XRDUG Session II

XRD Facilities Organization and Important Procedures

Edward Laitila
2/7/2009

DMSNT-Software and Computer Basic Information:

DMS-NT Software:

- All bold words in this handout and contained in the operation manual refer to actual software menu items.

- The data collected using this software is in a proprietary format that can only be read by the DMSNT software. To observe this data in other programs a text file must be created of the collected data: **File**, **Export**, **Raw**, select **Export to File, Browse** to input a file name. It is highly recommended that you indicate that this file is the raw data since the extension is txt as well with all other exported data.

- The software requires a hardware dongle in order for it to operate; therefore the software cannot be installed on computers outside the lab.

- Please be familiar with Windows Explorer (or My Computer), and Network Neighborhood before using the software.

Computer Organization:

- There are currently 4 computers in the XRD lab that have the DMSNT software available. These are named Warren, Bragglaw, Guinier, and Averbach.

- Computer Warren is dedicated to operating the Scintag θ/θ diffractometer, all collected data (and analysis files) are stored in drive X of this system. You must access this drive (using Network Neighborhood) when analyzing collected data using other computers in the lab. **Only files on this drive are backed up.**

  - **Do not use Warren** for data analysis if a scan is currently running on the instrument, if the software crashes (which happens frequently) the data for the scan will be lost. If you need to use this

computer for analysis open another DMSNT window session to prevent crashing of the current data collection.

- **Do not log off Warren** while data collection is occurring, logging off will cause a loss of all data.

- There is a computer with no name on it and printer attached that is exclusively for accessing the database using a search program, this is temporary until another solution is found.

Printer Organization:

- There are two printers for use with the diffraction software, one to print graphs, the other for printing data such as peak files.
- Graphs: Use the HP Office Jet Pro K8600 ink jet printer which is set up to print on 11X17" paper. When printing graphs select this printer, then go to **Properties** and select **XRD Graph** under Printing Shortcuts.
- Text: Use the Brother HL-4070CDW color laser printer for printing text files such as peak files. Select **Properties** then select **Paper Size – Legal** then select **Orientation - Landscape**. This allows all columns from the peak files to be displayed with reasonable size text.

Important Aspects of XRD Operation:

Calibration of Goniometer θ and Ω Positions:

- Goniometer positions must be physically checked to make sure it exactly matches with the computer positions prior to operation.

- Computer does not dynamically measure the goniometer position.

- Calibration procedure allows the user to synchronize the goniometer and computer positions.

- Computer calculates and maintains the goniometer position by counting pulses to the stepper (drive) motors for each goniometer axis.

- Refer to calibration procedure in the operation manual for the Scintag θ/θ Diffractometer.

- The θ position is the detector position.

- The Ω position is the x-ray tube position.

- In normal operation actual θ and Ω positions are equal to 2θ divided by 2.

    In general: $2\theta = \theta + \Omega$

- ☠ **Do not enter manual goniometer operation or attempt a calibration until you receive proper training by Ed Laitila.**

X-Ray Generator Operation:

- In order to improve longevity of the x-ray tube users should plan diffraction scans so that the turning on and off of the generator is minimized.

    - Leave x-rays on if another user will be using the instrument within 12 hrs, the tube power should be set to the minimum settings.

- X-ray tube parameters:

    - Decrease in intensity over time due to W deposits on the window and the target material. **This may cause the appearance of W lines on low angle shoulders of high intensity peaks.**
    - Power rating of tube, maximum limit of heat dissipation, exceed and target can melt.
    - Water cooling is vital to operation of x-ray tube.
    - Tube power equals the kV's times the mA's. Maximum tube power is 2000 watts; however, maximum power I allow is 1575 watts. You must see me if you need to operate at conditions that exceed this power rating.
    - Finite life, typically 3000-5000 hrs, we get >10,000 hrs.
    - Thermal shock of filament typical failure mechanism.

Diffraction Scan Checklist:

- Check to see that sample is inserted properly within the holder. The two black lines on the sample holder indicate the beam center.

- Always make sure the goniometer moves to the proper starting angle after a scan is started. An incorrect angle indicates a serious problem.

- Make sure the shutter opens. If the shutter stays closed, this may mean that the red safety circuit switch was not pressed.

- When operating in a step scan mode, verify that the goniometer is stepping the proper increment of angle. Note that movement about the goniometer axis for the Scintag θ/θ diffractometer is in degrees θ not 2θ.

Make sure the x-rays are on and the generator is at the proper kV and mA settings.

General XRD Facility Usage:

Logbooks:

- Please fill out the logbooks, failure to enter data in log books can result in loss of privileges.

- Please have an account number, if you do not have an account number you must talk with me prior to using the equipment.

- Sign the log book, note tube hours (start time entry obtained from hour meter located on the front panel) and any problems in the log book. If you change the standard configuration of the diffractometer (e.g. slits) leave a note on the instrument to warn other potential users that parameters have been changed.

- Please enter the total scan time of the data collection during your XRD session. This is the time you are charged, not tube operation time.

Scheduling XRD Time:

- Please see the on-line web scheduling within in the ACMAL facilities page. Go to the Scintag XDS-2000 θ/θ page to access the calendar (http://mcff.mtu.edu/acmal/scixdspd.htm).

- When scheduling XRD time please attempt to schedule next to time already scheduled to help minimize the number of times the instrument is turned on and off.

- Try to do as many samples as possible in succession to again alleviate the on-off cycles of the x-ray tube.

- Please allow for extra time for sample mounting, etc. when scheduling time.

Costs to Use the XRD Facility:

- The current rate (2008) is $5.25 per hour of scan time.

Other Software:

- Crystal Studio (crystal structure analysis create 3-D structures).
- PCPDFWIN allows access to the JCPDS-ICDD powder diffraction data base, currently available only on the unnamed computer in the lab.
- Origin (plotting and peak fitting software) – Currently only available on Guinier.
- Star Office (freeware similar to Microsoft Office)
- Volfract (volume fraction analysis software).
- Diffraction based freeware:
    - Full Proof (general anlaysis and Rietveld software)
    - GSAS (Rietveld software)
    - Dicvol (indexing powder patterns)
    - Crysfire (indexing powder patterns)CMPR
    - Checkcell (indexing powder patterns and spacegroups)
    - Powder X (file convertor and general diffraction software)
    - CMPR (multipurpose diffraction software)
    - Celref (lattice parameter refinement)