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Report:

In this experiment, we have studied dried-pressed samples of fluorohectorite clays consisting of a population of fluorohectorite clay particles possessing a preferential orientation due to dehydration coupled to a uniaxial pressure load. A short introduction to clay minerals in general and fluorohectorite in particular has been given in the proposal for beamtime for this experiment, nr. SC-1784. The pore space inside the samples consists of a microporosity, inside the clay stacks, and a mesoporosity between the clay particles. When humidity is supplied, each fluorohectorite particle behaves as an intercalation compound inside of which water molecules intercalate mono-layer by mono-layer (microporosity). The swelling of individual clay particles through water intercalation modifies the relative positioning, and in particular, the relative orientations, of the particles, and as a consequence it modifies the mesoscopic pore space around the particles.

The goal of this experiment was to monitor the changes in mesoporosity in situ, as a consequence of a change in ambiant humidity and temperature. These measurements were planned to complement WAXS measurements performed earlier that year on the same samples.

The data treatment consisted in analyzing the 2D diffractograms both in terms of q dependence at a given azimuthal angle and in terms of azimuthal intensity change. The latter feature was particularly investigated.

The data was treated and exploited in the beginning of 2006, and the results presented at the XIIIth Conference on Small-angle Scattering, in Kyoto, in july 2006. A proceeding ([1]) was published in a special volume of the Journal of Applied Crystallography devoted to contributions to that conference. The paper is now referenced in the ESRF publications database.

The abstract to that article summarizes our findings well. Here it is:

"Weakly hydrated samples of platelet-shaped nano-particles obtained by dry-pressing suspensions of the synthetic Na fluorohectorite clay are studied. The particles consist of stacks of several tens of 1 nm-thick nanosilicate platelets. They form a compound of quasi-two-dimensional particles whose average director is aligned with the direction of the uniaxial stress applied at dehydration. Small-angle X-ray scattering images from these samples are either isotropic or anisotropic, depending on the sample orientation with respect to the X-ray beam. From anisotropic images, changes in the scattering objects orientation distribution probability (ODP) function are investigated as the temperature is lowered, thus triggering swelling of the individual particles by water intercalation. This is done, on the one hand, by inferring the width of the ODP function from the eccentricity of quasi-elliptic iso-intensity cuts of the small-angle scattering images, and, on the other hand, by obtaining the ODP function from azimuthal profiles of the images. The decays of the scattering intensity as a function of momentum transfer along the two principal directions of the images exhibit power law behaviors. A crossover scale between two power law regimes is observed on the profiles recorded along the horizontal axis; it corresponds to the typical pore size along the direction of the initially applied load. These results are compared with a previous study of similar systems."

References

[1] Y. Méheust, S. Dagois-Bohy, K. D. Knudsen, and J. O. Fossum. Mesoscopic structure of dry-pressed clay samples from saxs measurements. *J. Appl. Cryst*, 40:s286–s291, 2007.