Chapter 8.35

Siemens D5000 X-Ray Diffractometer

(xdif) ***(380)***

1.0 Equipment Purpose

1.1 This X-ray diffractometer is used to characterize material properties on an atomic scale. Measurements to gain information on crystal structure, lattice strain, state of ordering, and chemical composition are taken with this instrument.

2.0 Material Controls & Compatibility

2.1 This tool supports piece work (when mounted on clay) and wafers up to 6" in diameter.

2.2 Powder samples are allowed only under special conditions. Please consult with staff if you have powder samples.

3.0 Training & Applicable Documents

3.1 Members who are not explicitly listed on the RUA (Radiation Use Authorization) and who have not been issued dosimetry rings may not operate or touch any part of this tool. Observation (at a distance) is permitted; members without dosimetry rings must **never** insert any part of their body into the tool enclosure. Training and qualification is done by staff to ensure all EH&S radiation safety requirements are met before members are allowed to independently operate the tool.

3.2 Before gaining access to the tool, all users must have:

3.2.1 Completed the Radiation Safety Training course from EH&S via the UC Learning Center. Contact staff if you do not have access to the UC (Berkeley) Learning Center. Take the online course; we recommend choosing 401.3 as it covers material from both 401.1 and 401.2. At the end of the course be sure to save your completion certificate as a pdf. We recommend taking a picture/screenshot of your completion certificate and then save that picture to a pdf file (don't try to save the screen pix to pdf… this doesn't work correctly).

3.2.1.1 Note: If you do not have a @berkeley email address, you will need to request a guest account in order to be added to the RUA. Other UC schools (ie. UC Davis, UCSF) may request access for a particular course. Contact process engineer 1 to begin this process.

3.2.1.2 Affiliate members may request a guest account to access the UC Learning Center. Staff will assist with this process.

3.2.2 Once you complete the online course, email the RUA holder (adove@berkeley.edu) and send her your course completion screenshot so that she can add you to the RUA.

3.2.3 Fill out the Radiation User Information Form

3.2.4 EH&S will reach out once you submit the above form to arrange for you to get fitted for your dosimetry ring. If you don't hear from EH&S within 72 hours of submitting the form or need immediate assistance, email radsafety@berkeley.edu to determine the status of your request. The EH&S office is on the West side of campus: **Environment, Health & Safety, 2199 Addison, Room 317, University Hall.**

3.3 Specialty Tool

3.3.1 This tool is a **Radiation Producing Machine (RPM)**.

3.3.2 This tool requires equipment specific training with staff. Once you have been added to the RUA, you may submit a training request in Mercury to request training on this tool.

3.3.3 Timeline:

3.3.3.1 Be notified by the RUA holder that you have been added to the RUA.

3.3.3.2 Submit a training request in Mercury.

3.3.3.3 Be trained by staff.

3.3.3.4 Take the online exam in the Nanolab office (open 8A - 12P, 1P-5P).

3.3.3.5 Arrange a qualification session with staff to show competency on the tool.

3.4 EH&S Radiation Safety Program Information at www.ehs.berkeley.edu/rs.html.

3.5 Diffrac-AT Manual D5S325, located in white binder next to the instrument.

3.6 PDF cards for common materials in the Nanolab, located in black binder next to the instrument.

3.7 Bartels Four-Bounce monochromator Design; J.W. Bartels, J. Vac. Sci. Technol. B 1(2), pp. 338-345, 1983.

3.8 Precision Lattice Constant Determination; W.L Bond, Acta. Cryst. 13, pp. 814-814, 1960.

3.9 Quantitative X-Ray Diffraction from Superlattices, E.E. Fullerton, I. K. Schuller, Y. Bruynseraede, MRS Bulletin, 33-38, 1992.

3.10 Notes on the XRD Commander software came in part from “Basic Operation of the Bruker D8 HRXRD and XRD Commander”, S. A. Speakman, http://prism.mit.edu/xray/oldsite/Bruker%20HRXRD%20Basic%20Operation.pdf, Retrieved 11/30/2016.

4.0 Definitions & Process Terminology

4.1 **Bartels Four Bounce Monochromator**: A device which selects x-rays of a specific energy, traveling along a specific axis. Two channel cut germanium crystals are mounted as periscopes in opposition. The radiation diffracts from the first surface to the second, where it diffracts again with direction unchanged but position displaced by the projected width of the channel. The second crystal reverses the displacement, so the beam direction and position are identical to the original beam. Refer to the following paper for more detailed information: J.W. Bartels, J. Vac. Sci. Technol. B 1(2), Apr-June 1983, pp. 338-345.

4.2 **Detector Angle (2θ)**: The angle between the projection of the x-ray source and the detector (see Figure 10.1).

4.3 **Eucentric Point**: The point at which all four goniometer axes intersect in space. The instrument is properly aligned when the sample surface coincides with the eucentric point, as does the point illuminated by the x-ray source and the point observed by the detector.

4.4 **Goniometer**: A stage that supports the sample and detector, allowing precise movement. The goniometer has four degrees of motion to adjust sample and detector positions:

4.4.1 1^{st} – rotation of sample about a vertical axis

4.4.2 2^{nd} – rotation of detector about a vertical axis

4.4.3 3^{rd} – tilt of sample about a horizontal axis (χ)

4.4.4 4^{th} – rotates the sample about the axis normal to the sample surface through the center of the sample holder (φ) (see Figure 10.1).

4.5 **RUA**: Radiation Usage Authorization. A permit issued by the University's Office of Environment, Health & Safety (EH&S) allowing a radiation-producing instrument to be operated on campus. Both the instrument and all operators must be explicitly identified on the RUA that is posted by the tool.

4.6 **Sample Angle (θ)**: The angle between the plane of the sample and the incident x-ray beam (see Figure 10.1). This is also referred to as the Bragg angle and is called theta or omega by the XRD software..

4.7 **Omega**: The XRD software uses the terms theta and omega interchangeably.

4.8 **High Resolution Mode**: A measurement that makes use of a Bartels four-bounce monochromator to reduce the beam spread FWHM from 0.2° to 0.0033°. This measurement, due to the Bertels monochromator, greatly reduces the intensity of the incident radiation and works best on highly crystalline samples. This mode is often used with rocking curve and coupled mode scans and can also be used to measure thickness of thin films in a stack. It is dependent on sample angle, θ. The slits and Ni filter are not used in this mode.

4.9 **High Throughput Mode**: A measurement that does not make use of a Bartels four-bounce monochromator. This mode is commonly used on polycrystalline materials because the higher intensity incident radiation yields a detectable signal at the detector. This mode follows Bragg reflection and is often used for normal coupled (θ-2θ) scans.

4.10 **XRD Commander Scantypes**:

4.10.1 **Locked Coupled**: moves 2θ and θ, forcing $\theta = 1/2 \times (2\theta)$

4.10.2 **Unlocked Coupled**: moves 2θ and θ, preserving any initial offset between the two angles, $\theta = 1/2 \times (2\theta) + offset$

4.10.3 **2Theta/Omega**: the same as unlocked coupled

4.10.4 **Omega/2Theta**: same as 2Theta/Omega, but the start and stop angles are specified in units of θ, not 2θ

4.10.5 **Rocking Curve**: moves only the incident-angle omega (i.e. θ)

4.10.6 **Detector Scan**: moves only the detector-angle 2θ

4.10.7 **Chi**: moves the tilt of the sample around the x-axis

4.10.8 **Phi**: moves rotation of the sample around the sample normal

4.11 **XRD Commander Scanspeed**: This determines whether data will be taken in constant time or constant step mode. The current setting is displayed on the button.

4.11.1 **Sec/Step** sets a constant time for taking data at each step. The range and the increment will determine the total scan time.

4.11.2 **Deg/Min** forces the total scan time to be constant over a given range and changing the increment will change the scan intensity.

4.11.3 Users should typically use **Sec/Step**.

4.12 **XRD Commander Continuous/Step Scan**: This determines the mode of the data collection. The current setting is displayed on the button.

4.12.1 **Continuous**:This mode counts the x-ray photons at all positions of the detector, and integrates them into discrete data points as indicated by the increment value. The detector is continuously moving. This is preferred for faster scans.

4.12.2 **Step Scan**:This mode counts the x-ray photons while the motor is stationary at discrete positions as determined by the increment value. Photons scattered between those data points are not measured. This is preferred for slower scans.

4.13 **XRD Commander Cont Button**: This button allows you to adjust your scan in real time.

4.13.1 **New Stop Value**: After the scan you can adjust the stop value and press the **Cont** button. The scan will resume from where it finished and run until the new stop value.

4.13.2 **Repeat**: If you click the **Cont** button without changing the stop value, the previous scan will be repeated and the two scans will be averaged together. You can also use auto-repeat at the top of the menu to automatically repeat scans to improve statistics.

5.0 Safety

5.1 **High Voltage Hazard**: The x-ray tube may be biased up to 40000 VDC. The tube, located in the back left side of the main chamber, is in an enclosure with interconnects for electrical and water cooling. Never touch the enclosure or associated cables.

5.2 **Ionizing Radiation**: This instrument is a radiation producing machine as defined by the State of California. All users must complete training and meet all standards set forth by the University's Office of Environment, Health & Safety (EH&S).

5.3 Users of this instrument must be assigned to Radiation Usage Authorization (RUA) 1501. Ensure a current RUA, listing all current equipment users, is posted by the instrument before use.

5.4 A current copy of the RUA must be in plain sight on the tool at all times.

5.5 Always wear your personally assigned dosimetry ring when operating the instrument.

5.5.1 The dosimetry ring must be worn at all times while operating the tool, even if the x-ray source is off. Your dosimetry ring should be worn under your glove, on the hand that will be in closer proximity to the x-ray source.

5.5.2 When not using the tool, dosimetry rings must be kept in the drawer near the tool at all times away from radiation fields.

5.6 Never over-ride or defeat interlocks at any time. If you notice an interlock is not working correctly, immediately turn off the x-ray source, discontinue use of the tool, and report a fatal problem.

5.7 Never rely explicitly on interlocks to protect you; they are in place as a backup when user error occurs.

5.8 Always close the x-ray shutter before opening the enclosure door. Check to make sure the X-ray Shutter Open Indicator Light is off before opening the enclosure door.

5.9 A Geiger Mueller survey meter (labeled GM PROBE 1) located next to the tool must be used as a redundant check to ensure the x-ray shutter is closed before any body part passes the plane of the enclosure door. If you are unable to locate the meter, or if it does not pass the battery test, immediately discontinue use of the tool and report a red fault. More info on the GM can be found in Appendix 11.2.

5.10 A survey meter (labeled GM PROBE 2) must be placed inside the enclosure and used as a redundant check to ensure the x-ray shutter is closed before opening the enclosure door. If you are unable to locate the meter, or if it does not pass the battery test, immediately discontinue use of the tool and report a red fault. More info on the GM can be found in Appendix 11.2.

5.11 Once you have determined it is safe to open the enclosure door, proceed with opening the door and immediately use the survey meter to verify that the shutter to the x-ray source is closed and that there is no ionizing radiation in the cabinet (see Appendix 11.2).

5.12 Never make manual adjustments to your sample while the shutter to the x-ray source is open. This is often referred to as “hot” or “live” adjustments/alignments. Such practice is not permitted on this instrument at any time.

6.0 Process Data

6.1 Refer to appendix for Marvell Nanofabrication Laboratory XRD Material Data Tables.

7.0 Available Processes, Gasses, Process Notes

7.1 General Process Information

7.1.1 The instrument uses copper K-alpha (Cu Kα) radiation with a weighted average wavelength of 0.154184 nm (Cu Kα1 = 0.154056, Cu Kα2 = 0.154439 nm). A Ni foil is used to filter Cu Kβ radiation.

7.1.2 The x-ray power supply has an idling condition of 20 kV and 5 mA. Normal operating conditions during measurements are 40 kV and 30 mA.

7.1.3 Solid samples ranging in size from ~ 1 mm^2 to 6 inches in diameter can be measured.

7.1.4 Measurement types include 2-theta/omega (θ-2θ), rocking curve, phi, and chi scans. Other measurement types may be possible, contact adove@berkeley.edu with questions.

8.0 Equipment Operation

(Note: If you find the system in an OFF condition, do not proceed - report it on Mercury!)

8.1 Software Setup and Goniometer Initialization

8.1.1 Enable the instrument on Mercury and put on your personalized dosimetric ring. Your dosimetry ring should go under your glove.

8.1.2 Open the DIFFRAC plus XRD Commander software by clicking the shortcut on the Desktop.

8.1.3 Initialize the goniometer by selecting the **Init Drives** button (yellow goniometer symbol) or clicking **Diffractometer > Direct Mode**, typing **IN** and hitting **Send Command**.

8.2 Preparation for Equipment Use

8.2.1 Verify that you are wearing your assigned dosimetry ring.

8.2.2 Set up the external GM survey meter:

8.2.2.1 Turn on the meter, located on the cart to the left of the system.

8.2.2.2 Position both toggle switches forward to the **ON** and **F** positions.

8.2.2.3 Ensure that the batteries are OK by switching the dial to **BAT** and verifying the needle gauge has a needle position in the **Bat. test** region.

8.2.2.4 Turn the dial to the **X1** position.

8.2.3 Verify that the RED X-ray Shutter Open indicator light (inside the chamber) is off.

8.2.4 Before opening the enclosure door, scan around the door and across the face of the system with the GM survey meter to ensure that the shutter is closed and no radiation is present. The presence of radiation will be indicated by a continuous audible tone from the survey meter. If you detect radiation at this point, discontinue use of the instrument and report a fatal problem on Mercury. (Read 11.4.1 for a more thorough description of proper scan technique.)

8.2.5 If no radiation is present, you may now open the door - but be sure to always break the plane of the door to the enclosure with the survey meter first. The hand that is holding the survey meter should be the hand with your dosimetric ring. Do not place body parts into the enclosure before checking with the survey meter.

8.2.6 Turn on the enclosure light -- there is a light switch inside the door frame immediately behind the door handle.

8.2.7 Set up the internal GM survey meter:

8.2.7.1 Turn on the meter, located just inside the door.

8.2.7.2 Position both toggle switches forward to the **ON** and **F** positions.

8.2.7.3 Ensure that the batteries are OK by switching the dial to **BAT** and verifying the needle gauge has a needle position in the **Bat. test** region.

8.2.7.4 Turn the dial to the **X1** position.

8.2.7.5 Do not remove the meter from inside the enclosure.

8.2.7.6 You may now turn off the external GM survey meter and put it back on the cart.

8.3 Collimator Setup

8.3.1 If the measurement requires the collimator (see Figure 10.3 for a picture of the collimator), make sure you have hands-on training and a complete understanding of how to set it up. If the measurement does not require the collimator proceed to section 8.5.

8.3.2 If the monochromator is installed, remove it by loosening the two (very tiny and hard-to-turn) metallic colored shafts hidden under the large prominent (and not-to-be-disturbed black knobs). Lift the monochromator off its platform and put it gently down in the cabinet.

8.3.3 Place the collimator on the platform and begin to tighten the screws only enough to give slight friction, such that it can still be moved by hand. Push it all the way to the back of the cabinet and rotate it counterclockwise (as viewed from above) as far as it will go. See Figure 10.3 for a picture of the collimator mounted on the platform.

8.3.4 Turn it clockwise about one degree. Do not tighten the screws yet.

8.3.5 Slip the phosphor screen onto the sample stage and adjust the stage so that z is 6.75 mm and the mark centers under the z-height dial indicator arm. See Figure 10.4 for a picture of the indicator arm being installed.

8.3.6 Remove the indicator arm, close the enclosure door, and begin the measurement by opening the shutter.

8.3.7 Bring the stage into working position and look through the cabinet window at the mirror. The bright green spot of fluorescence should be uniform and centered on the center mark of the phosphor screen. If not, close the x-ray shutter, open the enclosure when safe, and adjust the collimator.

8.3.8 Repeat until you have a well-centered spot, then tighten the screws (two fingers on the driver are enough) and re-check.

8.4 Monochromator Setup

8.4.1 If the measurement requires the monochromator (see Figure 10.5 for a picture of the monochromator), make sure you have hands-on training and a complete understanding of how to set it up. If the measurement does not require the monochromator skip to section 8.5.

8.4.2 If the collimator is installed remove it by unscrewing the two bolts holding it down and lifting it out of position. Store it somewhere safe inside the cabinet.

8.4.3 Take note of the two small steel pins sticking up in the plate that supports the monochromator. They fit into cavities in the monochromator's baseplate where they are engaged by stops and set screws that fix the location.

8.4.4 Pick up the monochromator by the handle (see Figure 10.5 for a picture of the monochromator), and gently lower it onto the two pins. It should settle down easily, requiring no extra force. Tighten the two very small and hard-to-touch shafts directly below the large (and not-to-be-disturbed) black plastic knobs, gently with your fingers, turn them clockwise to tighten. The shafts, which drive set screws, sometimes jam, wiggle them when they seem difficult to turn. Tighten them only enough to prevent the monochromator from moving from side to side under the gentle pressure of two fingers.

8.4.5 Ensure the 2 mm slit, Ni filter, and 0.2 mm slit are out of the sample-to-detector path.

8.5 Loading Samples

8.5.1 Centimeter-sized pieces are usually mounted over clay on a plastic sample holder. Whole wafers and large pieces of wafers, greater than about 2 cm, are more conveniently held with three adjustable clamps. Use of the adjustable clamps is strongly preferred over the use of the clay mounting technique.

8.5.2 Mounting Small Samples with Clay

8.5.2.1 There are three sample holders; black, clear and grey. The grey sample holder is the best because it contributes the lowest background signal to measurements. The black holder has a strong peak at ~22°, and weaker peaks at ~ 34°, 48°, 53°. The clear holder has weak peaks at ~15° and 29°.

8.5.2.2 Place a small ball of clay in the well of the sample holder, center your sample on top and press the sample into the clay using a flat, clean surface (see Figure 10.6).

8.5.2.3 Try to make the top surface of your sample parallel with the top surface of the sample holder. Tilt in the sample can easily shift peaks enough to make them very hard to find.

8.5.2.4 The clay should be completely under the sample. Otherwise, additional diffraction peaks from the clay might be detected.

8.5.2.5 The sample holder slides into a fixture that can be installed on the goniometer and is supported by its upper surface under spring pressure. This way one has only to measure the z height of the stage; if the sample is correctly mounted in the holder it will come out at the right height automatically.

8.5.2.6 Install the sample mount onto the goniometer.

8.5.2.7 Hold the z-height dial indicator in your right hand at the top of the mounting arm. **Gently** use your left hand to pull the probe up. While still holding the probe in the raised position. **Very gently** slip the mounting pin of the indicator arm into the socket at the top of the goniometer frame. While still holding the probe up, insert the locking cam pin and turn it clockwise until firm.

8.5.2.8 **Slowly and gently** release the dial indicator probe onto the surface of the stage. The indicator should settle to a reading of 6.75 mm. Turn off the gauge when finished.

8.5.2.9 Lift the probe of the dial indicator with your right hand and use your left hand to slip the sample holder into the stage; push down on the spring-loaded plate

and guide the holder in. Keep the probe of the dial indicator raised and move the sample until centered under the indicator probe. There is no need to let the probe touch the sample (you can if you want, just be careful to avoid tilting the sample in the clay).

8.5.2.10 Once the sample is centered, continue to lift the probe while using your right hand to release the lock cam and take out the dial indicator. Be very gentle, the alignment pins jam easily. The cam can be left in place.

8.5.3 Mounting Samples with the Adjustable Clamps

8.5.3.1 Slide the clamps within the tracks of the stage to approximate the size of a sample such that it will hold it in the center of the chuck.

8.5.3.2 Tighten the two rear clamps onto the sliding track by turning the bottom turn dial clockwise. If you are loading a 6-inch wafer, make sure they are located at the very end of the track, almost hanging out of the track.

8.5.3.3 Remove the clamp from the track that is closest to you.

8.5.3.4 Slide the sample into the top slots of the two adjustable clamps that are tightened on the stage and tighten the upper turn dial counter-clockwise until they clamp the sample.

8.5.3.5 Place the third adjustable clamp back into the stage, position it to hold the sample, and tighten the lower track dial followed by the upper clamp to hold the sample.

8.5.3.6 Use caution when tightening the clamps. Align and tighten the clamps such that the sample is not under strain. Deformation of samples can be detected and will affect measurements.

8.5.3.7 Hold the z-height dial indicator in your right hand at the top of the mounting arm. **Gently** use your left hand to pull the probe up. While still holding the probe in the raised position. **Very gently** slip the mounting pin of the indicator into the socket at the top of the goniometer frame. While still holding the probe up, insert the locking cam pin and turn it clockwise until firm. **Slowly and gently** lower the probe onto the surface of your sample, making sure not to damage the surface.

8.5.3.8 Adjust the z-height axis knob on the lower bottom portion of the stage (labeled “Z”) until the z-height indicator reads 6.75 mm. Turn off the gauge when finished.

8.5.3.9 Once the correct z-height is set, remove the indicator by gently lifting the probe with your left hand and use your right hand to remove the locking cam pin. Slide the top of the indicator mounting pin out of the socket while continuing to hold the probe tip in the up position.

8.6 Performing a Locked Coupled Scan

8.6.1 If using the collimator, verify that the **2 mm slit**, **Ni filter**, and **0.2 mm** slit are installed, in this order, between the sample and detector. If using the monochromator, verify no slits or filters are installed.

8.6.2 Close the enclosure door.

8.6.3 Verify that the x-ray generator is at 40kV and 30mA. If not, input these values into the target values and click “set” to adjust the generator.

8.6.4 In the **Scantype** menu, select **Locked Coupled**.

8.6.5 Adjust the following parameters

8.6.5.1 **Theta**: Does not require adjusting, but for faster set-up, set to half of the **Start** value..

8.6.5.2 **2Theta**: Does not require adjusting, but for faster set-up, set equal to the **Start** value.

8.6.5.3 **Phi**: Set the grey value to 0.000 for now. This may be optimized at another time.

8.6.5.4 **Chi**: Set the grey value to 0.000 for now. This may be optimized at another time.

8.6.5.5 **Start** and **Stop**: Located at the lower left and right corners of the box where the collected data is graphed. If the desired detector range is unknown, start with 20 - 130°.

8.6.5.6 **Increment**: If the desired step size is unknown, start with 0.100°.

8.6.5.7 All other values should remain constant and not be changed by users. They are as follows; Measuring channel = 1, Amplification = 2, High voltage = 800, Base level = 0.300, Upper level = 2.000, Tube voltage = 40, Tube current = 30. Scanspeed is determined by the software, Sec/Step should be the units. Continuous mode should be used.

8.6.6 Verify the enclosure door is securely closed.

8.6.7 Open the x-ray shutter by clicking on **Shutter: Open**. The icon should turn RED to indicate danger, radiation present. Ensure the X-ray Shutter Open Indicator Light is now on.

8.6.8 Click on **Start** to begin data-taking. Data will appear in the graph window as it is collected. To collect additional data and average scans together, click the red repeat arrow at the top of the screen to auto-repeat scans.

8.6.9 To stop the measurement, click on **Stop**.

8.6.10 Close the x-ray shutter by clicking on **Shutter: Close**. Verify the shutter is closed by listening for the audible click of the shutter moving, ensuring that the X-ray Shutter Open Indicator Light is off, and that the GM PROBE 2 is not emitting a continuous audible sound. The icon should turn GREEN to indicate a safe condition.

8.6.11 To save the collected data click on File > Save to save as a comma delimited file.

8.7 Performing a Rocking Curve Measurement

8.7.1 If using the collimator, verify that the **2 mm slit, Ni filter**, and **0.2 mm slit** are installed, in this order, between the sample and detector. If using the monochromator, verify no slits or filters are installed.

8.7.2 Close the enclosure door.

8.7.3 Click on Scanning mode, select **Rocking Curve Scan**.

8.7.4 Adjust the following parameters

8.7.4.1 **Detector Angle**: Located inside the Goniometer menu. Set the value to the peak location (2θ) determined in the normal coupled scan. This is the peak of interest that you are trying to optimize the signal strength of.

8.7.4.2 **Chi**: Set the grey value to 0.000 for now. This may be optimized at another time.

8.7.4.3 **Phi**: Set the grey value to 0.000 for now. This may be optimized at another time.

8.7.4.4 **Sample Angle Scan Range**: Located at the bottom left and right corners of the blank box where the collected data is graphed. A tight range, typically 5 – 10°, that covers only the peak of interest, should be used.

8.7.4.5 **Step Size**: If the desired step size is unknown, start with 0.010°.

8.7.4.6 All other values should remain constant and not be changed by users. They are as follows; Measuring channel = 1, Amplification = 2, High voltage = 800, Base level = 0.300, Upper level = 2.000, Tube voltage = 40, Tube current = 30.

8.7.5 Verify the enclosure door is securely closed.

8.7.6 Open the x-ray shutter by clicking on **Shutter: Open**. The icon should turn RED to indicate danger, radiation present. Ensure the X-ray Shutter Open Indicator Light is now on.

8.7.7 For a single scan, click on **Start**. Data will appear in the graph window as it is collected. To collect additional data and average scans together, click on **Continuous**.

8.7.8 To stop the measurement, click on **Stop**.

8.7.9 Close the x-ray shutter by clicking on **Shutter:Close**. Verify the shutter is closed by listening for the audible click of the shutter moving, ensuring that the X-ray Shutter Open Indicator Light is off, and that the GM PROBE 2 is not emitting a continuous audible sound. The icon should turn GREEN to indicate a safe condition.

8.7.10 To save the collected data click on File >Save to save as a comma delimited file.

8.8 Optimization of Signal with Phi and Chi Scans

Phi and Chi scans may be used to optimize signal strength and improve peak detection. Peak location can be determined using a normal coupled scan (see section 8.5) first, and then, without removing the sample from the stage, Phi and Chi scans are used to increase signal strength further.

8.8.1 Phi Scan Optimization

8.8.1.1 Immediately after identifying the peak of interest with the 2Theta/Omega scan, proceed with the Phi scan, do not adjust the sample or stage position.

8.8.1.2 Click on Scanning mode and select **Phi circle scan**.

8.8.1.3 Adjust the following parameters:

8.8.1.3.1 **Detector Angle**: Located inside the Goniometer menu. Set the value to the peak location determined in the normal coupled scan. This is the peak of interest that you are trying to optimize the signal strength of.

8.8.1.3.2 **Sample Angle**: Located inside the Goniometer menu. This value should read half of the detector angle (the value directly above). Users will need to calculate the appropriate sample angle manually.

8.8.1.3.3 **Chi**: Set the grey value to 0.000 for now. This may be optimized at another time.

8.8.1.3.4 **Phi Angle Scan Range**: Located at the bottom left and right corners of the blank box where the collected data is graphed. If the desired range is unknown, start with 0° to 359°.

8.8.1.3.5 **Step Size**: If the desired step size is unknown, start with 0.500°.

8.8.1.3.6 All other values should remain constant and not be changed by users. They are as follows; Measuring channel = 1, Amplification = 2, High voltage = 800, Base level = 0.300, Upper level = 2.000, Tube voltage = 40, Tube current = 30.

8.8.1.4 Verify the enclosure door is securely closed.

8.8.1.5 Open the x-ray shutter by clicking on **Shutter: Open**. The icon should turn RED to indicate danger, radiation present. Ensure the X-ray Shutter Open Indicator Light is now on.

8.8.1.6 For a single scan, click on **Start**. Data will appear in the graph window as it is collected. To collect additional data and average scans together, click on **Continuous**.

8.8.1.7 To stop the measurement, click on **Stop**.

8.8.1.8 Close the x-ray shutter by clicking on **Shutter:Close**. Verify the shutter is closed by listening for the audible click of the shutter moving, ensuring that the X-ray Shutter Open Indicator Light is off, and that the GM PROBE 2 is not emitting a continuous audible sound. The icon should turn GREEN to indicate a safe condition.

8.8.1.9 To save the collected data click on File>Save to save as a comma delimited file.

8.8.1.10 Choose the appropriate peak with the strongest signal and use this as the new Phi value during subsequent scans of the peak of interest.

8.8.2 Chi Scan Optimization

8.8.2.1 Immediately after identifying the peak of interest with the normal coupled scan and the optimal Phi angle, proceed with the Chi scan, do not adjust the sample or stage position.

8.8.2.2 Click on Scanning mode and select **Chi circle scan**.

8.8.2.3 Adjust the following parameters:

8.8.2.3.1 **Detector Angle**: Located inside the Goniometer menu. Set the value to the peak location determined in the normal coupled scan. This is the peak of interest that you are trying to optimize the signal strength of.

8.8.2.3.2 **Sample Angle**: Located inside the Goniometer menu. This value should read half of the detector angle (the value directly above). Users will need to calculate the appropriate sample angle manually.

8.8.2.3.3 **Phi**: Set the grey value to the optimized Phi value determined in section 8.7.1.

8.8.2.3.4 **Chi Angle Scan Range**: Located at the bottom left and right corners of the blank box where the collected data is graphed. Set a range of -5 to +5°.

8.8.2.3.5 Step Size: If the desired step size is unknown, start with 0.050°.

8.8.2.3.6 All other values should remain constant and not be changed by users. They are as follows; Measuring channel = 1, Amplification =

2, High voltage = 800, Base level = 0.300, Upper level = 2.000, Tube voltage = 40, Tube current = 30.

8.8.2.4 Verify the enclosure door is securely closed.

8.8.2.5 Open the x-ray shutter by clicking on **Shutter: Open**. The icon should turn RED to indicate danger, radiation present. Ensure the X-ray Shutter Open Indicator Light is now on.

8.8.2.6 For a single scan, click on **Start**. Data will appear in the graph window as it is collected. To collect additional data and average scans together, click on **Continuous**.

8.8.2.7 To stop the measurement, click on **Stop**.

8.8.2.8 Close the x-ray shutter by clicking on **Shutter:Close**. Verify the shutter is closed by listening for the audible click of the shutter moving, ensuring that the X-ray Shutter Open Indicator Light is off, and that the GM PROBE 2 is not emitting a continuous audible sound. The icon should turn GREEN to indicate a safe condition.

8.8.2.9 Choose the appropriate peak with the strongest signal and use this as the new Chi value during subsequent scans (along with the optimized Phi value) of the original peak of interest.

8.9 Analyzing Data

8.9.1 After you have completed your measurement, ensure that the x-ray shutter is closed, before attempting any analysis.

8.9.2 To resize the graph: click and drag over the new region you wish to graph, followed by a second click.

8.9.3 To reset the graph: click on Display Options, followed by ZOOM RESET.

8.9.4 To extract a peak location, double click on the window in the area of interest. A “peakstick” will appear at the location of the cursor.

8.9.5 When you are finished using the tool remember to turn off and remove GM PROBE 2 from the enclosure and to also enter the date, member login name, RUA Holder, minutes spent on the instrument, voltage/current settings, and any relevant comments in the logbook next to the instrument.

8.10 Saving Data

8.10.1 Data can be saved in a ***.TXT** format from **File > Save**.

8.10.2 The data has metadata in a header at the top of the file, providing the configuration of the tool at the time the data was taken. The diffraction data is found below this header following:

[Data]

Angle, Det1Disc1,

15, 419,

15.01, 420,

⋮ ⋮

8.11 Finishing Your Session

8.11.1 Initialize the goniometer to return the system to the loading position.

8.11.2 Verify that the x-ray shutter is closed.

8.11.3 Verify that the external GM survey meter is on and verify that no x-rays are present before opening the door.

8.11.4 Remove your sample.

8.11.5 Leave the chuck that you used in the tool (there is no default chuck).

8.11.6 Turn off the light.

8.11.7 Turn off both GM survey meters.

8.11.8 Close the door.

8.11.9 Return your ring to the rack.

9.0 Troubleshooting Guidelines

9.1 General Hints and Tips

9.1.1 The D5000 operates stepwise, moving the goniometer, accumulating detector counts and then moves again. It does not measure between steps. If the steps are too big, it is possible to completely miss a peak. For example, a highly crystalline silicon sample may have a peak width less than 0.007°. This peak may never be detected if you scan at 0.1° per step. Keep in mind the smaller the step size, the longer the scan will take. Typically, 100 steps per scan is a good starting point.

9.1.2 The sample always turns exactly half as much as the detector, but you have the opportunity to correct offsets by unlocking the sample via manual entry of an initial value.

9.1.3 When a mode is changed, say from normal coupled scan to rocking curve the previous parameters from the last time the mode was used are recalled. Make sure all the settings are correct before taking your measurement.

9.1.4 Parameters are changed by clicking on the value, for example Normal, coupled scan, and either dragging the mouse or hitting the arrow keys. Numerical parameters are selected by clicking, and can be incremented by dragging the mouse or by typing in new values.

9.2 Failure to Locate Diffraction Peaks

9.2.1 Is the yellow radiation warning light on top of the cabinet lighted? If not get help, it means the x-ray generator is off.

9.2.2 Is the sample mounted flush with the top surface of the sample holder? Any sample tilt will make it difficult to find a peak.

9.2.3 Are you certain that you know the sample cut and orientation? Occasionally samples get mixed up or mislabeled.

9.3 When I use the IN command, the phi axis will not stop nodding back and forth. What should I do?

9.3.1 Issue the command RC0 (zero, not O) to make xdif pay attention to the pull-out keyboard.

9.3.2 Go to the keyboard and press SHIFT, then CIRCLE 4:3 [ENTER] to put the phi axis in manual mode. Note that the shift-key is sequential, not like a typewriter.

9.3.3 While **holding** the tuning button down, press the right arrow three times. The arrow key determines the speed of the motion. This will rotate the phi axis 10-15º ccw (as seen from above), past the initialized position.

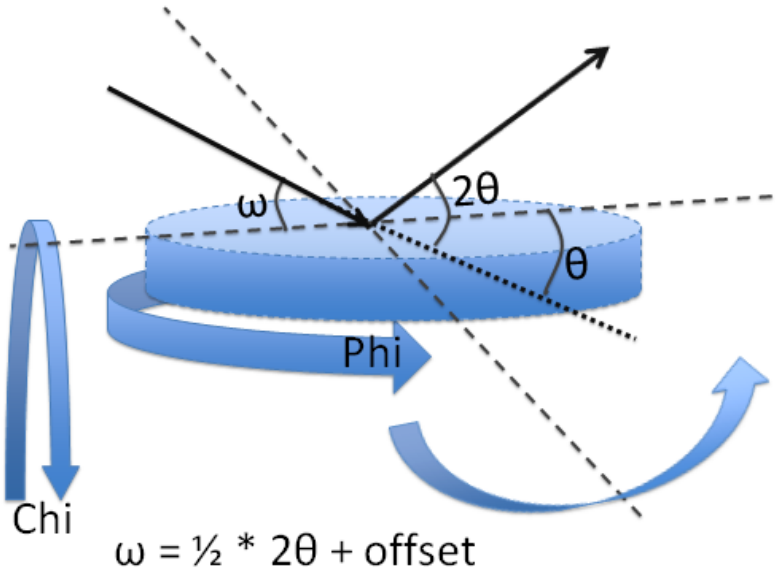
9.3.4 Go back to the computer and issue RC1, then the IN command. If this does not do the trick, ask for help.

9.4 The x-ray power supply will not enter HV mode (turn on).

9.4.1 Issue the command SHIFT, X-RAY, 1, [ENTER] on the pull-out keyboard.

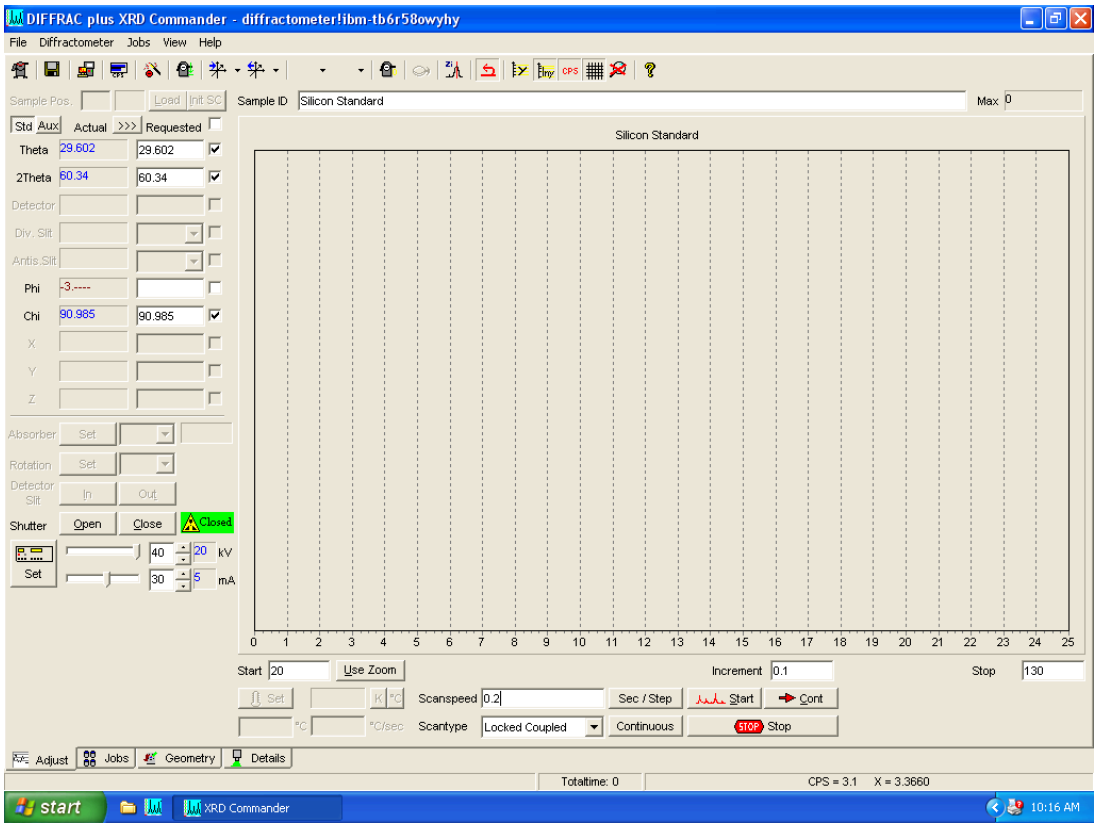
9.4.2 Note, the shift-key is sequential, not like a typewriter.

10.0 Figures & Schematics

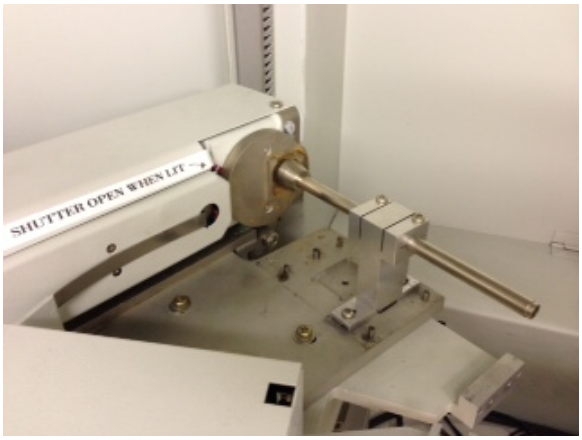


10.1 Degrees of Freedom

10.2 Screenshot of the XRD Commander Software:



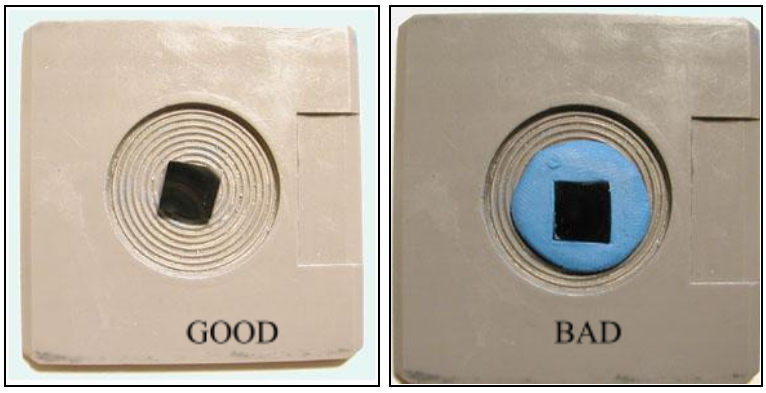
10.3 Collimator installed on the platform.



10.4 Z-height dial indicator being installed.

10.5 Bartels four-bounce monochromator.

10.6 Sample Mounting with Clay



10.7 EH&S Slide on Interlock Safety

Radioactive Beam Interlocks

Do ***NOT*** rely on an interlock to turn off the beam for the purposes of accessing the sample area.

- Although safety interlocks are designed to prevent direct exposure to the beam or to scatter radiation, you should **ALWAYS** use a calibrated survey meter to check for radiation fields before reaching into the machine enclosure, just to make sure that the beam port shutter is really closed!

11.0 Appendices

11.1 Quick Guide for System Startup and Shutdown (FOR STAFF ONLY)

11.1.1 Startup (Assuming facilities are hooked up, and you have already enabled the system)

11.1.1.1 Turn the MAINS switch to the I (on) position - (Netz - I or O).

11.1.1.2 Slide out the drawer and enter the following sequence:

11.1.1.2.1 SHIFT, X-RAY, 1, ENTER (this enables the HV circuits)

11.1.1.3 Turn the KEY to the I position (if not already there).

11.1.1.4 Press (& HOLD) White Button until the Green Button Light comes on (~5 sec)

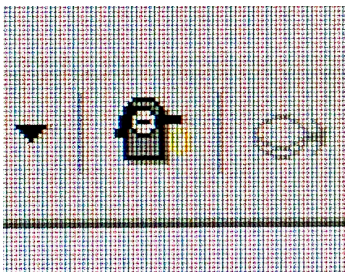
11.1.1.5 Press Green Button – the Orange X-RAY ON will illuminate. Should the X-RAY ON light not stay on, you haven't turned the HV circuit properly - repeat step **11.1.1.2.1** again - you may hear a light click as the HV circuit latches. Now hold the Green Button again…X-RAY ON should stay illuminated.

11.1.1.6 DO NOT open the door until you know it's safe - verify that the shutter-open light is off, and use the geiger counter to test for the presence of x-rays!.

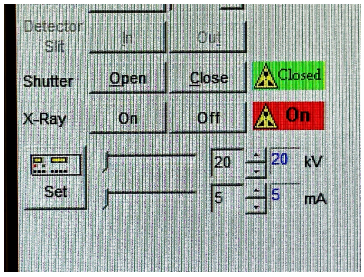
11.1.1.7 Turn on the computer and once booted, start the XRD Commander program (double-click the icon).

11.1.1.8 Look into the system to make sure the stages are in a safe state to initialize (no items blocking or disassembled)...then proceed to initialize the stages.

11.1.1.9 On the computer screen, the INIT Icon is on the top bar, just above the #2 vertical dashed line from scale below. (Be sure to select all 4 stages - Theta, @Theta, Phi, & Chi to be initialized)



INIT Button



SHUTTER/X-RAY

11.1.1.10 Also on the computer screen - verify that X-RAY is ON - this should always be ON (RED)…DO NOT turn it off, as it will turn off the HV latching circuit, and you will need to go back to **11.1.1.2.1** again to re-engage it.

11.1.1.11 The system is now ready for use.

11.1.2 Shutdown

11.1.2.1 From the computer screen, Close the X-Ray shutter and turn the X-RAY Off. Verify X-RAY safety shutter light is off.

11.1.2.2 Close the XRD Commander program.

11.1.2.3 You can now press the Mains (Netz) power switch into the O position (OFF).

11.1.2.4 Note: you will still see the Green READY led still lit, and the LCD readout will still be active.

11.2 Useful Information on the Geiger Mueller Survey Meter

The following originated from UC Davis Safety Services (http://safetyservices.ucdavis.edu/ps/rs) and has been modified.

A hand held radiation survey meter is used to perform routine and periodic inspections for radiation leaks. The operator should inspect the instrument at the beginning of each and several times during the session. This article describes how to perform this inspection.

The radiation survey meter is composed of two parts: the monitor and the probe. The monitor houses the electronics, the batteries, the controls and the meter which displays the reading. This meter might be calibrated in milliroentgens per hour (mR/hr) or counts per minute (cpm). The probe is a wand which is attached to the meter via a coax cable. Probes differ in shape and in the type of radiation (alpha, beta or gamma) they were designed to detect. Please note the type of probe you are using so that you can be sure you are using it correctly.

The following provides a quick reminder on how we describe the amount of radiation measured:

Roentgen R - Radiation exposure in air as ionizations per unit mass of air.

(1 R = 2.58x10-4 Coulomb/kg air)

Radiation Adsorbed Dose rad - The amount of radiation deposited per unit mass of the absorbing material. (1 rad = 0.01 J/kg)

Roentgen Equivalent Man rem - A measure of the biological effect of radiation. For x-rays, 1 rad = 1 rem.

Some common sources of radiation exposures and the dose equivalents are:

- Coast-to-coast airline flight 3 mrem
- Natural background radiation in the U.S. 150-300 mrem/yr
- Chest radiograph 15-65 mrem/view
- Full body CT scan (20 slices) 3000-6000 mrem

11.3 Inspection Procedure

During the inspection you will be placing the detector in all areas where radiation leaks might be found. Start the inspection at some distance from the x-ray source and work your way towards the source. In general, you will start by inspecting the instrument's cabinet and shielding for leaks followed by inspecting the area near the x-ray tube, checking for leaks around the tube's chamber and shutter. The basic procedure is:

1. Turn the main selector switch to battery test. The dial should indicate a good charge on the battery. If not, discontinue use of the tool and report the problem to staff.

2. Turn the main selector switch to the most sensitive range.

3. Turn the audio off/on switch to on.

4. Note the level of background radiation.

5. Hold the meter in one hand and the probe in the other

6. Slowly snake the probe around in the area being surveyed. If you are using the beta-probe then make sure that the probe is turned perpendicular to the field so that ionization can occur. Listen to and watch the meter for changes in the count rate.

7. A reading significantly above background indicates a serious leak. Discontinue use of the tool and report the problem to staff.

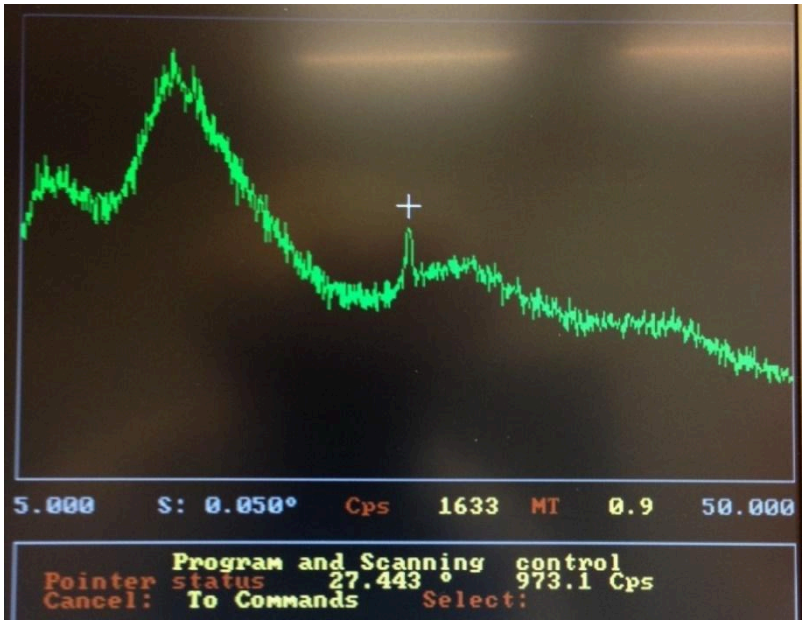
11.4 Table of Common Peaks

More peaks are in the binder located at the tool. Members can also use the Engineering Library's PDF (Powder Diffraction Files) database resource. There is a dedicated computer in the Engineering Library to access the database. More information can be found on OskiCat or you can contact a reference librarian.

Material	Orientation	Cu Kα	Cu Kβ
GaN	002	34.605	31.160
AlGaN(43%)	002	35.222	31.710
AlN	002	36.041	32.440
Al2O3	110	37.785	34.000
Al2O3	006	41.685	37.480
Si	400	69.132	61.630
GaN	004	73.000	64.970
AlGaN(43%)	004	74.481	66.250
AlN	004	76.445	67.930
Al2O3	220	80.695	71.550
Al2O3	0012	90.708	79.950
Mo	110	40.749	

11.5 Background Noise During Small Sample Measurements

It is likely that scans making use of chip sample holders will include a signal from both the chip and holder itself. The grey sample holder is known to have the lowest background signal and is typically the best choice for chip-based measurements. Below is a normal, coupled scan of the holder from 5 – 50 degrees.



11.6 Sample Calculation of Cu K-beta Peak Locations on Sapphire (Al2O3)

Despite the use of Ni film filters, Cu Kβ signal is still present within measurements on the tool. This results in the detection Cu Kα and Cu Kβ peaks during θ-2θ measurements. Take the expected Cu Kα peaks of Sapphire for example (JCPDS PDF card 42-1468):

Al2O3 (006) → 2θ = 41.685° (Cu Kα, λ = 0.1541838 nm)

Al2O3 (0012) → 2θ = 90.708° (Cu Kα, λ = 0.1541838 nm)

We can use Bragg's Law (λ = 2dsinθ) and the known Cu Kα peaks to determine what the expected Cu Kβ peaks will be:

Determination of Al2O3 (006) peak with Cu Kβ radiation:

Cu Kα, λ = 0.1541838 nm

$d_{(006)} = (\lambda_{CuK\alpha}) / (2\sin(\theta_{(006)})) = 0.1541838 / (2\sin(20.8425)) = 0.216672$ nm

Cu Kβ, λ = 0.1392218 nm

$\theta_{(006)} = \sin^{-1}((\lambda_{CuK\beta})/(2d_{(006)})) = \sin^{-1}(0.1392218 / 2(0.216672)) = 18.74°$

$2\theta = 2(18.74) = 37.48°$

Determination of Al2O3 (0012) peak with Cu Kβ radiation:

Cu Kα, λ = 0.1541838 nm

$d_{(0012)} = (\lambda_{CuK\alpha}) / (2\sin(\theta_{(0012)})) = 0.1541838 / (2\sin(45.354)) = 0.108357$ nm

Cu Kβ, λ = 0.1392218 nm

$\theta_{(0012)} = \sin^{-1}((\lambda_{CuK\beta})/(2d_{(0012)})) = \sin^{-1}(0.1392218 / 2(0.108357)) = 39.97°$

$2\theta = 2(39.97) = 79.94°$

There are two additional sapphire peaks from Cu Kα at 2θ = 37.48° and 2θ = 79.94°.