Revisiting elastic anisotropy of biotite gneiss from the Outokumpu scientific drill hole based on new texture measurements and texture-based velocity calculations

H.-R. Wenk a,⁎, R.N. Vasin b, H. Kern c, S. Matthes b, S.C. Vogel d, T.I. Ivankina b

a Department of Earth and Planetary Science, University of California, Berkeley, CA 94720, USA
b Frank Laboratory of Neutron Physics, Joint Institute for Nuclear Research, Joliot-Curie 6, 141980 Dubna, Moscow Region, Russia
c Institut f. Geowissenschaften, Universitaet Kiel, Olshausenstr. 40, D-24098 Kiel, Germany
d Department of Earth and Planetary Science, University of California, Berkeley, CA 94720, USA

⁎ Corresponding author. Fax: +1 510 643 9980.
E-mail address: wenk@berkeley.edu (H.-R. Wenk).

1. Introduction

Many rocks composing the Earth’s crust are anisotropic with respect to propagation of seismic waves (e.g. Lloyd et al., 2009). This anisotropy is particularly evident in rocks containing significant fractions of phyllosilicates. Most obvious are shales which have been studied in great detail because of their relevance for seismic prospecting (e.g. Johansen et al., 2004). In shales an alignment of phyllosilicates occurred during sedimentation, compaction and diagenesis. In metamorphic rocks alignment of mica was produced during crystallization under stress and ductile deformation. The alignment of platelets of phyllosilicates in shales, slates and schists is very strong, compared to other mineral fabrics such as calcite, quartz and feldspar, with maxima often exceeding ten times random distribution (m.r.d.) (e.g. Wenk et al., 2010a). While much is known about preferred orientation in monomineralic rocks such as marble, limestone, quartzite and dunite, information on polynemineralic rocks is more limited, even though they compose most of the Earth’s crust.

There has been particular interest in characterizing rocks from continental drilling projects to explore the composition of the deep continental crust and relate fabric properties to geophysical aspects such as seismic wave propagation. It has led to improvements in experimental techniques such as the advancement of neutron diffraction methods to determine mineral preferred orientation patterns on large samples that are representative of bulk rocks (e.g. Kern and Wenk, 1990; Siegesmund et al., 1994). This was initiated with the KTB (Kontinentales Tiefbohrprojekt) in Bavaria, Germany (Kern et al., 1991; Siegesmund et al., 1993), followed by the Kola deep drilling project, Russia (Ivankina et al., 2005; Kern et al., 2001). More recently the Outokumpu project in Finland was initiated (Kukkonen et al., 2006) and produced a wide range of samples of metamorphic rocks which were analyzed in detail, including mineral composition, microstructure, preferred orientation and seismic anisotropy (e.g. Kern et al., 2008).

For us a sample of biotite gneiss from 818 m depth was of most interest (Kern et al., 2008). This sample was analyzed for preferred orientation at the time-of-flight (TOF) neutron scattering facility of the Joint Institute for Nuclear Research (JINR) in Dubna, Russia, with the SKAT diffractometer. On the same material acoustic velocities were measured with different instruments at Kiel University, Germany and at the Institute of Geology AS CZ in Prague, Czech Republic. The two different velocity measurements show excellent agreement. However, elastic anisotropy of P-waves predicted from elastic tensor averaging based on crystallographic texture data (7.9%) is far lower than that which was measured (13–15%), even at 200 MPa confining pressure (Table 1 in Kern et al., 2008). Is this discrepancy caused by inadequacies in texture measurements, or by data analysis? Is it...
caused by the averaging scheme? A simple Voigt average used in the work may not be applicable for composites with platy particles. These issues were addressed by reanalyzing the old Dubna Outokumpu data with modern methods, and by measuring the same sample at a different neutron scattering facility, the HIPPO texture diffractometer at Los Alamos. We then apply self-consistent averaging methods to explore the importance of grain shape and conceivably residual microporosity. We assume that the reader is familiar with the previous papers (Kern et al., 2008, 2009) which give details about composition and microstructures of the sample as well as texture measurements and seismic velocity determinations.

The sample is a homogeneous biotite gneiss with pronounced foliation and lineation (Fig. 1). It is composed of 39.9 vol.% quartz, 37.4 vol.% andesine plagioclase and 22.6 vol.% biotite, as determined by mass balance calculations (Kern et al., 2009, Table 2). This table also illustrates that mass balance volume fractions are virtually identical with those based on independent neutron diffraction data analysis as will be discussed later (Section 2). Fig. 1 in Kern et al. (2008) was used to estimate grain dimensions. The thickness of biotite platelets perpendicular to the foliation Z is about 5–200 μm. Biotite grains are elongated subparallel to the lineation direction X, ranging in length from 0.4 to 2 mm. In the intermediate direction Y, lengths are between 0.4 and 1 mm. Based on this average, aspect ratios of 1:0.2:0.05 were assumed. Quartz and plagioclase are more or less equiaxed with an average grain size of 0.1 mm. Thus a cube of 1 cm³ contains about one million grains. There are no alternating quartz-, plagioclase- and biotite-rich layers to be seen in thin sections of the investigated sample (see Fig. 1 in Kern et al., 2008). The platy and elongated minerals (biotite, quartz), defining foliation and lineation, are almost isolated and do not form compositional layers

2. Neutron diffraction texture analysis

A round robin project established that neutron diffraction is the favorite method of texture analysis for fairly coarse-grained rocks (Wenk, 1991). The reason is the high penetration depth of thermal neutrons, which allows analyzing large volumes, rather than surfaces with a conventional X-ray pole figure goniometer or electron backscatter-diffraction, thus acquiring the necessary grain statistics for the quantitative texture analysis of all mineral components. Neutron texture analysis has become firmly established and has usually been performed with monochromatic neutrons at reactor facilities. More recently polychromatic neutrons at pulsed sources have been employed, with the possibility to measure diffraction spectra simultaneously, based on resolution of the time-of-flight (TOF) of diffracted neutrons (e.g. Wenk, 2006). In a TOF diffraction experiment polychromatic neutrons with a range of wavelengths λ scatter on the sample and the detector, positioned at an angle θ to the primary beam, measures the time t it takes the neutron to travel from the source to the detector at a distance L. Bragg’s law can then be formulated as d=(hλ)/(2mL sin θ), where d is the lattice spacing, h is Planck’s constant and m is the mass of the neutron. Since all parameters are constant, except for t, the d-spacing is directly proportional to t (TOF). We will show such spectra below.

A first TOF diffractometer, NSHR, dedicated to texture analysis was developed at the Joint Institute for Nuclear Research in Dubna, Russia, with neutrons produced by the pulsed nuclear reactor IBR-2 (e.g., Walther et al., 1995). It was followed up by the newer diffractometer SKAT (Ullemeyer et al., 1998). The thermal neutron pulse width is 320 μs and the flightpath is 100 m. Another TOF diffractometer is HIPPO at LANSCE, Los Alamos, which uses neutrons produced by a spallation source (Wenk et al., 2003). For HIPPO the pulse width is 20–30 μs (Mocko et al., 2011) and a flightpath of 10 m produces about the same energy resolution as for SKAT. For details about the instruments the reader is referred to these papers.

Both systems have many detectors, 19 in case of SKAT on a single ring at 2θ=90°. In the case of HIPPO 720 3He detector tubes are arranged on 30 panels (with the panels referred to as “detectors” in the following discussion), distributed over three rings at different Bragg angles (2θ=40°, 90° and 140°). On both instruments, each detector records spectra originating from differently oriented crystals. Fig. 2 displays the pole figure coverage for SKAT (Fig. 2a) and HIPPO (Fig. 2b). The size of the symbols in this equal area projection indicates the angular range each detector sees. For SKAT it is ~3° due to the use of parallel collimators on detectors with a divergence 90°±0.15° (Ullemeyer et al., 1998). For HIPPO detectors cover a much larger area with the advantage of increasing scattering intensity. For HIPPO the pole figure and lattice resolution also depend on the scattering angle of detectors. For 140° detectors the maximum angular range is 10°, for 90° detectors it is 15°, and for 40° detectors it is 19° (Matthies et al., 2005). Information from all detectors is used for the data analysis.

In order to improve the pole figure coverage, the sample is rotated around a single axis on both instruments. In the case of SKAT the axis is oblique and is in the horizontal plane, at an angle of 45° to the incident beam. The sample in the SKAT goniometer is rotated to 72 positions in 5° increments, producing 72×19=1368 spectra (Fig. 2c). In the case of HIPPO the rotation is around a vertical axis perpendicular to the incident neutron beam to 4 positions resulting in 4×30=120 spectra (Fig. 2d). The coverage for SKAT is more regular than for HIPPO but not uniform, with a much higher density of points in the center of the pole figure, which may introduce artifacts (Matthies and Wenk, 1992). For HIPPO it is just the opposite: coverage in the center is very poor and only viewed by low-resolution 40° detectors (see also Fig. 4c in Matthies et al., 2005). The total of the angular area covered by detectors is about 10% for SKAT and 40% for HIPPO.

Samples are of roughly cubic shape, in the case of SKAT more than 100 cm³ and in the case of HIPPO 2.5 cm³. The cubes have average edges of 4.6 cm and 1.3 cm. Thus the travel distance for diffracted neutrons along the shortest and the longest paths in the sample is 2.3 and 4.0 cm for SKAT and 0.65 and 1.12 cm for HIPPO. While the ratio is the same, the attenuation along 4 cm is substantially different from 2.3 cm, and SKAT, with large samples, could be biased toward higher intensity along the axes of the cube and weaker intensities along the diagonals unless this is corrected by the data analysis. In this case no absorption corrections were performed. Thus, even though the HIPPO sample was cut from the same large SKAT cube which appears homogeneous, they are not identical and there may be small variations on pole figures.
Fig. 3 shows single diffraction patterns for a 90° detector of both instruments plotted as function of d-spacing with peaks corresponding to diffractions on lattice planes. In TOF experiments intensity typically decreases with increasing d-spacing. Counting times for SKAT were 0.5 h per sample orientation (and 0.5 × 72 = 36 h for the whole measurement) versus 2 h per sample orientation for HIPPO (resulting in 4 × 2 = 8 h for the whole measurement). The thermal neutron flux at the sample position is about $10^6 \text{neutrons cm}^{-2} \text{s}^{-1}$ for SKAT and $2.4 \times 10^7 \text{neutrons cm}^{-2} \text{s}^{-1}$ for HIPPO. It is obvious that counting statistics are much better for HIPPO (Fig. 3b) than for SKAT (Fig. 3a), even though the SKAT sample was much larger. The main reason for the difference is attributed to detector size and collimation in the case of SKAT. Based on neutron flux, sample size and counting time the number of neutrons scattered by the sample is very similar but only a few reach detectors in the case of SKAT. The spectra in Fig. 3 have been normalized with scattering from a calibration standard, giving more intensity to high d-spacings. Here we will discuss data acquired on SKAT in 2006 and on HIPPO in 2010.

TOF diffraction spectra such as those in Fig. 3 are routinely analyzed with the Rietveld method using the program MAUD (Lutterotti et al., 1997) and hands-on procedures described in Wenk et al. (2010a). We will apply this method to both data sets available from SKAT and HIPPO. For the Rietveld method information about the crystal structure of the mineral phases is introduced as “crystal information files” (cif). For quartz we use Hazen et al. (1989, amc015462, spacegroup P3121, conventional setting for right-handed quartz), for andesine Fitzgerald et al. (1986, amc001053, spacegroup P1), and for biotite Brigatti et al. (2000, amc002386, spacegroup C2/m). For quartz, handedness does not affect diffraction intensities but the correct setting needs to be used to ensure rhombohedral reflections have the correct intensities such as $1011 > 0111$ and $1012 > 0112$ for neutrons. Biotite is monoclinic and for texture analysis the first setting ($Z' = [001]$, parallel to the 2-fold axis) needs to be used (Matthies and Wenk, 2009). This means that (100) is the cleavage plane and not (001) as for the second setting, which is more conventional in the mineralogic literature. In both cases the correct setting of the coordinate system is of extreme importance. In further modeling of the elastic properties, all the tensors are set in this crystal coordinate system. Also, the Brigatti et al. (2000) biotite uses a 10 Å d-spacing for (100) planes while others use a 20 Å spacing.

It should be noted that so far SKAT data were always analyzed with the conventional pole figure method, extracting intensities from individual diffraction peaks. This is inefficient if whole spectra are available as in the case of TOF. It is particularly limited for samples with many overlapping diffraction peaks as for this gneiss sample. Positions of diffraction peaks are indicated by the tick marks below the spectra in Fig. 3b. For pole figure extraction generally non-overlapping peaks are chosen. Here we reanalyze the old data (Kern et al., 2008) with the Rietveld method. In order to transfer standard SKAT data into MAUD a simple software SKAT2MAUD was developed which transforms SKAT data files into ASCII-format that can be loaded into MAUD with correct pole figure angles and correct monitor coefficients.

For this comparative analysis we choose a d-range from 1.5 to 4.3 Å in case of HIPPO and 1.1 to 3.5 Å in case of SKAT where the high d-spacing range is further restricted because of poor counting statistics.
The Rietveld method refines with a least squares method lattice parameters, phase volume fractions (Table 1) and orientation distributions for all phases to obtain the best fit with all the included diffraction spectra. For HIPPO the results are based on data from 120 spectra, whereas for SKAT the number of spectra was 1368. No atomic positions or individual thermal motion parameters were refined. The presence of 0.1% of iron sulfide (Kern et al., 2008, 2009) in the material was neglected. Phase fractions shown in Table 1 compare well with Kern et al.’s (2008) estimates based on mass balance calculations. In contrast to point counting and image analysis on thin sections, the mass balance calculation is based on large rock volumes (about 300g). After repeated homogenization and volume reduction, the chemical analyses used for the calculation thus represent the bulk rock. Importantly, with mass balance calculations heterogeneities and shape preferred orientation (SPO) are taken into account. For texture analysis we apply the E-WIMV method (Matthies and Vinel, 1982). For HIPPO data we use an angular grid of 7.5° for biotite with a strong texture, 10° for quartz with a weaker texture, and 15° for plagioclase with an almost random orientation distribution. For the analysis of SKAT data, an angular grid of 5° has been used for all three phases.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Kern et al. (2009)</th>
<th>SKAT</th>
<th>HIPPO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biotite</td>
<td>22.6</td>
<td>23.0</td>
<td>20.0</td>
</tr>
<tr>
<td>Quartz</td>
<td>39.9</td>
<td>39.5</td>
<td>40.7</td>
</tr>
<tr>
<td>Plagioclase</td>
<td>37.4</td>
<td>37.5</td>
<td>39.3</td>
</tr>
<tr>
<td>Biotite (100) max</td>
<td>8.7</td>
<td>21.2</td>
<td>23.2</td>
</tr>
<tr>
<td>Biotite (100) min</td>
<td>0.32</td>
<td>0.10</td>
<td>0.03</td>
</tr>
</tbody>
</table>

Fig. 3. Individual TOF spectrum of one 90° detector. (a) SKAT and (b) HIPPO. Dots are measured data and line is the Rietveld fit. Diffraction peak locations for individual phases are indicated at the bottom. Arrows in (a) point to biotite peaks used in the original pole figure extraction and adjacent quartz diffraction peaks.
From orientation distributions selected pole figures for biotite and quartz were calculated and are represented in Figs. 4–6. For pole figures we use the same Cartesian coordinate system as applied by Kern et al. (2008) with X = lineation and Z = pole to foliation (Fig. 1). All pole figures are projected on the X-Y plane. Pole densities are expressed in multiples of a random distribution (m.r.d.).

On these figures we show the new textures obtained by the Rietveld method from SKAT data (Figs. 4b, 5b and 6b) and from HIPPO data (Figs. 4c, 5c and 6c) and compare them with the old textures obtained from individual pole figure extraction (Figs. 4a, 5a and 6a) (Kern et al., 2008). The old orientation distributions were obtained with the program BEARTEX (Wenk et al., 1998) with the WIMV algorithm and using (11 1), (22 0) and (10 00) for biotite (monoclinic first setting, 20Å 001 d-spacing); (2 0 1), (1 1 1), (13 0), (2 2 0), (13 1), (15 0) and (2 4 0) for plagioclase; (10 10), (11 20) and (02 21)+(20 21) for quartz. These textures (Kern et al., 2008, Fig. 4) were rotated to bring the pole to the foliation (Z) into the center.

For Rietveld analysis, biotite (100) pole figures (monoclinic first setting) are similar, though with a somewhat stronger and broader maximum for HIPPO (23.1 m.r.d.) than for SKAT (21.2 m.r.d.) (Fig. 4 and Table 1). They are much stronger than those reported previously (8.7 m.r.d., Kern et al., 2008). The pole figures are considerably asymmetric with the biotite (100) maximum inclined 20° to the foliation normal and of elliptical distortion.

Quartz textures (Fig. 5), especially (10 11) and (01 11) pole figures, are similar and display a strong trigonal variant selection. The old (00 01) pole figure is stronger than the Rietveld pole figures (1.8 vs. 1.4 m.r.d.).

Feldspar textures vary (Fig. 6). HIPPO pole figures are basically random. Old feldspar pole figures display a strong (100) maximum in Z (>1.8 m.r.d) and SKAT-Rietveld pole figures are more similar to HIPPO-Rietveld pole figures. While old data show a (001) maximum in Y, the SKAT-Rietveld data show an (001) maximum in Z, which is absent in the HIPPO results. We will discuss these differences and likely reasons for them below.

3. Discussion of texture results

The analysis of the Outokumpu gneiss sample with a different instrument and different data analysis produced some revealing results. Contrary to the single phase limestone round robin experiment (Wenk, 1991), there are big differences indeed. Using the Rietveld method on the old SKAT data greatly increased the texture strength for biotite compared with the individual pole figure method. For quartz the results are similar. The reason is that for this polymineralic rock with three major phases, there is a multitude of overlapping diffraction peaks (Fig. 3) which prevents a satisfactory individual pole figure extraction. This is particularly critical for poor counting statistics which makes it very difficult to estimate the background on each spectrum and the background is directly related to texture strength. This effect is more pronounced in the case of weakly scattering minerals or minerals with low volume fraction such as biotite. The (100) pole figure maximum of biotite was originally 8.7 m.r.d. (Fig. 4a) and increased to 21.2 m.r.d., using the same diffraction data (Fig. 4b). More significantly, in the old biotite texture there was a large random component (0.32 m.r.d. in the (100) pole figure) which is reduced to 0.1 m.r.d. with the Rietveld analysis, and correspondingly the texture maximum is much larger.

The biotite texture obtained with HIPPO has an even stronger (100) maximum (23.2 m.r.d.) and a lower minimum (0.03 m.r.d.) (Table 1), with a large region below 0.25 m.r.d. Similar to SKAT, the biotite maximum is asymmetric to foliation and lineation, but it is less elongated. It is difficult to decide if this elongation is due to better coverage and resolution of the SKAT diffractometer, due to the much...
larger sample size, or if it is an artifact. Clearly, counting statistics with HIPPO are about a factor of ten better than for SKAT which improves the statistical analysis on which the Rietveld method relies (Toby, 2006). Based on SKAT data, pole figures for (010) and (001) are very similar, with a maximum in the lineation direction (X). For HIPPO the pole figures are distinctly different with a (010) maximum in X and a (001) maximum in Y (Fig. 4).

For quartz pole figures are rather similar (Fig. 5). This is not surprising because quartz contributes intense peaks to the diffraction spectrum. The quartz texture based on peak extraction appears stronger (Fig. 5a) than based on the Rietveld analysis (Fig. 5b,c). This can again be attributed to erroneous background estimates, in this case higher, because of contributions from neighboring peaks. It is assuring to see similar patterns for rhombohedral pole figures (1011) and (0111). These diffraction peaks occur at the same d-spacing and are thus overlapped. They are separated based on diffraction intensity differences. Normals to (1011) are close to the elastically softest direction in quartz and normals to (0111) planes close to the stiffest direction. It appears that they have distinct distributions which are commonly observed in metamorphic rocks (e.g. Pehl and Wenk, 2005; Wenk et al., 2010b), but the patterns are quite asymmetric relative to foliation and lineation. It is not impossible that this pattern could have been produced by mechanical Dauphiné twinning during the drilling operation (e.g. Tullis, 1970).

The case is most confusing for plagioclase. HIPPO data suggest a very weak texture, close to random (Fig. 6c). Pole figure extraction suggests a sharp maximum of (100) in Z and the Rietveld analysis of SKAT data a (001) maximum in Z. Plagioclase is a triclinic mineral with many diffraction peaks and it is a relatively weak scatterer. It is almost impossible to separate individual peaks and adequate Rietveld analysis can only be done with excellent counting statistics (e.g. Xie et al., 2003). Thus HIPPO data are probably most realistic. This would agree with observations in other deformed granitic rocks, where mica show strongest textures, quartz moderate textures and plagioclase is close to random (e.g. Pehl and Wenk, 2005).

A glance at a SKAT diffraction spectrum (Fig. 3a) and the large number of overlapping diffraction peaks (bottom of Fig. 3b) clearly identify this gneiss sample as a complex material, where it would be extremely difficult to isolate individual diffraction peaks. In fact the (120) biotite peak (at d = 2.64Å), used for the pole figure extraction, is right next to the strong (1120) quartz peak and several plagioclase peaks. The (500) biotite peak at 2.02Å is next to the (2001) quartz peak. These peaks are indicated by arrows in Fig. 3a. It highlights the advantage of the Rietveld texture analysis which relies on the full diffraction spectra, rather than on individual peak intensities for each phase.

As far as the raw data are concerned, both instruments have advantages and disadvantages. SKAT has very poor counting statistics (Fig. 3a) which makes it difficult to establish a background function, especially for high d-spacings. If backgrounds are underestimated, it results in lower texture strength because part of the background intensity is added to the diffraction peak, especially for phases with weak diffraction peaks such as biotite. On the other hand, the much larger sample size at SKAT produces better grain statistics but attenuation may cause problems. We should keep in mind that for texture analysis the sample volume has to be representative for a homogeneous region.

SKAT pole figure angular resolution in excess of <3° (Fig. 2c) may be exaggerated for most texture analysis, especially of geological materials. The collimation geometry and the excellent angular resolution of this instrument are more suitable to investigate extremely strongly textured aggregates, such as misorientations of bicrystals (e.g. Marks et al., 2004). Also, SKAT 90° detectors demonstrate better d-resolution than HIPPO 90° detectors; the strong quartz (1011) reflection at 3.4Å is much sharper for SKAT (Fig. 3a) than for HIPPO (Fig. 3b). The irregular detector coverage for HIPPO (Fig. 2b,d) imposes limitations. Particularly
the central region of the pole figure is only covered by low resolution 40° detectors.

The high resolution for SKAT comes at the cost of reduced counting statistics. For crystallographic textures, not just of geological materials, but metals and ceramics as well, generally a 5°–10° resolution is satisfactory, but for textures it is essential to have an accurate estimation of background to obtain accurate sample direction dependent peak intensities and accurately assess the random component in orientation distributions (known as “phon”). This random component is critically important for the derivation of mechanical and physical properties.

A newer version of SKAT on the modernized IBR-2 reactor will become available to users in the future and is expected to have a several times higher flux at the sample due to a new neutron guide. At that time it may be interesting to launch another round robin experiment on the same sample to explore the differences, e.g. in the biotite (100) pole figures (Fig. 4). For texture analysis, rather than misorientation studies, it may be advantageous to use at SKAT fewer sample rotations but longer counting times.

For HIPPO more detectors have been added since these measurements were done (120°, 60° banks) but for high resolution results and more regular pole figure coverage, it may be advantageous not just to rotate about a single axis but also tilt the sample. A new robotic sample changer with more degrees of freedom for sample positioning will become available in the future.

4. Elastic property calculations

Elastic properties of polycrystalline materials have been calculated based on averages over orientation distributions of components. Simple averages, assuming constant strain (Voigt, 1928) or constant stress (Reuss, 1929) establish bounds that may be similar for polycrystalline aggregates composed of elastically fairly isotropic and equiaxed crystals but can vary greatly in cases when grains possess high elastic and shape anisotropy such as sheet silicates. Often an arithmetic (Hill, 1952) or geometric (Reuss, 1929) mean is used. They are reasonably successful in predicting elastic properties of non-porous metals (e.g. Turner and Tomé, 1993) and there are numerous applications to rocks from Crosen and Lin (1971) and Babuska (1972) to Hurich et al. (2001) and Ji et al. (2004). Depending on the system, a particular average works better, indicating that important ingredients are missing. These averages have been used for a long time because they are simple and straightforward. None of them take grain size, grain shape, grain connectivity or porosity into account and have therefore largely failed to explain elastic anisotropy in phyllosilicate-rich porous shales (e.g. Hornby et al., 1994; Kanitpanyacharoen et al., 2011; Valcke et al., 2006; Voltolini et al., 2009). The influence of additional factors on elastic properties, such as compositional layering, chemical composition of constituent phases, defect structures in crystals as well as alteration has been discussed by Ji et al. (2003).

More sophisticated averaging methods have been developed. Some are based on Eshelby’s (1957) self-consistent concept of ellipsoidal anisotropic inclusions in a homogeneous medium (e.g. Castaneda and Willis, 1995; Willis, 1977). Recently, finite element models have been employed (e.g. Shen et al., 1994; Srinivasan et al., 2008). There are several applications of self-consistent methods to shales (e.g. Sayers, 1994) and phyllosilicate-rich schists (Le Ravalec and Guéguen, 1996; Nishizawa and Kanagawa, 2010; Nishizawa and Yoshino, 2001). Here we are using a version of self-consistent averaging developed by Matthies (2010, 2012).

This GMS-method (‘GEO-MIX-Self’) is a multiphase generalization of the conventional self-consistent approach that uses the Eshelby’s tensor. The circumstance (usually not mentioned in literature) is that in the case of non-spherical and not strongly orientation fixed inclusions there are two (stiffness C- or compliance S-related)
The elastic properties for albite are close to monoclinic, with (010) as mirror plane. For plagioclase the symmetry of elastic properties is close to hexagonal, though there is a distinct maximum in $Y'$. Note that plagioclase in the Outokumpu gneiss is andesine, not albite but#$\Sigma^{\alpha}$-quartz at ambient conditions. This is easily available for quartz and we use stiffness coefficients from Heiliger et al. (2003) for $\alpha$-quartz at ambient conditions. For plagioclase experimental data that assume monoclinic symmetry are available (Ryzhova, 1964). Recently elastic constants of triclinic albite were measured with modern techniques, suggesting a much higher elastic anisotropy (Brown et al., 2006). This has been confirmed by ab initio simulations (Militzer and Kaercher, unpublished) and we use here the simulated results. Also for biotite only old experimental data exist, assuming hexagonal symmetry (Aleksandrov and Ryzhova, 1961) and we use ab initio values for ideal phlogopite (Militzer and Kaercher, unpublished). Single crystal stiffnesses are listed in Table 2 and the corresponding elastic wave propagation surfaces are shown in Fig. 7. The wave surfaces express the crystal symmetry.

The elastic properties for albite are close to monoclinic, with (010) as mirror plane. For plagioclase the symmetry of elastic properties is close to hexagonal, though there is a distinct maximum in $Y'$. Note that plagioclase in the Outokumpu gneiss is andesine, not albite but#$\Sigma^{\alpha}$-quartz at ambient conditions. This is easily available for quartz and we use stiffness coefficients from Heiliger et al. (2003) for $\alpha$-quartz at ambient conditions. For plagioclase experimental data that assume monoclinic symmetry are available (Ryzhova, 1964). Recently elastic constants of triclinic albite were measured with modern techniques, suggesting a much higher elastic anisotropy (Brown et al., 2006). This has been confirmed by ab initio simulations (Militzer and Kaercher, unpublished) and we use here the simulated results. Also for biotite only old experimental data exist, assuming hexagonal symmetry (Aleksandrov and Ryzhova, 1961) and we use ab initio values for ideal phlogopite (Militzer and Kaercher, unpublished). Single crystal stiffnesses are listed in Table 2 and the corresponding elastic wave propagation surfaces are shown in Fig. 7. The wave surfaces express the crystal symmetry.

For orientation averaging standard conventions have to be used (i.e. $Z' = -c$, $Y' = (c \times a)$, $X' = (Y' \times Z')$; where $X', Y', Z'$ are axes of the right-handed Cartesian crystal coordinate system and $a, b, c$ are crystal axes). For monoclinic phlogopite, the first setting ($Z' = -c = [001]$) applies (Matthes and Wenk, 2009).

For this publication we show elastic property calculations based on HIPPO texture data and compare some results with SKAT data obtained with the Rietveld method. For the Outokumpu gneiss different polycrystal averages over composing phases biotite, quartz and plagioclase were used, taking experimentally determined phase volume fractions into account (Table 1). Results of polycrystal averages for stiffness coefficients are listed in Table 3 for wave velocity minima and maxima, as well as P-wave anisotropy in Table 4. P-wave anisotropy (in percent) is defined as $A = 200 \times (P_{\text{max}} - P_{\text{min}})/(P_{\text{max}} + P_{\text{min}})$ or $A = 200 \times (P_{\text{max}} - P_{\text{min}})/(P_{\text{max}} + P_{\text{min}})$ where $P_{\text{max}} = 300 \times (P_{\text{max}} - P_{\text{min}})/(P_{\text{max}} + P_{\text{min}})$. The reason is that, especially for asymmetric textures, an average velocity is difficult to define. Thus values in Table 4 deviate slightly from those in Table 1 of Kern et al. (2008). Wave surfaces for P-waves are displayed in Fig. 8 and for shear-wave splitting in Fig. 9. For some of these calculations and representations programs in the BEARTEX package were used (Matthes, 2008). Others rely on new computer programs described below. The sample coordinate system is defined as $X$, lineation and $Z$ perpendicular to the foliation (Fig. 1) and corresponding to the pole figures (Figs. 4–6). To be consistent, we use for all averages a bulk density of 2.75 g cm$^{-3}$ when calculating velocities from stiffness coefficients (Table 3).

Several simple averages were calculated (Reuss, Voigt, Hill). First, for each phase, single crystal elastic tensors are averaged over corresponding orientation distributions. Then the polycrystal averages for each phase are combined, taking volume fractions into account. Here we show the results for the Voigt stiffness average (arithmetic mean), both for orientation average and phase average, which produces a P-wave anisotropy of 11.1% (Tables 3 and 4, Model A) (Figs. 8A, 9A).

Next we advance to a self-consistent average which allows to consider grain shape and porosity and satisfies both stress (upper bound) and strain equilibrium (lower bound). For the self-consistent averaging the program GeoMixSel3 (Matthis, 2010) was used. This program can average three phases, one of which is an anisotropic matrix (with shapeless or effectively “spherical” grains) into which two other anisotropic phases with ellipsoidal shapes are embedded. For each phase the crystal orientations in the sample are given by the orientation distribution, based on the Cartesian crystal coordinate system $XYZ$ where $Z' = [001]$ and $Y' = pole to [010]$ (cf. Fig. 7).

In GeoMixSel3 it is assumed that the axes of the shape ellipsoids are parallel to these Cartesian coordinates. This is not the case for plagiopipite/biotite where platelets are parallel to (100) (first setting) and inclined about 10° to the $X'$ axis. Thus rotations are required in order to prepare the input data for GeoMixSel3 for this case. First the single crystal elastic tensor is transformed into the ‘nonstandard’ ellipsoid coordinate system (program ELACInKE). Secondly the program (FGtoOMEGA) transforms the lattice orientation distribution $f(\Omega)$ into the corresponding shape orientation distribution $F(\Omega)$.

Before performing self-consistent averages, elastic properties of the quartz–plagioclase matrix are calculated using the geometric mean approximation (Matthis and Humbert, 1995). Since for these two phases the single crystal anisotropies are moderate and their textures are weak, the resulting matrix is only weakly anisotropic. The self-consistent averaging in GeoMixSel2, based on the exact Eshelby solution that describes the elastic fields for an inclusion in a homogeneous but anisotropic medium, proceeds until convergence is reached. A first self-consistent average assumes equiaxed biotite grains in a homogeneous anisotropic quartz–plagioclase matrix (Model B). The resulting P-wave anisotropy increases slightly to 11.4% (cf. Tables 3, 4, and

**Table 2**

Single crystal elastic stiffness coefficients. All coefficients are in standard setting ($Z' = -c$, $Y' = (c \times a)$, $X' = (Y' \times Z')$, where $X', Y', Z'$ are Cartesian system for (c) and $a, b, c$ are crystal axes). Values are in GPa. Elastic stiffnesses of phlogopite and albite are simulated (Militzer and Kaercher, unpublished); quartz elastic coefficients are from Heiliger et al. (2003).

<table>
<thead>
<tr>
<th></th>
<th>Phlogopite</th>
<th>Quartz</th>
<th>Albite</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\rho$ g cm$^{-3}$</td>
<td>2.80</td>
<td>2.65</td>
<td>2.61</td>
</tr>
<tr>
<td>C11</td>
<td>46.60</td>
<td>87.26</td>
<td>63.60</td>
</tr>
<tr>
<td>C12</td>
<td>17.15</td>
<td>65.7</td>
<td>31.70</td>
</tr>
<tr>
<td>C13</td>
<td>15.85</td>
<td>11.95</td>
<td>26.80</td>
</tr>
<tr>
<td>C14</td>
<td>0</td>
<td>-17.15</td>
<td>5.00</td>
</tr>
<tr>
<td>C15</td>
<td>0</td>
<td>-3.70</td>
<td>-0.60</td>
</tr>
<tr>
<td>C16</td>
<td>2.27</td>
<td>0</td>
<td>-0.60</td>
</tr>
<tr>
<td>C22</td>
<td>177.50</td>
<td>C11</td>
<td>159.20</td>
</tr>
<tr>
<td>C23</td>
<td>38.40</td>
<td>C10</td>
<td>14.90</td>
</tr>
<tr>
<td>C24</td>
<td>0</td>
<td>-C14</td>
<td>-10.40</td>
</tr>
<tr>
<td>C25</td>
<td>0</td>
<td>0</td>
<td>-6.00</td>
</tr>
<tr>
<td>C26</td>
<td>-22.90</td>
<td>0</td>
<td>-6.00</td>
</tr>
<tr>
<td>C33</td>
<td>178.50</td>
<td>105.80</td>
<td>165.70</td>
</tr>
<tr>
<td>C34</td>
<td>0</td>
<td>0</td>
<td>3.90</td>
</tr>
<tr>
<td>C35</td>
<td>0</td>
<td>0</td>
<td>7.00</td>
</tr>
<tr>
<td>C36</td>
<td>-6.88</td>
<td>0</td>
<td>-8.70</td>
</tr>
<tr>
<td>C44</td>
<td>69.80</td>
<td>57.15</td>
<td>28.30</td>
</tr>
<tr>
<td>C45</td>
<td>-12.90</td>
<td>0</td>
<td>-1.40</td>
</tr>
<tr>
<td>C46</td>
<td>0</td>
<td>0</td>
<td>-5.00</td>
</tr>
<tr>
<td>C55</td>
<td>12.90</td>
<td>C44</td>
<td>22.75</td>
</tr>
<tr>
<td>C66</td>
<td>0</td>
<td>C14</td>
<td>0.10</td>
</tr>
<tr>
<td>C66</td>
<td>12.95</td>
<td>(C11-C12)/2</td>
<td>34.15</td>
</tr>
</tbody>
</table>

For orientation averaging standard conventions have to be used (i.e. $Z' = -c$, $Y' = (c \times a)$, $X' = (Y' \times Z')$; where $X', Y', Z'$ are axes of the right-handed Cartesian crystal coordinate system and $a, b, c$ are crystal axes). For monoclinic phlogopite, the first setting ($Z' = -c = [001]$) applies (Matthes and Wenk, 2009).
Fig. 8B). By introducing a platelet shape for biotite (with ellipsoid axes 0.05:1:0.2), the anisotropy increases significantly to 13.2% (Tables 3 and 4, Model C, Fig. 8C). The P-wave anisotropy is still slightly lower than the experimental values at 200 MPa (15.3%). Therefore a final step was to introduce micropores of similar shape as biotite platelets but five times thinner (with ellipsoid axes 0.01:1:0.2). It is assumed that pores are parallel to biotite platelets (same orientation distribution) (Model D). Assuming a 0.1% volume fraction of pores the anisotropy reaches 15.7% (Tables 3 and 4, Model D, Table 4 and Fig. 8D).

The calculated patterns for shearwave splitting are similar as for P-waves with a distinct minimum and a girdle perpendicular to it (Fig. 9). As shape and porosity are introduced the amount of shear-wave splitting increases (Table 4, Fig. 9A–D).

Table 3

<table>
<thead>
<tr>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td>C11</td>
<td>119.01</td>
<td>106.79</td>
<td>106.69</td>
<td>105.50</td>
</tr>
<tr>
<td>C12</td>
<td>23.01</td>
<td>22.10</td>
<td>22.37</td>
<td>21.54</td>
</tr>
<tr>
<td>C13</td>
<td>20.61</td>
<td>20.30</td>
<td>20.68</td>
<td>19.38</td>
</tr>
<tr>
<td>C14</td>
<td>−1.04</td>
<td>−0.81</td>
<td>−0.67</td>
<td>−0.88</td>
</tr>
<tr>
<td>C15</td>
<td>1.19</td>
<td>1.07</td>
<td>1.13</td>
<td>1.15</td>
</tr>
<tr>
<td>C16</td>
<td>0.38</td>
<td>0.50</td>
<td>0.77</td>
<td>0.87</td>
</tr>
<tr>
<td>C22</td>
<td>22.73</td>
<td>22.60</td>
<td>22.04</td>
<td>21.44</td>
</tr>
<tr>
<td>C24</td>
<td>−5.03</td>
<td>−5.08</td>
<td>−5.44</td>
<td>−0.59</td>
</tr>
<tr>
<td>C25</td>
<td>−0.69</td>
<td>−0.68</td>
<td>−0.63</td>
<td>−0.60</td>
</tr>
<tr>
<td>C26</td>
<td>0.34</td>
<td>0.41</td>
<td>0.56</td>
<td>0.67</td>
</tr>
<tr>
<td>C33</td>
<td>96.57</td>
<td>85.43</td>
<td>82.71</td>
<td>78.12</td>
</tr>
<tr>
<td>C34</td>
<td>−1.41</td>
<td>−0.76</td>
<td>−0.75</td>
<td>−1.38</td>
</tr>
<tr>
<td>C35</td>
<td>0.26</td>
<td>0.19</td>
<td>0.21</td>
<td>0.25</td>
</tr>
<tr>
<td>C36</td>
<td>0.30</td>
<td>0.05</td>
<td>−0.09</td>
<td>−0.04</td>
</tr>
<tr>
<td>C44</td>
<td>40.24</td>
<td>34.14</td>
<td>32.61</td>
<td>31.75</td>
</tr>
<tr>
<td>C45</td>
<td>0.68</td>
<td>0.55</td>
<td>0.47</td>
<td>0.48</td>
</tr>
<tr>
<td>C46</td>
<td>−0.43</td>
<td>−0.39</td>
<td>−0.30</td>
<td>−0.28</td>
</tr>
<tr>
<td>C55</td>
<td>39.67</td>
<td>33.37</td>
<td>31.93</td>
<td>31.25</td>
</tr>
<tr>
<td>C56</td>
<td>−2.77</td>
<td>−2.98</td>
<td>−3.18</td>
<td>−3.31</td>
</tr>
<tr>
<td>C66</td>
<td>45.98</td>
<td>40.13</td>
<td>39.65</td>
<td>39.23</td>
</tr>
</tbody>
</table>

5. Discussion of the elastic properties

Using the old Dubna pole figure data and old elastic properties for biotite a Voigt average produced an anisotropy of 7.9% which is strikingly different from 15.3% measured on a cubic sample and 13.0% measured on a sphere (cf. Table 1 in Kern et al., 2008) but adjusted for the different definition of “anisotropy.” This was the stimulus for our reinvestigation of the Outokumpu sample. The application of a more advanced texture analysis (Rietveld method) improves that discrepancy. But even with the stronger HIPPO biotite texture, only an anisotropy of 10.4% is obtained by the Voigt approximation (Table 4).

The ability of this method is demonstrated in Matthies (2012) for the description of the elastic properties of a sample of reactor graphite, consisting of extremely anisotropic and a complicated pore structure. Taking into account a flat shape for biotite grains, a better agreement is obtained (12.6% P-wave anisotropy).

The anisotropy increases if the mentioned −10° declination correction...
of the ellipsoids relative to the Cartesian crystal coordinate system is taken into account.

Note that the Voigt average is an upper bound and therefore the corresponding P-wave velocities are greater than those for the self-consistent averages (different scales are used in Fig. 8A and B–D). In averaging and calculating the velocities from elastic stiffness coefficients, there are some uncertainties. As mentioned above we have used the stiffness coefficients for phlogopite and albite, because reliable values for biotite and andesine are not available. This will somewhat affect magnitudes, but hardly the anisotropies. Also, we use the same shape for all biotite grains and pores. In reality it is a distribution which may depend on orientation.

In order to match the polycrystal averaging elastic properties with those of the acoustic measurements at 200 MPa, it is necessary to introduce small amounts of pores, even for a metamorphic rock such as gneiss. After the addition of 0.1% pores parallel to the biotite flakes a better agreement is obtained (Table 4, 14.9%). This suggests that in the velocity measurements, even at 200 MPa, not all pores are closed. Interestingly, at 600 MPa the P-wave anisotropy was reduced to 13.2% (Table 5 in Kern et al., 2009) which is identical to the self-consistent value without pores. It suggests that perhaps elastic properties at 200 MPa do not reflect the properties of the rock matrix (intrinsic properties) but some cracks may exist even at a pressure of 200 MPa. Christensen (1974) has shown that small amounts of low aspect ratio cracks are not completely closed, even at pressures of 1 GPa and cracks may also exist in crustal rocks at depth (Rasolofosaon et al., 2000).

Model E is closely related to Model D, except that it uses the orientation distribution for biotite, obtained from SKAT data with the Rietveld method (Fig. 4b). Stiffness coefficients are similar (Table 3) but P-wave anisotropies are substantially reduced (Table 4, 11.4%), even though the biotite (100) maximum for SKAT (21.2 m.r.d.) is very similar to that for HIPPO (23.2 m.r.d.) and is sharper, with a narrow width at half-maximum of only 8° versus 15° for HIPPO. This sharp orientation distribution is smoothed by the fourth-rank elastic tensor (Fig. 7). The reason for the lower anisotropy for SKAT data is the much higher random component (0.10 versus 0.03 m.r.d. in biotite (100) pole figures, Table 1).

So far we have only discussed the “anisotropy” of the P-waves along the sample coordinate axes X,Y,Z where acoustic measurements were performed. But we should also discuss the impact of the texture
asymmetry on the anisotropy of P-wave velocity surfaces. This is best done by comparing data in Figs. 4 and 8, and Table 4. Like biotite (100) pole figures, P-wave surfaces are inclined. The broad minimum of P-waves corresponds to the maximum in the (100) pole figure and is inclined about 20° away from the sample direction Z. The maximum is very close to X. Since the minimum is a broad depression and the maximum is aligned with a sample direction X, the anisotropy A = 200×(P_X − P_Z)/(P_X + P_Z) corresponds very closely to the true anisotropy based on actual maximum and minimum (Table 4). However, velocities in the Y direction are much more ambiguous and a small sample rotation has a significant effect. The plane with velocity maxima does not correspond to the sample coordinate X-Y plane that was defined as “foliation”. This explains the relatively low value for measured P-velocities in Y (Table 4).

Velocities on this sample were also measured on a sphere (Fig. 3 in Kern et al., 2008). In this figure the velocity pattern looks symmetrical and their coordinate system is therefore not quite the same as that of the cube on which velocities have been measured, or the symmetry of the pole figures. The measured P-velocity surface for 200 GPa is very similar to that for Model D (Fig. 8) if this rotation of coordinate system is taken into account.

For shear waves there is no shear-wave splitting in the Z direction, both for model and experiment (Table 4) and there is maximum shear-wave splitting in the X direction, in contrast to the experiment where it is in Y. As for P-waves, the Y direction is complex because of the asymmetry (Fig. 9) and weaker splitting is observed than would be expected. The Y shear-wave velocities depend critically on sample alignment and the asymmetry may be enhanced by a more complex crack distribution (Ivankina et al., 2005). It also should be noted that, though the modeling based on HIPPO and SKAT data give very similar values of elastic constants and similar symmetry of P-wave propagation surfaces (Fig. 8), the shear wave splitting predicted from HIPPO measurements is significantly stronger than that of SKAT (Fig. 9). This suggests that shear-wave measurements in multiple directions may be required to correctly determine elastic properties of such complex materials.

The porosity required to explain the P-wave velocity anisotropy is relatively small (<0.1%). We can not determine if this is the intrinsic porosity which is also present in the deeper crust, or if it has been introduced during sample excavation and sample preparation and did not close at 200 GPa. Porosities in the 5–10% range occur in shales. Thus differences between measured and calculated velocities (based on orientation distributions) are much larger (e.g. Karatapanicharaen et al., 2011; Voltonlini et al., 2009; Wenk et al., 2008) and in shales the significance of pores and microstructures is much more important (Anderson et al., 1974; Dewhurst and Siggins, 2006; Hornby et al., 1994). It raises concerns, both about the value of experimental data, as well as the applicability of different averaging models (e.g. Ji et al., 2003). It appears that both experiments and models are necessary to define the true parameters which contribute to the bulk elastic properties of the material (e.g. Popp and Kern, 1994). Averaging methods based on textural and microstructural features are not sufficient, even though there is a direct link.

6. Conclusions

The investigation of the preferred orientation of component minerals in a homogeneous biotite gneiss without compositional layering and no alteration retrieved from the Outokumpu borehole in Finland at 818 m depth confirms the advantages of neutron diffraction for texture analysis of relatively coarse-grained polymorphanic rocks. But this new analysis of an old sample also cautions that applications of sophisticated instruments do not always provide correct answers. The Rietveld method, which relies on full diffraction spectra, is essential to separate overlapping peaks and provide quantitative orientation distributions in polymorphanic rocks. The SKAT diffractometer at JNIR has the possibility to measure very large samples to achieve representative grain statistics and possesses high resolution that may be necessary to define very sharp grain orientation distributions. The HIPPO diffractometer at LANSE has better counting statistics which is a prerequisite for most quantitative texture studies, and makes it possible to perform measurements in reasonable time. The weakness of HIPPO is the irregular and sparse pole figure coverage (Fig. 2b) and for SKAT, poor counting statistics (Fig. 3a). Knowing the textures of the minerals biotite, quartz and plagioclase, the anisotropic elastic properties of the gneiss have been calculated. A comparison with experimental velocity measurements reveals that even for a compact, low porosity material as a gneiss examined at 200 MPa, observed velocities can only be matched with models that include grain shape and assume a small percentage of cracks, confirming suggestions made by Kern et al. (2008). Such micro pores, associated mainly with phyllosilicates, may also be present in crustal rocks at depth and an important contributing factor to observed seismic anisotropy. Experiments and modeling give clear evidence that shape preferred orientation of platy minerals such as biotite contribute to bulk anisotropy in strongly foliated rocks. Because of its asymmetric texture and microstructure this sample is quite complex and was a good case to compare advantages and disadvantages of different methods.

Acknowledgments

We acknowledge access to the HIPPO diffractometer at the Lujan Neutron Scattering Center at LANSE in Los Alamos, which is funded by the Office of Basic Energy Sciences of DOE. HRW is appreciative for support from the NSF (EAR-0836402) and the DOE (DE-FG02-05ER15637), RV for support from the Federal Targeted Program “Scientific and Scientific-Pedagogical Personnel of the Innovative Russia”, and TI for support from the RFBR (10-05-00722-a). We thank B. Miltzer and P. Kaercher for letting us use unpublished single crystal elastic constants for plagiopelite and albite. We also are grateful to M. Savage and an anonymous reviewer for constructive comments that helped improve the manuscript.

References


