X-ray Specular Reflectivity Study of C32 Thin Films Adsorbed on the Si(100) Surface

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Introduction: The wetting and growth of thin dotriacontane [n-$C_{32}H_{66}$ or C32] films adsorbed on Si(100) substrates have been studied by x-ray specular reflectivity and high-resolution ellipsometry.[1] The C32 films are of interest from a fundamental standpoint in that they serve as prototypes for the structure of more complex polymer films at solid interfaces. They are also of considerable technological interest in their own right, since intermediate-length alkanes are the principal constituents of commercial lubricants.

Methods and Materials: C32 films were prepared by dipping the Si(100) substrates, cut from commercial wafers, into a solution of C32 in heptane (C7) and allowing the C7 to evaporate. All the samples were examined using high-resolution ellipsometry to determine the C32 film thickness prior to the x-ray experiments. The measured specular reflectivity scans were fit to a three-slab model consisting of a semi-infinite Si substrate, a slab of SiO_2, and a slab of C32.[2]

Results: Figures 1 and 2 show the measured x-ray specular reflectivity as function of Q for two samples at room temperature after normalizing to the Fresnel reflectivity. The C32 films of both samples were previously measured by ellipsometry to have a thickness of ~97 Å. Fits to the period of the Kiessig fringes observed in our first measurement of Sample 1 (blue curve in Fig.1) also yielded a thickness of ~97 Å. This is about twice that expected from earlier x-ray specular reflectivity measurements by Merkl *et al*.[3] on intermediate-length alkanes adsorbed on Si(100) substrates. These authors inferred adsorption of only a single solid layer in which the molecules were oriented with their long axis perpendicular to the surface. The alkane monolayer thicknesses were approximately equal to the all-*trans* length of the molecule which for C32 would be ~44 Å. After a heating/cooling cycle to 346 K for 1.5 h (the C32 film melting point is 345 K as determined ellipsometrically), we remeasured the reflectivity of Sample 1 at room temperature and observed fewer Kiessig fringes (red curve in Fig. 1) indicating a rougher alkane/air interface. Also, fits to the fringe period indicated a decrease in film thickness to ~58 Å but still larger than the all-*trans* length of the molecule. Sample 2 (Fig. 2) was annealed twice during the ellipsometry experiment: first to just below the film's melting point and then to 346 K. The fewer number of Kiessig fringes again indicates a rougher C32 film than found initially for Sample 1, while a best fit of the reflectivity curve to the three-slab model gives a thickness of only ~37 Å.

Conclusions: The measured specular x-ray reflectivity curves indicate that it is possible to grow thicker C32 films than previously observed[3] but that the film thickness and roughness depend sensitively on the heat treatment of the films.

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References:

1. U. G. Volkmann, M. Pino, and H. Taub, Bull. Am. Phys. Soc. **45**, 996 (2000); to be published.
2. I. M. Tidswell *et al*., Phys. Rev. B **41**, 1111 (1990).
3. C. Merkl, T. Pfohl, and H. Riegler, Phys. Rev. Lett. **79**, 4625 (1997).

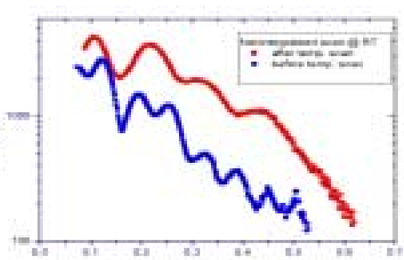

Figure 1

Specular X-ray reflectivity of Sample 1 at room temperature before and after being subjected to a heating/cooling cycle to 346 K.

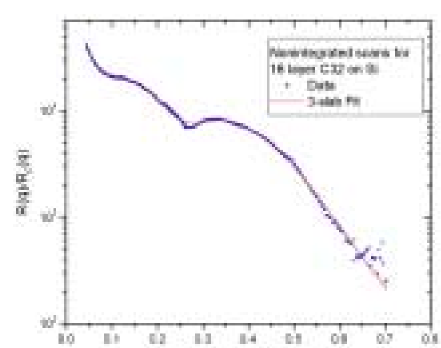

Figure 2

Specular X-ray reflectivity of Sample 2 at room temperature after two annealing cycles.