

Cornell Center for Materials Research

Bruker D8 Discover Powder

Diffractometer

Standard Operating Procedures

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2 INTRODUCTION

The Bruker D8 Discover Powder diffractometer is designed for efficient collection of theta-2theta powder x-ray diffraction patterns to measure lattice parameters of crystalline materials or to find the mass ratios of multiple crystalline phases in a sample. Diffraction is measured using Cu K- α radiation with a wavelength of approximately $\lambda=1.54 \text{ \AA}$ and photon energy of $E=8.04 \text{ keV}$.

The source is a water-cooled fixed Cu target excited by a 40 kV x 25 mA electron gun. A Ni filter inserted near the end of the source arm is used to filter out Cu K- β radiation, leaving Cu K- α_1 and Cu K- α_2 and some remaining Bremsstrahlung radiation. A silicon strip detector (SSD160) is used to record the photon count rate (intensity) as a function of diffraction angle. This detector has a limited degree of energy discrimination, allowing it to ignore photons with energy far away from Cu K- α and Cu K- β , reducing the background level of measurements.

3 PREREQUISITES

3.1 SAFETY TRAINING

Before beginning training on the Bruker D8 Discover, users must have completed the following safety training courses through CULearn:

- EHS 2555 - Laboratory Safety
- EHS 2716 - Chemical Waste Disposal
- EHS 5330 - Fire Safety
- EHS 5033 - Radiation Producing Equipment Safety

The Radiation Producing Equipment Safety course must be current, meaning that it must have been taken in the past year. Users are required to keep this safety training current in order to be permitted to continue using radiation producing equipment at Cornell University.

3.2 CORAL ACCOUNT

Users must have a currently active CCMR Coral account for billing purposes. Users may not use anyone else's Coral account. If multiple collaborators are on an instrument together, only one needs to have an account. For more information on obtaining a CCMR Coral account, please see the CCMR webpage.

4 ACKNOWLEDGEMENTS

This instrument was purchased with support of the NSF MRSEC program. All publications and patent applications which include work performed on this instrument must acknowledge this support, including the current NSF grant number. For recommended wording, please see <https://www.ccmr.cornell.edu/research/acknowledging-ccmr-funding/>.

5 EQUIPMENT

5.1 COMPUTERS

There are two computers associated with the powder diffractometer station. The computer on the right can be used for Coral, data analysis, and general internet access.

The computer on the left, nearer to the diffractometer, must be enabled through Coral. For more information on obtaining a CCMR Coral account, please see <https://www.ccmr.cornell.edu/facilities/becoming-a-ccmr-facility-user/>

For information about using Coral, please see <https://www.ccmr.cornell.edu/facilities/ccmr-coral-faqs/>

5.2 BRUKER D8 DISCOVER POWDER DIFFRACTOMETER

When not in use, the Bruker D8 Discover is left in an enabled state with the source at low power and the radiation shutter closed. The source enabled is indicated by an amber light on the source (inside the hutch) and an amber illuminated radiation symbol on the outside of the hutch. A red light on the source is illuminated only when the radiation shutter is open. The shutter is automatically opened before a scan and closed at the end of the scan. For safety, both the shutter and the motorized motions of the diffractometer are interlocked with the door of the hutch.

6 PROCEDURE

6.1 ENABLING THE CONTROL COMPUTER

The instrument control computer must be enabled through CCMR Coral by a user with an active Coral account. This software can be found on the data analysis computer, or downloaded from the internet and run on most computers. For more information, see <http://www.ccmr.cornell.edu/facilities/ccmr-coral-faqs/>

6.2 CONFIGURING THE DATA ACQUISITION SOFTWARE

Start the software *Bruker Diffrac.Suite* on the instrument control computer. There should be an icon for it on the left corner of the status ribbon at the bottom of the screen. The user name is 'Lab manager' and there is no password. Once the software has started, select the 'Commander' tab.

In the upper left portion of the window is a list of variable physical on the instrument. The left column contains the names of variables. The middle column shows values (with gray background) read from the diffractometer. The right column contains editable values (with white background).

6.3 SETTING THE SOURCE TO OPERATING POWER

The standard source used in this instrument is water cooled Cu source. Edit the source voltage and current to **40 kV** and **25 mA** for 1000 W (see Figure 1). The standby power is 20 kV and 5 mA for 100 W. After editing the voltage and current fields click the 'Set' button nearby. It takes under 30 seconds for

the source to ramp to full power, during which time the control software is grayed out and the illuminated radiation symbol on the front of the D8 Discover blinks to indicate that the source power is not stable. Once the source power is stable, the instrument is ready for use.

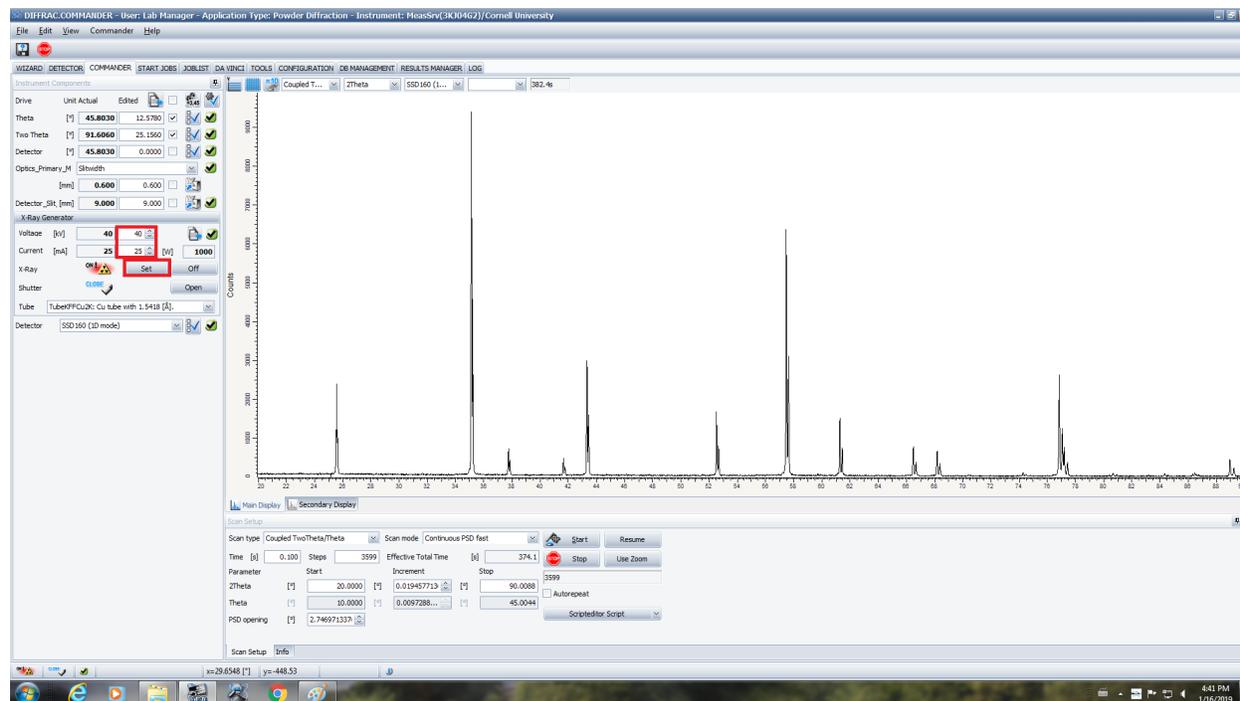


Figure 1 Setting the x-ray source voltage and power.

6.4 PREPARING AND MOUNTING A SAMPLE

Powder diffraction assumes a random angular distribution of a very large number of crystallites illuminated by the x-ray beam. Traditionally this is satisfied by using a powdered sample with roughly 10 μm or smaller extent. Larger crystallites (100 μm) result in a pattern which is not representative of scattering from all orientations, but it in some cases may still be useful for phase identification. Polycrystalline solids may also be used as samples for powder diffraction, but partial orientation of grains may affect the relative intensities of Bragg peaks, potentially completely eliminating some diffraction peaks.

6.4.1 Choose sample well

There are a few different wells which can hold powdered samples. The powdered sample should completely fill the well so that the surface of the powder is level with the surface of the holder. The small quartz wells have a volume of 0.1 mL. If the sample does not completely fill the well, the surface of the sample will not be at the correct height when mounted in the diffractometer, resulting in an angular error in the measurement. The round sample wells are glass, which will give a broad background diffraction signal below roughly 30° 2-theta. The square sample wells are quartz and have no diffraction peaks in the vertical diffraction plane.

6.4.2 Grind sample if necessary

Wear gloves when handling powder both for safety concerns and for general cleanliness of the laboratory. Gloves are provided at the sample preparation table.

Powder x-ray diffraction assumes that the sample contains an ensemble of crystals with every possible orientation. Samples composed of a relatively small number of large crystals will not give a representative diffraction patterns. If your sample has large grains (greater than 100 microns) then data quality will be improved by crushing your sample using the mortar and pestle provided. There is no absolute rule for maximum particles size – it depends on what information is needed and how complex the diffraction pattern is. If the goal is to distinguish between two possible phases which have completely different diffraction patterns, then large grain size may not be detrimental. If the sample is a combination of multiple phases, and the diffraction pattern will be used to differentiate the sample between possibly compositions which have similar diffraction patterns, then more care must be taken in sample preparation.

6.4.3 Fill sample well and smooth surface

The sample wells should be found in a clean state. If you are highly concerned about cross-contamination of samples, particularly if you will use the same material for further analysis, clean the sample with alcohol and laboratory wipes.

There are options for sample wells and sample pucks.

1. The square wells are quartz and must be slid into the sample pucks with slots. These sample wells are crystalline but do not have diffraction peaks in the vertical plane. They are therefore good general purpose sample mounts. Greater care needs to be taken in aligning the sample puck when mounting it (see next section), and the puck should be carried level because the well can slide rather freely in the slot.
2. The round wells are glass, and as such they produce a broad diffraction peak at low angle. For many experiments, particularly with highly crystalline materials and large diffraction angles (over 30°) this is usually not a concern. These wells sit in the sample pucks with round receptacles. Note that there are a few sample well thicknesses and receptacle depths. Before filling a sample well, make sure that there is a sample puck that that sample well fits in with their two surfaces flush.

Place a sheet of weigh paper under the sample well before transferring your sample. The sample should completely fill the sample well. If there is enough material available, a smooth sample can be obtained by overfilling the sample well and using the edge of a glass microscope slide to scrap across the surface of the well, pushing excess material onto the weight paper.

Place the sample well in the appropriate sample puck.

6.4.4 Mount sample puck

The diffractometer includes three pins which serve as the height reference for the sample (see Figure 2). When a sample is correctly mounted and inserted into the system, the surface of the sample is coplanar with the three reference pins.

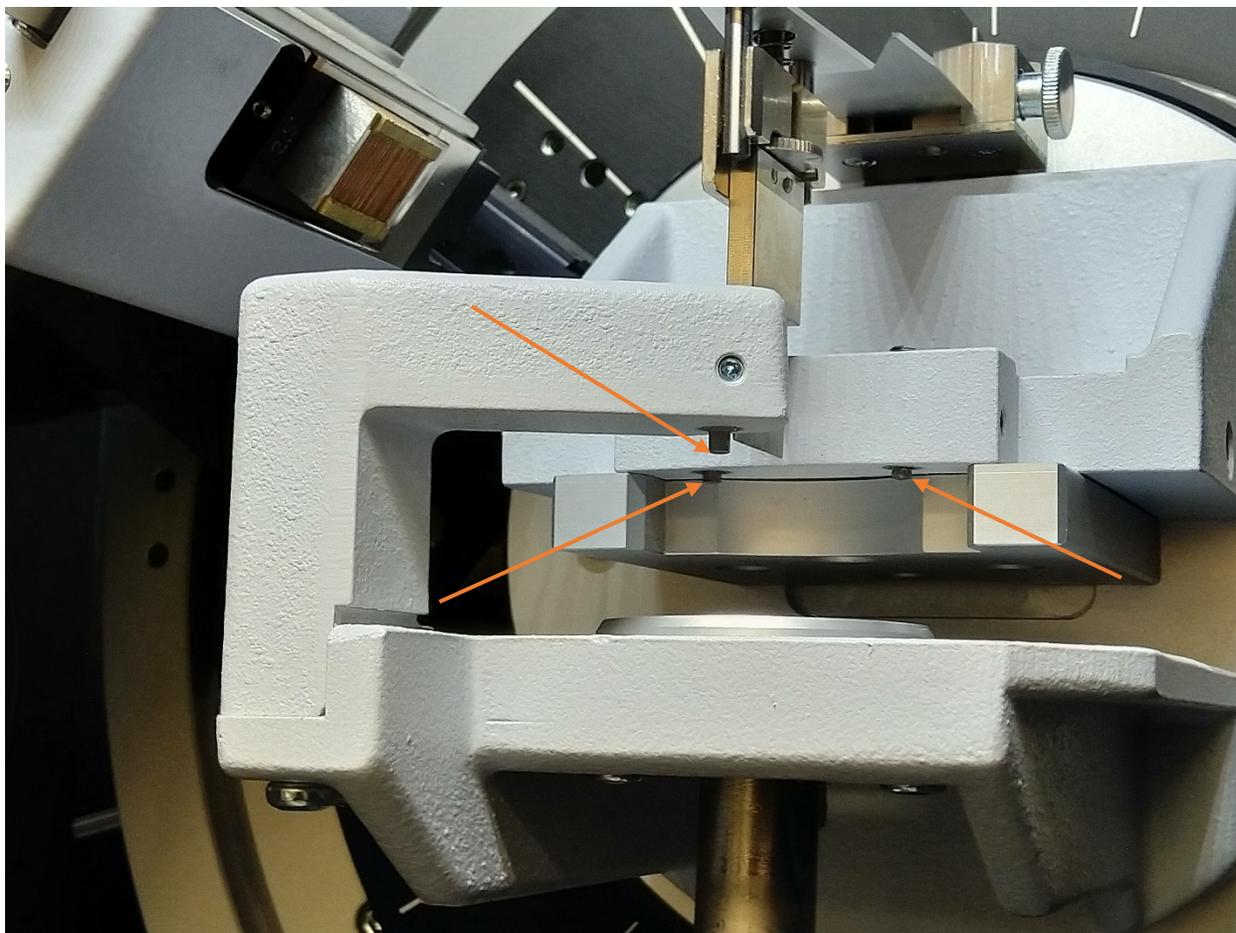


Figure 2 Sample mounting platform and three height reference pins (indicated by arrows).

Place the sample puck on the round aluminum platform in the center of the diffractometer. Visually check that the puck is centered on the platform. Using the post that extends below the platform, push upwards until the top of the sample puck is just touching the three reference pins. Never raise the platform without a sample puck in place, because when doing so it is possible to push the stage higher than it is designed to go. Do not pull down on the post; lowering should be done by supporting the post from below, and pulling the lever that is behind the post. The lever should be pulled towards the front of the instrument. Keep the lever pulled towards you as you slowly lower the post, by lowering the hand which is supporting it from below. Again, you should not need to pull on the post, and your hand should only be needed below it to keep it from falling too quickly.

After raising the platform to the height where the top face touches the three reference pins, visually check that it is touching all three. If it does not, slide the puck until it is centered under them. Failure to do this will cause you to record invalid data and likely cause the instrument to jam. Once the top of the sample puck is touching all three reference pins, push the sample post up, compressing a spring, until it stops – approximately 1 cm above the position where the puck initially makes contact with the

reference pins. A properly mounted sample is shown in Figure 3.

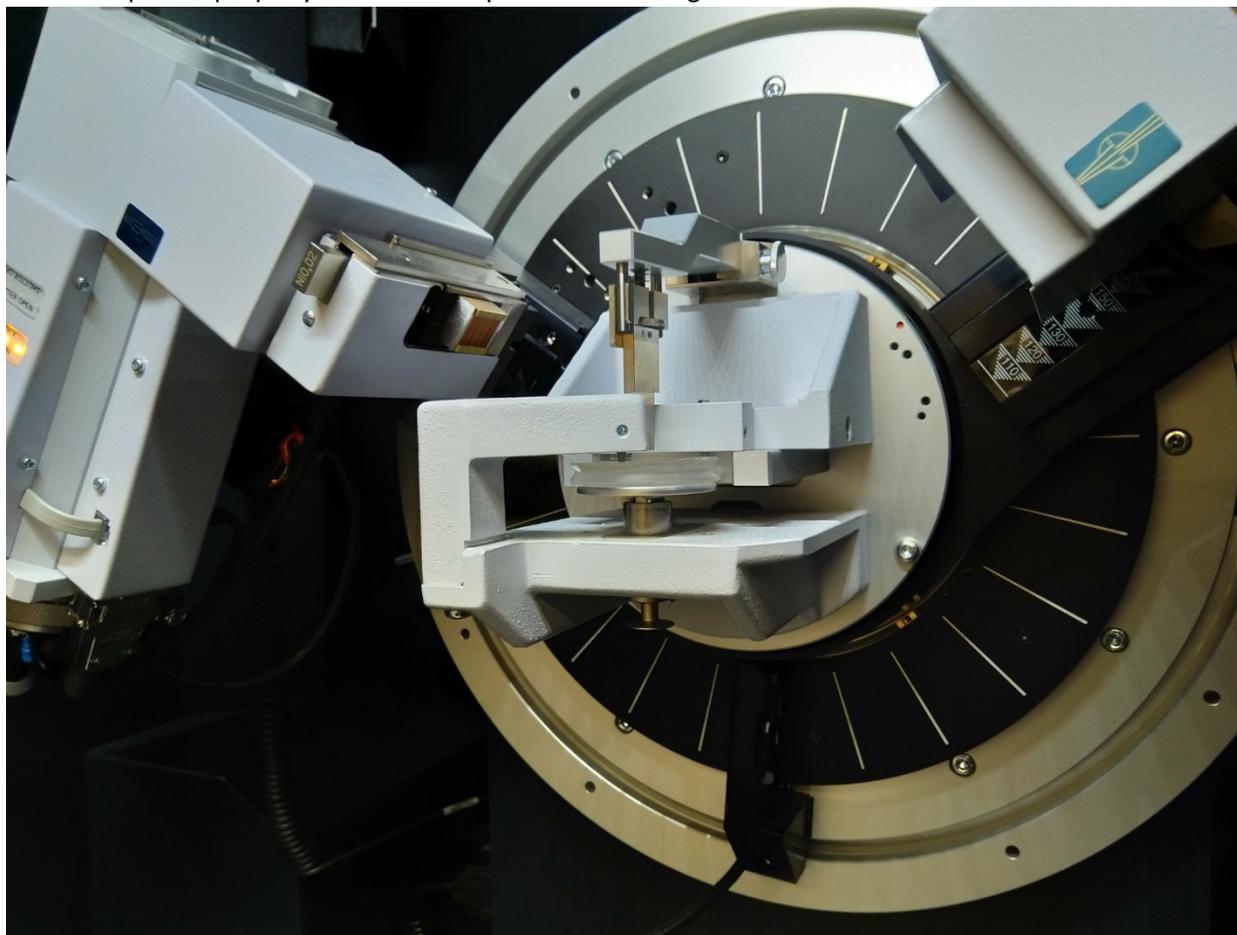


Figure 3 Sample properly mounted in diffractometer.

6.5 MEASUREMENT

6.5.1 Setting the slits

The incident beam slit, detector slit, and active detector height (PSD opening) should be set based on the type of measurement to be made (see Figure 4 and Figure 5). Decreasing the slit opening reduces the low angle scattering which causes a background in low angle scans. This should be considered when the minimum scan angle is below 15°. Reducing slits also decreases the signal available to a measurement; this is often the cause for lower than expected signal during a measurement. The PSD opening only needs to be decreased when the starting angle is below 5° to prevent exposing the detector to the direct beam.

	Standard	Low angle	Lower angle
Starting 2θ	>15°	5.5°-15°	<5.5°
Divergent beam slit (unlabeled)	0.6-1.0 mm	0.1-0.2 mm	0.1-0.2 mm
Detector slit	9 mm	3 mm	3 mm
PSD opening	~2.747°	~2.747°	½ of starting 2θ

Figure 4 Slit settings for standard and low angle scans.

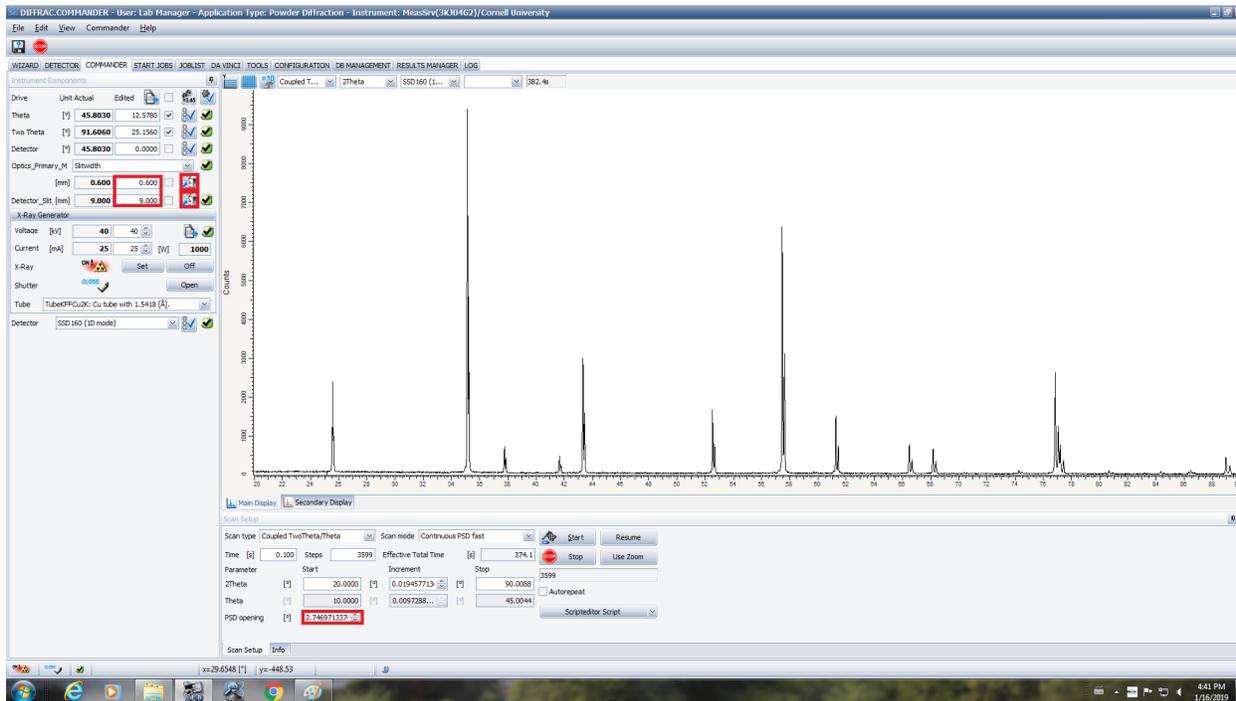


Figure 5 Setting the slits and PSD opening.

6.5.2 Scan design

Instrument users typically know in advance the general position for peaks which might be representative of their sample. Using Cu K-alpha radiation, minerals and metallic samples can typically be characterized by measuring peaks between 20° and 80°. Polymers and glasses may have peaks in the 5° to 30° range.

Measurement noise is generally dominated by statistical noise in the counted number of photons, which is given by the square root of the number of counts. For a diffraction peak which is very strong relative to the background level, the signal to noise is therefore proportional to square root of the counting time; in order to improve signal to noise by a factor of 2, counting time (per data point) needs to be increased by a factor of 4. Where the background is large (often due to a non-crystalline phase), the background also contributes to the statistical noise.

In the lower portion of the 'Commander' tab, confirm that the scan type is set to 'theta - 2 theta' (see Figure 6). Set the 'Start angle' to the desired beginning of the scan range. The default step size (approximately 0.019°) is reasonable for most measurements, and needs only to be decreased if the goal is to characterize peak shape, or increased for efficiency if the user is characterizing very broad peaks, for example from glassy phases or nanocrystals. This step size will always be a multiple of the detector pixel height. Set the 'End angle' to the desired end position for your scan. After changing the cursor focus, that value will be replaced by a very similar value which is an integer number of steps away

from the start angle.

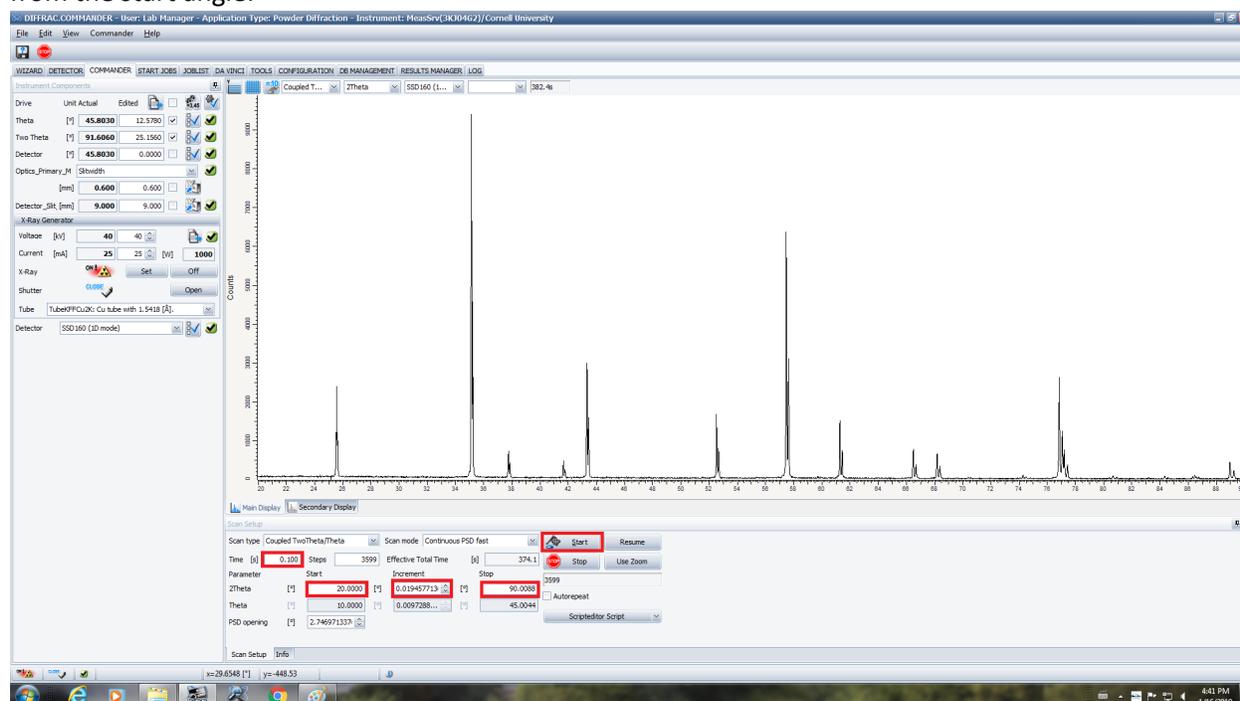


Figure 6 Typical scan configuration for highly crystalline mineral.

The software should have updated the number of steps in the scan. Click on the box for 'Time per point'. Increasing this value will improve the signal to noise at the cost of making the measurement take longer. For most mineral and metallic samples, 0.1 s is a reasonable starting value. After changing this value, you may need to click in another box, such as number of data points, to see the calculated measurement time updated by the software.

Click 'Start scan'. The source and detector angles will move to the start position. The shutter will automatically open and the instrument will continuously sweep the source and detector angles while recording diffracted intensity.

6.6 SAVE DATA

Scans are not automatically saved by the *Diffraction Suite* software. To save a scan select File->Save Result. Navigate to the '\\rs-ccmr-coral-a\Share\Users\' directory, and create a directory with your NetID if one does not already exist. Save your data in this directory in .brml format. If you wish to export data in a format you can analyze with your own software, open *Diffraction Eva* (on either the instrument control computer or the analysis computer) and choose File->Import Data. With a single data set selected in the software, select Export Data and save as a .xy file, which includes 1 line of header information (including the source type) and contains data in a two column (two-theta and intensity) which space separated format.

6.7 ANALYZE DATA

The CCMR maintains licenses for *Jade 10* and ICSD database of crystallography data. They can be accessed from the analysis computer in the room with the Bruker Powder diffractometer or the analysis computers at the Bruker GADDS or Rigaku SmartLab. CCMR diffraction users may use any of these computers for data analysis without having to log on through Coral. This document only gives a basic introduction to using *Jade*.

Start *Jade* using the icon on the desktop. Navigate to your data directly by selecting Start->Open New Folder. Files in that directory will be listed in the panel on the left side of the window, organized by data format. *Jade* is capable of reading many data formats used in x-ray diffraction, including .brml, .raw, .asc, and .xy. Select a data file by double clicking on it in the left panel. Many functions in *Jade* are organized by tab: Scans, Phases, Peaks, and Profiles.

6.7.1 Background fitting

Jade automatically applies background correction to loaded data. To see and modify the fit background, select the button labeled 'BC' with a red line passing through points. This will add to the plot window a red line passing through a series of points which the software chose as background points. For data from highly crystalline samples with sharp peaks more than 10x the background level, automatic background fitting is typically satisfactory. For lower signal levels and more complex signals, the background is often overfit by the software.

The background is modeled by a cubic spline passing through a set of fixed points, with a minimal curvature constraint. Right clicking on one of these points eliminates it from the model, left clicking on an open portion of the plot adds a new constraint point. Constraint points can be moved either by clicking and dragging, or hovering and moving the scroll wheel (holding Ctrl for horizontal movement).

6.7.2 Measuring lattice parameters

Select the 'Peaks' tab. The software will attempt to locate peaks within the data. The parameters *H*, *A*, *PK*, and *Filter*, are used in the automatic identification of peaks. Peaks can be manually added (or removed) by selecting 'Edit Peaks' from the right side of the plot and left (or right) clicking on the plot. The peak list in the bottom panel includes statistics for each peak, including d-spacing, relative height, and peak full width at half maximum.

6.7.3 Phase identification

Select the 'Phases' tab. Select 'Chemistry' to modify the chemistry filter. Select 'Toggle All' to select or unselect all elements. Either manually select each element which might be present, or select sets of elements (such as 'Light elements') from the drop-down menu and manually add elements you expect to be present. Check the 'Unit cell' tab and unselect all check boxes unless you want to apply constraints based on additional information known about your sample.

Click 'S/M' (Search/Match) to select an appropriate database for the materials expected to present in the sample. For a power sample of polycrystalline material without orientational ordering (texture), 'Severely Oriented' should not be selected. For thin films or oriented crystallites, select 'Severely Oriented'. When analyzing data recorded on the Bruker powder diffraction system or Bruker GADDS, ensure that 'K-alpha 2 present' is selected. When analyzing data recorded on the Rigaku SmartLab unselect 'K-alpha 2 present' if the Ge monochromator was used during data collection.

After configuring the search/match function, click on 'One-Click Analysis'. If the software is able to interpret the measured diffraction pattern as a sum of patterns from the selected crystallography databases, it will return a list of phases with relative compositions expressed as weight percentages. In other cases, the software will return a list of potential fits.

6.7.4 Strain and particle shape fitting

Through the 'Profile' tab it is possible to fit the profiles of individual peaks to obtain a more accurate measure of their relative areas and full-widths at half-maxima. Click on the position of a peak in the data to add a peak to the curve fitting function, then click and drag to change the peak height or position. Hover over the peak and use the scroll wheel on the mouse to change the peak width. When the curve is a reasonable estimate of the data, click on 'Fit', which will refine the curve. The table below the plot will display information about the peak area, width, and an estimate of the crystal domain size which gave rise to the diffraction pattern, if the domains are smaller than 100 nm.

More accurate fits of peak shape can be obtained through the 'Whole Pattern Fitting' function. The button to enter this function is labeled with small line plot and the letters 'WPF'.

6.8 END OF EXPERIMENT

6.8.1 Cleaning up workspace

The sample preparation area is a shared workspace. It is critical that users do not leave residue on any surface – countertop, slides, sample wells, mortar and pestle, spatulas. The user after you will have no idea what you were working on, and whether sample residue they encounter is toxic. Remove all samples and sample containers before leaving the laboratory.

6.8.2 Setting the source to standby power

When you are done using the instrument, set the source to **20 kV** and **5 mA** for 100 W (see Figure 7).

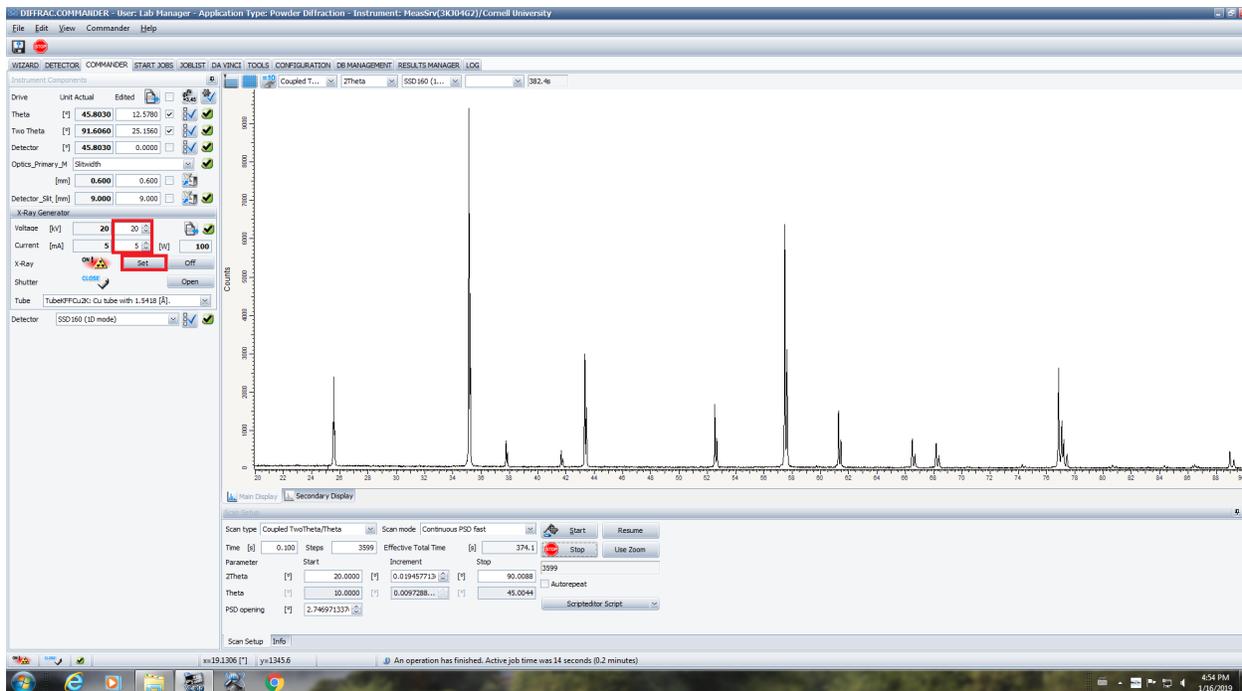


Figure 7 Setting the x-ray source to standby voltage and current at end of experiment.

6.8.3 Disabling the control computer through coral

Disable the instrument through Coral. Ensure that the correct instrument is selected before choosing 'Disable', since it is possible to accidentally disable the wrong instrument.

7 QUICK REVIEW GUIDE

1. Enable the instrument though Coral.
2. Set the source to **40 kV x 25 mA**.
3. Use weigh paper below the sample slide when filling with powder. Mount your sample so that it is level with the surface of the puck.
4. Put the sample puck in the instrument and carefully raise it until the plastic surfaces meet all three reference pins. This is the easiest place to make a mistake, so check that it the puck meets all three pins before pushing the post further to compress the spring.
5. Check the slit settings, the scan range, and time per step (0.1 s typical).
6. Run your scan.
7. Save you data on the shared drive in your own folder.
8. Support the post to keep the sample from dropping quickly, but **DO NOT** pull on the post. Pull the lever or trigger behind the sample post, and keep the lever pulled as you lower the post.
9. Remove your sample and clean the work surface.
10. Set the source to **20 kV x 5 mA**.
11. Disable the instrument through Coral.
12. Make sure you cleaned the work surface and remove all of your materials.