The aim of this experiment (SC 436) was to characterize, using the coherent beam of ID10A, low frequency dynamics inside cubic mesophases formed by close packing of microemulsion droplets in a water/oil/SDS butanol system [1]. For that purpose, we planed to measure the time autocorrelation function of the intensity <I(t)I(0)> determined with spatially coherent x-ray, in a q-range corresponding to the typical length scale of individual droplet (~ 150 Å droplet diameter). Such measurements required coherent x-ray in the range 10^{-2} – 10^{-1} Å^{-1} (Fig. 1).

In the cession corresponding to the allocated beam time, the beam line was configured to provide maximum coherent flux in range 10^{-3} – 10^{-2} Å^{-1}. Consequently, droplet dynamics were difficultly accessible, but through measurements in the vicinity of the first order diffraction peak of low intensity (Fig. 1). However, this intensity turned to be too low to achieve successfully XPCS measurements.

We have taken advantage of this configuration to perform during the attributed beam time structural and dynamic investigations of these cubic mesophases in the very small angle range. These investigations have provided interesting preliminary results:

- in addition to previously known diffraction patterns in range 10^{-2} – 10^{-1} Å^{-1} reflecting the droplet crystalline packing, we have observed unexpected diffraction patterns at much smaller angle (in range 1×10^{-3} – 5×10^{-3} Å^{-1}).

- such diffraction patterns at very small angle seem a general property of these systems since we have observed the same property for other samples prepared at lower surfactant concentration (the concentration range 20 – 8 wt % in surfactant has been explored).

- intensity scans performed on these low angle diffraction peaks have revealed high oscillatory intensity fluctuations with a time period of ~ 100 s.

We spent much time after that ID10 experiment to verify that such striking diffraction patterns were not artefacts. First, performing a test on the SAXS camera of ID2, we did not succeed to find an appropriate beam configuration to observe these diffraction patterns at small angle. To clarify this point, we recently performed another test on ID10 (08/2003). This new test (4 hours beam time) unambiguously confirms the existence of diffraction peaks in the very small angle range (1×10^{-3} – 5×10^{-3} Å^{-1}) when samples are illuminated in coherent configuration.

It seems now clear that a larger structural level than that previously recognized in these mesophases does exist. Furthermore, these previous tests have shown that the observation of such large-scale structures necessary requires combined high angular resolution and very small beam sizes such as those required to provide coherent x-ray (a 12 micron pinhole aperture at the distance of 45 m from the source is used on the beamline ID10). Further measurements on the beam line ID10 will be necessary to understand and characterize more into details such new structural features.

We have also progressed in characterizing dynamics in these mesophases, by combining neutron spin echo (NSE), dynamic light scattering (DLS), pulsed field gradient NMR, and rheological measurements. These results are described into details in a PhD thesis [2] and recent articles [3,4].

References:

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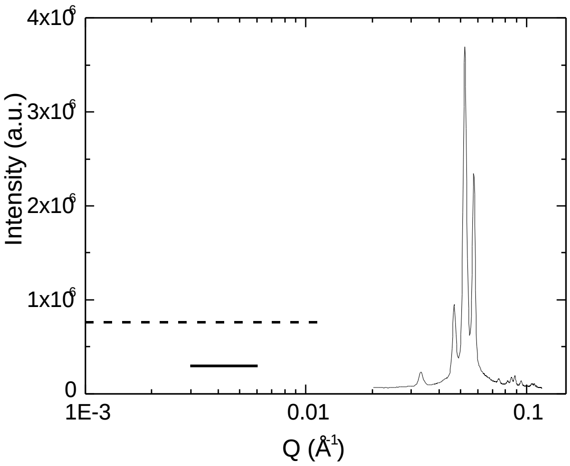

Fig.1: *X-ray diffraction pattern of a cubic phase (here, Pm3n symmetry) formed by close-packing of microemulsion droplets. Bragg peaks in range 10^{-2} – 10^{-1} Å^{-1} reveal the droplet crystalline packing (lattice parameter 268 Å). The dotted line indicates the q-range showing maximum coherent flux for the ID10A beam line configuration. The full line corresponds to the q-range where novel diffraction patterns have been observed when the mesophase is illuminated in coherent condition (see Fig. 2).*

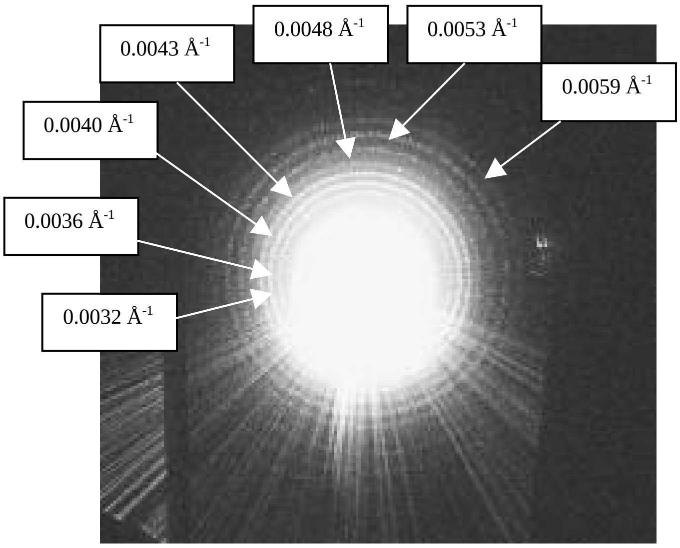

Fig. 2: *Typical small angle x-ray diffraction pattern measured in coherent condition with high resolution sensitive x-ray films, by using a 12 µm pinhole aperture on the beamline ID10A, with a sample-detector distance of 2225 mm and λ = 1.5464 Å. At least 7 strikingly well defined diffraction peaks can be observed for q values ranging between $3x10^{-3}$ - $6x10^{-3}$ Å^{-1}. Magnification of the image size by a factor 6.3.*