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MicroHRXRD: Small Area High-Resolution X-ray Diffraction Analysis

EAG has a new highly customized XRD/XRR instrument to help our clients solve challenging problems that require the analysis of small areas or volumes.

Background

The Eurofins EAG XRD/XRR lab routinely analyzes both polycrystalline and epitaxial thin films. XRR data provides information about film density, thickness, and roughness. Typical XRD analyses include qualitative phase identification, semiquantitative phase analysis, %crystallinity, crystallite size, texture, residual stress and much more. Due to film thickness, data for many of these analyses are acquired by grazing incidence XRD (GIXRD) to maximize the X-ray signal from the film and minimize any interference from the substrate. High-resolution XRD (HRXRD) is used to measure composition and strain/relaxation of epitaxial thin films.

Due to the very shallow incidence angles used in both XRR and GIXRD data acquisition, the irradiated area is large, i.e. often more than 30mm in length on traditional XRD/XRR instrumentation. While this is generally not a problem when analyzing blanket wafers or larger cleaved pieces, it is increasingly of interest to our customers to be able to make the same measurements on small areas on device wafers or on very small samples where currently the irradiated area is much larger than the desired small sample dimensions.

Our existing microdiffractometer has a spot size as small as 25µm and a 2D area detector. This system achieves a small incident X-ray spot size by using a convergent X-ray beam. This is great for many samples, but unfortunately, a convergent beam is not appropriate for either XRR or for GIXRD and does not have sufficient energy resolution to perform HRXRD analyses. The current instrument also does not have enough energy discrimination to reduce fluorescence effects. With these limitations in mind, a search began for an instrument design that would allow us to make typical thin film analyses on samples as small as 2×2mm or at this restricted dimension within larger samples. We call these analyses microXRR, microGIXRD and microHRXRD to differentiate them from more standard work we currently do on much larger areas.

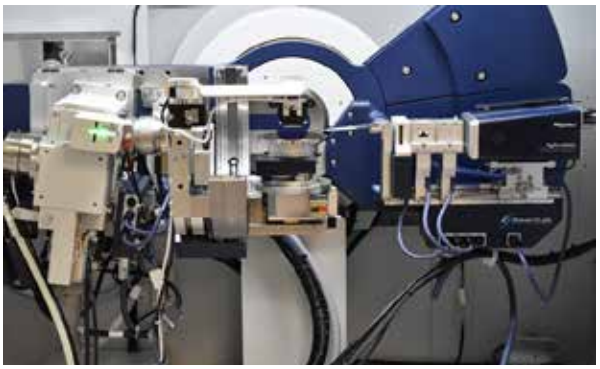

Figure 1 – Micro High Resolution System

After a lengthy evaluation, we selected a micro high resolution system (Figure 1). Working from left-to-right, this system is equipped with a high-brilliance microfocus rotating anode with a 70micron spot size. To the right of the rotating anode is a parallel confocal max-flux (CMF) mirror which produces an X-ray beam that is parallel in both meridian and equatorial directions. The CMF is compatible with both standard receiving slits for XRR and GIXRD and high-resolution monochromators for HRXRD. Multiple sample stages are available that allow full x-y-z-chi (tilt)-phi(rotation) motion used for both mapping experiments (e.g. thickness maps by XRR) and HRXRD reciprocal space maps. On the detector

side, we have standard receiving slits for XRR, parallel slit analyzers for GIXRD, and a 2-bounce monochromator for HRXRD rocking curves and reciprocal space maps. Finally, we have a 2D detector which can be operated in either 0D, 1D or 2D modes and incorporates energy discrimination to reduce fluorescence. This is a unique tool. As of this writing, there are only two instruments with this configuration in the world: this one at Eurofins EAG and the other is at the vendor's applications lab!

The purpose of this application note is to demonstrate the capabilities of this tool for microHRXRD. Other notes in this series will cover microXRR and microGIXRD applications.

MicroHRXRD analysis

Figure 2 shows the alignment picture of the HRXRD sample. Each tic mark is 1mm. The sample is a multilayer $Si_{1-x}Ge_x$ thin film on a Si substrate.

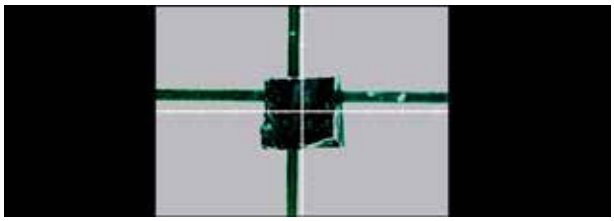

Figure 2 - Sample alignment of the SiGe thin film sample

Figure 3 shows n Omega:2-Theta rocking curve data acquired near the Si (004) substrate peak. The data shows that the $Si_{1-x}Ge_x$ thin film is made up of at least nine individual layers with differences in Ge content between each layer. Overall X-ray intensities are typically very high for symmetric reflections like this one so reasonable quality data may be acquired even on a traditional diffractometer. However, it is not possible to separate layer composition from strain/relaxation using symmetric reflection data (see HRXRD app note for details) so Reciprocal Space Maps (RSMs) on weaker asymmetric reflections are often needed. These scans, which are built up from a series of Omega:2-Theta scans taken with different omega offsets, can take a long time to acquire.

Figure 4 shows a RSM acquired on this sample near the Si (224) reflection using a conventional diffractometer. The locations and intensities of the peaks are shown in the table beneath the figure.

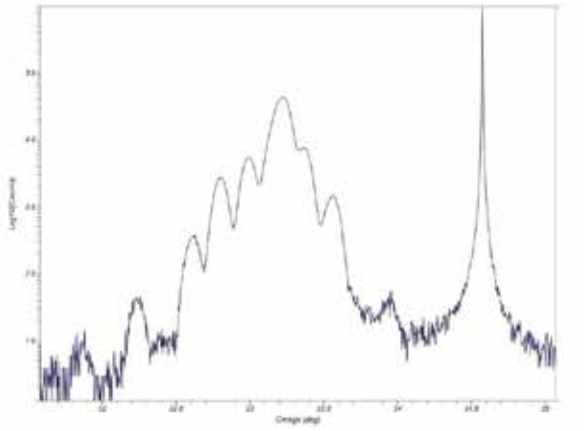

Figure 3 – High resolution Omega:2-Theta scan near the Si (004) reflection

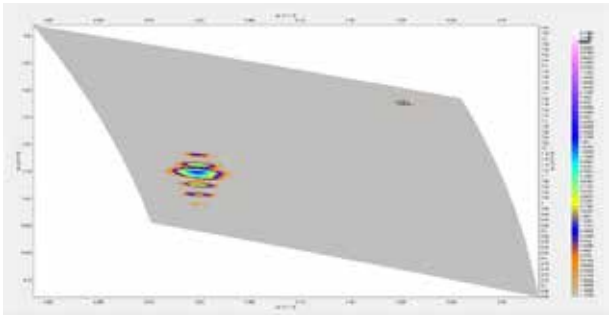

Figure 4 - Si (224) RSM acquired on a conventional diffractometer

Peak	qx (rlu)	qz (rlu)	Intensity (counts)
Sub	5.2082	7.3658	2499.5
1	5.0211	7.1767	13.2
2	5.0186	7.1406	52.2
3	5.0194	7.1131	240.5
4	5.0211	7.1011	91.3
5	5.0211	7.0684	40.5
6	5.0194	7.0323	21.5
7	5.0202	6.9963	3.5

Note that only seven of the nine layer peaks identified in the symmetric rocking curve scan were detected.

Figure 5 shows a similar scan taken using the micro high-resolution system. The table beneath the figure shows that eight of the nine layer peaks were detected with the eighth peak being the very weak one above the other layer peaks on the left side of the figure and which corresponds to the peak near

33.9 degrees in Figure 3. Peak positions agree between the two measurements within about +/- 0.02 reciprocal lattice units (rlu) which is excellent given the difficulty in finding the exact center of these fairly diffuse layer peaks.

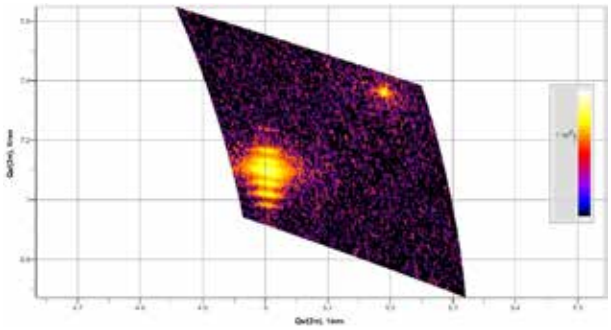

Figure 5 -Asymmetric RSM near Si (224) on the micro high-resolution system

Peak	qx (rlu)	qz (rlu)	Intensity (counts)
Substrate	5.1907	7.3598	3454.9
1	5.0036	7.2385	0.6
2	5.0037	7.1635	13.1
3	5.0048	7.1285	66.6
4	5.0022	7.1021	341.3
5	5.0049	7.0907	118.1
6	5.0053	7.0565	50.9
7	5.0037	7.0202	28.6
8	5.0030	6.9844	5.3

The intensities from the micro high-resolution system are on average 31% higher than those from the conventional diffractometer even though the time/point for the conventional diffractometer was 2.5 times longer. This means that intensity from the micro high-resolution system is actually 77% higher than the conventional diffractometer even though the conventional diffractometer's X-ray tube operates at twice the power. This is due to the high brilliance of the microfocus rotating anode. Higher intensity means that high quality data on these very small samples can be acquired much more rapidly.

Conclusion

An asymmetric RSM acquired near the Si (224) reflection on a 2x2mm multi-layer $Si_{1-x}Ge_x$ epitaxial thin film sample using our micro high-resolution XRD system has 77% higher intensity than data collected on a conventional diffractometer. This means that higher quality data may be acquired in less time on small samples or on small areas of larger samples.

At Eurofins EAG, our XRD team has collectively decades of XRD expertise. We have a collaborative group coming from different backgrounds, industries and research specialties. With our recent addition of a Small Spot XRD tool along with our extensive instrument base, we offer a wide variety of analytical options that can be tailored specifically to our clients' needs.