Liquid Confined between Two Solid Substrates: An X-ray Reflectivity Study

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Introduction

The behavior of nanoscopically confined liquids between two solid substrates has been studied extensively in recent years to help understand the role of confinement in lubrication, adhesion, catalytic reactions at surfaces, etc. [1-5]. Although there has been significant progress, many important questions are still left unanswered, in large part because of the relatively slow development of direct probes of confined liquid structure. Only one paper reporting a few experimental results was published last year [5].

We report here on our first reflectivity data from tetrakis(2-ethylhexoxy)silane (TEHOS) confined between two smooth silicon substrates. TEHOS is a roughly spherical, nonpolar, nonconducting, nonentangling van der Waals liquid. Our group has previously found interesting static and dynamic structural behavior of TEHOS near a single solid substrate [6-8]. These findings and the techniques that we have developed are believed to be relevant to the behaviors of confined liquids between two solid substrates.

Methods and Materials

We cleaned 100 silicon substrates from Silicon Processing Company at a thickness of $5 \times 25 \times 2.5$ mm in a strong oxidizer (sulfuric acid and 30% hydrogen peroxide in a volume ratio of 7:3) between 80° and 90^oC for 30 minutes. They were then rinsed in pure water many times and left in pure water before use. For each experiment, two silicon pieces were dried under fast nitrogen flow, and a small amount of TEHOS was placed between the two polished silicon surfaces. We aligned the 5-mm side in the direction of the x-ray beam to reduce background scattering from the silicon substrates.

X-ray reflectivity measurements were made at the MR-CAT sector 10 beamline at the APS. The x-ray energy was 27 keV and the incident beam size was 0.2 mm vertically and 2 mm horizontally. We attempted to make nanoscopically thick TEHOS films by shearing the liquid (i.e., by rubbing the two silicon pieces against each other).

Results and Discussion

Figure 1 shows reflectivity data taken from four different locations from a sample containing TEHOS confined between smooth silicon substrates.

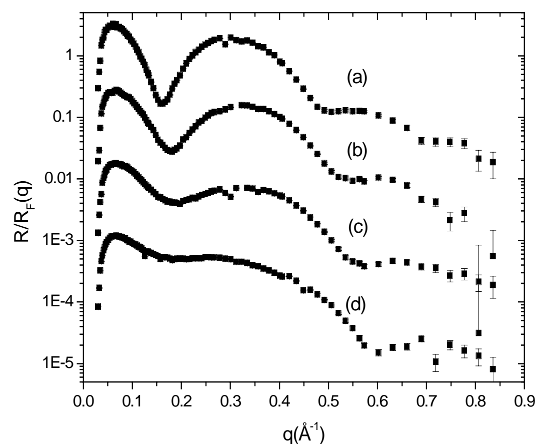

FIG. 1. X-ray reflectivity data from TEHOS confined between two smooth silicon surfaces. The data are shifted vertically for clarity.

These data show that a nanoscopically thick liquid film can be obtained by the simple method of rubbing the two substrates against each other. The two minima near 0.16 and 0.5 in Fig. 1(a) imply two different thicknesses, 40 and 25 Å. The other data from different regions shown in Fig. 1(b-d) show minima in the same positions, so this result is not caused by different liquid film thicknesses in different regions within the x-ray footprint. The large "hump" in some scans may be a diffuse diffraction peak. We have not obtained a full interpretation of the data, and further studies are underway.

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