INTRODUCTION TO X-RAY DIFFRACTION

Introduction

This experiment provides an introduction to the experimental side of x-ray diffraction. It is an opportunity to put into practice much of what you have been taught about the continuous and characteristic spectra produced by the x-ray tube, x-ray optics, detector technology, x-ray - specimen interactions and the crystalline structure of solids. You will receive an introduction to a modern diffractometer and will learn how to use it to obtain reliable data from your specimen. The specimen will be a familiar material, one which is well known, well characterized and yet offers some interesting features.

Objective

There are two simple objectives for this experiment. The first one is to teach you how to operate the Scintag x-ray diffractometer, including how to plan and execute your own x-ray diffraction analysis. If you succeed in this objective you will be in a good position to complete the following diffraction experiments with minimal interference from your instructor.

The second objective is to learn how to interpret the powder diffraction profile. There is a lot more to real data than is generally described in classroom lectures. The complexity of the real data comes in part from the complexity of the non-ideal specimen and from the many factors associated with the use of real diffractometers. The non-ideal aspects of the data actually reveal much more about the material and the diffraction process and often become the basis of new experimental techniques.

Materials

Specimens to analyze will be provided. Other specimens may be available to show you how to prepare powder samples.

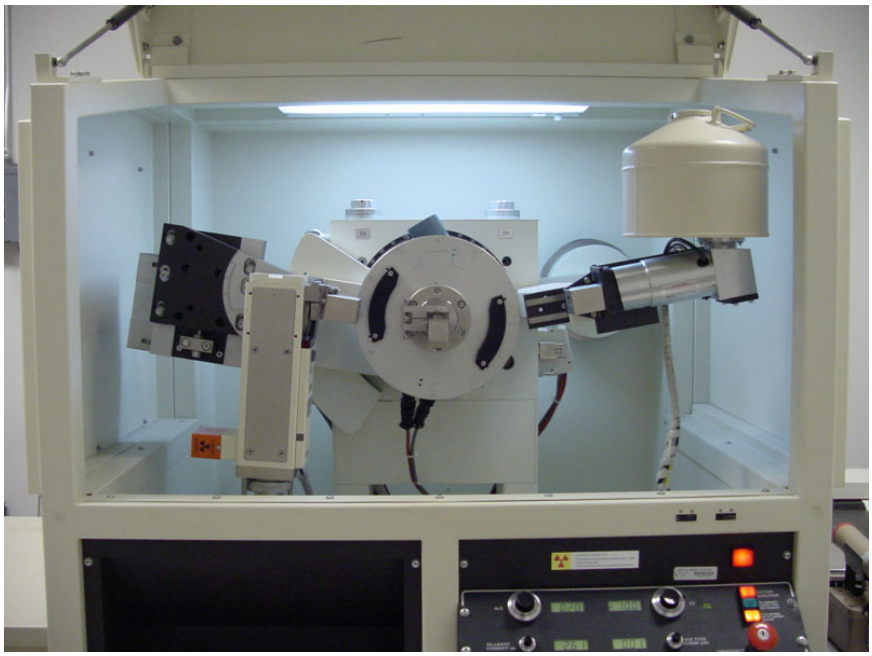

Figure 1 The Scintag x-ray diffractometer.

Equipment

The only equipment used during this experiment is the Scintag XDS 2000 x-ray diffractometer. This is a modern high-resolution diffractometer which features a 2.2 kW long-line fine-focus x-ray tube, a solid state Germanium detector and a three-axis goniometer. It is operated via a computer which contains numerous data analysis programs and the complete ICDD powder diffraction database.

Procedure

This procedure is designed to show you how to set up and execute an analysis of a specimen you are not familiar with. Using the specimen provided (quartz stone) you will start by performing a quick qualitative analysis, illustrating how one can, within 5 minutes, get a good overview of the specimen's diffraction profile. Next, you will perform a slower scan on an particularly interesting region of the same specimen (the five fingers of quartz). For both sets of data you will run basic data correction and analysis programs. Finally, the data will be plotted, allowing you to compare the results of each scan and draw your own conclusion regarding the tradeoffs between scan rate and the quality of the results.

1. Your instructor will show you how to start up the system, log on to the computers, start the Scintag's software and make sure the system is ready to use.

2. Search the JCPDS database for the card file of your specimen. Display the data as 2θ versus intensity for the wavelength being used in this experiment and then print out this file.

3. Perform a quick scan over wide range of 2θ where interesting results might be obtained. Refer to the powder diffraction file for the appropriate 2θ range. Use the "continuous scan" mode to perform the scan and use a scan rate of 10 degrees per minute or higher.

4. Perform a slower scan over region called the "five fingers of quartz" (67-69 degrees 2θ). Use the "continuous scan" mode again but this time use a scan rate of 1 degree per minute or slower.

5. Perform background corrections and then run the peakfinder program on both sets of data. Get a printout of the results of the peakfinder program and review it to see what type of information it provides.

6. Display the results of both scans. Set the scaling to show only the "five fingers" region and overlap the raw data from the first (fast) scan and the raw data and the corrected (net intensity) data from the second (slower) scan. Study the differences in the three sets of data.

7. Optional: Use the "Profile Fitting" program to obtain a best fit of the "five fingers" raw data. Use the split Pearson profile. Notice how many peaks are found and how well the fit is. Can you can explain why five peaks were not fit?

8. Optional: Perform one more scan of the quartz stone. Use the "Step Scan" program and scan from 26 to 27 degrees 2θ. Use a step size of 0.01 degrees and a preset time of 2.5 seconds and replace the detector slits with the 0.3 mm scatter slits and the 0.1 mm receiving slits. When finished overlap the 26-27 degree portion of the first (fast) scan and this scan and note the differences.

Results

At the end of this experiment you should be able to recognize and explain all of the major features of you diffraction profiles. You should also be able to explain what the background correction program does and how to modify your scan parameters to obtain higher quality data.

Additional

1. Bring an diskette or Zip drive so you can take copies of your data files with you. If you'd rather, you can ftp your files to your account on another computer. The data can be used to prepare quality figures for your reports.

2. Pay very close attention to what your instructor tells you during this experiment. His/her goal is to make you a competent (but not independent) user of this diffractometer in only one session. Learn as much as you can about operating the equipment so that you will be able to perform the next couple experiments yourself.

3. Keep copies of all printouts. They will be needed when you write your report.

4. Your Operator's Guide for the Scintag diffractometer contains a lot on information about this system that you will find useful when writing your report. The section on "A Typical Session ..." will remind you of the details of each program you used, what each did, etc. The section on the specifications of the diffractometer will be helpful when describing the equipment you used in this experiment.

5. Bring your homework assignment to this laboratory session. Several of the questions can only be answered by examining the diffractometer itself.