A Novel Sample Preparation Method for Thin-Film Transmission SAXS Measurements

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Introduction

Small-angle x-ray scattering (SAXS) is widely used to determine the size distribution of nanocrystals formed within a variety of matrices including SiO_2 [1-4]. This oxide is typically grown on a Si substrate for compatibility with electronic and photonic device fabrication and SAXS measurements are thus commonly performed in a grazing-incidence geometry [5-7]. Transmission measurements are also possible with the appropriate photon energy and flux to penetrate the thick Si substrate though scattering and diffraction from the latter can degrade image quality. For the present report, we have developed a novel sample preparation method for transmission SAXS measurements to determine the size distribution of metallic nanocrystals in SiO_2.

Methods and Materials

Nanocrystals of Cu, Pt and Au with an average diameter of 3-5 nm were first formed in a thin SiO_2 layer (~2 μm) on a Si substrate (~500 μm) by ion implantation and thermal annealing. 300 μm of Si was then removed from the underlying substrate by mechanical grinding. A concavity of 180 μm depth was then formed using a dimple grinder normally utilized for transmission electron microscopy sample preparation. Finally, as shown in Figure 1, a hole of diameter ~1 mm was opened in the remaining Si below the nanocrystal-containing SiO_2 layer using selective chemical etching (KOH/H_2O).

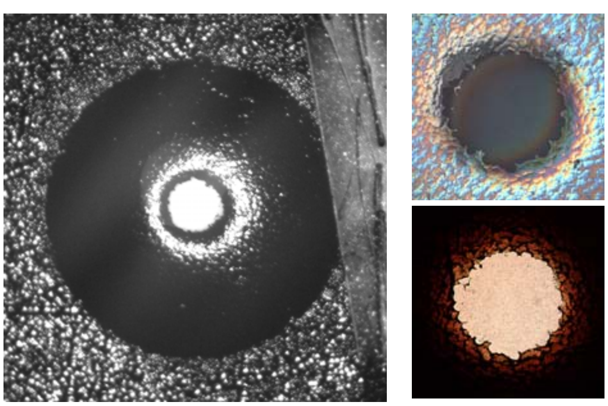

Fig. 1: Optical micrographs of (left) an entire sample with the hole visible in the center and (right) higher magnification views of the hole in both reflection (top) and transmission (bottom) geometries.

SAXS measurements were performed at the ChemMat-CARS beamline (15ID-D) of the Advanced Photon Source, USA, using x-rays of wavelength 1.09 Å. The scattered intensity I(Q) was measured with a Bruker 6000 CCD detector with 5 s exposures and a camera length of 1881 mm.

Results

Using the method described previously, we were able to prepare self-supporting thin-film samples ideal for transmission SAXS measurements. As an example, Figure 2 compares the SAXS intensities as a function of the scattering vector Q for an isolated SiO_2 layer with and without Pt nanocrystals.

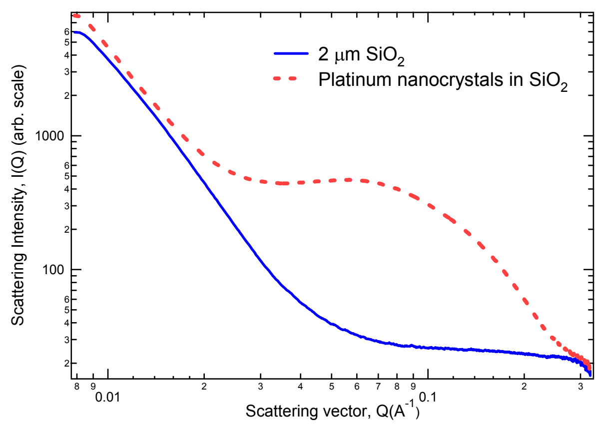

Fig. 2: SAXS data of a Pt nanocrystal-containing SiO_2 layer and the scattering contribution from a SiO_2 layer only.

Discussion

As apparent from Figure 2, the scattering contribution from the SiO_2 layer can be easily isolated and quantified, enabling an absolute determination of the Pt nanocrystal size distribution. Precision alignment for grazing-incidence geometry is no longer required and scattering and diffraction from the underlying Si substrate are eliminated. Furthermore, given our ability to form nanocrystals with oriented, non-spherical shapes (such as ellipsoids with the major axis aligned along the ion-beam direction), this sample preparation method will also enable incident-angle-dependent SAXS measurements to better extract both the nanocrystal size and shape distributions.

References

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