A device for sample rotation under external load for the simultaneous measurement of strain and orientation dependent material properties by means of TOF neutron diffraction

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Abstract. The time-of-flight diffractometer EPSILON-MDS at the modernised pulsed reactor IBR-2M at Dubna is designed for the detection of residual and applied strains in polycrystalline materials. The instrument allows the recording of diffraction patterns with high resolution ($\Delta d/d = 5 \times 10^{-3}$ to the best) over a wide wavelength band ($\lambda_{\text{max}} = 7.8 \text{ Å}; d_{\text{max}} = 5.6 \text{ Å}$). Complicated polyphase materials like rocks consisting of minerals with large unit cells and low crystal symmetry can be investigated. The diffractometer is equipped with 81 detectors positioned behind nine radial collimators at a unique Bragg angle of $2\theta = 90^\circ$ for the central detector and at different radial angles allowing the simultaneous measurement of nine sample directions. A pressure device for the in situ investigation of intra-crystalline strain under uniaxial load up to 150 MPa on cylindrical samples is available. The macroscopic strain on the sample surface can be determined simultaneously by means of a laser extensometer. The sample can be rotated around its axis, i.e. limited information on texture can be obtained. The simultaneous strain and CPO measurements are helpful for a better understanding of the deformational behaviour of polycrystalline materials, e.g. to investigate elastic processes in rocks before, during and after rock failure.

1. Introduction
The moving plate boundaries of the Earth’s crust are marked by the concentration of volcanism and earthquakes with focus depths almost between 2 and 10 km, occasionally up to 60 km. Earthquakes may have enormous consequences for humanity as well as economics. That’s why countries like Japan, China, and Russia make large effort to understand the mechanisms of earthquake generation in order to predict earthquakes. Nevertheless, up to now, geoscientists agree that it will be impossible to
predict location, time, magnitude, focus depth or any other parameter of seismic events, because the processes of earthquake generation are poorly understood.

By the end of 2011, the reconstructed pulsed reactor IBR-2M of the Frank Laboratory of Neutron Physics at JINR (Dubna, Russia) will be available for physical experiments. The neutron time-of-flight (TOF) diffractometers EPSILON and SKAT are especially designed for strain and texture investigations on geological samples, which may open new possibilities for the investigation of the mechanical properties of rocks. In addition to the simultaneous investigation of the textural and strain development with time, it will be possible to determine the strain storage capacity and strain release characteristics of rocks. Information on the loading capacity of rocks in the deep crust is expected, as well as information on strain-stress redistribution processes acting within the crust during seismic activity.

The mechanical properties of rocks like the deformational behaviour are strongly influenced by the composition, the grain size, the grain size distribution, the grain orientation, the internal strain, the strain distribution and other parameters. Non-destructive methods are favoured to characterize the internal structure of materials. TOF neutron diffraction offers the possibility to measure the crystallographic preferred orientation and the internal strain on bulk samples with sample volumes up to several cm³, in addition, all diffraction peaks in the available range of lattice spacings can be measured at the same time. This is of interest for in situ load experiments in order to investigate the strain development and the strain relaxation, because the diffraction geometry has not to be changed in the course of the experiment. The TOF neutron diffraction technique has been successfully used to characterize the deformational behaviour and the structure of polyphase rocks [1, 2]. A pressure device for in situ texture investigations by conventional neutron diffraction method has been presented by [3].

2. TOF neutron diffraction as a tool for strain and texture determinations
The TOF neutron diffractometer EPSILON-MDS at beamline 7A of the pulsed neutron source IBR-2M at JINR (Dubna) has been designed for high resolution measurements of applied and residual differential strains \( \varepsilon = (d - d_0)/d_0 \), including in situ strain/stress experiments as well [4]. The research reactor IBR-2M generates neutron pulses with a frequency of 5 Hz, corresponding to a maximal accessible neutron wavelength of \( \lambda_{\text{max}} = 7.8 \) Å. This allows the investigation of large unit cells up to a maximal lattice spacing of \( d_{\text{max}} = 5.6 \) Å. Furthermore, a good spectral resolution of \( \Delta \lambda/\lambda = 5 \times 10^{-3} \) at \( d = 2\) Å is achieved, mainly due to collimation of the diffracted beam. The high resolution is advantageous for the investigation of polyphase materials with many Bragg reflections, whereas the wide \( d \)-range allows the investigation of materials with large unit cells. Both these properties apply to geological materials.

The diffractometer EPSILON-MDS (Fig. 1) is a multi-detector system equipped with 81 \(^3\)He-detectors. The detectors are arranged in nine banks, each bank locates behind a radial collimator. The detectors of a bank cover the 20-range from \( 82^\circ \leq 20 \leq 98^\circ \), with the central detector at \( 20 = 90^\circ \). The detector banks are arranged around the incident beam at radial angles of \(-21^\circ, 0^\circ, 21^\circ, 69^\circ, 90^\circ, 111^\circ, 159^\circ, 180^\circ, \text{and} 201^\circ\), i.e., the angular coverage is 222°. This arrangement allows the simultaneous detection of Bragg diffraction patterns for nine different sample directions. Because the individual detectors of a detector bank are arranged at slightly different Bragg angles and at different secondary flight paths, the data of the nine detectors will be summarized applying 'on-the-fly' time focusing method.

3. Pressure device and rotatable sample
For the investigation of applied load sample states, an electrically operated uniaxial pressure device is mounted on the sample goniometer (Figs. 1a, 1b). The pressure device allows the in situ investigation of intracrystalline strains to a force of up to \( F = 100 \) kN, corresponding to a pressure of \( p_{\text{max}} = 150 \) MPa [5]. The corresponding cylindrical sample dimensions are \( d = 30 \) mm and \( l = 60 \) mm. The deformation axis is oriented at \( 45^\circ \) with respect to the incident neutron beam. This allows the detection of the intracrystalline strain in different sample directions at the same time, e.g., in axial (detector 2) and radial direction (detector 8, Fig. 2). Poisson's ratio, \( \nu \), can be determined, which is strongly
depending on the sample composition and the crystallographic preferred orientations (CPOs = textures) of the constituents. An arrangement of incident beam diaphragms determines the gauge volume. Mostly, a gauge volume of 2 cm$^3$ is used.

The macroscopic strain on the sample surface is measured simultaneously. Using a laser extensometer, any sample-sensor contact can be avoided and the bulk strain can be determined with an accuracy of 0.5 μm. The combined measurement of macro stresses and micro stresses allows conclusions on the sample fabric, e.g., the pore volume. Acoustic emissions, generated by microcracks during deformation, can be detected in the course of the in situ deformation experiment (Fig. 1b). The intensity as well as the local and chronological distribution of acoustic emissions characterises the formation of microcracks.

Fig. 1a: The neutron time-of-flight diffractometer Epsilon-MDS for the in situ investigation of residual and applied strains, mainly of geological materials. 1b: Uniaxial pressure device ($p_{\text{max}} = 150$ MPa) with the deformation axis oriented at 45° with respect to the incident beam.

Fig. 2: Schematic sketch of the experimental geometry at the Epsilon-MDS.
A sufficient number of recorded sample directions is required to get information on the orientation dependence of applied and/or residual strain. For the investigation of residual strain and its distribution within the sample, the sample can be translated and/or rotated using a four-axis goniometer. The range of horizontal $x$- and $y$-translation is 120 mm, vertical translations up to 40 mm are possible ($z$-axis). The rotation axis $\Phi$ covers the whole $2\pi$ range. The accuracy of sample movements is 0.0025 mm and 0.0025°, respectively. For the investigation of applied strain, the pressure device has been equipped with a mechanical gear to rotate the sample under load in order to measure the strain at different orientations (Figs. 3, 4). Sample rotation can be done in arbitrary steps. This allows access to further orientation dependent information, which one can get by analysis of the neutron diffraction pattern relating the Bragg diffraction peak position for strain characterization and the Bragg diffraction peak intensity for the characterization of crystallographic preferred orientation. High precision of the rotation mechanics is required to avoid eccentric moments of force.

As the diffractometer EPSILON-MDS is optimized for strain/stress investigations, the suitability for crystallographic preferred orientation measurements is limited. The better CPO information can be obtained applying specialized equipment like the SKAT texture diffractometer [6], which is operated at the neighbouring beamline.

Fig. 3: Schematic sketch of the rotatable sample chamber inside the uniaxial pressure device.

Fig. 4: Photograph of the rotatable pressure device.
Tab. 1: Technical parameters of the pressure device for sample rotation.

| Motor                     | - High torque step motor with a micro step-by-step control  |
|---------------------------|-------------------------------------------------------------|
|                           | - maximal moment of a torque: 39.6 Ncm                      |
|                           | - resolution: < 0.36°                                        |
| gear mechanism            | - 3 stage gear                                              |
|                           | - overall multiplication factor: 100.3:1                    |
|                           | - stage 1: planetary gear with gear ratio 100:1, max. outgoing unit 2000 Ncm |
|                           | - stage 2: synchronically load distribution on the intermediate shaft, gear ratio 1:1 |
|                           | - stage 3: gear ratio 1.3:1                                 |
| encoder                   | - optical                                                   |
|                           | - directly flanged on the metering shaft                    |
|                           | - resolution: 500 pulses per rotation                        |
| Max. possible axial load  | $F = 265 \text{ kN}$, corresponding to $p = 400 \text{ MPa}$ |
| of the rotation plane     |                                                             |

4. Conclusions
Nondestructive intracrystalline strain investigations on geological materials can be carried out by means of neutron diffraction method. The description of the mechanical behaviour of polycrystalline materials requires the analysis of the crystallographic structure, especially the crystallographic texture and the intracrystalline strain. The combined analysis of applied and/or residual strain and texture can be done only by a rotatable sample in the deformation device. The strain storage capacity of mineral lattice planes, which have an effect on the crustal stability, can be determined by time-resolved experiments. Applied and/or residual strains under variable load states, e.g., cyclic loading of the sample, can be recorded.

The uniaxial pressure device at the neutron time-of-flight strain diffractometer EPSILON-MDS has been extended by a rotatable sample chamber. The deformation axis is oriented at 45° with respect to the incident beam, allowing access to a maximal number of sample directions by means of sample rotation, and to achieve minimal geometrical restrictions for the incident and backscattered beam paths. By this way, information on crystallographic preferred orientation of the rock constituents is obtained simultaneously to the time-depending variation of applied strain.

High precision of the rotation mechanics is required to avoid the eccentric moment of force. The simultaneous strain and CPO measurements are helpful for a better understanding of the deformational behaviour of polycrystalline materials, in particular rocks.

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