EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:
https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

	Experiment title: **Understanding extreme anisotropy in slate**	**Experiment number:** ES-698
Beamline: ID11	**Date of experiment:** from: 28 March 2018 at 08:00 to: 29 March 2018 at 08:00	**Date of report:** 15/3/19 *Received at ESRF:*
Shifts: 3	**Local contact(s):** GIACOBBE Carlotta	

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Report:

Large parts of the Earth display strong elastic anisotropy, as revealed by seismic data. There is consensus that crystal preferred orientation (or texture) plays a crucial role. There is a broad effort to quantitatively link microstructures to macroscopic properties with physical models. The microstructural characterization relies on X-ray diffraction to quantify crystal preferred orientation. Sheet-silicate-rich rocks occur in a wide range of geologic environments. Clay-rich shales are estimated to comprise over 75% of the rocks in sedimentary basins and sheet silicate minerals become aligned during sedimentation, compaction and diagenesis. Slates form by recrystallization of shales at low grade conditions. The goal is to learn to understand the mechanisms that lead to this alignment with a systematic investigation of the famous slate localities in N Spain.

Our original idea was to develop a better strategy for quantitative texture analysis of complex slates at ESRF, and compare the result with previous data in ALS (Berkeley) and new experiments along 2020 in APS

(Chicago). In this first round of experiments we established an optimal procedure by performing tests at ID11 beamline. Optimal samples are cylinders, which can be rotated to any orientation. Cylinders are 2-3mm in diameter and 10mm long. They were analyzed in transmission with a beam 0.5 mm. Hard X-rays (40keV) were finally provided what to some extend limited the resolution of some phases. A second drawback to overcome was the small size of the Frelon detector which reduce the final d-spacing range per measurement. Samples were aligned on the goniometer to have the cylinder axis centered in the beam. Acquisition strategy included translation of the sample along the axis to improve grain statistics. Images will be collected at different rotations (from -75° to +75° in 15° intervals) to obtain good pole figure coverage.

Diffraction images (Fig. 1A) from each rotation were corrected for FReLon geometrical distortion and merged into a single one in TIFF format, using Fit2D [1] Some issues came out as the geometrical distortion increase with the rotation angle, making +/-75° images unusable in some cases, a point that need to be explored and fixed in future experiments with Frelon detectors. Diffraction images were decomposed with Rietveld program MAUD (Material Analysis Using Diffraction; Lutterotti et al., 1997) into 72 azimuthal sectors of 5°, over which intensity was integrated, providing a total of 216 diffraction spectra per sample analyzed with the Rietveld method in MAUD as described in our tutorials [3,4] (Fig. 1B).

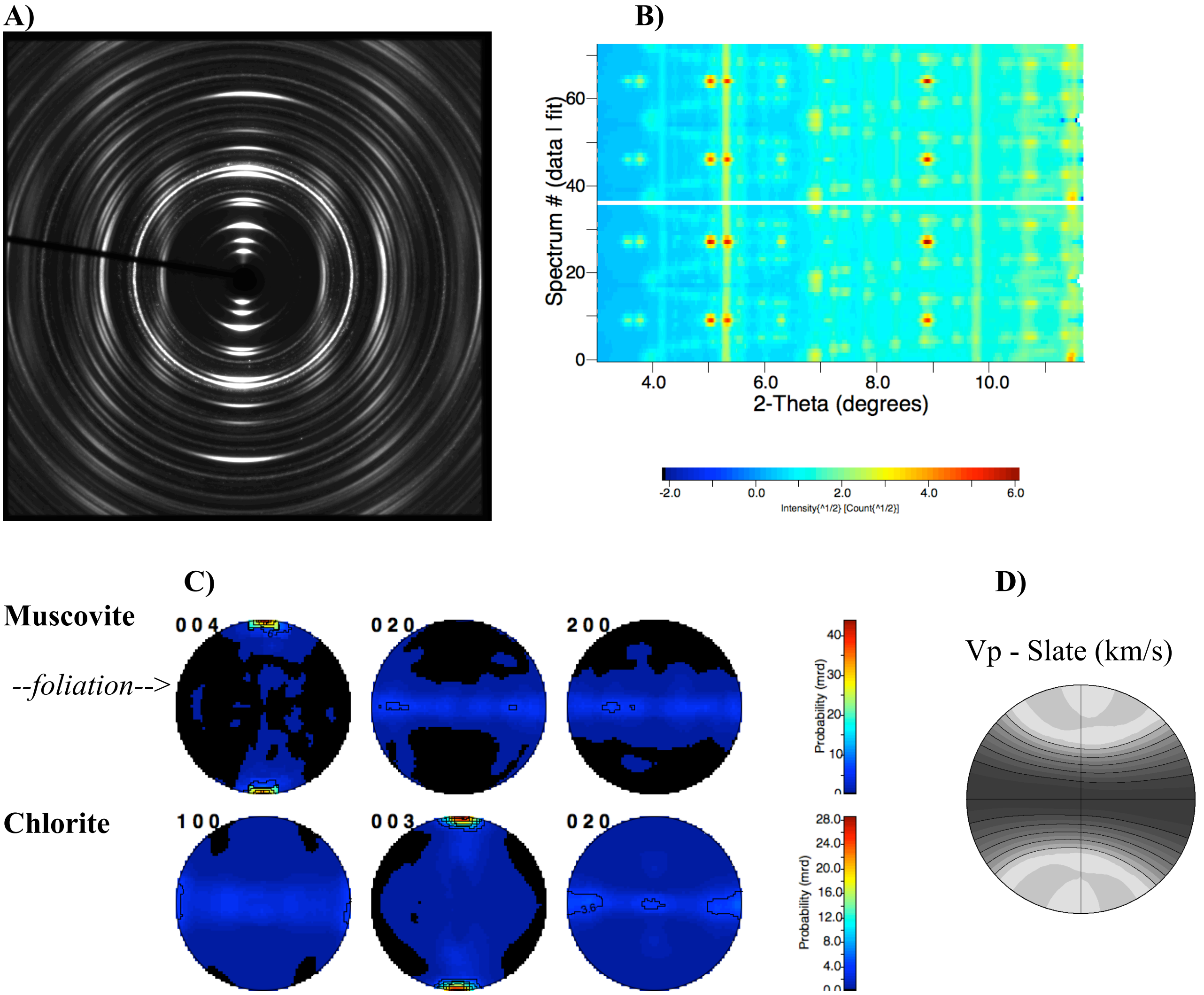

Figure 1: *A) Diffraction image from a slate. Strong intensity variation along Debye rings is due to texture. B) 'Unrolled' diffraction image (data) and Rietveld fit after complete texture analysis on MAUD. The Rietveld processing results in a good data fit which ensure a convergence of the refined parameters. C) Recalculated pole figures for Muscovite and chlorite. D) P-wave velocity distribution calcualted after the orientation distribution function, where fast propagation is along the foliation plane.*

Pole figures were recalculated after the orientation distribution (OD) of each phase (Ms, Chl, Qtz, Pl). Only phyllosilicates showed a texture, with values between 40-25 m.r.d. Some texture components were preliminary identified and tentatively related to SEM microstructure as part of a composite fabric developed during regional scale folding of the unit. However, more samples are needed to confirm this point. OD were used to calculate polyphasic elastic tensors per sample and seismic anisotropy (Fig. 1D).

References

[1] Hammersley, A. P., Svensson, S. O., & Thompson, A. (1994). Calibration and correction of spatial distortions in 2D detector systems. *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment*, *346*(1-2), 312-321.

[2] Lutterotti, L., Matthies, S., Wenk, H. R., Schultz, A. S., & Richardson Jr, J. W. (1997). Combined texture and structure analysis of deformed limestone from time-of-flight neutron diffraction spectra. *Journal of Applied Physics*, *81*(2), 594-600.

[3] Lutterotti L., Vasin R., Wenk H.-R. (2014) Rietveld texture analysis from synchrotron diffraction images: I. Basic analysis. *Powder Diff.* 29, 76

[4] Wenk H.-R., Lutterotti L., Kaercher P., Kanitpanyacharoen W., Miyagi L., Vasin R. (2014) Rietveld texture analysis from synchrotron diffraction images: II. Complex multiphase materials and diamond anvil cell experiments. *Powder Diff.* 29, 220