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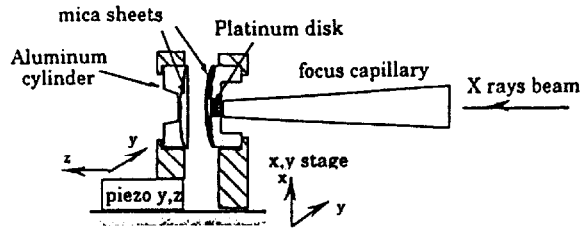
Report A first step towards the coupling of a Surface Force Apparatus (SFA)[1] with x-ray solution scattering has been started. The long term project is to provide an ***in situ*** structural information on the structure and orientation of the confined system between two surfaces or in their vicinity. Capturing such an information will enable us to relate the force-distance profile as measured in a SFA with the structure for a given state of the system.

The crossed-cylinder geometry, on which the surface force apparatus technique is based, has been adapted for allowing the X-ray beam to irradiate the confined sample between the two surfaces. Thin molecularly smooth mica sheets (thickness l-3 $\mu$m), as traditionally used in a SFA, were glued down on two polished Aluminum cylinders (radius of curvature about 2 cm), The axes of the cylinders were then crossed upon mounting on holders. The separation between the two surfaces could be varied (from tens of micrometers down to mechanical contact) with a piezo element superimposed on the coarse positioning range of a micrometer. For a given separation, both surfaces could be translated jointly in the vertical plane by means of a (x,y) stepper-motor driven translation stage (step resolution > 0.1 $\mu$m). This allows the confined gap to be scanned in two dimensions with respect to the incident X-ray beam (Figure).

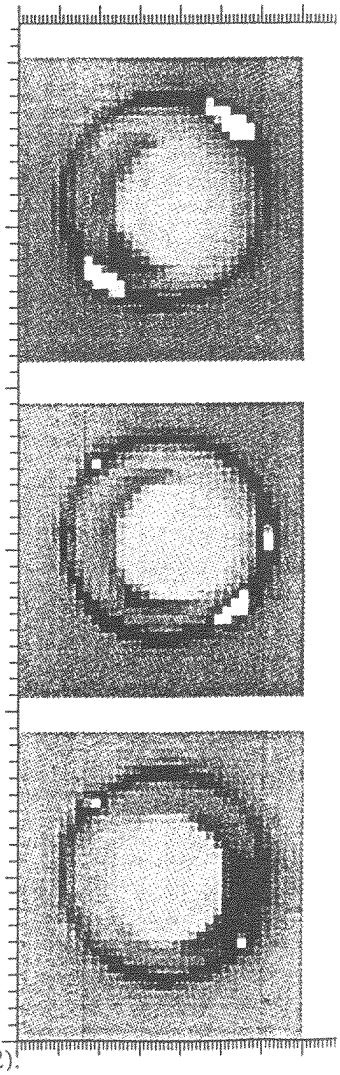
A 2 $\mu$m beam size (full width) at 0.092 nm wavelength (13 keV) was obtained at the exit of a borosilicate-glass capillary. The flux was about $10^{10}$ photons / sec / $\mu m^2$ /100mA

with a divergence of about 2.3 mrad. The background scattering was reduced by placing a Platinum aperture (minimum aperture 5 μm) in front of the capillary exit[3]. The upstream Aluminum cylinder was hollow which allowed to place the focus glass capillary and the Platinum aperture close to the mica surface. This minimizes the irradiated section extent (typically : about 4 μm) of the confined sample.




As an illustration we present the first results obtained with a thermotropic liquid crystal. We investigated the effect of confinement upon the orientation of 4'-n-octyl-4-cyanobiphenyl (8CB) which is in a smectic phase at room temperature. These rod-like molecules associate in dimers and orientate with their long axis parallel to the mica surfaces. The smectic phase is thus a one-dimensional array of stacked layers (periodicity 3.2 nm) oriented perpendicular to the confining walls. In the crossed-cylinder geometry the medium is of non-uniform thickness; thus there is a conflict between the planar alignment and the need for a constant layer spacing. As a result defects must arise. Disinclinations separating domains of different orientations are expected.

The main advantage of our set up with use of a microbeam is that it allows a systematic exploration of the full succession of oriented domains present within the confined gap. When the two surfaces are far apart (hundreds of micrometers), similar diffraction patterns are observed for any position across the micas relatively with the incident beam. Indeed in the case of large gaps, the X-ray beam passes through successive domains of different orientations giving rise to complex diffraction patterns. In the limiting case where the domains are randomly oriented, the superimposition of all the Bragg peaks would give powdered diffraction rings. Conversely, when the separation is reduced, single domains of given orientation can be explored individually. This is illustrated opposite where we present a selection of diffraction patterns obtained on a 2d-CCD camera with an exposure time of 8 s. The micas were held at a separation of about 3 μm, and a 2-d scan was carried out with the incident beam with a step size of 2 μm between individual frames. Note that on muscovite mica, which has a ternary symmetry, the 8CB molecules can align along three different directions of the director making angles of 60" to each other. The distribution of Bragg peaks reflectsthe sampling by the Ewald sphere of reciprocal lattice points corresponding to the different crystallites. Thus only a few domains will contribute to the scattering collected by the planar detector. Domains located in the central part of the confined gap around the common normal to the surfaces have all their reticular planes parallel to the incident beam. Thus 6 spots with a six-fold symmetry, corresponding to the first Bragg diffraction order, are revealed (central frame). When the irradiated volume is moved away from the centre, then only the scattering by the tilted domains is collected by the detector (upper and lower frames).



1. Israelachvili, J. N. *Intermolecular and surface forces*. Acad. Press, London (1992).
2. Engström, P. & Riekel, C. *J. Synchrotron Rad.* 3, 97-100 (1996).
3. Engström, P. & Riekel, C. *Rev. Sci. Instrum.* 67 (12), 1 (1996).