A Cell for Confined Liquids Investigated with X-rays

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The studies of the liquid films deposed on the surface show that close to the interface the liquid molecules arrange itself in layers [1]. The effect could be stronger pronounced if the liquid is confined in gap with size comparable to the molecular dimensions. In the frame of the current project, the experimental environment for x-ray reflectivity measurements of confined liquids confined has been probed. The setup is developed on the base of a diamond anvil cell (DAC). The previous studies [2] show that the X-ray reflectivity is perfectly suited to investigate molecular layering along the surface normal, as it is sensitive to the averaged electron density profile. For reflectivity the spatial accuracy is usually better than 0.1 nm and the sensitivity to the contrast (density changes in the profile) is in the range of a few percent. However, reflectivity cannot monitor in-plane ordering of molecules. For this reason, the setup is also designed for out of incidence plane diffraction and/or small angle scattering (SAXS).

The experiment is carried out at P08 Beamline, PETRA III with photon energy of 18 keV. The liquid used in the measurement is Benzene (Sigma-Aldrich Chromasolv Plus, for HPLC). Diamond culets similar to that used in the DAC are used as substrates. The liquid is injected in bottom part of the sample cell and heated. During the injection the gap is open with distance of ca. 20 µm. The liquid evaporates and condenses mostly on the surface of the top diamond, after that the gap is closed by means of the pieso stage under the bottom diamond.

The good evidence that the gap is well closed is the missing of the total reflection region on the reflectivity curve (the critical angle at used energy, appears at qz ca. 0.04 $Å^{-1}$). The thickness of the film is calculated through the oscillation observed on the curve (see Fig. 1). The further pressure on the diamond surface, created by pieso devise, decreases the film thickness, as it can be seen on the Fig. 1. The gap size changes of 200 Å to 160 Å with increasing of Pz of 67.5 µm to 74 µm.

The layering of the liquid on the surface due to the confinement would had spacing with size in the region of the of the structure factor of liquid. Therefore the scanning of the structure factor angular region is also of interest. For Benzene the structure factor peak appears in reciprocal space at value of ca. 1.4 $Å^{-1}$. The observation of the structure factor has been possible after focusing the x-ray beam down to 4x40 µm in vertical and horizontal direction, respectively. The micro focused enhances the efficiency of the x-ray beam. It improves the signal to nose ratio and resulting in stronger scattered signal from the gap. The reflectivity curve, measured with micro focus beam, has two orders of magnitude stronger intensity and it could be prolonged over the liquid structure factor angular region (see Fig. 2). In this rage a large week peak is observed on the reflectivity curve. The peak arises from the layering of the liquid molecules on the surface.

The experiment verifies that the setup can be used for confinement of liquids dawn to molecular levels. In-plane and out-off-incidence plane x-ray measurements give very interesting results that could contribute to the understanding behavior of the liquid at the interface.

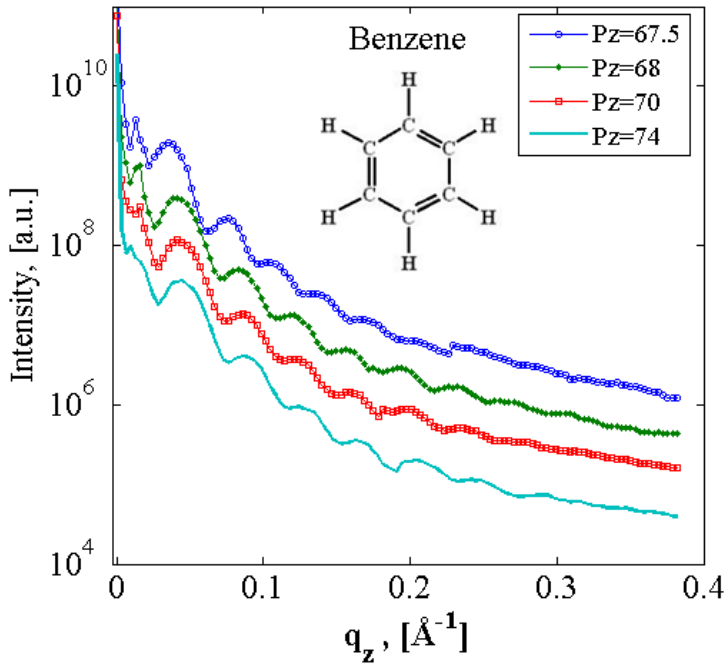

Figure 1: Reflectivity curves of confined benzene film, Energy 18keV; beam size: V x H - (4 x 40) µm. Pz notes the position of the vertical travel of the piezo devise.

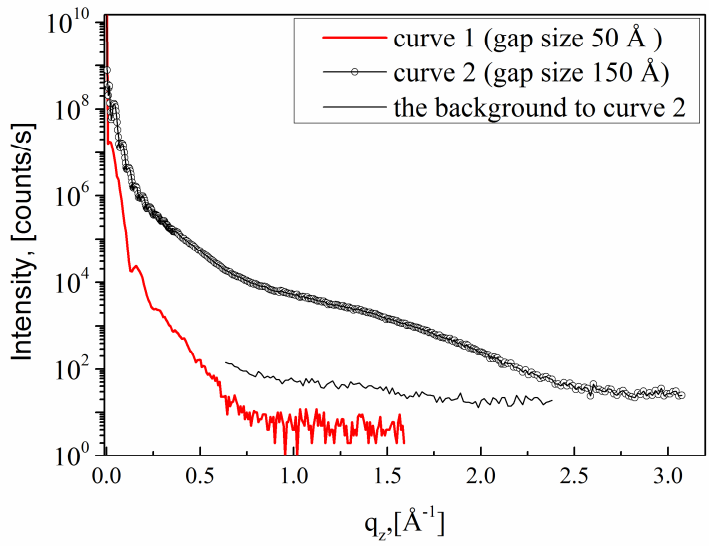

Figure 2: Confined reflectivity curves measured with moderately focused and micro focused beam. Curve 1: beam size V x H - (30 x 200) µm; curve 2: beam size V x H - (4 x 40) µm.

References

[1] C.-J Yu, A.G Richter, A Datta, M.K Durbin and P Dutta, Physica B, **283**,27 (2000).

[2] O.H. Seeck , Hyunjung Kim, D.R. Lee, D. Shu, I.D. Kaendler, J. Basu and S.K. Sinha, Europhysics Letters **60**, 376 (2002).