



	Experiment title: Quantitative Texture Analysis of Naturally and Experimentally Compacted and Deformed Clays and Claystones - Implications for Seismogenic Fault Zone	Experiment number: ES 246
Beamline: ID11	Date of experiment: from: 16 Jul 2015 to: 19 Jul 2015	Date of report:
Shifts: 9	Local contact(s): Vadim Diadkin (email: vadim.diadkin@esrf.fr)	<i>Received at ESRF:</i>

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Report:

As demonstrated in [1], Synchrotron diffraction is the most suitable method for quantitative texture analyses of fine-grained, partly water-containing and partly soft samples of clays and claystones. In this experiment we continued our measurements with a series of about 40 naturally and experimentally deformed samples from an accretionary (Nankai trench, Japan; project NanTroSEIZE) and an erosive continental margin (Costa Rica; project CRISP). Depending on the origin of samples, i.e. soft or solid material directly from drilling cores or from different deformation experiments, the size of the cylindrical-shaped samples varied from 10 to 40 mm in diameter. While the solid material could be directly mounted on the sample holder, the soft material was placed in especially manufactured acrylic, non-diffracting sample holders.

A slightly modified perpendicular set-up of the beam line ID11 was used. It consisted of an X-Y-Z-stage, and an omega-rotation stage. This set-up was appropriate to record diffraction data with a Perkin Elmer 1621 detector in order to calculate Quantitative Texture Analyses (QTA). A step width in omega of 5° and an omega-range of 180° was applied. The cylinder axes of the samples were mounted parallel to the Y-direction of the sample holder, so that the sample geometry was constant during rotation around omega and a following geometrical correction is not necessary. The beam energy has been adjusted to 80 keV and the beam size to 0.5 mm x 0.5 mm.

Exposure time per frame varied between 1 to 2s depending on the sample diameter and the material composition. To ensure that we consider possible heterogeneities of the natural material and to improve statistical data representation to the best, especially of the mineral

phases of low portion, we run three to fifteen texture measurements per sample at different positions along the cylinder axis.

The textures are calculated from the data with MAUD (Material Analysis Using Diffraction [2]), a Rietveld-based code.

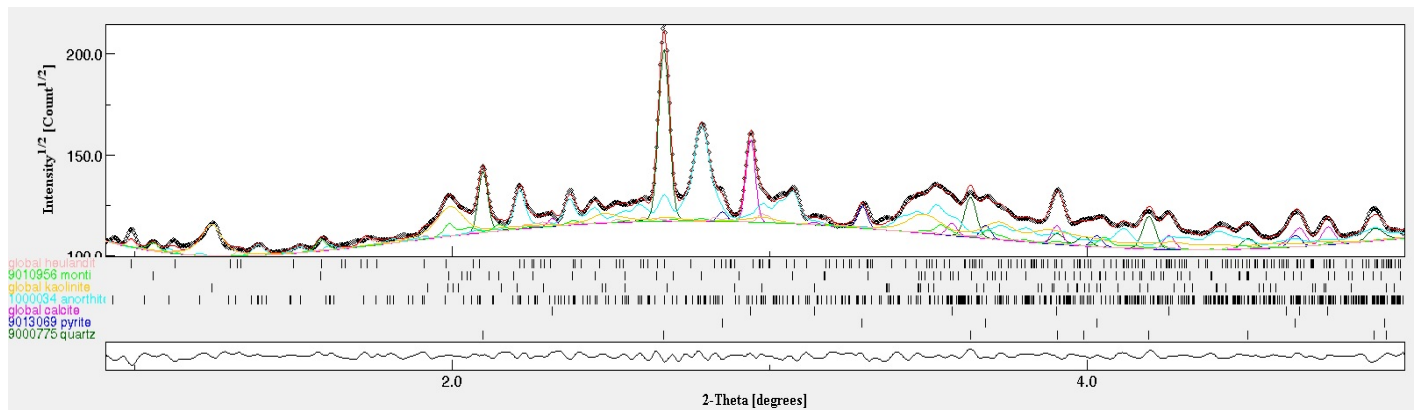


Figure 1 Exemplary diffractogram of a measured clay sample with identified phases with the Rietveld-based software program MAUD, showing the complex composition with a resulting very complex peak overlapping.

All scheduled samples could be measured during the proposed beam time. The data sets are complete for publications and will also complement existing sample series. However, the quantitative texture analyses by means of MAUD are very time consuming, also due to long computer calculation times. This duty will be completed within a running DFG-project within the next year.

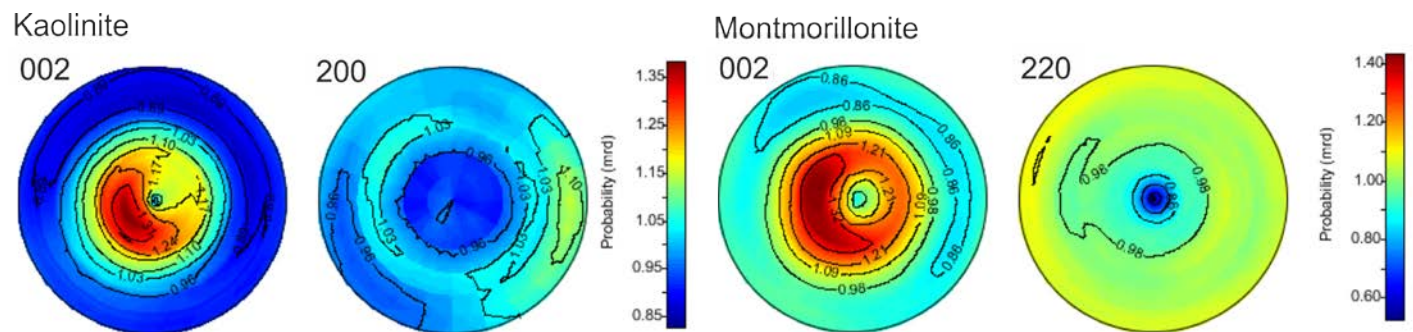


Figure 2 Exemplary recalculated pole figures from the QTA for the clay minerals kaolinite and montmorillonite

Acknowledgement: We want to emphasize the extremely enthusiastic support of the beam line scientist during our beam time.

[1] K. Schumann, M. Stipp, B. Leiss, and J. Behrmann, Texture development of naturally compacted and experimentally deformed silty clay sediments from the Nankai trench, Japan (NanTroSEIZE, IODPExpeditions 315, 316, 333), *Tectonophysics*, 18 p. (2014) (doi: 10.1016/j.tecto.2014.08.005)

[2] Lutterotti, L., Mathies, S., Wenk, H.R., Schultz, A.J., Richardson, J., Texture and structure analysis of deformed limestone from neutron diffraction spectra. *J. Appl. Phys.* 81(2), 594–600, (1997).