ESRF	**Experiment title:** Origin of the flat phase of lipid-divalent cations complexes	**Experiment number:** SC-1070
Beamline: ID10 B	**Date of Experiment:** from: November 13th 2002 to: November 18th 2002	**Date of Report:** September 3, 2003
Shifts: 18	**Local contact(s):** O. Konovalov	*Received at ESRF:*

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Report:

Some divalent ions (e.g. Mn^{2+}, Cd^{2+}, Pb^{2+} and Mg^{2+}) dissolved in the aqueous subphase cause the formation of an inorganic superlattice under a fatty acid monolayer (Kmetko et al., *J.Phys. Chem. B*, 2001, pp 10818-10825). The aim of the experiment was to study the fluctuations of these monolayers: contrary to standard fatty Langmuir film, elasticity of the inorganic superlattice does change the height-height fluctuation spectrum. In such "polymerised membranes", this is suspected to come from a coupling between height fluctuations and in-plane phonons.

According to the phenomenological description of Helfrich (1973), the (Fourier transformed) Hamiltonian for interface deformation $\mathcal{H}(q)$ can be written as $\mathcal{H}(q) = \Delta\rho g + \gamma q^2 + \kappa q^4$, where the bending rigidity κ is q dependent due to non-linear couplings with in-plane elastic parameters (modulus of area compressibility and shear modulus). According to a renormalisation theory of the polymerized membranes, κ behaves like:

$$\kappa \sim q^{-\eta}.$$

Numerical simulations give $\eta \simeq 0.6 - 0.7$ for zero surface tension polymerized membranes (tethered membranes). Only a very small number of experimental studies of the coupling have been carried out due to the lack of potential experimental realisations of polymerized membranes.

$\mathcal{H}(q)$ (and $\kappa(q)$) can be determined by grazing-incidence surface scattering following the method developed in previous experiments: Grazing incidence x-ray scattering allows the precise measurement of the surface fluctuation spectrum $< h(q)h(-q) >$, which is the Fourier transform of the height-height correlation function. Since $\mathcal{H}(q) = k_B T / < h(q)h(-q) >$, κ can therefore be precisely determined as a function of q.

The experimental setup was quite similar to those of the previous experiments (SC-441 and SC-688).The detector was a vertically mounted gas-filled position sensitive detector (PSD), and the energy was fixed to 8 keV. The grazing angle of incidence was set below the critical angle for total external reflexion in order to minimize penetration depth and to be sensitive to interface fluctuations. Scanning the horizontal scattering angle up to 20 degrees allowed us to reach wave vector transfers on the order of $2.10^{10}\mathrm{m}^{-1}$ and therefore to detect the interface short-length scale structure.

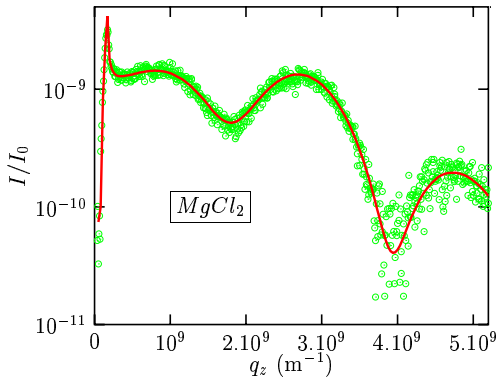

Fig.1- *X-ray diffuse scattering yields information on the vertical structure of monolayers. Calculations (solid line) show that the vertical structure is well understood.*

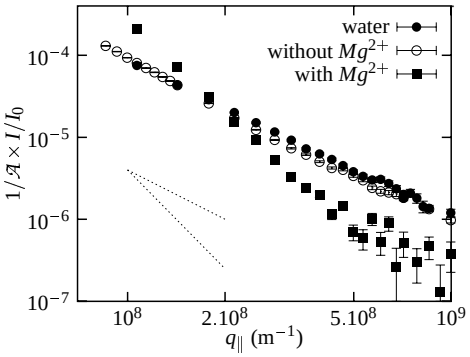

Fig.2- *Scattered intensity as a function of the in-plane component of the momentum transfer vector for three interfaces. Pure q^{-2} and q^{-4} behavour are indicated by dashed lines.*

Measurements were performed for water and behenic acid with Mg^{2+}, Cd^{2+} or Pb^{2+}. We checked that the superlattice diffraction peaks are still present after the measurement. Radiation damage of the superlattice was the slowest for Mg^{2+} and films with this ion lead to very reproductible measurements. The vertical structure of the monolayers can be well described using a simple model (see Fig.1). Fig.2 shows a dramatic change in the scattered intensity (which is proportional to the surface fluctuation spectrum $< h(q)h(-q) >$) from a q^{-2} spectrum (water or monolayer without divalent ions) to a much steeper decrease with wave-vector with the Mg^{2+} ions. η is determined from the slope of the curve in Fig.3. One finds $\eta = 0.35 \pm 0.05$.

The resolution of the beamline's crystal analyser was good enough to determine the intrinsic shape of different Bragg peaks (see Fig. 4). The shape is expected to be $|q_{\|} - G|^{\eta_G}$ where G is the Bragg vector and $\eta_G \propto G^{2\eta}$. In consequence, exponent η could be determined by analysing the shape of several Bragg peaks. Unfortunately we didn't have time to investigate different peaks in detail and check that prediction.

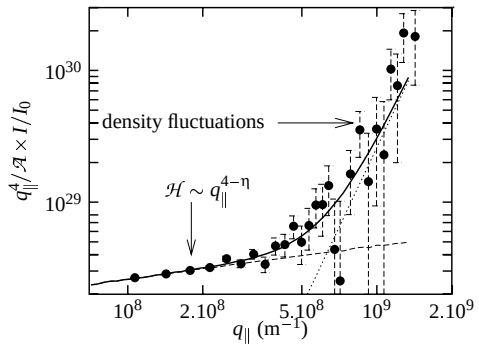

Fig.3- *Scattered intensity (behenic acid with $MgCl_2$) being multiplied by q^4 (and a geometric factor $\mathcal{A}$), the slope obtained for small $q_{\|}$ is the exponent η.*

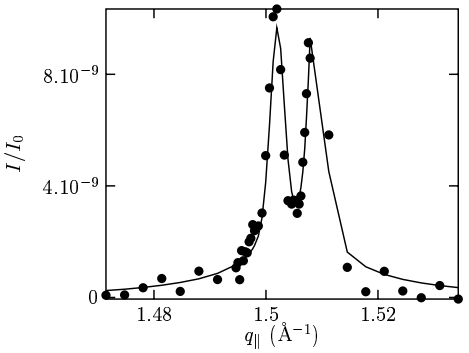

Fig.4- *Diffracted intensity versus $q_{\|}$. These two peaks (0 1) and (1 $\overline{1}$) correspond to the reciprocal lattice of the Langmuir monolayer*