Our experiment was scheduled in the period 12-16 July 2003 at the ID01 beamline. During this period not all the requested equipment was available at the beamline, then we could not perform temperature dependent measurements as described in the proposal. Considering that the beamtime was less than requested we focused our attention on X-ray reflectivity and X-ray diffuse scattering measurements.
The incident beam energy was 16 KeV. The samples were in air at room temperature with flowing nitrogen on top for preventing them from oxidation.
During this beamtime we were able to study 5 samples:
two highly oriented lamellar lipids (the cationic DOTAP and the neutral one DOPC) deposited, their mixture with molecular weight fraction = DOPC/DOTAP+DOPC = 0.50, and the lipid mixture/DNA complex at the isoelectric point (zero total charge). All the samples were deposited by spin coating on Si <100> and in the case of DOPC also on GaAs <100>.
Here we present some of the measured data, with no fits. Since the short delay between the experiment dates and the submission deadline it has not been possible to simulate the data.
In Fig. 1a we show the reflectivity measurements obtained from neutral DOPC lipid on Si <100>. The curve shows well-defined interference fringes and the presence of a superlattice peak. These features indicate that the lipid is organized as a bilayer whose thickness is about 39 Å, and that the whole film has a well defined thickness of about 13 bilayers.

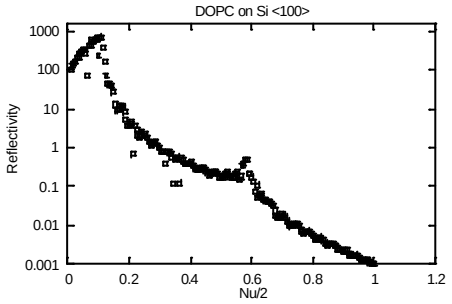

Fig. 1a

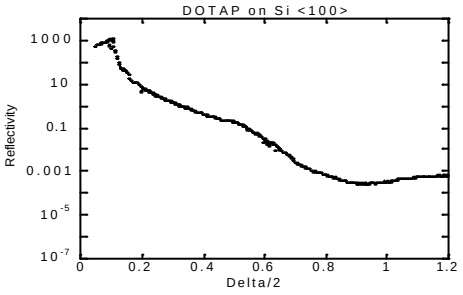

Fig. 1b

On the contrary the cationic lipid DOTAP on Si <100> results to be less ordered. The film is not well organized in bilayers and the roughness of the film is more important as observable by the dumping of the interference fringes near the critical edge (Fig. 1b).

When the lipids are mixed together we have the simultaneous presence in the spectrum of structures which can be associated to the two lipids.
The mixture seems to be characterized by the coexistence of the two lipids, which form different domains (Fig. 2a). This indicates that the mixture is unstable and the two components seem to segregate. This effect has already been observed in lipid mixtures as recently reported in literature (L. Bagatolli and E. Gratton, Biophys. J. 79, 434 (2000); F. Tokumasu et al., Biophys. J. 84, 2609 (2003)).

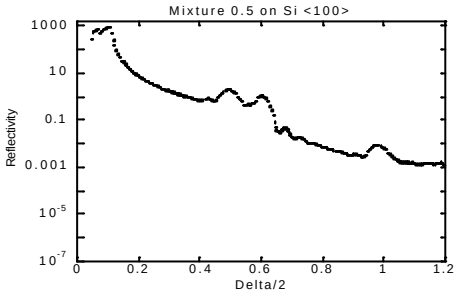

Fig. 2a

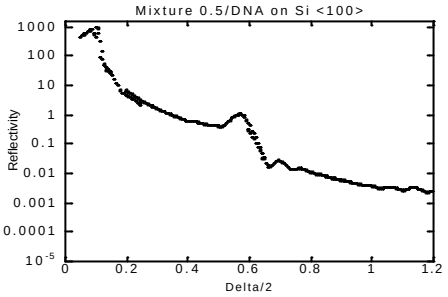

Fig. 2b

Important modification in the structure is induced by the presence of DNA. In this case (Fig. 2b) we observe only one bilayer peak at the average value of the two peaks in Fig. 2a. DNA seems to facilitate the mixing of the two lipids which now coexist with an average structure. More detailed information on this phenomenon will be extracted by a further analysis of the diffuse scattering.

Finally we have been able also to show the influence of the substrate on the lipid film structure. In the case of DOPC deposited on GaAs substrate we observed a reflectivity curve shown in Fig. 3. This data set should be compared to the plot reported in Fig. 1a. The film is more disordered as the absence of Bragg peaks indicates.

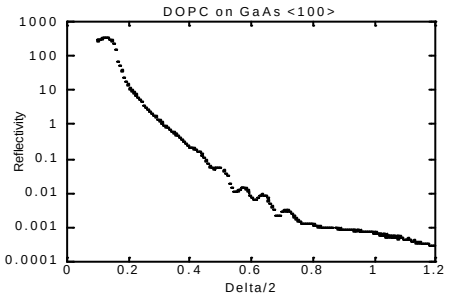

Fig. 3

Beside the reflectivity curves, we have also obtained a reciprocal space map in order to have information about the roughness of the sample surface.
The data analysis will be completed within the next month.

We would stress that the number of samples measured is much lower than foreseen due to some technical and configuration problems of the beamline not dependent from the local contact present during the experiment.
In particular we have met a bad operation of the automatic filters and, the absence of a PSD detector. Both resulted in much longer acquisition time for each sample than expected.
Moreover the impossibility to use the cryostat as requested in our proposal due to beamline set up problems has prevented us to perform measurements as a function of temperature and to clarify the structural changes foreseen by quasi-elastic neutron scattering data.