

Residual strain analysis by neutron diffraction on granites from the drilling EPS1 of the geothermal site in Soultz-sous-Forêts, France

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Background:

The porphyritic granite of the Soultz-sous-Forêts geothermal site has been intensely altered during several hydrothermal alteration events. These events caused the dissolution of primary minerals and the precipitation of secondary minerals, e.g. clay minerals (Figure 1). The presence of clay inside the rock matrix and fractured zones is affecting the mechanical strength. Mechanical contrasts in rock masses are supposed to change the ambient stress field locally (e.g. Valley 2007). The intrinsic strain parameters of the drill core samples reflect the local stress field to which the samples were exposed in-situ. Strain develops in rocks and minerals as a response to the applied stress field. Rock deformation is reflected by a change in the lattice constants of the minerals, which can be measured by X-ray scattering or by neutron scattering. The advantage of neutron scattering is its larger penetration depth into samples. For multi-phase rock material, a large penetration depth (i.e. large measured gauge volume) is essential to measure a representative number of lattice constants from different mineral phases and directions. The applicability of neutron-TOF-diffraction for strain analysis has been demonstrated earlier by Siegesmund *et al.* (2008) and Scheffzük *et al.* (2014).

The determination of strain parameters of samples with different alteration grades will show inasmuch the intrinsic stress in altered samples with a large mechanical contrast to the surrounding rock deviates from the global stress field.

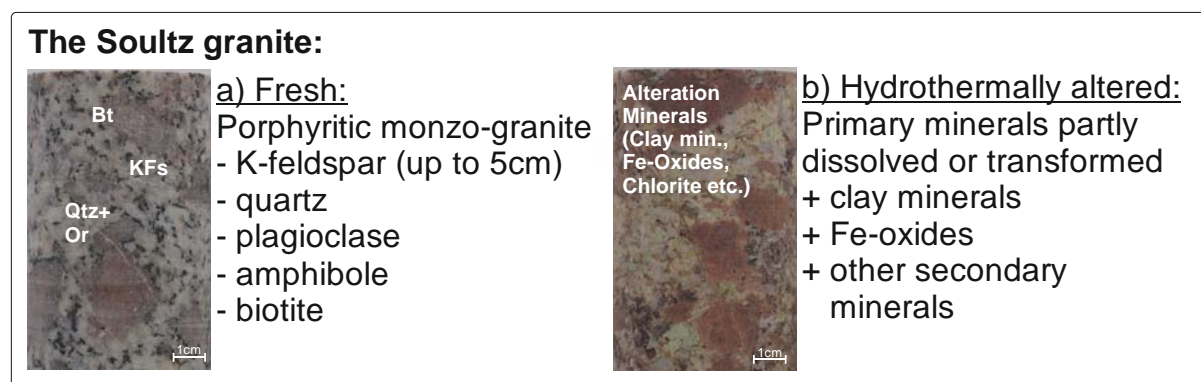


Figure 1: a) Core sample from the fresh Soultz granite with large K-feldspars. b) Core sample of intensely altered granite with secondary mineral formation.

Methods:

Our study focuses on the determination of residual strains on the EPSILON Strain diffractometer in Dubna, Russia. We selected core samples from the well EPS1 with 3 different alteration grades. Neutron diffraction patterns are measured with beam dimensions of HxW: 20x10 mm with a gauge volume of 2 cm³. The characterization of the residual strains will be carried out by two defined perpendicular directions (Fig. 2). Because of the possible different mineralogical content of the samples reference powder samples are prepared by grinding up to 63 µm and tempering on 600°C to achieve a strain-free (in 1st and 2nd order stress) reference sample.

Afterwards, the samples are measured at elevated temperatures (ca. 145°C) to generate the *in situ* thermal conditions at the original sample drilling depth. *In situ* stress and temperature values of the samples have been determined by:

K96-2525, depth: 1,568 m, 140°C, $\sigma_1 = 22.39$ MPa; $\sigma_2 = 21.94$ MPa; $\sigma_3 = 4.04$ MPa; $P_p = 16.25$ MPa
 K112-2833, depth: 1,654 m, 143°C, $\sigma_1 = 23.68$ MPa; $\sigma_2 = 23.21$ MPa; $\sigma_3 = 4.39$ MPa; $P_p = 16.25$ MPa
 K114-2870, depth: 1,663 m, 143°C, $\sigma_1 = 23.90$ MPa; $\sigma_2 = 23.43$ MPa; $\sigma_3 = 4.45$ MPa; $P_p = 16.25$ MPa

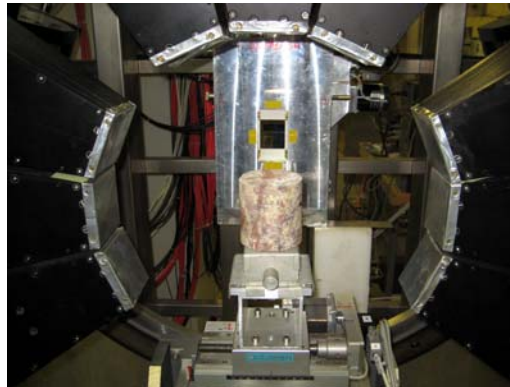
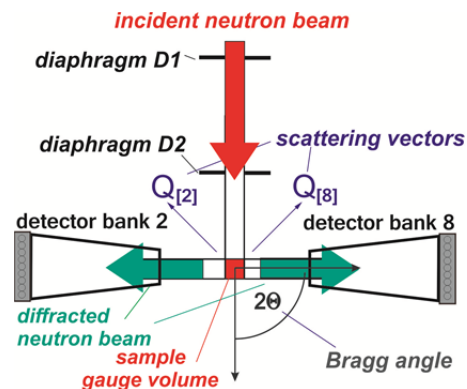


Figure 2: Measuring geometry of the EPSILON diffractometer to measure 2 perpendicular directions (characterized by the scattering vectors Q_2 and Q_8), simultaneously and arrangement of the nine detectors of the strain diffractometer around the sample at different scattering angles ϕ at $2\theta = 90^\circ$.

Results:

Rietveld Analysis:

In total, 9 diffraction patterns were measured, 3 of which are taken from powdered samples as reference values for the lattice constants, 3 at room temperature and 3 at 140°C *in situ* temperature. Analysis of the measured diffraction patterns is conducted with the Rietveld method using the software MAUD (Mineral Analysis Using Diffraction, Lutterotti *et al.* 1999). The main mineral phases detected with MAUD are Oligoclase, Microcline, Orthoclase, and Quartz (+Illite and Calcite for altered samples).

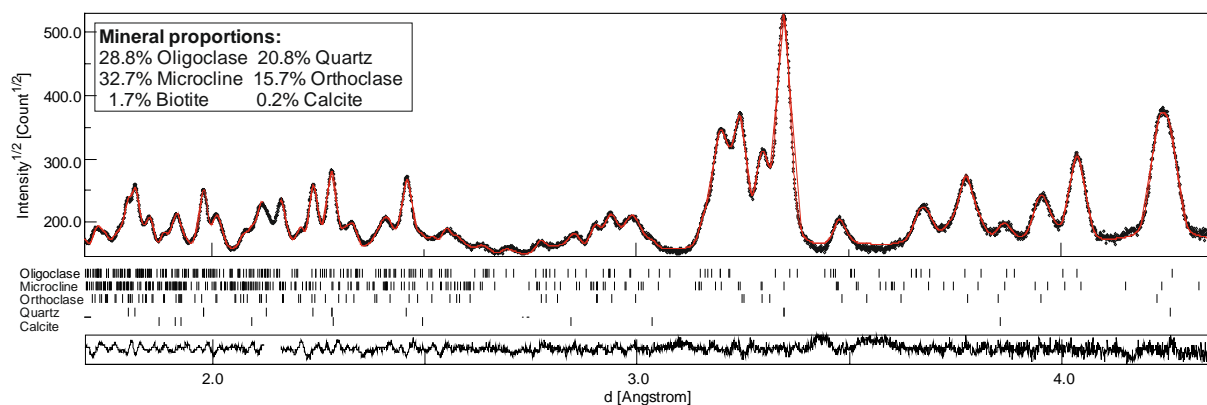


Figure 3: Neutron-TOF-diffraction pattern (black line) of the powder sample of K96_2525 (depth: 1568 m). The diffraction pattern represents the sum of all nine detector banks, mounted at EPSILON. Mineral analysis was done by Rietveld refinement using the Java software MAUD (Lutterotti *et al.* 1999). The line patterns indicate peak positions. The lowermost curve shows the deviation of the computed data (red line) from the measured pattern.

Strain Analysis:

Fitting of the diffraction patterns of core samples turned out to be rather challenging for the core samples. The nine detectors of the strain diffractometer register diffraction pattern at different scattering angles ϕ at $2\theta = 90^\circ$ (Fig. 2). Due to the porphyritic character of the Soultz granite, the nine detectors obtain information from different mineral assemblages, which results in large differences between the patterns obtained from the nine detectors. The applicability of Rietveld analysis on such diffraction patterns is limited. Hence, the determination of lattice constants is still ongoing work and remains challenging.

Conclusion:

It is principally possible to fit neutron diffraction spectra of multiphase granite powder can by Rietveld analysis using MAUD and the applicability of neutron-TOF-diffraction on a crystalline rock has earlier been demonstrated (Scheffzük et al. 2015). The neutron-TOF-diffraction method in combination with a large beam cross section (HxW: 60x50 mm²) is suitable to analyze multi-phase rock material, but it is limited to analyze very coarse grained samples with grain sizes in the cm range like the large K-feldspars in the Soultz granite. For better results selection of adequate samples relating the grain size would be recommended or by focusing on small grained phases which make up statistically high proportion of the sample.

References

- Lutterotti, L., Matthies, S. & Wenk, H.-R. (1999) MAUD: a friendly Java program for material analysis using diffraction. IUCr: Newsletter of the CPD **21**, 14-15.
- Meller, C., Genter, A. & Kohl, T., 2014. The application of a neural network to map clay zones in crystalline rock, *Geophys. J. Int.* **196**, 837-849.
- Scheffzük, Ch., Ullemeyer, K., Vasin, R., Naumann, R. & Schilling, F.R. (2014): Strain and Texture Investigations by Means of Neutron Time-of-Flight Diffraction: Application to Polyphase Gneisses. *Materials Science Forum* **777**, 136-141.
- Siegesmund, S., Mosch, S., Scheffzük, Ch. & Nikolayev, D.I. (2008): The bowing potential of granitic rocks: Rock fabrics, thermal properties and residual strain. *Environ. Geol.* **55**, 1437-1448.
- Valley, B.: The relation between natural fracturing and stress heterogeneities in deep-seated crystalline rocks at Soultz-sous-Forêts (France). Dissertation. Zürich, E. T.H., Zürich. ETH Reprozentrale Hönggerberg, HIL C45, Zürich.