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Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

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- 1st March for experiments carried out up until June of the previous year;
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Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** X-ray scattering experiments of thin liquid films in confined geometry (SFA)	**Experiment number**: Si 1013
Beamline: 10C	**Date of experiment**: from: 26.3.04 to: 6.4.04	**Date of report**: 2.Sept.2004
Shifts: 18	**Local contact(s)**: Federico Zontone	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The mechanical properties of ultrathin lubricants layers are of crucial importance to the function and life time of microelectromechanical systems (MEMS). Without suitable lubrication they fail quickly due to excessive friction and wear. Both experiments and numerical simulations showed that thin film friction can deviate strongly from the extrapolated behavior of the bulk liquid when the thickness reaches molecular dimensions. For instance, it is well established that the molecules of sufficiently simple liquids arrange in layers parallel to the solid liquid interfaces if confined between two atomically smooth surfaces at a separation of few molecular diameters. Experimental evidence for this so-called 'layering' of liquids was first found in the Surface Forces Apparatus (SFA), in which two single cristalline mica surfaces in crossed cylinder geometry are approached with the liquid of interest confined in between. The thickness of the liquid layer is measured with Angstrom resolution by multiple beam interferometry using visible light reflected from Ag mirrors that were evaporated onto the back sides of the mica sheets. While the correlation between the layered structure of the liquid and the (oscillatory) normal force profile is well established, the in-plane structure of the confined liquid, which is of crucial importance for the shear properties, is largely unknown. The main challenge in order to resolve this question is to obtain structural information from an extremely small scattering volume. In the present experiment, we demonstrated that scattering data can indeed be obtained for buried lubricant layers thinner than 10nm with a diameter of the probe volume of only ≈ 20 µm.

Originally, we planned to investigate the silicone oil OMCTS. It turned out, however, that either the scattering intensity was too low and/or that radiation damage prevented prevented the acquistion of sufficiently strong scattering signal. Therefore, we investigated liquid crystalline 8CB in its smectic A phase. 8CB has the advantage of being a well characterized system with a rich phase behavior. The molecules form roughly rod-like dimers, which align frequently with specific directions on substrates (anchoring). It was confined between two mica sheets (with controlled orientation of the atomic lattices) inside an SFA setup specifically built to perform x-ray scattering experiments and optical interference microscopy simultaneously. The SFA was mounted on a micro diffraction stage inside a vacuum chamber in order to reduce air scattering. The x-ray beam was transmitted perpendiculary through liquid film and the confining surfaces, i.e. two mica substrates (thickness d≈ 5µm + 50nm Ag mirrors), two glue layers (d ≈ 20µm), and two glass slides (d = 80µm). Diffraction peaks of the smectic phase (layer spacing: 32Å) are well separated in q-space from the substrate induced scattering signal. Due to the small lateral dimensions of the sample (<100µm), the x-ray beam had to be focused. 29 Compound Refractive Lenses (CRL; Beryllium) were used to produce a focused beam with a full width at half maximum of 29.4µm (vertically) by 19.3µm (horizontally) at the sample position. In order to increase the photon flux a pink beam (13.1 keV) instead of a monochromatic beam was used. Low energy contributions with energies of 8keV and 10.3keV were attenuated by a 10µm Co foil in front of

the CRLs to <5% and to 19%, respectively, of the intensity at 13.1keV. The x-ray scattering signal was detected with a two-dimensional CCD detector (Princeton). The SFA was positioned relative to the x-ray beam with two motorized translation stages using a careful alignment strategy involving knife edge scans with both the x-ray beam and the visible light.

Fig. 1 shows an optical image of the sample recorded with monochromatic illumination. Sharp Newton fringes, corresponding to a single domain of perfectly ordered 8CB, appear centered around the roughly circular contact area with a diameter of ≈ 150µm. (Dark spots in the image correspond to beam damage in the Ag layers on the back sides of the mica sheets. While the sample is not noticably affected, this effect demonstrates first the success of the alignment procedure and second the fact that the film thickness varies only little across the small diameter of the x-ray beam.) Note that the fringes become blurred approximately 10 to 15 fringes away from the contact area, corresponding to a film thickness of approx. 2µm and above. Beyond that film thickness, several liquid crystalline domains are present between the two surfaces and disturb the optical image due to birefringence. This result is also recovered in the x-ray scattering data (Fig. 2): The data were recorded at three different locations, as indicated in Fig. 1 with decreasing film thickness from left to right. At d=3.87µm, there are two azimuthal domains present, whereas the two peaks begin to merge at d=1.92µm, which is also the range where the Newton fringes become sharp. (Note that each maximum is seen twice along the radial direction, with the inner smectic peak corresponding the 13.1keV and the outer one to 10.3keV.) Closer to the contact area, only one azimuthal domain is observed (data not shown) with the integrated intensity decreasing linearly with film thickness. Even at the smallest film thickness (8.4nm), there is still finite scattering signal. This is a factor of 50 thinner than for the thinnest layers investigated before [1]. From the size of the molecules and the scattering volume we deduce that this signal originates from merely 2-4x10^9 8CB dimers.

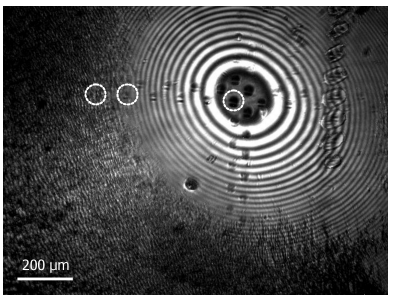

Fig. 1: Interference microscopy image (l=497nm). Scale bar: 200µm

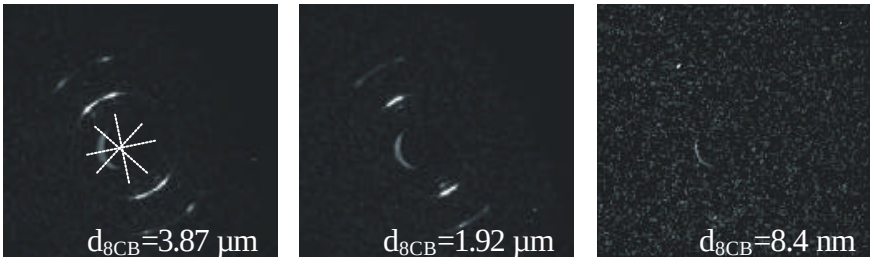

Fig. 2: Scattering signal obtained at the locations indicated in Fig. 1. The solid and dashed lines in left image indicate the orientations of the mica optical axes of the top and bottom surfaces, respectively, as determined optically.

Fig. 3 shows the azimuthal intensity distribution for d = 2.82µm. There are two peaks separated by a plateau. The orientations of the peaks correspond to the directions of the optical axes of the two mica substrates, which were rotated by 37° with respect to each other (see Fig. 2). Obviously, the director of the liquid crystal is preferentially aligned along the optical axes of mica. To explain the profile in Fig. 3, we propose the following preliminary model: The director of the liquid crystal is equal to the anchoring direction (i.e. 104° or 131°, respectively) within a certain range of each surface. In between, there is a layer where the director orientation changes linearly from one orientation to the other [2].

In summary, these results demonstrate that our technique is well suited to study the structure of molecularly thin liquid films. The measurements indicate that the phase diagram for 8CB should not only depend on the confinement but also on the relative orientation of the substrate lattices.

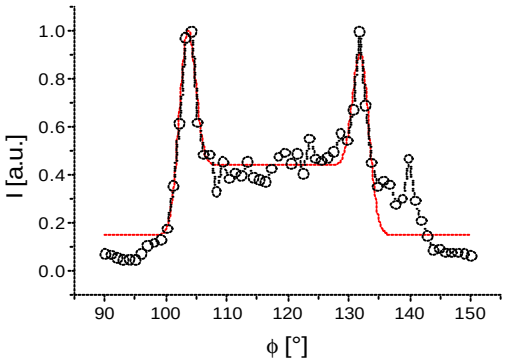

Fig. 3 Scattered intensity vs. azimuthal angle.

[1] S.H.J. Idziak et al., Science **264**, 1915 (1994).
[2] F. Mugele et al., in preparation