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**X-ray Specular Scattering Study of C32 Thin Films and Bulk Particles Adsorbed on the Si(100) Surface**

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Beamlines: X18A

**Introduction:** Films of intermediate-length alkanes such as dotriacontane (*n*-$C32H_{66}$ or C32) are of interest as prototypes for more complex polymer films at solid interfaces. They are also of technological interest for their lubricating properties. We have investigated the structure and growth of C32 films on $SiO_2$/Si(100) substrates using x-ray specular reflectivity measurements. Here we report how the structure of three different films depends on the age of the solution from which they were deposited.

**Methods and Materials**: Commercial Si(100) wafers were cleaned in an $H_2O_2$ + sulfuric acid mixture [1]. The C32 films were deposited by dip-coating the wafers in a solution of C32 in heptane (*n*-$C_7H_{16}$) [2]. All samples were examined by VHRE to determine the C32 film thickness before the x-ray measurements. The method used for collecting x-ray data was similar to that in Ref. 1.

**Results**: Figure 1 shows x-ray reflectivity scans for the three "acid cleaned" samples. The data for sample Ar42 was collected at the MUCAT sector of the Advanced Photon Source, and data for the other two samples were collected at beamline X-18A at the NSLS. The reflectivity curve for sample Ar42 clearly shows Kiessig fringes corresponding to a film thickness of about 54 Å. The detailed structure of this film has been discussed elsewhere [3]. For samples Ar02 and Aps10, scattering from bulk particles on top of the film significantly reduced the amplitude of the Kiessig fringes. However, one can still see several minima in the Kiessig fringes at low wave vector transfer *Q* by comparing their reflectivity scans with that of sample Ar42. Sample Ar42 was made out of a fresh solution of C32 in heptane. Interestingly, sample Aps10 was deposited from the same solution but nine months later and had a thickness 20 Å greater than Ar42 as determined by ellipsometry. The third sample Ar02 was deposited from a different solution that was three months old. These results indicate that the older solutions yield C32 films having a larger and less uniform thickness. The older solutions also favor the nucleation of bulk particles as evidenced by the Bragg peaks marked by arrows in Fig. 1. Two bulk phases are present: a recently discovered surface-stabilized orthorhombic structure [3] and the previously known "free standing" monoclinic phase for which the Bragg peak intensities are much weaker. Both phases grow preferentially with their *ab* basal planes parallel to the film plane [3].

**Conclusions**: The measured specular x-ray reflectivity curves indicate that the structure of C32 films deposited from a solution of C32 in heptane depends strongly on the age of the solution. The older solutions yield thicker films as measured ellipsometrically, which may be due to the evaporation of some heptane from the solution. In addition, the films grown from the older solutions are less uniform in thickness (weaker Kiessig fringes) and favor the nucleation of bulk C32 particles on top of the film.

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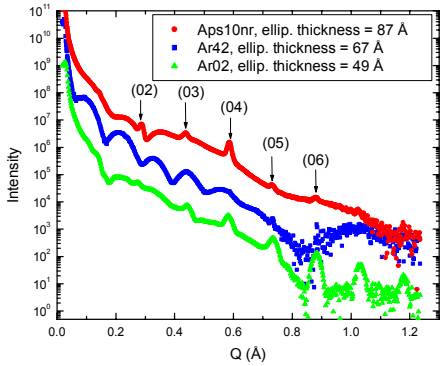


**Figure 1**. Specular x-ray reflectivity of the three samples. The data for samples Ar02 and APS10 were multiplied by a constant factor to separate the curves from each other.