Grazing Incidence Angle Thin Film Analysis

Background

It is sometimes very difficult to analyze thin films due to their small diffracting volumes, which result in low diffracted intensities compared to the substrate and background. This combination of low diffracted signal and high background make it very difficult to identify the phases present. Accordingly, special techniques must be used when analyzing thin films.

The most common technique for analyzing thin films as thin as 100Å is to use a grazing incidence angle arrangement combined with a parallel beam geometry. By increasing the path length of the incident X-ray beam through the film, the intensity from the film can be increased so that conventional phase identification analysis can be run.

Applications

Thin film phase analysis	Plating
Coatings	Thin film deposition
Oxidation	Near-surface depth profiling
Corrosion products	

Grazing Incidence Geometry

Figure 1 compares the Grazing Incidence Angle geometry used for thin film with the more common θ/2θ geometry used for bulk analysis. In the conventional θ/2θ geometry, a para-focusing arrangement is used where the X-ray source and the detector slit are at the focal points of the incident and diffracted beams, respectively. For the thin film arrangement, on the other hand, the incident and diffracted beams are made nearly parallel by means of a narrow slit on the incident beam and a long Soller slit on the detector side. In addition, the stationary incident beam makes a very small angle with the sample surface (typically 1° to 3°), which increases the path length of the X-ray beam through the film. This helps to increase the diffracted intensity, while at the same time, reduces the diffracted intensity from the substrate. Overall, there is a dramatic increase in the film signal to the background ratio. Since the path length increases when the grazing incidence angle is used, the diffracting volume increases proportionally. This is the reason for the increased signal strength.

During the collection of the diffraction spectrum, only the detector rotates through the angular range, thus keeping the incident angle, the beam path length, and the irradiated area constant. The long Soller slit on the receiving side allows only those beams that are nearly parallel to arrive at the detector. This has an added advantage of reducing sensitivity to sample displacement from the rotation axis.

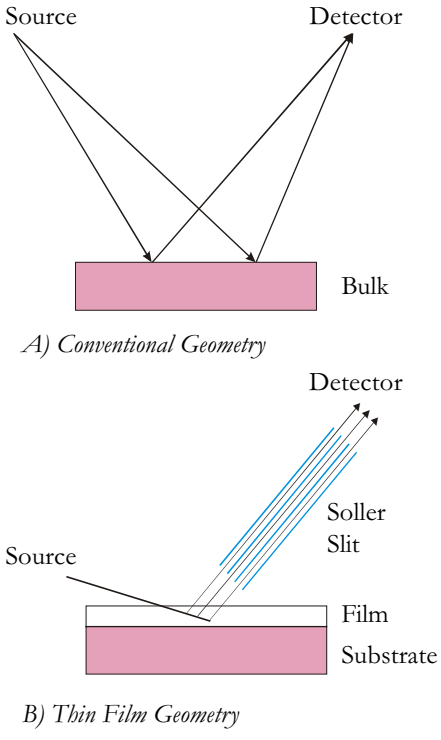

Figure 1 Geometries for *A*) bulk analysis and *B*) thin film analysis showing differences in the beam path lengths.

Example

Figure 2 illustrates the much improved signal strength from a thin film of TiO_2 deposited onto a glass substrate when the spectrum is collected with a thin film geometry compared with the conventional Q/2Q geometry. With the conventional geometry, the Rutile form of TiO_2 can be seen clearly, but it would be hard to confirm the presence of the Anatase form since its strongest peak coincides with the amorphous hump arising from the glass substrate. But when the thin film geometry is used with an incidence angle of 1.5°, both forms of TiO_2 are now evident. At this point, conventional XRD analysis methods can be used to identify the phase, determine the relative amounts of each phase (30% Anatase & 70% Rutile), and their grain sizes (26-28nm). Without the glancing angle geometry, these analyses would not have been possible.

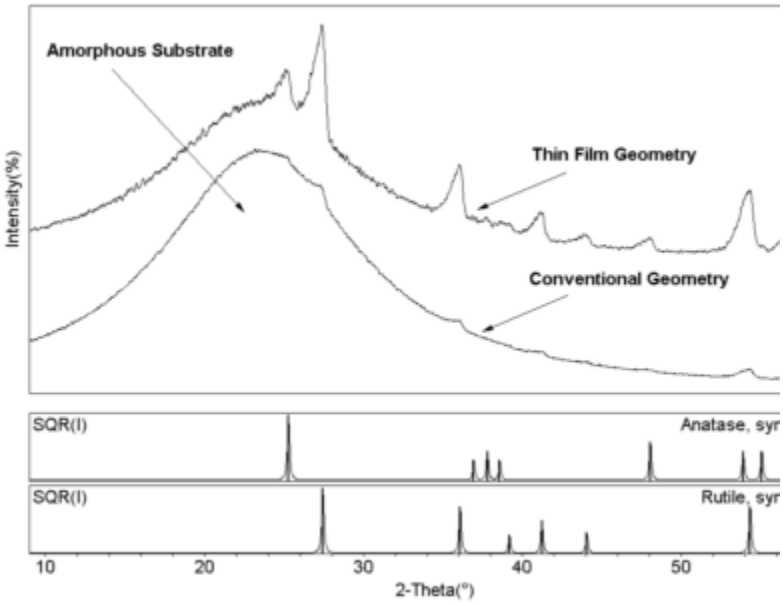

Figure 2 Comparison of XRD spectra of TiO2 on a glass substrate taken with a) Conventional Bragg-Bretano geometry, and b) Grazing Incidence geometry

Applicability

For films in the range of 100Å to 2000Å, glancing incidence analyses should be used. The lower detection limit is strongly dependent on the film chemistry, so the 100Å is only suggestive of the method's limitations under optimal conditions. Finally, if the film has a high degree of preferred orientation (such as an epitaxial film), the method is not suitable.

Our Pledge

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- crystal orientation
- grazing incidence angle
- retained austenite analysis

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