Applied, residual strain and texture investigations by means of neutron time-of-flight diffraction combined with acoustic emission detection: Application to a gneiss sample from Forsmark, Sweden

Ch Scheffzük1,4,a, A Zang2, O Stephansson2, K Ullemeyer3 and FR Schilling1

1 Karlsruhe Institute of Technology, Institute of Applied Geosciences, Kaiserstr. 12, 76131 Karlsruhe, Germany
2 Helmholtz Centre Potsdam, GFZ German Research Centre for Geosciences, Section 2.6 Seismic Hazard and Stress Field, Telegrafenberg, 14473 Potsdam, Germany
3 Institute of Geosciences, University of Kiel, Otto-Hahn-Platz 1, 24098 Kiel, Germany
4 Joint Institute for Nuclear Research Dubna, Frank Laboratory of Neutron Physics, ul. Joliot-Curie 6, 141980 Dubna, Russia

Abstract. The mechanical behaviour of a gneiss sample from the Forsmark drilling site (Sweden) consisting of quartz, plagioclase (oligoclase), microcline and biotite, is characterized by residual strain and crystallographic preferred orientation measurements. Cyclic in situ uniaxial deformation experiments were carried out on a cylindrical sample at four uniaxial stress levels (55, 84, 114 and 141 MPa) and examined using time-of-flight neutrons and simultaneous recording of acoustic emissions. At each stress level applied, a residual strain scan was performed around the sample axis and residual strains were determined. The residual compressional strain detected for quartz was -1.0 x 10^{-3}, the maximum value of residual strain approached 1.2 x 10^{-3}. The onset of acoustic emissions is an indicator of the previous in situ maximum stress. Comparison of acoustic emission data with applied and residual stress values at different load levels is the key to understand the freezing-in mechanism of residual stress and the Kaiser Effect in rock.

1. Introduction and sample characterization

The combination of neutron strain analysis and acoustic emission measurements aims to gain a better understanding of the physics behind the Kaiser effect [1]. ‘Kaiser effect’ describes the phenomenon that a material undergoing cyclic load/unloading with increasing stress level emits acoustic emissions only after the previous maximum stress (PMS) has been exceeded. If the recalled maximum stress under cyclic load equals the PMS, the Kaiser effect par excellence is confirmed. The experiments presented in this study are important to gain insight into the freezing-in mechanism of rock stress in situ. This process is closely related to the conservation of residual stress in rocks in general and the actual origin of acoustic emission during laboratory cyclic loading.

For the investigation of the physical properties of polycrystalline rocks the neutron diffraction has advantages due to the large penetration depth of neutrons [2, 3]. Time-of-flight neutrons allow the simultaneous registration of a large number of Bragg diffraction lines over a wide lattice plane d-spacing range. Hence, the texture diffractometer SKAT [4] and the strain diffractometer EPSILON-MDS [5] at the IBR-2M pulsed reactor at JINR (Dubna) were used for our investigations.
The gneiss sample investigated was taken from the Forsmark area (Sweden) and was selected because of high residual stresses to be expected. Sample KFM01-D1 shows a foliation in the xy-plane and a coarse-grained granoblastic fabric consisting of quartz, plagioclase (oligoclase), alkali feldspar and biotite. The phase content has been determined by X-ray phase analysis on powder sample. Large quartz grains (< 5 mm in diameter), which exhibit undulose extinction, are surrounded by smaller recrystallized neoblasts (< 200 µm). The neoblasts show homogenous extinction, indicating post-deformational growth at high temperatures. The plagioclase is affected by intense hydrothermal alteration and widely replaced by fine-grained sericite and/or saussurite (Fig. 1).

**Fig. 1**: Microstructure of the gneiss sample KFM01-D1. Quartz-feldspar matrix: a) quartz (qz) grain showing undulose extinction (ue) and marginal recrystallization, b) saussuritized large plagioclase grain (ep - epidote), xy-plane.

2. Textures
For the texture evaluation from the time-of-flight patterns gained at SKAT the ‘Rietveld Texture Analysis’ (RTA) [6] was applied using the MAUD software [7]. In contrast to X-ray phase analysis, RTA revealed potassic feldspar (microcline) and slightly different volume fractions of the rock constituents (quartz 39%, oligoclase 42%, microcline 17%, biotite 2%; Fig. 2).

**Fig. 2**: Normalized neutron TOF diffraction pattern of the sample measured at SKAT. Blue color: experimental data, black color: approximated data. The line patterns below indicate peak positions.

The quartz texture is well-pronounced and characterized by a single maximum in the [0001] pole figure close to the z-axis and 120° pole density distributions of the other lattice directions around the [0001] cluster (Fig. 3). These are hints for post-
kinematic recrystallization. In contrast, the textures of the other phases are very weak, see example pole figures in Fig. 3.

Fig. 3: Textures of quartz (top), oligoclase, microcline and biotite (bottom). Lower hemisphere, equal area projection, linear scale contours.

3. Residual and applied strain

The residual strain data gained at Epsilon-MDS are related to a ground (< 62 μm) and heated powder of the sample serving as reference for the strain-free state. The cylindrical sample (d = 30 mm, l = 60 mm) has been rotated around the cylinder axis z to detect the residual strain in steps of 15° in the xy-plane (Fig. 4a). The residual strains vary between -1 x 10⁻³ and 1.2 x 10⁻³.

Fig. 4: a) Residual strain of the quartz (10-10), (10-11)/(01-11), (11-20), (01-12), (11-21) and oligoclase (20-1) lattice planes. The sample was rotated around the Z-axis in steps of 15°. b) Applied strain of the quartz (10-11)/(01-11), (11-20), (01-12), (11-21) and oligoclase (20-1) lattice planes. The Z-axis is the main stress direction (σ₁), the x-axis is oriented perpendicular to σ₂.

For applied strain measurements, inclination of the z-axis with respect to the incident beam was 45°, i.e., σ₁ and σ₃ were measured simultaneously with two detectors in opposite position [8]. The experiments were carried out at loads of 40, 60, 80 and 100 kN (55, 84, 114 and 141 MPa). The negative strain in z direction corresponds to Hooke’s law, the positive strain in x direction is lower and depends on Poisson’s ratio (Fig. 4b). Simultaneously, grain-scale cracking processes have been detected by eight piezoceramic transducers positioned on two rings around the z axis (preamplifier: 40 dB, filter: 95-850 kHz, threshold: 31 dB, sensor frequency: 200 kHz). In each cycle, the
onset of acoustic emission (cracking events) was close to the previous applied maximum stress. In Figure 5, the results for load step 40 kN (Fig. 5a) and 60 kN (Fig. 5b) are shown for reference.

**Fig. 5:** Force and rate of acoustic emissions versus time of uniaxial deformation experiment. Presented are only two of four loading steps: a) at 40 kN, and b) at 60 kN at previous maximum load.

4. Conclusions
The Kaiser effect on a granite rock sample has been investigated using uni-axial *in situ* deformation of the sample in four steps up to 100 kN. The strain analysis has been carried out in two perpendicular directions parallel to \(\sigma_1\) and parallel to \(\sigma_3\) using neutron TOF diffraction. Texture and residual strain measurements indicate that the material is characterized by a quartz recrystallization texture, and preserved residual strains. In the next step, the ratio of acoustic emission crack strain and applied and residual strain from neutron diffraction will be used together with pre- and post-mortem microstructural analysis of rock cores to identify the freezing-in mechanism of residual stresses and the physics of the Kaiser effect in rock.

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