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Orthogonal switching of AMS axes during type-2 fold interference: Insights from integrated X-ray computed tomography, AMS and 3D petrography

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Abstract

We applied X-ray computed microtomography (μ -CT) in combination with anisotropy of magnetic susceptibility (AMS) analysis to study metamorphic rock fabrics in an oriented drill core sample of pyrite-pyrrhotite-quartz-mica schist. The sample is extracted from the Paleoproterozoic Martimo metasedimentary belt of northern Finland. The μ -CT resolves the spatial distribution, shape and orientation of 25,920 pyrrhotite and 153 pyrite grains localized in mm-thick metapelitic laminae. Together with microstructural analysis, the μ -CT allows us to interpret the prolate symmetry of the AMS ellipsoid and its relationship to the deformation history. AMS of the sample is controlled by pyrrhotite porphyroblasts that grew syntectonically during D1 in subhorizontal microlithons. The short and intermediate axes (K3 and K2) of the AMS ellipsoid interchanged positions during a subsequent deformation (D2) that intensely crenulated S1 and deformed pyrrhotite, while the long axes (K1) maintained a constant position parallel to the maximum stretching direction. However, it is likely that all the three AMS axes switched, similar to the three principal axes of the shape ellipsoid of pyrite porphyroblasts from D1 to D2. The superposition of D1 and D2 produced a type-2 fold interference pattern.

Keywords: microtomography; AMS; microtectonics; magnetic fabric; pyrrhotite; strain

1. Introduction

X-ray computed micro-tomography (μ -CT) is increasingly being applied in structural geology and ore petrology due to its ability to resolve the three-dimensional (3D) shape and spatial distribution of minerals and associated textures in metamorphic rocks (e.g., Sayab et al., 2015; Macente et al., 2017). Sulfides and oxides yield brighter gray values than rock-forming silicates owing to high X-ray

33 attenuation or higher density. They can be segmented or separated, for example, using the threshold
34 limits of the gray level histogram, and rendered and quantified in 3D volume (e.g., Sayab et al., 2016a;
35 Hanna and Ketcham, 2017). The resulting 3D views provide a spatial distribution of high-density ore
36 minerals (ranging from ca. 4 to 22 g/cm³) inside a given rock specimen, which is otherwise difficult to
37 determine using serial thin sectioning methods (Hayward, 1990; Aerden, 2003; Bell and Bruce, 2006).
38 The μ -CT technique is non-destructive and can be integrated with a number of conventional and
39 unconventional microstructural methods to investigate regions of interest in extraordinary detail
40 without destroying the whole sample (Kyle and Ketcham, 2015). As a holistic 3D visualization method
41 of analyzing rock fabrics (Sayab et al., 2015), μ -CT is ideal for integrating with measurements of bulk
42 rock anisotropy of magnetic susceptibility (AMS).

43 AMS measures the directional variations of magnetic susceptibility (K) present in a rock sample,
44 and the AMS magnitude ellipsoid defines the bulk rock orthogonal principal susceptibility axes (K1:
45 maximum, K2: intermediate, K3: minimum; Tarling and Hrouda, 1993). These axes can be correlated
46 with the finite strain axes ($X \geq Y \geq Z$) and structural features, for example mineral stretching and
47 intersection lineations, and, therefore, provide a mean to study deformation in 3D (e.g., Borradaile,
48 1988; Riller et al., 1996; Ferré et al., 2014; Parsons et al., 2016). However, since AMS measures the
49 sum of different magnetic carrier minerals in a sample, its interpretation requires knowledge of
50 amounts and relative abundances of these minerals, their magnetic susceptibilities, shape preferred
51 orientation (SPO), lattice preferred orientation (LPO) and relationship with tectonic fabrics (e.g.,
52 Kruckenberg et al., 2010; Borradaile and Jackson, 2010). Thus, a good understanding of the internal
53 microstructure behind AMS measurements is crucial. High-resolution μ -CT can fulfill this requirement
54 by allowing 3D visualization and determination of statistically volumetric abundances of magnetic
55 carrier minerals and their SPOs. We have explored this possible nexus using an oriented drill core of a
56 pyrrhotite rich micaschist sample that preserves two tectonic foliations. Apart from applying AMS and
57 μ -CT techniques, we studied the sample in three perpendicular thin sections oriented with reference to
58 the matrix foliation. Through this integrated approach, the deformation history of the sample is
59 reconstructed and considered in the regional tectonic framework as revealed by high-fidelity

aeromagnetic image (Fig. 1a). The application of non-invasive (AMS and μ -CT) followed by invasive (thin sections) methods illustrated herein offers a means of determining the magnetic lineation and foliation in ore-bearing rocks, which is highly relevant to the exploration of structurally controlled mineral deposits as will be briefly discussed.

2. Tectonic setting and sample description

The Martimo metasedimentary belt forms a part of the Peräpohja region, which lies in the center of the Fennoscandian shield. The area is characterized by lack of topography, where structural field relations in the 3rd dimension are difficult to reconstruct. Five incremental, but heterogeneously distributed deformation events have been proposed for the Martimo belt in a timeframe ranging from 1.92 to 1.77 Ga (Lahtinen et al., 2015). The belt hosts metapelites interbedded with metapsammites and preserves a sequence of near-orthogonal tectonic fabrics that are not fully developed elsewhere in the region (Lahtinen et al., 2015). In particular, the area is passing through a period of extensive geological research because of the recent discoveries of Rompas Au-U and Rajapalot Au (e.g., Nykänen et al., 2017; Ranta et al., 2015; Ranta et al., 2016). Metamorphism in the area is characterized by upper greenschist facies, however, the grade varies from low greenschist facies rocks in the southwest to kyanite bearing migmatites in the northeast (Hölttä and Heilimo, 2017).

An oriented sample (sample 57; Fig. 1; 66° 16' 53.43" N, 24° 29' 24.69" E) was drilled from the middle of the Martimo belt. The drill core sample is characterized by two near-orthogonal foliations that correspond to the D1 and D2 deformations of Lahtinen et al. (2015). S1 is subparallel to S0 (S0//S1) and on average strikes N-S. It is folded with E-W striking axial planes and gently east plunging axes, and overprinted by a steeply dipping, finely-spaced S2 foliation (Fig. 1b). The drill-core sample consists of pyrrhotite-pyrite-quartz mica schist with two distinct foliations (S1 and S2), and thus ideal for integrated μ -CT and AMS studies. Before extracting it from the bedrock using a portable mini-drill, the sample was marked with a north pointing groove on the top surface so that it could be reoriented in the lab. The sample has a 2.5 cm diameter and is 8 cm long.

3. X-ray computed micro-tomography (μ -CT)

3.1. Scanning configuration and processing

The sample was scanned using the high-resolution Phoenix|X-ray Nanotom 180 (GE) scanner at

the University of Helsinki. Before scanning the drill core, two brass pins were attached, about 40° from north counting towards the east for reference. The tungsten target X-ray tube was operated with an acceleration voltage of 140 kV and tube current 175 μ A. The radiation was filtered with 1.0 mm of Cu. Six hundred views per 360° were acquired, with 1.75 s total exposure time per view. The virtual 3D volume is composed of 1152 horizontal slices with a voxel size of 33 μ m $\times$ 33 μ m $\times$ 33 μ m. The projections were captured with a Hamamatsu flat panel sensor C7942SK-05. The 3D reconstructions were computed with *datos|x* – reconstruction software provided by Phoenix|X-ray.

3.2. 3D visualization

After reconstructing the 3D distribution of X-ray attenuations, the contrasting gray shades of pyrrhotite and pyrite allowed us to semi-automatically separate them from the rest of the silicate matrix by mapping gray levels to specific colors using Avizo software (<http://www.fei.com/software/avizo-3d-for-materials-science/>). Volume rendering further allowed to visualize the dataset in 3D (Figs. 2 and 3). Besides looking at the microtextures in 3D in all directions, it is possible to digitally cut the sample at any angle and to make certain surfaces transparent while rendering others opaque for better visual analysis. A virtual 3D tour inside the sample, at 33 μ m voxel resolution, allowed us to closely examine petrographic details and the spatial distribution of sulfides.

To show the penetrative nature and spatial distribution of pyrrhotite and pyrite grains, the drill core was virtually chopped at different horizontal (Fig. 2) and vertical (Fig. 3) levels. The 3D images clearly show that pyrrhotite is present throughout the core but localized within millimeter-scale metasedimentary laminae or bands (S0). Pyrite porphyroblasts are mainly localized along layers in the center (Figs. 3c,e). For better visualization from the top, two such layers were isolated and rendered in red and blue (Fig. 3g). The fold shape, defined by these layers, match the fold pattern observed in outcrop (cf. Fig. 1b).

Most of the pyrrhotite are standalone grains in the 3D visuals, whereas some pyrite grains are intergrown or touching each other (Figs. 2a and 3e). In horizontal and E-W striking vertical gray slices (Figs. 3a,f) as well as the 3D rendered images (Figs. 3c,e), pyrrhotite grains mimic the bulk finite

114 strain ellipsoid with their long (X) and short (Z) axes being aligned E-W and N-S, respectively.

115 Turning the core ($\sim 90^\circ$) and looking west, the pyrrhotite grains appear flattened or oblate in the Y-Z
116 plane (Figs. 3i,j; see also 7f).

117 3.3. *Segmentation of pyrrhotite and pyrite*

118 Segmentation of pyrrhotite and pyrite grains was performed with ImageJ and in-house written
119 software as follows. In order to reduce imaging noise, the reconstructed tomographic image was
120 filtered with variance weighted mean filter (noise variance = 500, radius = 2 pixels; Gonzalez and
121 Woods, 2002). After filtering, contrast to noise ratio between mineral crystals and background was
122 high ($> \sim 8$), and thus the crystals could be segmented using simple thresholding. The threshold value
123 was determined using Tsai's moment preserving thresholding method (Tsai, 1985). After thresholding,
124 the image contained pyrrhotite and pyrite grains as white regions, and additionally some small white
125 regions caused by imaging noise that is typically found in tomographic images and caused by various
126 processes such camera noise and variance in the count of X-ray photons received by the camera
127 (Stock, 2008). Regions corresponding to imaging noise were suppressed by removing all white regions
128 whose volume was less than 20 voxels, a cut-off that seemed to remove noise regions efficiently but
129 preserve even the smallest crystals. Finally, pyrite and pyrrhotite grains were separated manually based
130 on the characteristic cubic or parallelepipedic shape of pyrite.

131 Pyrrhotite grains were separated from each other using watershed segmentation (Meyer and
132 Beucher, 1990) seeded with local maxima of distance map. In order to avoid over-segmentation near
133 locations with multiple nearby local maxima, only the largest of them was taken to be a seed point.
134 The resulting segmentation was checked by visually comparing the segmented regions to the original
135 data. The quality of the segmentation was found to be satisfactory. Each segmented grain or crystal
136 was analyzed separately. Grain volumes were calculated as the number of voxels in the grain. The
137 orientations of long (X), intermediate (Y) and short (Z) axes were determined by counting voxels
138 along three mutually perpendicular principal axes within the grain (Pearson, 1901). The aspect ratio
139 was determined as the ratio between the lengths of the X and the Z axes, and the Flinn parameters were
140 calculated as ratios between the X and Y, and Y and Z, respectively.

Finally, the orientation data for all grains were plotted in rose diagrams for the horizontal plane

and for vertical planes (Figs. 4a,b and 5a,b). Pyrite grains were separated from each other and analyzed similarly as pyrrhotite grains, but applying a closing operation with radius of 5 pixels before watershed segmentation. A flowchart summarizing the image processing operations is shown in Appendix 1, and provided as an electronic supplement.

3.4. 3D quantitative analysis

We collected quantitative data for volume, surface area, aspect ratio (X/Z and Y/Z), and orientation of pyrrhotite and pyrite grains. The volume fraction in the whole rock sample obtained through full segmentation of pyrite and pyrrhotite is $3.5 \pm 0.3\%$ and $4 \pm 2\%$, respectively, where the uncertainty estimates have been made according to Füsseis et al., (2012). A total of 25,920 pyrrhotite and 155 pyrite grains were encountered but grains touching the edges or boundary of the sample were discarded due to incomplete size and axes attributes.

Pyrrhotite X-axes plunge shallowly ($\sim 22^\circ$) towards the east, whereas metasedimentary layers (S0) dips about 45° in the same direction (Figs. 3c-f and 4a,b). The mean aspect ratio and volume of the pyrrhotite grains are ~ 14 and 0.019 mm^3 , respectively (Figs. 4c,e). A small fraction of pyrrhotite grain (about 4%) have a much higher aspect ratio as others reaching a maximum value of 292 (Fig. 4c). The Flinn diagram portrays dominantly prolate shapes of pyrrhotite grains, although with a significant fraction of oblate shapes (Fig. 4d). We interpret the latter as representing pyrrhotite microboudins (see micro-textural description below). Aspect ratio increases with increasing grain volume (Fig. 4e).

The horizontal rose diagram for the long axes of pyrite grains (Fig. 5a) shows a wide range of trends with a main E-W maximum and weaker N-S maximum (Fig. 5a) that can be correlated with L_2^1 and L_1^0 , respectively. The rose diagram of Fig. 5b shows that the long axes (X) of pyrite grains generally pitch shallowly in both EW and N-S vertical sections. The 3D images show that the larger pyrite grains are confined to a single layer located in the middle of the sample (Figs. 3c,e). The mean aspect ratio of pyrite grains is about 4 with a smaller spread as observed for pyrrhotite grains. The Flinn diagram for pyrite grains indicates a mixture of highly prolate to highly oblate shapes (Fig. 5d), whereas the volume to aspect ratio plot reveals a slightly inverse relationship between grain volume

168 and aspect ratio (Fig. 5e). We now proceed to compare the above described 3D data for the shape and
169 orientation of the two main sulfide minerals in the studied sample with AMS fabrics and
170 microstructural observations in thin section.

171 **4. Anisotropy of magnetic susceptibility (AMS) analyses**

172 *4.1. Pyrrhotite, AMS and tectonic fabric*

173 Pyrrhotite (density 4.6 g/cm³) is a magnetically ordered (ferromagnetic) mineral that, at ambient
174 temperature, has its atomic magnetic moments arranged at the lattice scale (e.g., Borradaile and
175 Jackson, 2010). Because of this magneto-crystalline property, a correlation between the AMS and rock
176 deformation fabrics has been proposed as a proxy for 3D strain geometry (e.g., Parsons et al., 2016).
177 The shape of the AMS ellipsoid has been shown to be a function of the deformation history in a rock
178 and the response of minerals to deformation in terms of deformation mechanisms (e.g., Borradaile and
179 Jackson, 2004).

180 AMS is defined by a symmetric second rank tensor (Nye, 1957) that gives the ratio ($K = M/H$)
181 between an applied magnetic field (M) and the induced magnetization (H) in different directions. The
182 principal axes of the AMS ellipsoid or principal susceptibility axes, K_1 , K_2 , and K_3 , define the
183 magnetic foliation (K_1 - K_2 plane), and magnetic lineation (K_1 ; Tarling and Hrouda, 1993). The mean
184 susceptibility (K_m) is the average of K_1 , K_2 and K_3 (Janák, 1965). The corrected degree of anisotropy
185 (P') is a measure of the deviation of the AMS ellipsoid ($P' > 1$) from a perfect sphere ($P' = 1$). The shape
186 of the AMS ellipsoid can be defined as prolate ($T = -1$, $K_1 > K_2 = K_3$) or oblate ($T = 1$, $K_1 = K_2 > K_3$)
187 end members depending on the strain conditions and mineral behavior (e.g., Borradaile and Jackson,
188 2004).

189 It has been shown that AMS is mainly controlled by ferromagnetic and paramagnetic minerals in a
190 rock, whereas diamagnetic minerals have only a minimal effect (e.g., Borradaile and Jackson, 2010;
191 Ferré et al., 2014). Strong SPO and LPO of ferromagnetic minerals produce a magnetic foliation and
192 lineation that reflect the bulk rock fabric. The principal susceptibility axes (K_1 , K_2 , K_3) may or may
193 not correspond to finite strain axes ($X \geq Y \geq Z$), and depending on crystallographic properties and

deformation mechanism, an inverse or blended relationship can result other than the expected strain axes (Rochette et al., 1999; Ferré, 2002; Borradaile et al., 2012).

To identify the origin of AMS in a given rock sample, both ferromagnetic and paramagnetic minerals can be decoupled by measuring and plotting their thermomagnetic or susceptibility-temperature (K/T) curves and Curie points (Hrouda et al., 1997). The thermomagnetic curves represent the variation with temperature (heating and cooling effects) of magnetic susceptibility (Hrouda, 1994) and can be used to constrain the mineral species that are the magnetic carriers in a sample (e.g. Skyttä et al., 2010; Karell et al., 2014). A detailed account on the AMS and petrofabrics of deformed and metamorphosed rocks can be found in seminal reviews by Borradaile and Jackson, (2004, 2010).

4.2. Analytical methods

For this study, AMS and temperature variation of the magnetic susceptibility (K/T) experiments were performed at the Research Laboratory, Geological Survey of Finland in Espoo. The 2.5 cm × 8 cm (diameter × length) drill core (sample 57) that was partly used for the μ -CT analysis was cut into three pieces of 2.1 cm length each. AMS was analyzed with a KLY-3S Kappabridge, operating at a frequency of 875 Hz and with a field intensity of 300 Am⁻¹, while rotating each subsample about three mutually perpendicular axes to construct its magnetic susceptibility axes and determine the corresponding magnetic parameters. Thermomagnetic measurements were performed using CS-3 furnace apparatus (Agico, Inc.). The AMS and thermomagnetic data were processed with the Anisoft (version 4.2) and Cureval (version 8) programs, respectively.

4.3. AMS results

The mean magnetic susceptibility (Km) of the 3 subsamples has a high value ranging from 17703 to 23308 μ SI, whereas the corrected degree of anisotropy (P') varies from 1.81 to 1.94 (Fig. 6). The shape of the AMS ellipsoid (T) is clearly prolate with T values of -0.14 to -0.23. The thermomagnetic curve shows a typical pattern of pyrrhotite with an abrupt drop in magnetization at ~325°C (Hrouda et al., 1997), suggesting that AMS is mainly controlled by this mineral. The orientation of the magnetic foliation matches that of the east-west striking and subvertically dipping S2 foliation (Fig. 6d; cf. Fig. 1b), whereas the short axis (K3) is oriented perpendicular to this plane. The orientation of the triaxial

magnetic ellipsoid also coincides with the principal X, Y and Z axes of pyrrhotite determined through the μ -CT (cf. Figs. 3 and 4).

5. Petrographic and SEM analyses

Three thin sections were cut from the sample normal and parallel to the main S2 matrix foliation and L^1_2 intersection lineation. The horizontal thin section shows a penetrative, highly differentiated and finely-spaced S2 foliation striking east-west (Fig. 7a). The long axes (X) of most pyrrhotite grains are well aligned with this fabric (Fig. 7b). Pyrite grains are wrapped by S2 suggesting pre- or syn-D2 growth (Zwart, 1962; Passchier and Trouw, 2005). No inclusions of pyrrhotite were found inside pyrite, at least in these three thin sections. Some pyrrhotite grains appear microboudinaged in east-west direction (Fig. 7c) with the boudin necks filled with silicate minerals. As mentioned earlier, oblate pyrrhotite grains in the Flinn diagram of Fig. 4d probably represent microboudins of originally larger prolate grain, or alternatively, grains weakly deformed during D2 and retaining their original (syn-D1) oblate shape (Figs. 7e,f).

The vertical N-S section cut normal to S2 and the mineral stretching lineation reveals a high-angle relationship between S2 overprinting S1 (Figs. 7d-f) and a stage 4 crenulation cleavage morphology in the scheme of Bell and Rubenach (1983). An important observation in this section is that the Y-axes of pyrrhotite grains are parallel to S1 but truncated, deflected or even folded by S2 (Figs. 7e,f) indicating that this mineral grew prior to D2. Close examination of pyrrhotite grains reveals variable shapes ranging from flattened parallel to S1, to oval shaped parallel to S2, to microfolded with axial planes parallel to S2 (Fig. 7f). Pyrrhotite grains with Y- and Z-axes parallel and perpendicular to the S1 foliation, respectively, are relatively less deformed. Whereas, highly deformed pyrrhotite grains have Y- and Z-axes perpendicular and parallel to the S1 in the same thin section.

The section cut parallel to S2 (Fig. 8) shows the finely laminated layers already seen in the μ -CT images (Fig. 3), wherein pyrrhotite grains plunge shallowly (25°) towards the east (Fig. 8d). Pyrite grains have euhedral outlines and are associated with symmetric fibrous quartz strain fringes more or less aligned E-W with the stretching lineation and L^1_2 intersection lineation.

(EDS) analyses were performed at the Research Laboratory, Geological Survey of Finland in Espoo, using an Oxford Instruments EDS-spectrometer X-Max 80 mm² (SDD) attached to a JEOL JSM 7100F Schottky field emission scanning electron microscope (representative FE-SEM images and EDS spectra are provided in Appendix 2 as an electronic supplement). The analytical conditions were as follows: high vacuum mode, a COMPO back-scattered signal (BSE), 20 kV accelerating voltage and 0.5 nA probe current. FE-SEM and EDS analyses showed that almost all pyrite grains are partially to completely pseudomorphed by pyrrhotite (Fig. 9), indicating prograde heating resulting in sulphur release from pyrite (Craig and Vokes, 1993). Skeletal textures mark this pyrrhotite to pyrite replacement (Fig. 9b). Besides, we detected inclusions of euhedral arsenopyrite and anhedral sphalerite around the median and rim regions of pseudomorphed pyrite (Figs. 9a,b). EDS analyses, conducted in an FE-SEM, of both intact and microboudinaged grains confirmed their pyrrhotite composition (Figs. 9c,d). Some of the cubic-looking grains have rhombic shape possibly due to replacement of diagenetic anhydrite by pyrrhotite (Hall, 1982).

6. Discussion

6.1. Implications for the D1 related structures

Microstructures commonly exhibit complex overprinting relationships that are only partially reflected by outcrop-scale structures (Aerden, 1998; Sayab et al., 2016b). This counts in particular for Proterozoic belts, where glacially eroded bedrock and sedimentary cover exacerbate field observations in the 3rd dimension. Under such limited exposure conditions, oriented drill cores are useful to understand multiple fabric overprinting relationships and microstructures (Sayab, et al., 2015). As described earlier, sample 57 was extracted from a D2 low-strain zone of a flat-lying folded surface, where the east-west striking pervasive fabric marks the axial planar S2 foliation. High-resolution tomographic 3D images accompanied by a vertical east-west oriented thin section revealed that metasedimentary layers (S0) dips ~23° more steeply than the S1 foliation, whereas the intersection lineation (L_1^0) is north-south trending and shallowly plunging (Fig. 10). This structural relationship suggests that the sample comes from the normal limb of an F1 recumbent fold, in which deformation

274 was partitioned between top-to-the west shearing components and perpendicular shortening
275 components (Fig. 10), consistent with the east-vergent D1 thrusting and folding event of Lahtinen et al.
276 (2015).

277 The secondary north-south preferred orientation seen in the rose diagram of Fig. 5a suggests that
278 pyrite porphyroblasts were aligned N-S during D1. During D2 they would have developed their main
279 E-W preferred orientation (Figs. 5a and 11), probably associated with pressure solution as that is the
280 dominant deformation mechanism of pyrite in greenschist facies conditions (McClay and Ellis, 1983)
281 (Fig. 5b). Pyrrhotite X-axes do not retain a primitive N-S direction, which is expected because as a
282 much weaker mineral, it would have experienced much stronger deformation during D2 as pyrite (cf.
283 Craig and Vokes, 1993).

284 The shape and spatial distribution of pyrite and pyrrhotite grains could also be influenced by the
285 preferential growth in S1 microlithons or low-strain lenses, given the control of deformation
286 partitioning on metamorphic reactions in general (Bell and Hayward, 1991). Additionally, the growth
287 of sulfide minerals was concentrated in alternate metasedimentary layers, probably because of a
288 chemically more favorable composition (Figs. 3c,d,e).

289 Microtextural relationships show that arsenopyrite and sphalerite are cogenetic with pyrite,
290 whereas the latter was partially to completely replaced by pyrrhotite. Thermodynamic modelling of the
291 arsenopyrite-sphalerite-pyrite and pyrrhotite assemblage in closed-system metapelites (e.g., Lynch and
292 Mengel, 1995) indicate upper-greenschist facies conditions at intermediate pressures of 5.5-6.9 kbars,
293 and these P-T estimates can potentially be applied to D1 in the study area.

294 6.2. *Implications for the D2 structures*

295 The north-south oriented vertical thin section, cut perpendicular to S2 and normal to L_2^1 reveals a
296 high-angle relationship between S1 and S2 (Figs. 7e,f). We already showed that pyrrhotite and pyrite
297 porphyroblasts formed syn-D1 because pyrrhotite grains are aligned parallel to S1, but truncated and
298 deformed by S2 (Fig. 7f), whereas pyrite grains are wrapped by S2 and developed syn-D2 strain
299 fringes. We, therefore, infer two possible tectonic scenarios: 1) that the K3 axes of the AMS ellipsoid
300 (pyrrhotite porphyroblasts) changed from subvertical (normal to a thrusting-related S1) to gently

301 south-dipping normal to S2. In other words, the K2 and K3 axes interchanged positions during D2,
302 while K1 was maintained constant (Figs. 11a,b), 2) alternatively, the K1, K2 and K3 axes all
303 interchanged positions during D2, similar to the pyrite porphyroblasts where all three principal axes of
304 the shape ellipsoid switched from D1 to D2 (Figs. 5a and 11). Thus, the SPO of pyrrhotite during D1
305 depends on the geometry of the AMS axes (Fig. 11b).

306 We have argued that the magnetic foliation reflects an originally north-south striking and shallowly
307 east dipping orientation of S1 and of axial planes of F1 recumbent folds (Figs. 10 and 11b). We further
308 propose that progressive bulk inhomogeneous shortening associated with foliation development (Bell
309 and Rubenach, 1983) controlled the growth, orientation and AMS properties of pyrrhotite and pyrite
310 porphyroblasts (Fig. 11a). Both minerals grew during D1 in low-strain microlithons bounded by S1
311 cleavage planes as manifested by the preferred orientation of pyrrhotite parallel to S1. With the
312 development of S2, porphyroblast growth ceased and symmetric strain fringes grew off pyrite grains
313 parallel to the E-W maximum extension direction.

314 Aeromagnetic image and field observations around the Martimo area show a type-2 fold
315 interference pattern, where the east-west striking upright F2 folds deform east-vergent, shallowly
316 dipping F1 folds locally resulting in crescent-mushroom or arrowhead surface patterns (Fig. 1a). The
317 general fold pattern is characterized by a major synform in the south and antiform in the north
318 (Lahtinen et al., 2015). This regional scale geometry fits well with our interpretation of fabrics in the
319 studied sample, which comes from the core of the synformal D2 structure (cf. Figs. 1a, 10 and 11c). A
320 more extensive description of the deformation sequence can be found in Lahtinen et al. (2015). Our
321 integrated approach and workflow of combining μ -CT, AMS and microstructures can be highly useful
322 and applied to unfold the complex structural history, especially for the mineralized bedrock such as the
323 Peräpohja region.

324 6.3. Complementary character of AMS and μ -CT techniques

325 μ -CT quantitative data provide 3D information for metamorphic textures that significantly
326 complements and allow a more rigorous interpretation of AMS results. More specifically, μ -CT data
327 add statistical mineral abundances, spatial distribution, size and shape of different mineral species with

328 a precision that cannot be achieved with other methods. In the case that concerns us here, the magnetic
329 lineation (K1) was shown to coincide with the X-axis of pyrrhotite grain volumes (cf. Figs. 4a,b and
330 6d), whereas a Flinn plot for these grains shows predominant prolate shapes in agreement with the
331 AMS shape parameter (T) and anisotropy (P'). In addition, our μ -CT data reveal that both pyrrhotite
332 and pyrite grains are localized along alternating metasedimentary layers suggesting that the sulfide
333 growth was chemically controlled by individual laminae, and were formed during D1 to form
334 metamorphic porphyroblasts. The study of thin sections is still indispensable for revealing
335 microstructural relationships between deformation fabrics and different minerals, as shown herein for
336 pyrite and pyrrhotite grains with respect to S1 and S2 foliations, the truncation, shape and micro-
337 folding of pyrrhotite by S2, or the microboudinage textures.

338 7. Conclusions

- 339 a. By adding the 3D shape and preferred orientations of sulfide and magnetic minerals in a rock, μ -
340 CT can significantly enhance the interpretation of bulk rock AMS, particularly in multiply
341 deformed rocks. In addition, integrating μ -CT and AMS methods are highly useful in interpreting
342 metamorphic textures in 3D.
- 343 b. In this study, we have shown that the AMS principal susceptibility axes experienced major changes
344 between two successive deformation phases associated with a horizontal (S1) and subvertical (S2)
345 foliations. K3 changed its orientation from normal to S1 to normal to S2 foliations, while K1 was
346 maintained. Alternatively, the long axes of both pyrite and pyrrhotite porphyroblasts changed from
347 N-S to E-W orientations during D1 and D2, respectively. The sample scale interpretations fit well
348 with the regional scale type-2 fold interference pattern.
- 349 c. Quantitative measurements of individual pyrrhotite grains demonstrate a predominance of highly
350 prolate shapes, whereas pyrite grains have more equant geometry reflecting the higher strength of
351 this less-deformed mineral. Our μ -CT images highlighted the localization of sulfide minerals along
352 alternating metasedimentary layers that crystallized as porphyroblasts during the D1
353 metamorphism.

d. 3D petrography using multiple oriented thin sections was crucial for unravelling the deformation history of the studied sample. We conclude that conventional thin section microstructural analysis cannot be substituted, but only complemented by more sophisticated techniques such as μ -CT and AMS.

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Supplementary data

Appendix 1 and 2 are provided as an electronic supplement.

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Fig. 1. (a) Aeromagnetic map of the study area showing the location of sample 57. The area shows distinct mushroom-shape or arrowhead patterns typical of type-2 fold interference. A main antiform and synform pair can be distinguished. Inset on the top left shows a schematic 3D model. (b) Field photograph and line diagram of S0/S1 overprinted by S2, and drill core location.

Fig. 2. Three-dimensional microstructures of sample 57 based on X-ray computed microtomography (μ -CT). The drill core is oriented and digitally chopped at three different levels to show the penetrative pyrrhotite fabric. Images on the left (a,c,e and g) are 3D side views, whereas those on the right (b,d,f, and h) are horizontal top-view slices. S denotes south. Pyrrhotite (po) and pyrite (py) are rendered yellow except for (b).

Fig. 3. Different 2D and 3D representations of μ -CT data. (a) Horizontal 2D grayscale slice showing east-west aligned (X-axis) pyrrhotite (po) grains. (b) Volume rendering of the same image as shown in (a). (c,d,e) Side views, looking to the north and perpendicular to the long axes (X) of the pyrrhotite grains. Prolate pyrrhotite grains and cubic pyrite (py) are localized in alternating mm-scale metasedimentary layers (S0). The latter dips 45° east, whereas pyrrhotite long axes plunge more shallowly ($\sim 22^\circ$) in the same direction. (f) Grayscale μ -CT slice showing textural characteristics of pyrrhotite, pyrite and S0 and S1 relationship. (g) Folded metasedimentary layers are segmented (isolated) in red and blue. (h) Top 3D view of the drill core showing pyrrhotite grains in the X-Z axes. (i) 3D rendered side view along the Y-Z plane. (j) The same Y-Z plane is shown as a grayscale μ -CT slice. Pyrrhotite and pyrite are rendered yellow in (b), (c), (e), (g), (h) and (i).

Fig. 4. Quantitative geometric analyses of pyrrhotite grains. (a) Rose plot showing the preferred east-west alignment of pyrrhotite long axes (X). (b) Plunge values of X-axes of pyrrhotite averaging 22° . (c) Aspect ratio plot with a mean ratio of about 14. (d) Flinn diagram showing the tendency of grains towards the prolate shape, with a minor population of oblate shape (see text for discussion). (e) Aspect ratio vs. grain volume plot showing a trend between logarithms of grain volume and aspect ratio, where volume increases with increase in the aspect ratio.

Fig. 5. Quantitative geometric analysis of pyrite grains. (a) Rose diagram for the long axes (X) of pyrite grains showing large spread with an E-W maximum, and a weaker N-S maximum. (b) Plunges of pyrite long axes are predominantly shallow (20 – 30°). (c) Aspect ratio diagram with an average value of about 4. (d) Flinn diagram for pyrite showing highly variable shapes of this mineral, but much lower aspect ratios than the pyrrhotite. (e) Aspect ratio vs. grain volume plot showing a minor volume increase with decreasing aspect ratio.

Fig. 6. (a) Mean susceptibility (Km) vs. anisotropy degree (P') plot showing typical values of pyrrhotite. (b) Shape parameter (T) vs. anisotropy degree (P') suggesting prolate shapes of pyrrhotite grains. (c) Heating (red) and cooling (blue) thermomagnetic curves showing a drop at about 325°C , typical of pyrrhotite. (d) AMS plot showing east-west striking magnetic foliation (K1-K2) and shallowly plunging magnetic lineation (K1) carried by pyrrhotite.

Fig. 7. Oriented photomicrographs showing microtextures in sample 57. (a) Horizontal thin section showing S2 deflecting around a pyrite (py) porphyroblast, crossed polarized light. Inset shows the μ -CT image marking the actual section plane. (b-c) Close ups from the horizontal thin section in (a) showing (b) intact, and (c) microboudinaged pyrrhotite (po) porphyroblasts elongated along S2, reflected light photomicrographs. (d) N-S vertical thin section showing change in the shape of pyrrhotite porphyroblasts from oblate to prolate. Strain fringes developed on the upper and lower faces of the pyrite porphyroblasts could be a cut effect (cf. Fig. 8b), crossed polarized light. (e) Close-up of the same thin section as 'd', showing pyrrhotite porphyroblasts truncated, deflected or microfolded by S2, plane polarized light. (f) Photomicrograph, plane polarized light image digitally rendered to

enhance the shape of pyrrhotite grains using image-processing tools, and associated sketch showing microstructures associated with the deformation of the pyrrhotite grains (N-S vertical section). The SPO of the pyrrhotite changes from oblate to prolate controlled by S1 and S2, respectively.

Fig. 8. (a) E-W vertical thin section and (b) sketch showing face-controlled syn-D2 strain fringes developed on the eastern and western faces of pyrite, crossed polarized light image. (c) Micro-CT image corresponding to the actual E-W vertical section shown in (a). (d-e) Close-ups of (a) showing (d) pyrrhotite (po) plunging towards the east, and (e) strain fringes developed on the edge of the pyrite porphyroblasts. Both close-ups are taken under the crossed polarized light.

Fig. 9. FE-SEM images of sample 57. Red dots are EDS spots (see Appendix 2). (a) Pyrite totally replaced by pyrrhotite. The rim contains two unaltered sphalerite grains. (b) Pyrite-pyrrhotite replacement front associated with transitional skeletal texture. Euhedral arsenopyrite inclusions were not replaced by the pyrrhotite. (c) Single pyrrhotite grain plunging towards the east in an E-W vertical section (half arrow points up and East). (d) Pyrrhotite microboudinaged in the E-W direction (cf. Fig. 7c).

Fig. 10. Interpreted large-scale tectonic context of the sample in the normal limb of an F1 recumbent fold based on S0-S1 relationships in the sample.

Fig. 11. (a) Conceptual model showing progressive bulk inhomogeneous shortening during D2 causing steepening of S1 and pyrrhotite grains. Change in the SPO of both pyrite (py) and pyrrhotite (po) is controlled by the S2. (b) Two possible D1-related SPO geometries are presented: 1) prolate, and 2) oblate. In the former case, K1 retained the original orientation from D1 to D2. In the latter case, K1 changed orientation from N-S (D1) to E-W (D2). In either case, the S1 is striking N-S and shallowly dipping towards the east, consistent with the orientation obtained from the μ -CT image (cf. Fig. 10). (c) Present day geometrical configuration forming type-2 fold interference pattern.

Fig. 1

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6

Fig. 7

Fig. 8

Fig. 9

Fig. 10

Fig. 11

Highlights

Bulk rock AMS is integrated with the X-ray computed μ -tomography and 3D petrography

Change in the SPO is seen, wherein AMS ellipsoid axes were switched from D1 to D2

Type-2 fold interference is deduced based on AMS and two near-orthogonal foliations