SETTING UP XRF EXPERIMENT

Join active session [link to Joining a Session]. Create/add experiment [link to Project Navigator] under session folder. Go to Experiment Setup tab window and view image of loaded sample [link to Loading a Sample]. Choose experiment from the dropdown experiment list. Move sample stage [link to Moving Sample Stage] to view sample. Look for any fiducial markings you may have put on the sample in order to help you locate the sample area you want to analyse. Move the sample stage so that the feature you want to test during setup is located in the centre of the image, under the green spot marking the x-ray beam position on the sample.

Before running an experiment, you need to set or optimize the detector parameters and also determine the sample area you want to analyse. The fields for detector parameter settings are accessible within the XRF tab window. Enter a time for collection of a spectrum, in seconds, in the preset time field. Enter a value for maximum energy in keV. Click on the 'Set' button to set the newly entered values.

Click on the Start button to start the 'test' scan. The elapsed time in seconds is shown in the detector status box below, as the scan progresses. Deadtime % is also shown. If the deadtime is greater than 10% you should consider filtering (attenuating) the incoming x-ray beam. If a filter is placed in the beam or there is a change in filtering, retest the detector response. If the counts, shown in the detector status box, are too low or very high, consider changing the preset time. Inspect the collected spectrum in the spectrum window. Note that spectra can be viewed in linear or log scale by choosing the desired scale from the drop down list in the Plot Style box. If the spectrum is weak and the counts are low, retest with a higher preset time. If the counts are high and minor peaks are apparent above the background consider retesting with a smaller preset time. The collection of spectra will be faster and more efficient.

Note that spectra collected during this testing or setup phase, while in the XRF tab window, are not saved in Science Studio and will not appear in the Project Navigator tree. Spectra can, however, be saved as txt, png or xml files by clicking on the desired file format in the Save Data box. Note that you may save only the current spectrum in the Spectrum window. Once you have determined the optimum detector parameters, you may also test as many spots on your sample as you wish in order to determine the area you want to run.

You are now ready to start your spot, line or area scan. Go back to Experiment Setup window. If you wish to do a simple spot scan, make sure that the green dot indicating the x-ray beam spot position on your sample, is located over the feature you want to analyse. For a simple spot scan, also make sure that there is no red outlined rectangular box (or a red line) within the sample image view. The red rectangular box or red line indicates the position of an intended area or line scan. Also, check that the Number of Points for H and V read 1 for both, as this would be consistent with a spot scan.

For a line or area scan, check the value for the x-ray beam spot size. Enter the step scan size [link to Setting scan step size] in mm in the Step Size field. Define the line or area that you wish to scan [link to Defining a Scan Region]. As you are defining the scan region, you will see the values for Number of Points for H and V change as the size of the region is changed [increased

or decreased]. The total number of points in the defined scan region can be calculated (#pts. H x #pts. V). Knowing the preset time for XRF data collection will also allow you to estimate the time it will take to collect the scan region. Note that collection of scan point data is greater that the preset time as the collection of a scan point involves moving the stage to a new sample position, allowing the stage movement to settle and then collecting the scan with the detector, repeat, etc.

STARTING EXPERIMENT

Once the experiment has been set up, the experiment is ready to be started. To start the experiment (spot/line/area scan) click on the 'Start' button on the 'Experiment Setup' page in the 'Scan Control' window on the right hand side under the 'Scan Setup' window. Once the start has been activated, a small window will pop up requesting a scan name. Enter a scan name with some context and click on OK.

The sample stage will move so as to position the desired start point on the sample under the x-ray beam. The green spot on the microscope image of the sample within the experiment setup window, representing the x-ray beam position, will appear to move so as to coincide with the start position of the scan on the sample. Note that the system is set up to start an area scan on the bottom left most point in the rectangle drawn out on the sample, representing the area to be scanned [link to Defining a Scan Region]. The scanning proceeds from this bottom left most point horizontally to the right. The sample stage is rastered or moved from left to right, from the bottom of the scan area towards the top most row.

Collecting scans may be viewed in the XRF tab window for XRF scans, and images in the XRD window for Laue XRD scans. The scans collected during a session will appear in the project navigator tree on the left under the associated session and experiment. At the moment, scans do not appear in the tree until they have been completed [link to Viewing and Downloading scans]. Future plans include the ability to download partial scans while they are still being collected and also to view maps of the collecting scans while they are being collected.