issue of the of the beam defining slit and other problems that arise with the modified Krantky apparatus.

X-Ray Diffraction

Theory: The basic theory involving X-ray diffraction is Bragg's law, which states that in works where two times the distance between the atoms times the sin of the angle to which the ray on the material is equal to an integer times the wavelength.

$$2d\sin\theta = n\lambda^2 \quad \text{Equation 1}$$

The integer value "n" is for the different orders of the refraction. Since we are only concerned about looking at first order then $n=1$.

When the material has a crystalline structure the prominent X-ray patterns have distinct peaks. On the other hand the more metamorphic or non-crystalline the material, the less prominent the pattern becomes and the graph has a single peak where the strongest reflection occurs in the material.

Note on safety: High energy X-rays can be dangerous to a person's health if exposed to too much of the radiation. The most prominent danger is to the organs and skin. Being aware of the risk is important. Radiation is commonly measured in units of " millirems". On a dally basis the

²Thorntion, p 154

human body is bombarded with around 360mrem per year³ due to radiation in rocks, cosmic rays and other natural sources as well as some man-made sources such as medical. The problem comes when the radiation is excess of what the body has evolved to withstand.

For individuals working with X-rays exposure is inevitable. About the only defense that the individuals have is to monitor the amount of radiation that they receive. This is usually done by wearing a device that detects the radiation. At Pittsburg State University, film badges are used and anyone working in close proximity to the X-rays must wear one. Governmental limitations on prolonged exposure limits workers to 5,000⁴ mrem per year, which works out to be around 13.9 mrem per day.

Methodology

Equipment: A General Electric Wide angle apparatus Model number: 11GN1 along with accompanying electrics.

The X-ray source is Copper K α (Cu K α).

Materials: The BTI mixed Polyurethane with differing amounts of nano-clay.

Material 1: LHT-2-R-MEK, LHT-3-R-MEK, LHT-4-R-MEK

³ *Understanding Radiation*

⁴ *Understanding Radiation*

Material 2: LHT-0-R, LHT-2-R, LHT-3-R, LHT-4-R

Material 3: G30-0-R, G30-3-R, G30-4-R

Material 4: G30-2-R-MEK, G30-3-R-MEK, G30-4-R-MEK

Procedure:

1. Turn water on under the sink

Note: If equipment has not been used in a while it is necessary to make sure that the water reservoir under the primary table is full.

2. Turn high voltage supply on. Press and hold the "on" button until it stays on.

3. Allow high voltage supply to power up. X-rays now available for use. Avoid turning on except when needed, to minimize excess exposure to radiation.

4. Set Counter electronics: window width, base line, course and fine gain, and high voltage for electronics. See appendix A for two set-up methods.

5. On the high voltage generator for the X-ray tube, set high voltage at 45 kV and set (not already done) the X-rays to be admitted on the left side of the table. There is a knob that reads "L", "Both", "R"; put it on the "L".

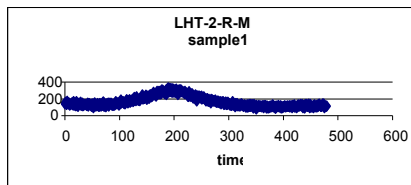
6. Set the sample in the wide-angle diffraction apparatus. Close the lid; the X-rays will not come on if the lid is not down.
7. Manually set the apparatus at 6.8 degrees. The reason being is that you need the .2 degrees to start the computer program.
8. Set the speed on High/High. There are two knobs one reads "High", "Manual" and "Low" and right next to that is a second smaller knob that reads just "Low" and "high". Set both on High.
9. Turn on the computer and open up the program "MCS-32"
note: program view must be on MCS to take data, and to recall data the view must be on buffer.
10. Set up time sweep at 460 seconds (about 8 min), and set cursor over the start button.
11. Turn on X-rays.
12. Turn knob wide-angle apparatus that says "decrease" "off" and "increase" to increase to start it.
13. As soon as the angle clicks over to 7 degrees start the computer program.
14. When run is finished, save off file and export data, and import into excel and graph. If counts vs. theta is needed, convert the time to degrees, appendix B.

15. For multiple samples clear out program and restart procedure from step 5.

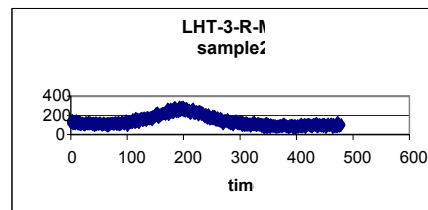
Results/Evidence

Data: all time is taken in seconds and can be transferred to degrees by the following conversation factor:
1degree=15seconds

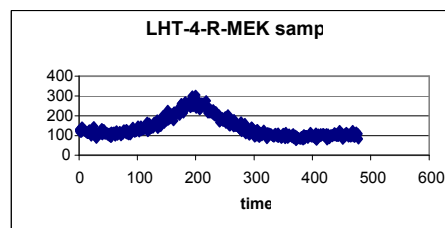
LHT-2-RMEK



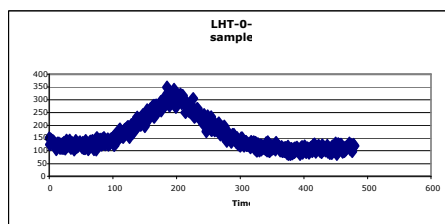
LHT-3-RMEK



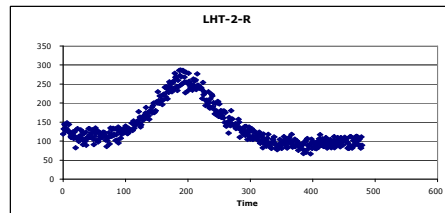
LHT-4-R-MEK



LHT-0-R

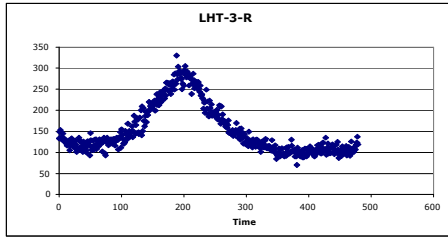


LHT-2-R

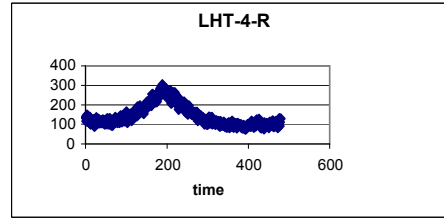


LHT-3-R

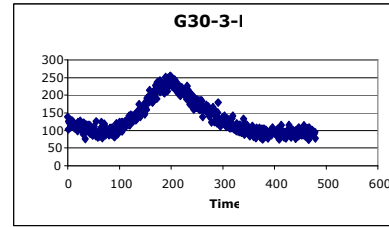
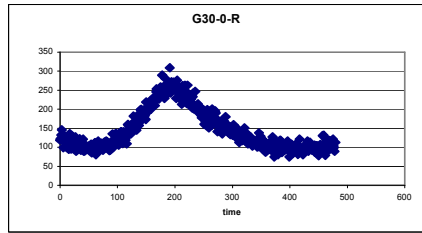
LHT-4-R



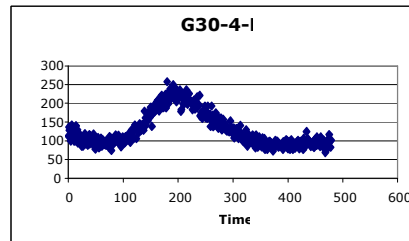
G30-0-R



G30-3-R

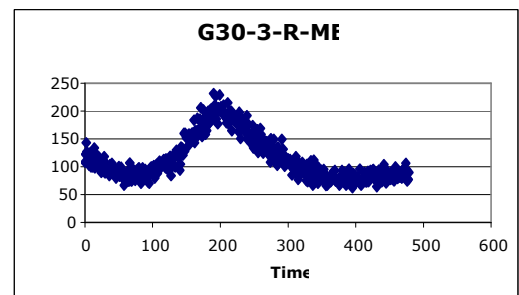
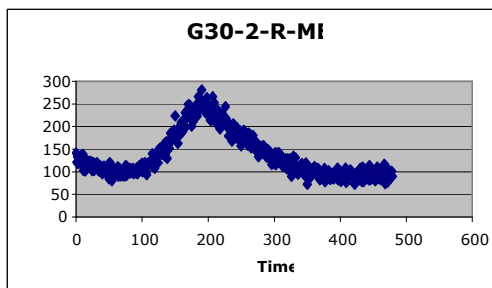


G30-4-R

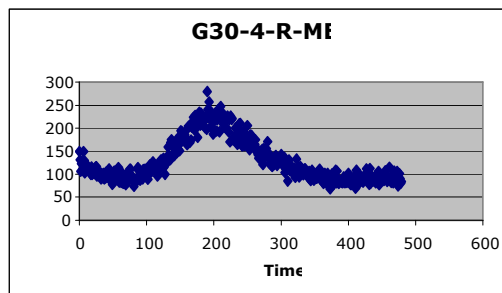


G30-2-R-MEK

G30-3-R-MEK



G30-2-R-MEK



Discussion: All of the graphs show there peaks around 200 seconds and all the graphs are the same there is no significant variation in the data. Also given that there is only the one peak in all the graphs, there is no discernable structure.

Conclusion

Given that there is no significant variation in the placement of the peaks, this indicates that there is no substantial change in the material. Secondly, given that no internal atomic structure is present, the material is metamorphic.

Improvements to the actual experiment that can be made are to find a way to automate the process between the starting of the wide-angle equipment and the computer. Other improvements are to have a wider sample to insure that the beam is properly striking the sample.

Recommendations

To supplement the work done on this project the small angle patterns will need to be obtained. Although that cannot be started until the rebuilding of the physics departments apparatus is complete or a new one is purchased. Both the small and wide angle are needed to completely analyze the samples.

Acknowledgments

I would like to thank Dr Jim Tomas for his involvement in the project with out his counted efforts things would not have progressed as far as it did.

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with both low and high angle capabilities.
Pittsburg state college, July 1966

Appendix A: setting up counter electronics.

There are two methods to set up the electronics the basic set up for both is the same. Using a Germanium (Ge) crystal lattice setting of (111) the main diffraction beam should be around 3 000 counts. Knowing that information is then possible to set up the counter electronics. The first method is to obtained data and graph and get a solid electronic settings. The other is to use trial and error to get roughly the best settings.

Basically two things need to be set the base line voltage (BL) and the window with voltage (window). The first is obtained by setting and holding fixed the fine and coarse gain, and window constant and varying the BL to see about where it levels off. Once the leaving off point is found the BL is held constant at that point. Then the window is varied to see where the maximum counts occurs.

Appendix B: to get the time to angle ratio all that needs to be done is to set the speed and time how long it takes to go through a given amount of degrees. The four speeds on the wide angle equipment are high/high, high/Low, Low/High and Low/Low. The following respectively are the time to angle ratios: 15s=1°, 30s=1°, 2.5minute=1°, 5minute=1°