ESRF	**Experiment title:** Temperature dependence of the dynamics of electrorheological chain formation in synthetic nano-layered silicates	**Experiment number:** 01-02-679
Beamline: BM01A	**Date of experiment:** from: 17/07/2003 to: 19/07/2003	**Date of report:** 26/09/2003
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Scientific background – motivation for the study :

Electrorheological fluids have the property of displaying drastic changes in their rheology when exposed to a sufficiently intense electric field. The simplest form of electrorheological fluid is a suspension of polarizable particules immersed in a non-polar fluid [1]; Dipolar interaction between polarized particles is responsible for their aggregation along chains parallel to the electric field [1]. Nano-layered silicate suspensions, or clay suspensions, on the other hand, are colloidal suspensions of platelets that display a complex phase behavior depending among other things on the concentration in colloids, ionic strength of the solvent, and particle sizes. These properties have been investigated theoretically and experimentally using many techniques [2], among which synchrotron X-ray scattering methods [3, 4].

Microscope observations carried out in our laboratory at NTNU in Trondheim (Norway) on suspensions of fluorohectorite clays showed evidence of an electrorheological behavior for an electric field of a few kV/mm [5]. Fluorohectorites are synthetic swelling clays with a formula X_x-$Mg_{3-x}Li_xSi_4O_{10}F_2$. The colloidal particles consist of a stacking of ~ 100 silicate platelets that are bonded together by an interlayer cation X, the nature of which influences the strength of the bonding and the interaction between particles in the solvent. Cations and water molecules can be intercalated between the platelets, which plays a role in the polarizability of the particles.

In the spring of 2003 we performed WAXS experiments using the 2D MAR detector at BM01A. From this data we determined the orientation of the particles in our electrorheological chains, and showed that they polarize along the nanosilicate sheets [5]. The series of WAXS presently reported addressed the temperature dependence of the electrorheological state and of the dynamics of its formation: we observed ordering within the chains as a function of time after application of the field, at several different well-controlled temperatures.

Experimental method:

The three types of samples consisted of fluorohectorite (X=Na, Ni and Fe) particles immersed in a silicone oil (viscosity 100 cSt).

They were investigated by Wide Angle X-Ray Scattering, using a wavelength $\lambda = 0.71$ Å. A cell that we specially designed allows to apply a high voltage E between 2 copper electrodes, between which the sample is contained. This cell is a development of that used last year. Its electrically-insulating parts are in teflon so that the cell be able to bear temperatures up to 200 degrees. Using a temperature-control system based on a Peltier element and controlled by a laptop placed in the experimental hutch and operated from outside the hutch through the LAN network, we were able to impose a given temperature between 5 and 140 degrees (Celsius scale).

At controlled temperature, an electric field ~ 1 kV/mm was applied between the electrodes, after which a series of 2D diffractograms was recorded until the system was observed to be stabilized in its electorheological "organized" state. This was repeated for the three types of initially "wet" (1 or 2 layers of intercalated water) samples.

The 6 shifts of beam time awarded were fully utilized. More time would have been needed in order to perform experiments at different magnitudes of the electric field.

Results obtained:

The analysis method is basically the same as that described in a previous report (experiment 01-02-634; see also [5]). When increasing temperature, two effects influence the electrorheological process. On the one hand, the silicon oil has a viscosity that is strongly dependent on temperature. Decreasing its viscosity makes the electrorheological response of the suspension much faster. On the other hand, intercalated water can escape the clay particles, whose polarization properties subsequently change. The latter effect is not likely except at high temperatures, because the water and the surrounding oil "do not like each other".

Monitoring the changes in the peak position in q-space, we can monitor the possible escape of water molecules. Monitoring the degree of ordering ("rocking curves") as a function of time after application of the field, we see how the characteristic time for electrorheological structure formation depends on temperature. If the hydration state of the particles remains unchanged over the temperature range, this change in characteristic time is mainly due to the changes in oil viscosity. Analyzes are presently ongoing.

References

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