

X-ray measurements:

Shutting down:

The idle position of the system is: 15 kV, 5 mA. Furthermore the aluminum plate needs to be inserted in front of the detector and the two theta angle should be 0 degrees. If you lower the X-ray source first lower the current and then lower the acceleration voltage. If you change the two theta angle back to zero first close the shutter. After you have moved it back to zero include the aluminum plate in front of the detector. The system is never switched off.

Alignment:

1. Alignment of the sampleholder is done with an aluminum plate in front of the detector in order to reduce the count. Furthermore the X-ray source needs to be at 30 kV, 20 mA. If the X-ray source is at a lower value, first increase the voltage and then increase the current. Count should be around 500,000 if the straight-forward position.
2. For alignment we first move the sample as far back as possible (positive z) so the beam is not obstructed by the sample and can reach the detector slit without any problem. After this run a two-theta scan in the scan window: single scan, two-theta, degree, centered (around zero), range= 1 degrees, points=81, counting method = continuous, count time = 0.5 sec. Click the graph-tab and push run on the right side in the window. The system will move the detector arm, measure, and register the count in a graph. After the measurement reset motor and check the position of the peak. Write down the value, for example 0.03 degrees. Go to the control panel and move the two-theta motor to the position of the peak. Push the C of calibrate and enter zero for the current position. The two-theta is now calibrated. Realize that the resolution of the motors is 0.001 degrees so smaller difference than this do not matter. The two-theta motor should now be at the zero position and the count should be high, i.e. over 500,000. Write down the exact count.
3. Now choose the z-motor. By hand move the position of the z-motor so that the count will be half of the registered count. Now select the omega angle, go to the scan window and let the machine measure the intensity as a function of the omega angle. Of course it should be maximum at zero degrees: single scan, omega, degree, centered, range= 2 degrees, points = 81, count time =0.5. Once the scan is finished determine the peak angle. Go to the control panel, move the omega angle to the peak angle, choose the C from calibrate, and type in zero for the new angle.
4. Now double check if the count is still at half of the initial value. If not adjust the z position so that the count will be half of the initial value for the unobstructed beam.
5. Scan omega again as outlined above to check if the peak appears at 0 degrees.

Measurements:

Two-theta measurements are done at 40 kV, 40 mA. If the X-ray source is at a lower setting first increase the voltage of the X-ray source and then its current setting. Before you start the measurement make sure to first move the two-theta to a value different from zero degrees, preferable the starting value of the scan, and remove the aluminum plate that is in front of the detector. Before you open the hood, be sure to close the shutter. After the aluminum plate is removed and you are ready to close the hood, make sure that you do that carefully since a big bang will change the alignment of the setup. Go to the

control panel to set the measurement conditions: two-theta-omega, single scan, degree, scan direction is positive, minimum angle = 30 degrees, maximum angle is 80 degrees, stepsize = 0.5 degrees, 1001 points, counting time = 3 seconds. Make sure the auto-save button is checked and that the right folder is selected. Choose the sample window and update the data on the sample. Select the scan window and select run.

Interpretation of data:

After the scan is finished, use the smoothing option to remove some of the noise, and then determine the position of the peaks and possibly the width and or the surface area under the major peaks. Make a print-out of the file and use the computer labeled X-ray, to analyze the data. Analyzing means looking in an available library for experimental two-theta data measured by others. Check the film material, the substrate material, the seed layer material, possible oxides of the film material, possible oxides of the substrate material, and possible alloys of the seedlayer with the thin film material, or the seedlayer with the substrate. Realize that the exact position of the peaks might have been shifted because of stress.