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EAG Laboratories

MicroXRR: Small Area X-Ray Reflectivity 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, semi-quantitative 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.

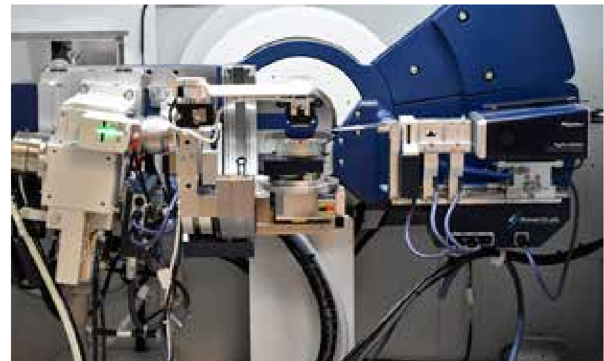

Figure 1: Micro High Resolution System

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 in Sunnyvale, CA 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 microXRR measurements. Other notes in this series will cover microGIXRD and microHRXRD applications.

MicroXRR analysis

Figure 2 shows the alignment picture of the XRR sample. Each tic mark is 1mm. The sample is a nominally 10nm HfO_2 thin film on a Si substrate. Figure 3 shows the experimental data acquired during a 35min scan and the best fitting model to the data. The model indicates that the film is actually only 4.06nm thick and has a thickness-averaged HfO_2 density of 8.87 g/cc.

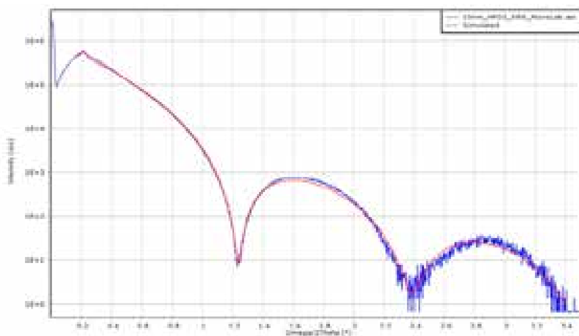

Figure 2: Experimental micro XRR data and best fitting model

Layer No.	Type	Material	Density (g/cm^3)	Thickness (nm)	Roughness (nm)
4	Single	HfO_2	8.7698	1.7041	0.4901
3	Single	HfO_2	9.6949	1.6665	0.01 Fixed
2	Single	HfO_2	7.1374	0.6905	0.01 Fixed
1	Single	SiO_2	2.7931	0.9941	0.1236
Sub.	Substrate	Si	2.328 Fixed	600000 Fixed	0.01 Fixed
GOF: 0.919					

However, the XRR results themselves are not as important as demonstrating whether the data is an improvement over what could previously be obtained on a standard diffractometer. Figure 4 compares the data acquired on the micro high resolution system in 35min with data acquired in 163min on a diffractometer equipped with a standard X-ray tube (operating at 1600watts), parallel-beam optics, a 0.3mm pinhole collimator and a scintillation detector. Intensity is plotted in counts per second to compensate for the differences in scan speed.

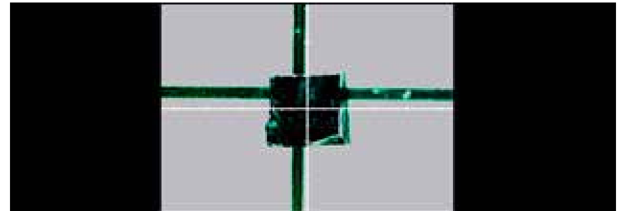

Figure 3: Alignment picture of the XRR sample

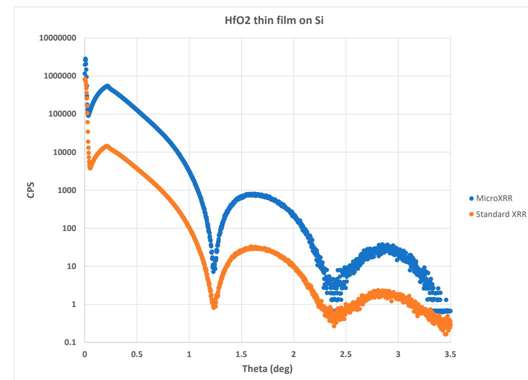

Figure 4: Comparison of XRR data from the SmartLab μHR with a conventional diffractometer

Conclusion

The results indicate that the XRR signal intensity on the micro high resolution system is at least a factor of 10 higher than on the standard instrument even though the standard tool'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 or restricted areas can be acquired much more rapidly. In addition, the small spot size is more appropriate for mapping film thickness across a whole wafer than the large irradiated areas on our standard diffractometers.

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