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TM005 – Depth profiling WiRE™ 5

This document aims to show the WiRE™ 5.0 user how to perform depth profiling measurements. It assumes that the WiRE™ software is installed and that a motorised z stage is fitted and working correctly.

Depth profiles

Also known as depth scanning, z-profiling or z-scanning, this feature allows the user to 'map' their sample at one point (XY co-ordinate) with depth, or in the z axis. Depth slicing is a variation that permits Raman spectra to be collected between two XY co-ordinates (i.e. a line) and at different 'depths' within the sample. Depth profiling of a Si wafer is the traditional method for expressing the confocal performance of a microRaman instrument. Spectra are collected sequentially with the stage moving in set intervals, i.e. the laser becomes focussed at different depths within the sample. Profiles are created from the datasets. As with area and line mapping measurements, the raw data (intensity at a point, signal to baseline, and signal between two points), curve-fitted data (intensity, FWHM, area, position) or components can be used to create profiles.

Confocal performance improves as the excitation wavelength decreases and microscope objective numerical aperture (NA) increases. Typically, the best depth discrimination is obtained with blue/green lasers and a 100× objective. Of course, this assumes that fluorescence is not an issue and that the feature to be analysed below the surface lies within the working distance of the 100× objective (around 330 micrometers). Renishaw's inVia Raman microscopes operate using the Easy Confocal method, whereby the spectrograph entrance slit and CCD area combine to create a virtual pinhole. Depth profiling measurements should be performed in 'confocal' mode.

Confocal performance and depth discrimination are also improved by the use of water or oil immersion techniques. Here, a refractive index-matching liquid is used between the sample's surface and the immersion objective. This method should be used with caution if it is possible that the liquid will adversely interact with the sample. Water immersion options are extensively used in the life science application area.

Setting up the measurement

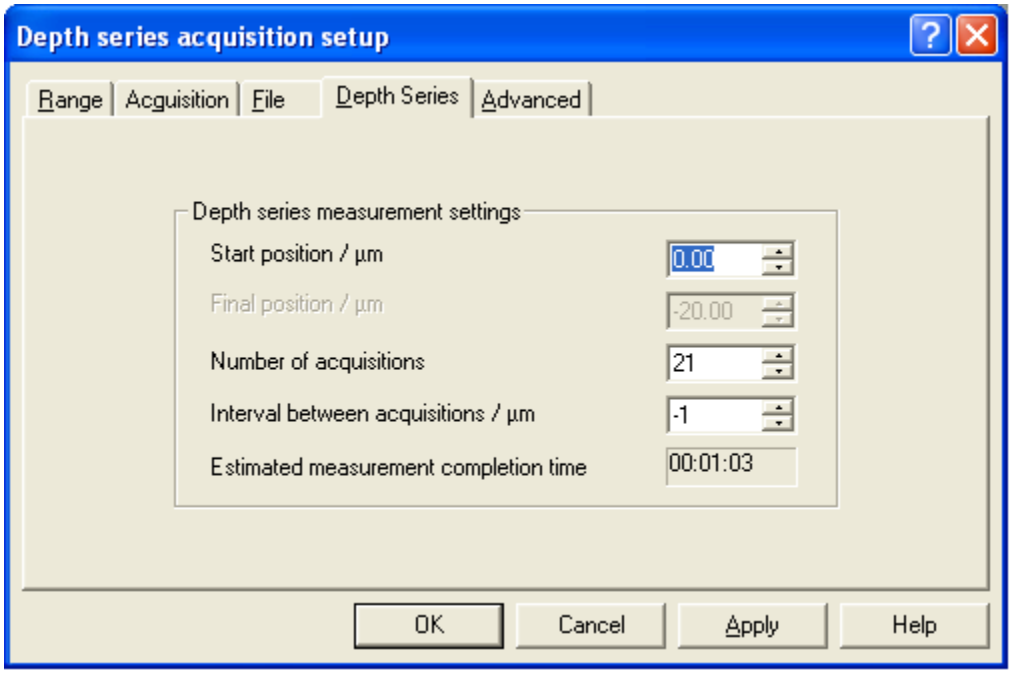
Best results are obtained from transparent samples. Samples that are coloured or absorb the laser are likely to be precluded from depth profiling or strictly limited to a very shallow depth. Since signal intensity will decrease as the laser is focused deeper into the sample, it may be a good idea to optimise the collection parameters (exposure time, accumulations, binning) at the deepest point within the sample before the depth profile measurement is performed. Check that these conditions are suitable for the surface of the sample (or point at which the sample is analysed closest to the surface), i.e. to prevent CCD saturation.

In the example that follows, a two layer polymer laminate is depth profiled.

1. With the sample's surface in focus with the 100× objective, the stage XYZ co-ordinates are set to 0 using the Set origin button

2. Check the direction of the Z travel in relation to the increasing positive or negative reading of the Z position.
3. Select **Measurement…New…Depth series acquisition**. Set the scan type and range in the Range tab and ensure that the correct Laser and Grating are set. Select the **Confocal** option; this will automatically adjust the slit width and CCD settings. In the Acquisition tab, set the laser power and number of accumulations determined from the test measurements at depth. Select a file name in the File tab if you wish to autosave the data.

4. In the Depth Series tab, set the Start position. This can be either at the surface (0 in this example) or at the maximum depth, in micrometers, within the sample. Having previously determined whether an increasing positive or negative stage movement adjusts the microscope stage up or down set the interval step size to move the stage up, effectively focussing the laser spot at successively deeper levels within the sample. The Start position, step size and number of acquisitions determine the Final position. In the example, 21 spectra will be acquired, at 1 micrometer intervals, beginning at the surface and finishing at 20 micrometers below the surface. The Estimated measurement completion time is calculated based on the number of acquisitions and the scan conditions set in the Range and Acquisition tabs.



Using the smallest interval step size (0.1 micrometer using Renishaw Z drive) allows better depth discrimination between layers. More acquisitions, however, will lead to longer total measurement times. Methods to maximise the efficiency of depth profiling include:

- Only profiling the region of interest. For example, if the interface you wish to analyse exists at 50 micrometers below the sample's surface, there may be little to be gained from ~45 spectra at 1 micrometer intervals above this interface.

- Using shorter scans. It may be unnecessary to perform extended scans when a sequence of static grating scans may be adequate to determine the changes in Raman spectra with depth.

5. Run the measurement. In the new viewer, the upper section shows the last spectrum acquired. The lower section provides information on the current position, number of spectra acquired and the updated estimated completion time.

At the end of the measurement, you can create maps using the Create maps button  .

For this polymer example, component maps (similarity of each spectrum in the dataset with a reference spectrum or spectrum from the depth profile against depth) were created (see TM15). The spectrum from the surface is least likely to have any contribution from the lower layer and the last spectrum (deepest level analysed) is least likely to have any contribution from the upper layer.

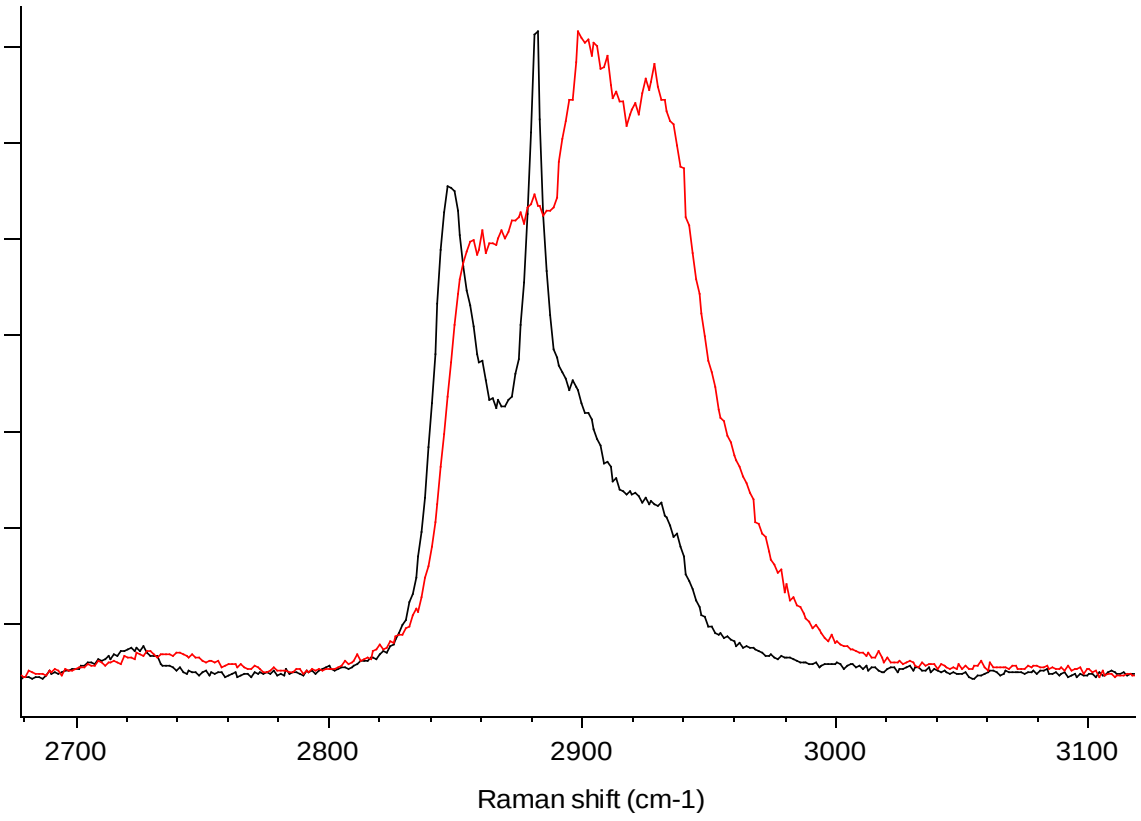


Figure 1. Representative spectra from two polymer species in a laminate sample.

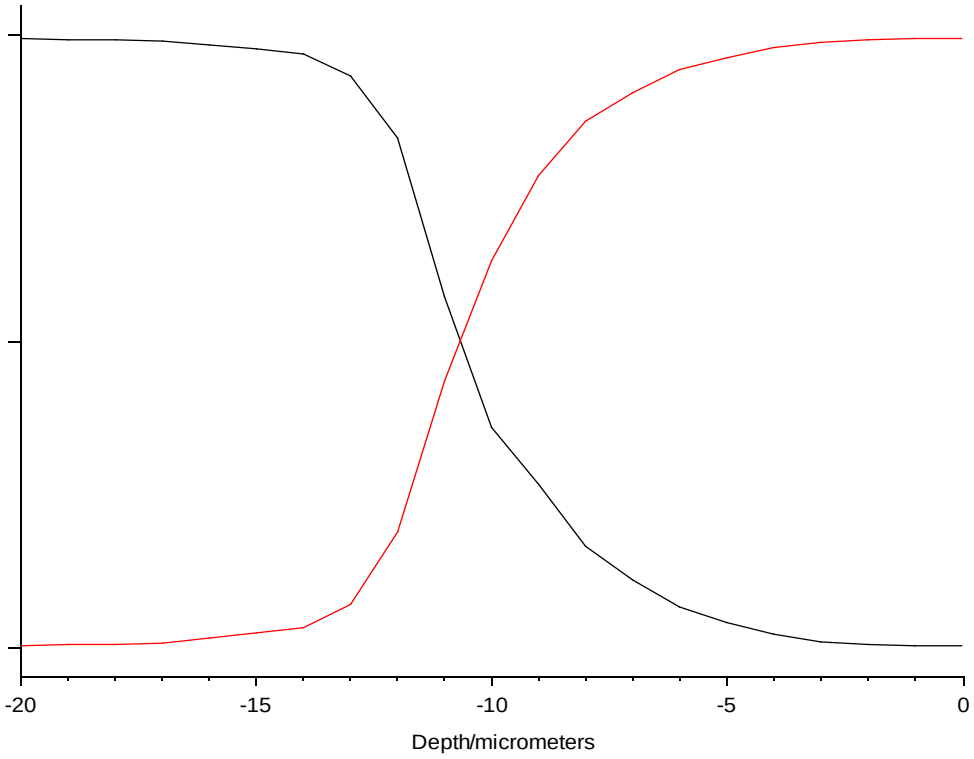


Figure 2. Overlay of component maps for a bilayer polymer laminate example. Data indicates that the upper laminate is ~10 micrometers thick.

Profiles can be saved separately. In the Navigator's Data tab, expand the branches of the file to reveal the Derived data branch which lists the maps (profiles) created. Right-click and select Save data as….You may also load the profile from here. It is possible to extract individual depth spectra from the measurement file using the Collected data branch. Select the Acquisition (expand its entry to see the depth at which it was acquired) and right-click and select Save data set as…

It is possible to view a stacked plot of the depth profile spectra from the Navigator. By right-clicking on **Collected data** and selecting **View original data in 3D** a new Image Data Viewer opens at the bottom of the screen with a 3D stacked plot.

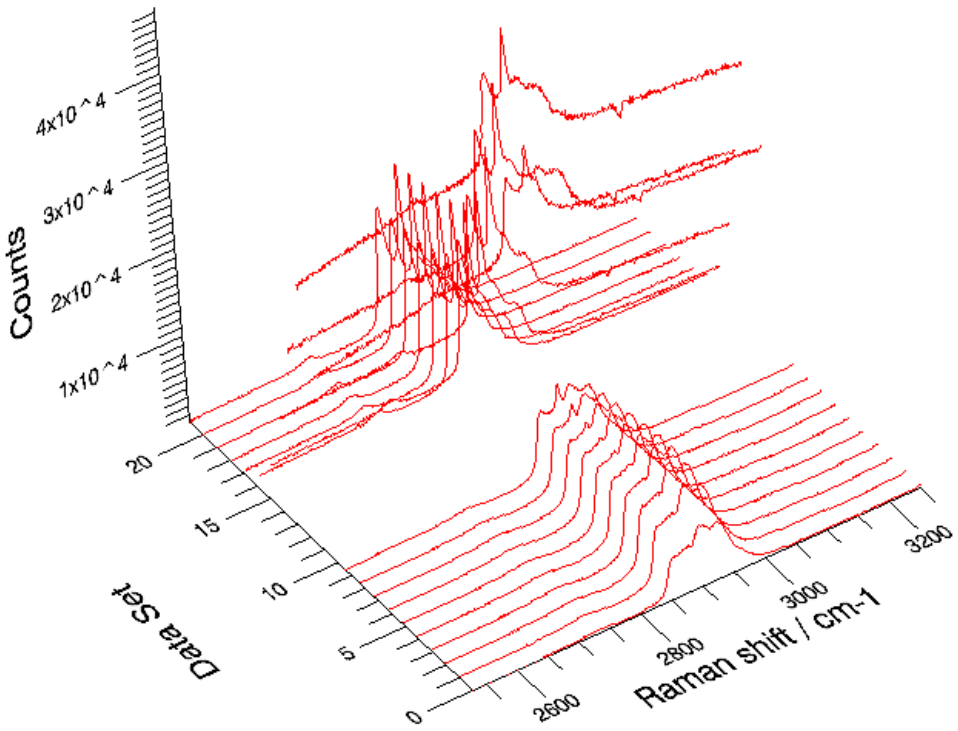


Figure 3. 3D stacked plot of bilayer polymer laminate depth profile.