Texture and Structure Analysis of Metagabbro by Neutron Diffraction

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Abstract. Crystallographic preferential orientation (CPO) of metagabbro mylonite from the eastern part of Stare Mesto belt, Bohemian Massif, is investigated by means of neutron diffraction method. The experiments were performed on the KSN-2 neutron diffractometer situated at the research reactor LVR-15 in the Nuclear Research Institute, plc. Rez, Czech Republic. Based on the collected diffraction patterns, the orientation distribution function of crystalline grains is determined by Rietveld harmonic method for the principal mineral phases - amphibole and plagioclase - and used to calculate (001), (020), (021), (110), (111) (plagioclase) and (001), (111), (020), (110), (200) (amphibole) pole figures. Lattice parameters of the phases have been obtained from diffraction patterns of a powder specimen. Main features of the observed CPO are compared with results of electron back-scatter diffraction measurements performed formerly on the same sample and with other known data.

1. Introduction

A substantial part of the lower continental and oceanic crust is formed by metabasic rocks, a heterogeneous and anisotropic microstructural nature of which is expected to have a pronounced effect on seismic properties. Consequently, the studies of crystallographic preferential orientation (CPO, texture) of such rock materials are of a high theoretical and practical importance because, for instance, the obtained texture results might be used in models of seismic wave propagation, the results calculated using such models can be used in prediction of seismic effects and further correlated with the laboratory data obtained by ultra-sound pulse transmission (UPT) method [1-3].

Since about three decades ago, neutron diffraction (ND) is increasingly applied to CPO analysis of rocks. Comparing with the other techniques, such as X-ray diffraction, electron back-scatter diffraction (EBSD) or special microscopic techniques, the ND method provides the unique possibility to integrate over a large specimen volume. The later feature is necessary for examination of coarse-grained, significantly heterogeneous materials and leads to statistically highly significant CPO characterization (cf. reviews [4-6]).

In our study, we present the results of investigation performed by ND on sample of metagabbro mylonite rock from the eastern part of the Stare Mesto belt, Bohemian Massif. The sample is part of a larger series used in complex studies of the seismic behaviour of meta-gabbros from the Stare Mesto belt [7, 8]. Methodically, the presented study links up to our former research devoted to CPO and structure of rocks [9].
2. Experimental

2.1. Samples
The investigated mylonite metagabbro rock was sampled from the eastern metagabbroic sheet of the Stare Mesto belt located in the eastern part of the Bohemian Massif. The rock underwent dynamic deformation under amphibolite facies conditions [7]. Two specimen types were prepared and investigated by the ND method: a powder specimen used to refine the lattice parameters of the constituting minerals and a spherical sample for CPO analysis (50 mm in diameter, further referred as GK3; the particular shape and size is used with aim to facilitate a consecutive juxtaposition of the results obtained by ND and UPT methods). The powder sample was prepared by grinding the raw rock material in a small ball mill to the final powder of average grain size ca 10 μm (the estimate obtained by means of optical microscopy).

2.2. ND measurements
The ND experiments were performed on the KSN-2 neutron diffractometer situated at the horizontal channel of the research reactor LVR-15 in the Nuclear Research Institute, plc. Rez, Czech Republic. The main experimental parameters were as follows: the wavelength of the monochromatic neutron beam $\lambda = 0.1362$ nm, the ultimate resolution $\Delta d/d = 7 \times 10^{-3}$ at the diffraction angle $2\theta = 19^\circ$, the diffraction patterns recorded within the $2\theta$ range 7.5° < 80° with $\Delta 2\theta = 0.1^\circ$. For the powder specimen measurement, the grinded rock material was placed in a cylindrical vanadium container. The bulk GK3 specimen employed in CPO characterization was fixed in a three axes texture goniometer and the latter was used to set the specimen gradually into 83 different positions covering uniformly one full hemisphere of the specimen orientations. At every specimen position, the full ND diagram was recorded. The coordination system of the GK3 specimen (which the texture analysis results are referred to) is defined by the normal to the plane of foliation (NF), the lineation direction (LD) and the third rectangular direction completing the right-handed set of axes.

3. Results
All the obtained ND patterns were corrected for non-linear background and then evaluated using the Rietveld method implemented in the software package GSAS [10]. At first, the powder specimen data were used to refine the structure parameters of the plagioclase (labradorite structure, triclinic space group C-1, further referred as Phase I) and the amphibole (monoclinic space group C2/m, referred as Phase II). Then, the GK3 specimen data were refined to reveal the CPO of the phases. The following procedure was applied in the latter case: At first, the scale factors, background coefficients and lattice parameters were determined for all diffraction patterns corresponding to the individual GK3 orientations. Then, the fixed average values of the lattice parameters were used to refine the orientation distribution functions (ODFs) of crystallites of both mineral phases. The ODF representation by spherical harmonics was employed (P1 sample symmetry, the harmonic expansion order limit L = 8).

The standard statistical descriptors related to the refinement process are given in Table 1. The calculated lattice parameters are summarized in Table 2. It is apparent that the refinement of the textured specimen GK3 converged to the final state statistically comparable with the fit of the powder specimen. There is also no significant difference between the lattice parameters obtained for both specimens. The calculated values of the lattice constants lie well within the common range expected upon the particular type of the mineral structures [11, 12].
Table 1. Statistical parameters of the Rietveld refinement.

| Specimen | Rwp (%) | Rp (%) | $\chi^2$ |
|----------|---------|--------|----------|
| Powder   | 4.25    | 3.40   | 1.88     |
| GK3      | 4.41    | 3.43   | 3.31     |

Table 2. Calculated lattice parameters.

| Specimen | Phase | a (Å)   | b (Å)   | c (Å)   | $\alpha$ (deg) | $\beta$ (deg) | $\gamma$ (deg) |
|----------|-------|---------|--------|---------|----------------|---------------|----------------|
| Powder   | Phase I | 8.146(5) | 12.83(6) | 7.110(5) | 93.76(6) | 116.22(4) | 89.89(4) |
|          | Phase II | 9.833(4) | 17.99(5) | 5.291(3) | 90      | 105.01(6) | 90           |
| GK3      | Phase I | 8.154(3) | 12.82(5) | 7.104(2) | 93.55(6) | 116.18(7) | 89.77(5) |
|          | Phase II | 9.837(4) | 18.01(3) | 5.296(6) | 90      | 105.00(5) | 90           |

Figure 1. Calculated pole figures of the Phase I (plagioclase).
Pole figures (PFs) of plagioclase (Phase I) and amphibole (Phase II) re-calculated from the corresponding ODFs are shown in figure 1 and figure 2, respectively. Scale of the contour lines is given in multiples of the random density (m. r. d.). Orientation of the stereographic projection is as follows: the NF points to the 'north' and the LD direction is in the 'zenith' of the projection. Thus, the foliation plane corresponds to the horizontal (equatorial) intersection of the projection.

In case of the Phase I (plagioclase), the PFs provide evidence about a complex multi-component CPO. Within the reconstructed PFs, the highest grain orientation population along LD and NF belongs to (021) and (001) poles, and (111) poles, respectively.

In case of the Phase II (amphibole), two distinguished texture components can be identified. The common feature of them is orientation of grains with the (001) pole parallel to LD, but they differ in orientation of the [100] and [010] directions. They can be considered as transient sub-types of the

**Figure 2.** Calculated pole figures of the Phase II (amphibole).
amphibole texture characterized by (001) poles parallel and (hk0) poles normal to LD observed with glaucophanite samples deformed under eclogite facies conditions and compatible with the (hk0)[001] slip system [13]. The more pronounced of the two components features orientation of the [010] direction within the plane of foliation and the [100] along the NF direction. It can be identified with the 'Type 1' texture introduced in [14] and agrees well with the results obtained by the EBSD method [7]. The second, weaker component shows [010] and [100] orientation out of the foliation plane, the directions inclined respectively ca. 30 and 60 degrees from NF and normal to LD. The second amphibole texture component was not observed in the upper mentioned EBSD measurements.

4. Conclusion

Neutron diffraction structure and texture analysis has been applied to investigate CPO of mylonite metagabbro sample. The obtained structural parameters agree well with the values expected for the particular forms of the plagioclase and amphibole minerals forming the rock. The results of the performed texture analysis suggest two-component nature of the CPO of the amphibole phase and a multi-component texture of the plagioclase phase. The amphibole CPO is in general features compatible with the published data and extends the results observed by EBSD method. Taking in account that the amphibole CPO is known to control anisotropy of ultrasound transmission [15], presence of the additional second texture component can possibly influence on the calculated seismic wave velocities of the rock and might correct to the formerly found discrepancy between the measured and calculated UPT data [7]. Verification of the latter effect will be subject of further research.

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5. References

[1] Pros Z, Babuska V 1968 Studia geoph. et geod. 12 192
[2] Mainprice D 1990 Computers and Geoscience 16 385
[3] Ullemeyer K, Siegesmund S, Rasolofosaon P N J, Behrman J H 2006 Technophysics 414 97
[4] Leiss B et al 2000 J. Struct. Geol. 22 1531
[5] Schäfer W 2002 Eur. J. Mineral. 14 289
[6] Kockelmann W, Chapon L C, Radaelli P G 2006 Physica B B385-386 639
[7] Baratoux L 2004 Petrology, Deformation Mechanisms, and Fabric Anisotropy of Metabasites Deformed at Natural Strain and Metamorphic Gradient - PhD Thesis (Prague: Charles University)
[8] Machek M 2011 Relation of Pore Space Geometry, Permeability and Microstructure in Low Porosity Rocks - PhD Thesis (Prague: Charles University)
[9] Vratislav S, Dlouha M, Kalvoda L 2009 Zeitschrift für Kristallographie Supl. 30 195
[10] Von Dreele R 1997 J. Appl. Cryst. 30 517
[11] Ribbe P H 1975 Chemistry, Structure and Nomenclature of Feldspars Feldspar Mineralogy (Reviews in Mineralogy vol 2 2nd edition) ed P H Ribbe (Chantilly: Mineralogical Society of America) chapter 1 pp 1-12
[12] Hawthorn F C 1981 Crystal Chemistry of the Amphiboles Amphiboles and Other Hydrous Pyrobes - Mineralogy (Reviews in Mineralogy vol 9A) ed D R Veblen (Chantilly: Mineralogical Society of America) chapter 1 pp 91
[13] Zucali M, Chateigner D, Dugnani M, Lutterotti L, Ouladif B 2002 Quantitative texture Analysis of Glaucophanite Deformed under Eclorige Facies Conditions (Sesia-Lanzo Zone, Western Alps): Comparison between X-ray an Neutron Diffraction Analysis Deformation Mechanisms, Rheology and Tectonics: Current Status and Future Perspectives (Geological
Society, London, Special Publications vol 200) ed S de Meer, M R Drury, J P H de Bresser, G M Pennock (The Geological Society of London) pp 239-253

[14] Leiss B, Groeger H R, Ullenmayer K, Lebit H 2002 Textures and Microstructures of Naturally Deformed Amphibolites from the Northern Cascades, NW USA: Methodology and Regional Aspects Deformation Mechanisms, Rheology and Tectonics: Current Status and Future Perspectives (Geological Society, London, Special Publications vol 200) ed S de Meer, M R Drury, J P H de Bresser, G M Pennock (The Geological Society of London) pp 219-238

[15] Barruol G, Kern H 1996 Physics of the Earth and Planetary Interiors 95 175