Coherent Grazing Exit X-ray Scattering Geometry for Probing the Structure of Thin Films

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Introduction

Evaporated metallic thin films contain a hierarchy of grain sizes distributed over the depth of the film. Because conventional x-ray scattering methods are not sensitive to the depth within the film, powerful methods based on the refraction of the beam near the critical angle for total external reflection α_c have been introduced [1]. When an x-ray beam impinges on a surface with its incidence angle α_i below α_c, it reflects totally and creates an evanescent wave inside the sample with a depth that can be varied from a few nanometers to a few hundred nanometers.

Reciprocity ensures that the same refraction behavior applies to a beam emerging from the sample at an angle of exit α_f near α_c. Using both α_i and α_f near α_c is a method for obtaining even stronger surface sensitivity. Here we demonstrate that the use of a beam with $\alpha_i > \alpha_c$ allows sufficient penetration to probe the full depth of a thin film yet, by analysis of the angular distribution in α_f still allows depth profiling of its internal microstructure. We show that the surface contributions result in a strong enough signal that coherence effects can be exploited to further enhance understanding.

Coherent diffraction from a granular microstructure results in a speckled modulation of what would otherwise be diffuse scattering with an overall width that is inversely related to the grain size. The size of the speckles is inversely related to the beam size but also includes the effects of refraction of the incident or exit beams. The speckles also provide information about the specific distribution of grains under illumination, which can be employed for detailed characterization of the sample.

Methods and Materials

Our experiments were carried out at undulator beamline station 34-ID-C at the APS by using monochromatic radiation of 8.92 keV. Coherence was achieved by the use of two roller-blade slits that cut down the beam to a typical size of 10 μm horizontally and vertically, to approximately match the transverse coherence of the source. The sample, oriented horizontally, was illuminated at an angle of α_i, as illustrated in Fig. 1. A direct illumination charge-coupled device (CCD) x-ray camera was placed on the detector arm of the diffractometer. The measurements described here were all made in the forward-scattering direction, below the reflected beam, with the CCD subtending an angular range $0 < \alpha_f < \alpha_c$ vertically, as shown. Two samples were used. The first was a 1000-Å evaporated film of gold on a quartz substrate. The second was an iron film of 940 Å, evaporated in the ultrahigh vacuum (UHV) sample environment of the 34-ID-C beamline station.

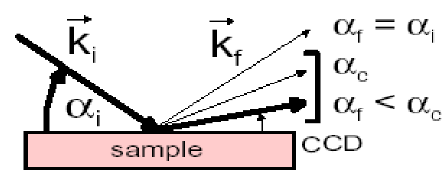

FIG. 1. Sketch (side view) of the geometry used in the experiment. The out-of-plane directions, which are referred to as the x-coordinate or ϕ-angle, lie perpendicular to the drawing plane and are not shown here.

Results and Discussion

Three general components to the scattering from an inhomogeneous sample with a well-defined surface are expected under these conditions. A specular beam, with the same shape and propagation properties as the incident beam, is expected, with an intensity given by the Fresnel law but reduced by any surface roughness present. A "ridge" of off-specular scattering, but lying within the scattering plane, is usually attributed to the missing specular intensity due to the roughness [2]. The extended ridge shape is due to the extreme grazing geometry. Finally, there is a generally diffuse scattering coming from the distribution of bulk grains. Both of these diffuse components acquire a speckled appearance when a coherent beam is used [3]. The intensity of both diffuse components becomes enhanced when $\alpha_f \cong \alpha_c$ by the electric-field transmission function of the interface [4] to produce a so-called "Yoneda" peak. All three of these features are seen in the data in Figs. 2 and 3, with the general (doubly off-specular) diffuse scattering taking a striking "triangular" shape.

The new geometry described here will find important applications in studies of the coalescence and maturation of thin films during growth, which are not readily accessible by any other technique.

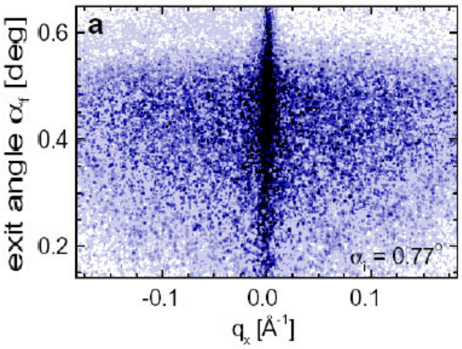

FIG. 2. Diffraction observed for the Au film sample. Linear contour plots of the measured intensity distribution in the detector plane (CCD) as a function of the lateral momentum transfer q_x and the exit angle α_f for fixed angles of incidence $\alpha_i = 0.77°$.

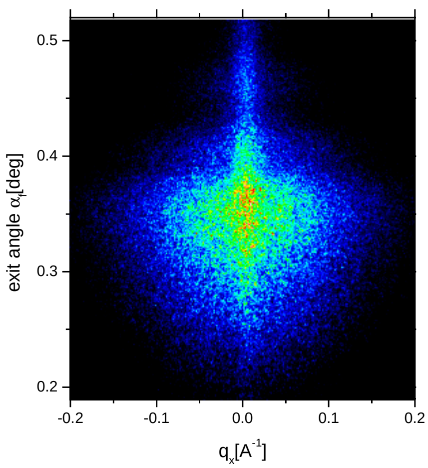

FIG 3. Diffraction observed for the Fe film sample. Linear contour plots of the measured intensity distribution in the CCD as a function of q_x and α_f for fixed angle of incidence $\alpha_i = 0.66°$. Thickness fringes are visible above the critical angle, $\alpha_f \cong \alpha_c$.

Acknowledgments

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