

Grazing incidence X-ray diffraction study of meso-structured silica thin films

Takashi NOMA*¹, Taihei MUKAIDE¹, Kazuhiro TAKADA¹, Hirokatsu MIYATA¹, Atsuo IIDA²
¹Canon Research Center
² KEK-PF, Tsukuba, Ibaraki 305-0801, Japan

Introduction

Self-assembled surfactant-templated silica thin films exhibit two-dimensional (2D) hexagonal, three-dimensional (3D) hexagonal, or 3D cubic mesostructures. A synchrotron x-ray microbeam has been utilized for analysis of these materials. The diffraction patterns of silica thin films have extra spots that are observed under the grazing incidence condition.¹ In this study we observed the incidence-angle dependences of the extra spots position.

Experimental

The x-ray diffraction experiments were performed at the Photon Factory on beam line 4A. Synchrotron X rays were monochromated with a W-Si multilayer monochromator. X rays tuned at 8 keV were used. Using the Kirkpatrick-Baez system consisting of a pair of elliptical mirrors formed the x-ray microbeam. The mirrors were made of platinum-coated fused quartz. The beam size was about $4 \times 4 \mu\text{m}^2$ at the sample position. The samples were mounted on XZ translation stages that were mounted on a ω -rotation stage with a vertical rotation axis and a ϕ -rotation stage with a horizontal rotation axis. The incidence angle of X-ray microbeam was regulated by the ω axis. A CCD X-ray detector with image intensifier was placed at a distance of 250mm from the sample to record the diffraction patterns.

Continuous silica thin films with highly oriented mesostructure were obtained on a silica glass substrate using a rubbing-treated thin polyimide coating on the substrate. The silica film was formed through a slow epitaxial growth of a self-organized mixture of hydrolyzed silicon alkoxide and the surfactant under acid conditions.

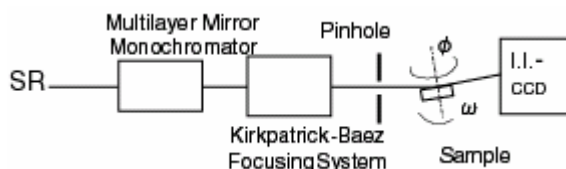


Fig. 1

Results and Discussion

An X-ray diffraction pattern measured under the grazing incidence condition ($\omega=0.2\text{deg}$) is shown in Fig.2(a). This sample has a well-ordered 2D hexagonal structure. The extra spots are observed near the main spots in Fig.2(a). The incidence angle dependence of a diffraction spot is shown in Fig.2(b). Figure 3 exhibits the intensity profiles of the diffraction spot at various incidence angles. The distances between the main spots and the extra spots increase with an increase of the incidence angle.

These results suggest that the x-ray beam reflected at the boundary of the silica film and the substrate.

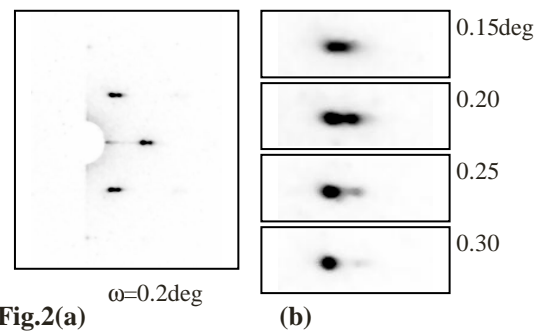


Fig.2(a)

(b)

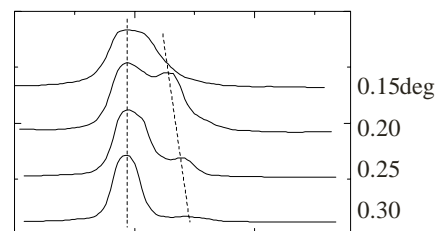


Fig.3

References

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 * noma.takashi@canon.co.jp