

**This is an electronic reprint of the original article.
This reprint *may differ* from the original in pagination and typographic detail.**

Author(s): Sayab, Mohammad; Miettinen, Arttu; Aerden, Domingo; Karell, Fredrik

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

Year: 2017

Version:

Please cite the original version:

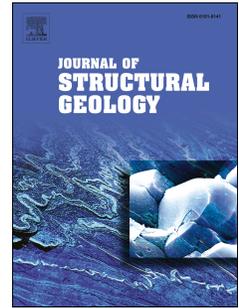
Sayab, M., Miettinen, A., Aerden, D., & Karell, F. (2017). Orthogonal switching of AMS axes during type-2 fold interference : Insights from integrated X-ray computed tomography, AMS and 3D petrography. *Journal of Structural Geology*, 103, 1-16.
<https://doi.org/10.1016/j.jsg.2017.09.002>

All material supplied via JYX is protected by copyright and other intellectual property rights, and duplication or sale of all or part of any of the repository collections is not permitted, except that material may be duplicated by you for your research use or educational purposes in electronic or print form. You must obtain permission for any other use. Electronic or print copies may not be offered, whether for sale or otherwise to anyone who is not an authorised user.

Accepted Manuscript

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

Mohammad Sayab, Arttu Miettinen, Domingo Aerden, Fredrik Karell



PII: S0191-8141(17)30173-6

DOI: [10.1016/j.jsg.2017.09.002](https://doi.org/10.1016/j.jsg.2017.09.002)

Reference: SG 3520

To appear in: *Journal of Structural Geology*

Received Date: 7 June 2017

Revised Date: 23 August 2017

Accepted Date: 1 September 2017

Please cite this article as: Sayab, M., Miettinen, A., Aerden, D., Karell, F., Orthogonal switching of AMS axes during type-2 fold interference: Insights from integrated X-ray computed tomography, AMS and 3D petrography, *Journal of Structural Geology* (2017), doi: 10.1016/j.jsg.2017.09.002.

This is a PDF file of an unedited manuscript that has been accepted for publication. As a service to our customers we are providing this early version of the manuscript. The manuscript will undergo copyediting, typesetting, and review of the resulting proof before it is published in its final form. Please note that during the production process errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.

1 **Orthogonal switching of AMS axes during type-2 fold interference: Insights from integrated X-**
2 **ray computed tomography, AMS and 3D petrography**
3
4

5 Mohammad Sayab^{1*}, Arttu Miettinen², Domingo Aerden³, Fredrik Karell¹

6 ¹Geological Survey of Finland, P.O. Box 96, FI-02151 Espoo, Finland

7 ²Department of Physics, University of Jyväskylä P.O. Box 35, FI-40014, Finland

8 ³Departamento de Geodinámica and IACT-CSIC, Universidad de Granada, Granada 18002, Spain

9 *Corresponding author: sayab.muhammad@gtk.fi
10

11 **Abstract**

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

26 **Keywords:** microtomography; AMS; microtectonics; magnetic fabric; pyrrhotite; strain
27

28 **1. Introduction**

29 X-ray computed micro-tomography (μ -CT) is increasingly being applied in structural geology and
30 ore petrology due to its ability to resolve the three-dimensional (3D) shape and spatial distribution of
31 minerals and associated textures in metamorphic rocks (e.g., Sayab et al., 2015; Macente et al., 2017).
32 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

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

64 **2. Tectonic setting and sample description**

65 The Martimo metasedimentary belt forms a part of the Peräpohja region, which lies in the center of
66 the Fennoscandian shield. The area is characterized by lack of topography, where structural field
67 relations in the 3rd dimension are difficult to reconstruct. Five incremental, but heterogeneously
68 distributed deformation events have been proposed for the Martimo belt in a timeframe ranging from
69 1.92 to 1.77 Ga (Lahtinen et al., 2015). The belt hosts metapelites interbedded with metapsammites
70 and preserves a sequence of near-orthogonal tectonic fabrics that are not fully developed elsewhere in
71 the region (Lahtinen et al., 2015). In particular, the area is passing through a period of extensive
72 geological research because of the recent discoveries of Rompas Au-U and Rajapalot Au (e.g.,
73 Nykänen et al., 2017; Ranta et al., 2015; Ranta et al., 2016). Metamorphism in the area is characterized
74 by upper greenschist facies, however, the grade varies from low greenschist facies rocks in the
75 southwest to kyanite bearing migmatites in the northeast (Hölttä and Heilimo, 2017).

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

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

86 *3.1. Scanning configuration and processing*

87 The sample was scanned using the high-resolution Phoenix|X-ray Nanotom 180 (GE) scanner at
88 the University of Helsinki. Before scanning the drill core, two brass pins were attached, about 40°
89 from north counting towards the east for reference. The tungsten target X-ray tube was operated with
90 an acceleration voltage of 140 kV and tube current 175 μ A. The radiation was filtered with 1.0 mm of
91 Cu. Six hundred views per 360° were acquired, with 1.75 s total exposure time per view. The virtual
92 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
93 projections were captured with a Hamamatsu flat panel sensor C7942SK-05. The 3D reconstructions
94 were computed with datos|x – reconstruction software provided by Phoenix|X-ray.

95 3.2. 3D visualization

96 After reconstructing the 3D distribution of X-ray attenuations, the contrasting gray shades of
97 pyrrhotite and pyrite allowed us to semi-automatically separate them from the rest of the silicate
98 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
99 (Figs. 2 and 3). Besides looking at the microtextures in 3D in all directions, it is possible to digitally
100 cut the sample at any angle and to make certain surfaces transparent while rendering others opaque for
101 better visual analysis. A virtual 3D tour inside the sample, at 33 μ m voxel resolution, allowed us to
102 closely examine petrographic details and the spatial distribution of sulfides.

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

111 Most of the pyrrhotite are standalone grains in the 3D visuals, whereas some pyrite grains are
112 intergrown or touching each other (Figs. 2a and 3e). In horizontal and E-W striking vertical gray slices
113 (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.

141 Finally, the orientation data for all grains were plotted in rose diagrams for the horizontal plane
142 and for vertical planes (Figs. 4a,b and 5a,b). Pyrite grains were separated from each other and analyzed
143 similarly as pyrrhotite grains, but applying a closing operation with radius of 5 pixels before watershed
144 segmentation. A flowchart summarizing the image processing operations is shown in Appendix 1, and
145 provided as an electronic supplement.

146 3.4. 3D quantitative analysis

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

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

160 The horizontal rose diagram for the long axes of pyrite grains (Fig. 5a) shows a wide range of
161 trends with a main E-W maximum and weaker N-S maximum (Fig. 5a) that can be correlated with L_2^1
162 and L_1^0 , respectively. The rose diagram of Fig. 5b shows that the long axes (X) of pyrite grains
163 generally pitch shallowly in both EW and N-S vertical sections. The 3D images show that the larger
164 pyrite grains are confined to a single layer located in the middle of the sample (Figs. 3c,e). The mean
165 aspect ratio of pyrite grains is about 4 with a smaller spread as observed for pyrrhotite grains. The
166 Flinn diagram for pyrite grains indicates a mixture of highly prolate to highly oblate shapes (Fig. 5d),
167 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

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

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

203 4.2. Analytical methods

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

213 4.3. AMS results

214 The mean magnetic susceptibility (K_m) of the 3 subsamples has a high value ranging from 17703
215 to 23308 μSI , whereas the corrected degree of anisotropy (P') varies from 1.81 to 1.94 (Fig. 6). The
216 shape of the AMS ellipsoid (T) is clearly prolate with T values of -0.14 to -0.23. The thermomagnetic
217 curve shows a typical pattern of pyrrhotite with an abrupt drop in magnetization at $\sim 325^\circ\text{C}$ (Hrouda et
218 al., 1997), suggesting that AMS is mainly controlled by this mineral. The orientation of the magnetic
219 foliation matches that of the east-west striking and subvertically dipping S_2 foliation (Fig. 6d; cf. Fig.
220 1b), whereas the short axis (K_3) is oriented perpendicular to this plane. The orientation of the triaxial

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

223 5. Petrographic and SEM analyses

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

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

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

247 In order to verify the chemical composition of sulfides, Energy-dispersive X-ray Spectroscopy
248 (EDS) analyses were performed at the Research Laboratory, Geological Survey of Finland in Espoo,
249 using an Oxford Instruments EDS-spectrometer X-Max 80 mm² (SDD) attached to a JEOL JSM 7100F
250 Schottky field emission scanning electron microscope (representative FE-SEM images and EDS
251 spectra are provided in Appendix 2 as an electronic supplement). The analytical conditions were as
252 follows: high vacuum mode, a COMPO back-scattered signal (BSE), 20 kV accelerating voltage and
253 0.5 nA probe current. FE-SEM and EDS analyses showed that almost all pyrite grains are partially to
254 completely pseudomorphed by pyrrhotite (Fig. 9), indicating prograde heating resulting in sulphur
255 release from pyrite (Craig and Vokes, 1993). Skeletal textures mark this pyrrhotite to pyrite
256 replacement (Fig. 9b). Besides, we detected inclusions of euhedral arsenopyrite and anhedral sphalerite
257 around the median and rim regions of pseudomorphed pyrite (Figs. 9a,b). EDS analyses, conducted in
258 an FE-SEM, of both intact and microboudinaged grains confirmed their pyrrhotite composition (Figs.
259 9c,d). Some of the cubic-looking grains have rhombic shape possibly due to replacement of diagenetic
260 anhydrite by pyrrhotite (Hall, 1982).

261 **6. Discussion**

262 *6.1. Implications for the D1 related structures*

263 Microstructures commonly exhibit complex overprinting relationships that are only partially
264 reflected by outcrop-scale structures (Aerden, 1998; Sayab et al., 2016b). This counts in particular for
265 Proterozoic belts, where glacially eroded bedrock and sedimentary cover exacerbate field observations
266 in the 3rd dimension. Under such limited exposure conditions, oriented drill cores are useful to
267 understand multiple fabric overprinting relationships and microstructures (Sayab, et al., 2015). As
268 described earlier, sample 57 was extracted from a D2 low-strain zone of a flat-lying folded surface,
269 where the east-west striking pervasive fabric marks the axial planar S2 foliation. High-resolution
270 tomographic 3D images accompanied by a vertical east-west oriented thin section revealed that
271 metasedimentary layers (S0) dips ~23° more steeply than the S1 foliation, whereas the intersection
272 lineation (L⁰₁) is north-south trending and shallowly plunging (Fig. 10). This structural relationship
273 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 inhomogenous 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.

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

358 **Acknowledgements**

359 We thank R. Lahtinen, F. Molnar and S. Mertanen for useful discussions, M. Lehtonen for help with
360 the FE-SEM imaging, A. Kallonen for acquiring and reconstructing the X-ray computed tomography
361 data at the Helsinki University, and P. Telkkälä for help with the mini drill in the field. We
362 acknowledge A. Käpyaho for initiating the research collaboration between the GTK and Jyväskylä
363 University. Constructive journal reviews were made by Eric C. Ferré and Florian Fousseis. We are
364 thankful to Toru Takeshita for his editorial handling and useful suggestions.

366 **Supplementary data**

367 Appendix 1 and 2 are provided as an electronic supplement.
369

370 **References**

- 371 Aerden, D., 2003. Preferred orientation of Planar microstructures determined via statistical best-fit of
372 measured intersection-lines: The “FitPitch” computer program. *Journal of Structural Geology* 25,
373 923–934. [https://doi.org/10.1016/S0191-8141\(02\)00119-0](https://doi.org/10.1016/S0191-8141(02)00119-0)
- 374 Aerden, D.G.A.M., 1998. Tectonic evolution of the Montagne Noire and a possible orogenic model for
375 syncollisional exhumation of deep rocks, Variscan belt, France. *Tectonics* 17, 62–79.
376 <https://doi.org/10.1029/97TC02342>
- 377 Bell, T.H., Bruce, M.D., 2006. The internal inclusion trail geometries preserved within a first phase of
378 porphyroblast growth. *Journal of Structural Geology* 28, 236–252.
379 <https://doi.org/10.1016/j.jsg.2005.11.001>
- 380 Bell, T.H., Hayward, N., 1991. Episodic metamorphic reactions during orogenesis: the control of
381 deformation partitioning on reaction sites and reaction duration. *Journal of Metamorphic Geology*
382 9, 619–640. <https://doi.org/10.1111/j.1525-1314.1991.tb00552.x>
- 383 Bell, T.H., Rubenach, M.J., 1983. Sequential porphyroblast growth and crenulation cleavage
384 development during progressive deformation. *Tectonophysics* 92, 171–194.
385 [https://doi.org/10.1016/0040-1951\(83\)90089-6](https://doi.org/10.1016/0040-1951(83)90089-6)

- 386 Bell, T.H., Rubenach, M.J., 1980. Crenulation cleavage development—evidence for progressive bulk
387 inhomogeneous shortening from “millipede” microstructures in the robertson river metamorphics.
388 *Tectonophysics* 68, T9–T15. [https://doi.org/10.1016/0040-1951\(80\)90003-7](https://doi.org/10.1016/0040-1951(80)90003-7)
- 389 Borradaile, G.J., Almqvist, B.S.G., Geneviciene, I., 2012. Anisotropy of magnetic susceptibility
390 (AMS) and diamagnetic fabrics in the Durness Limestone, NW Scotland. *Journal of Structural*
391 *Geology* 34, 54–60. <https://doi.org/10.1016/j.jsg.2011.10.008>
- 392 Borradaile, G.J., Jackson, M., 2010. Structural geology, petrofabrics and magnetic fabrics (AMS,
393 AARM, AIRM). *Journal of Structural Geology* 32, 1519–1551.
394 <https://doi.org/10.1016/j.jsg.2009.09.006>
- 395 Borradaile, G.J., Jackson, M., 2004. Anisotropy of magnetic susceptibility (AMS): magnetic
396 petrofabrics of deformed rocks. Geological Society, London, Special Publications 238, 299–360.
397 <https://doi.org/10.1144/GSL.SP.2004.238.01.18>
- 398 Craig, J.R., Vokes, F.M., 1993. The Metamorphism of Pyrite and Pyritic Ores: An Overview.
399 *Mineralogical Magazine* 57, 3–18. <https://doi.org/10.1180/minmag.1993.057.386.02>
- 400 Ferré, E.C., 2002. Theoretical models of intermediate and inverse AMS fabrics. *Geophysical Research*
401 *Letters* 29, 29–32. <https://doi.org/10.1029/2001GL014367>
- 402 Ferré, E.C., Gébelin, A., Till, J.L., Sassié, C., Burmeister, K.C., 2014. Deformation and magnetic
403 fabrics in ductile shear zones: A review. *Tectonophysics* 629, 179–188.
404 <https://doi.org/10.1016/j.tecto.2014.04.008>
- 405 Füsseis, F., Schrank, C., Liu, J., Karrech, A., Llana-Funez, S., Xiao, X., Regenauer-Lieb, K., 2012.
406 Pore formation during dehydration of a polycrystalline gypsum sample observed and quantified in
407 a time-series synchrotron X-ray micro-tomography experiment. *Solid Earth* 3, 71–86.
408 <https://doi.org/10.5194/se-3-71-2012>
- 409 Gonzalez, R., Woods, R., 2002. Digital image processing, Prentice Hall. <https://doi.org/10.1016/0734->
410 [189X\(90\)90171-Q](https://doi.org/10.1016/0734-189X(90)90171-Q)
- 411 Hall, A.J., 1982. Gypsum as a precursor to pyrrhotite in metamorphic rocks - Evidence from the
412 Ballachulish State, Scotland. *Mineralium Deposita* 17, 401–409.

- 413
414 Hanna, R.D., Ketcham, R.A., 2017. X-ray computed tomography of planetary materials: A primer and
415 review of recent studies. *Chemie Der Erde - Geochemistry* 1–26.
416 <https://doi.org/10.1016/j.chemer.2017.01.006>
- 417 Hayward, N., 1990. Determination of early fold axis orientations in multiply deformed rocks using
418 porphyroblast inclusion trails. *Tectonophysics* 179, 353–369. [https://doi.org/10.1016/0040-](https://doi.org/10.1016/0040-1951(90)90301-N)
419 1951(90)90301-N
- 420 Hrouda, F., 1994. A technique for the measurement of thermal changes of magnetic susceptibility of
421 weakly magnetic rocks by the CS-2 apparatus and KLY-2 Kappabridge. *Geophysical Journal*
422 *International* 118, 604–612. <https://doi.org/10.1111/j.1365-246X.1994.tb03987.x>
- 423 Hrouda, F., Jelínek, V., Zapletal, K., Jelinek, V., Zapletal, K., Jelínek, V., 1997. Refined technique for
424 susceptibility resolution into ferromagnetic and paramagnetic components based on susceptibility
425 temperature-variation measurement. *Geophysical Journal International* 129, 715–719.
426 <https://doi.org/10.1111/j.1365-246X.1997.tb04506.x>
- 427 Janák, F., 1965. Determination of anisotropy of magnetic susceptibility of rocks. *Studia Geophysica et*
428 *Geodaetica* 9, 290–301.
- 429 Karell, F., Ehlers, C., Airo, M.L., 2014. Emplacement and magnetic fabrics of rapakivi granite
430 intrusions within Wiborg and Aland rapakivi granite batholiths in Finland. *Tectonophysics* 614,
431 31–43. <https://doi.org/10.1016/j.tecto.2013.12.006>
- 432 Kruckenberg, S.C., Ferré, E.C., Teyssier, C., Vanderhaeghe, O., Whitney, D.L., Seaton, N.C.A.,
433 Skord, J.A., 2010. Viscoplastic flow in migmatites deduced from fabric anisotropy: An example
434 from the Naxos dome, Greece. *Journal of Geophysical Research: Solid Earth* 115, 1–18.
435 <https://doi.org/10.1029/2009JB007012>
- 436 Kyle, J.R., Ketcham, R.A., 2015. Application of high resolution X-ray computed tomography to
437 mineral deposit origin, evaluation, and processing. *Ore Geology Reviews* 65, 821–839.
438 <https://doi.org/10.1016/j.oregeorev.2014.09.034>
- 439 Lahtinen, R., Sayab, M., Karell, F., 2015. Near-orthogonal deformation successions in the poly-

- 440 deformed Paleoproterozoic Martimo belt: Implications for the tectonic evolution of Northern
441 Fennoscandia. *Precambrian Research* 270, 22–38.
- 442 Macente, A., Fuisseis, F., Menegon, L., Xianghui, X., John, T., 2017. The strain-dependent spatial
443 evolution of garnet in a high-P ductile shear zone from the Western Gneiss Region (Norway): a
444 synchrotron X-ray microtomography study. *Journal of Metamorphic Geology* 35, 565–583.
445 <https://doi.org/10.1111/jmg.12245>
- 446 McClay, K.R., Ellis, P.G., 1983. Deformation and Recrystallization of Pyrite. *Mineralogical Magazine*
447 47, 527–538. <https://doi.org/10.1180/minmag.1983.047.345.14>
- 448 Meyer, F., Beucher, S., 1990. Morphological segmentation. *Journal of Visual Communication and*
449 *Image Representation*. [https://doi.org/10.1016/1047-3203\(90\)90014-M](https://doi.org/10.1016/1047-3203(90)90014-M)
- 450 Nykänen, V., Niiranen, T., Molnár, F., Lahti, I., Korhonen, K., Cook, N., Skyttä, P., 2017. Optimizing
451 a Knowledge-driven Prospectivity Model for Gold Deposits Within Peräpohja Belt, Northern
452 Finland. *Natural Resources Research*. <https://doi.org/10.1007/s11053-016-9321-4>
- 453 Parsons, A.J., Ferre, E.C., Law, R.D., Lloyd, G.E., Phillips, R.J., Searle, M.P., 2016. Orogen-parallel
454 deformation of the Himalayan midcrust: Insights from structural and magnetic fabric analyses of
455 the Greater Himalayan Sequence, Annapurna-Dhaulagiri Himalaya, central Nepal. *Tectonics* 35,
456 2515–2537. <https://doi.org/10.1002/2016TC004244>
- 457 Passchier, C.W., Trouw, R.A.J., 2005. *Microtectonics*, Springer-Verlag Berlin Heidelberg.
- 458 Pearson, K., 1901. On lines and planes of closest fit to systems of points in space. *The London,*
459 *Edinburgh, and Dublin Philosophical Magazine and Journal of Science*.
460 <https://doi.org/10.1080/14786440109462720>
- 461 Ranta, J.-P., Hanski, E., Cook, N., Lahaye, Y., 2016. Source of boron in the Palokas gold deposit,
462 northern Finland: evidence from boron isotopes and major element composition of tourmaline.
463 *Mineralium Deposita*. <https://doi.org/10.1007/s00126-016-0700-x>
- 464 Ranta, J.-P., Lauri, L.S., Hanski, E., Huhma, H., Lahaye, Y., Vanhanen, E., 2015. U–Pb and Sm–Nd
465 isotopic constraints on the evolution of the Paleoproterozoic Peräpohja Belt, northern Finland.
466 *Precambrian Research* 266, 246–259.

- 468 Riller, U., Cruden, A.R., Schwerdtner, W.M., 1996. Magnetic fabric and microstructural evidence for a
469 tectono-thermal overprint of the early Proterozoic Murray pluton, central Ontario, Canada.
470 *Journal of Structural Geology* 18, 1005–1016. [https://doi.org/10.1016/0191-8141\(96\)00028-4](https://doi.org/10.1016/0191-8141(96)00028-4)
- 471 Rochette, P., Aubourg, C., Perrin, M., 1999. Is this magnetic fabric normal? A review and case studies
472 in volcanic formations. *Tectonophysics* 307, 219–234. [https://doi.org/10.1016/S0040-](https://doi.org/10.1016/S0040-1951(99)00127-4)
473 [1951\(99\)00127-4](https://doi.org/10.1016/S0040-1951(99)00127-4)
- 474 Sayab, M., Shah, S.Z., Aerden, D., 2016b. Metamorphic record of the NW Himalayan orogeny
475 between the Indian plate-Kohistan Ladakh Arc and Asia: Revelations from foliation intersection
476 axis (FIA) controlled P-T-t-d paths. *Tectonophysics* 671, 110–126.
- 477 Sayab, M., Suuronen, J.P., Hölttä, P., Aerden, D., Lahtinen, R., Kallonen, A.P., 2015. High-resolution
478 X-ray computed microtomography: A holistic approach to metamorphic fabric analyses. *Geology*
479 43, 55–58.
- 480 Sayab, M., Suuronen, J.P., Molnár, F., Villanova, J., Kallonen, A., O'Brien, H., Lahtinen, R.,
481 Lehtonen, M., 2016a. Three-dimensional textural and quantitative analyses of orogenic gold at the
482 nanoscale. *Geology* 44, 739–742. <https://doi.org/10.1130/G38074.1>
- 483 Skyttä, P., Hermansson, T., Elming, S.Å., Bauer, T., 2010. Magnetic fabrics as constraints on the
484 kinematic history of a pre-tectonic granitoid intrusion, Kristineberg, northern Sweden. *Journal of*
485 *Structural Geology* 32, 1125–1136. <https://doi.org/10.1016/j.jsg.2010.06.020>
- 486 Stock, S., 2008. *Microcomputed Tomography: Methodology and Applications*. CRC Press.
487 <https://doi.org/CAT#58762>
- 488 Tarling, D.H., Hrouda, F., 1993. *The Magnetic Anisotropy of Rocks*. Chapman and Hall, London.
489 <https://doi.org/10.1002/gj.3350300111>
- 490 Tsai, W.-H., 1985. Moment-preserving thresholding: A new approach. *Computer Vision, Graphics, and*
491 *Image Processing*. [https://doi.org/10.1016/0734-189X\(85\)90133-1](https://doi.org/10.1016/0734-189X(85)90133-1)

492

493

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

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

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

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

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

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

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

546 enhance the shape of pyrrhotite grains using image-processing tools, and associated sketch showing
547 microstructures associated with the deformation of the pyrrhotite grains (N-S vertical section). The
548 SPO of the pyrrhotite changes from oblate to prolate controlled by S1 and S2, respectively.
549

550 Fig. 8. (a) E-W vertical thin section and (b) sketch showing face-controlled syn-D2 strain fringes
551 developed on the eastern and western faces of pyrite, crossed polarized light image. (c) Micro-CT
552 image corresponding to the actual E-W vertical section shown in (a). (d-e) Close-ups of (a) showing
553 (d) pyrrhotite (po) plunging towards the east, and (e) strain fringes developed on the edge of the pyrite
554 porphyroblasts. Both close-ups are taken under the crossed polarized light.
555

556 Fig. 9. FE-SEM images of sample 57. Red dots are EDS spots (see Appendix 2). (a) Pyrite totally
557 replaced by pyrrhotite. The rim contains two unaltered sphalerite grains. (b) Pyrite-pyrrhotite
558 replacement front associated with transitional skeletal texture. Euhedral arsenopyrite inclusions were
559 not replaced by the pyrrhotite. (c) Single pyrrhotite grain plunging towards the east in an E-W vertical
560 section (half arrow points up and East). (d) Pyrrhotite microboudinaged in the E-W direction (cf. Fig.
561 7c).
562

563 Fig. 10. Interpreted large-scale tectonic context of the sample in the normal limb of an F1 recumbent
564 fold based on S0-S1 relationships in the sample.
565

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

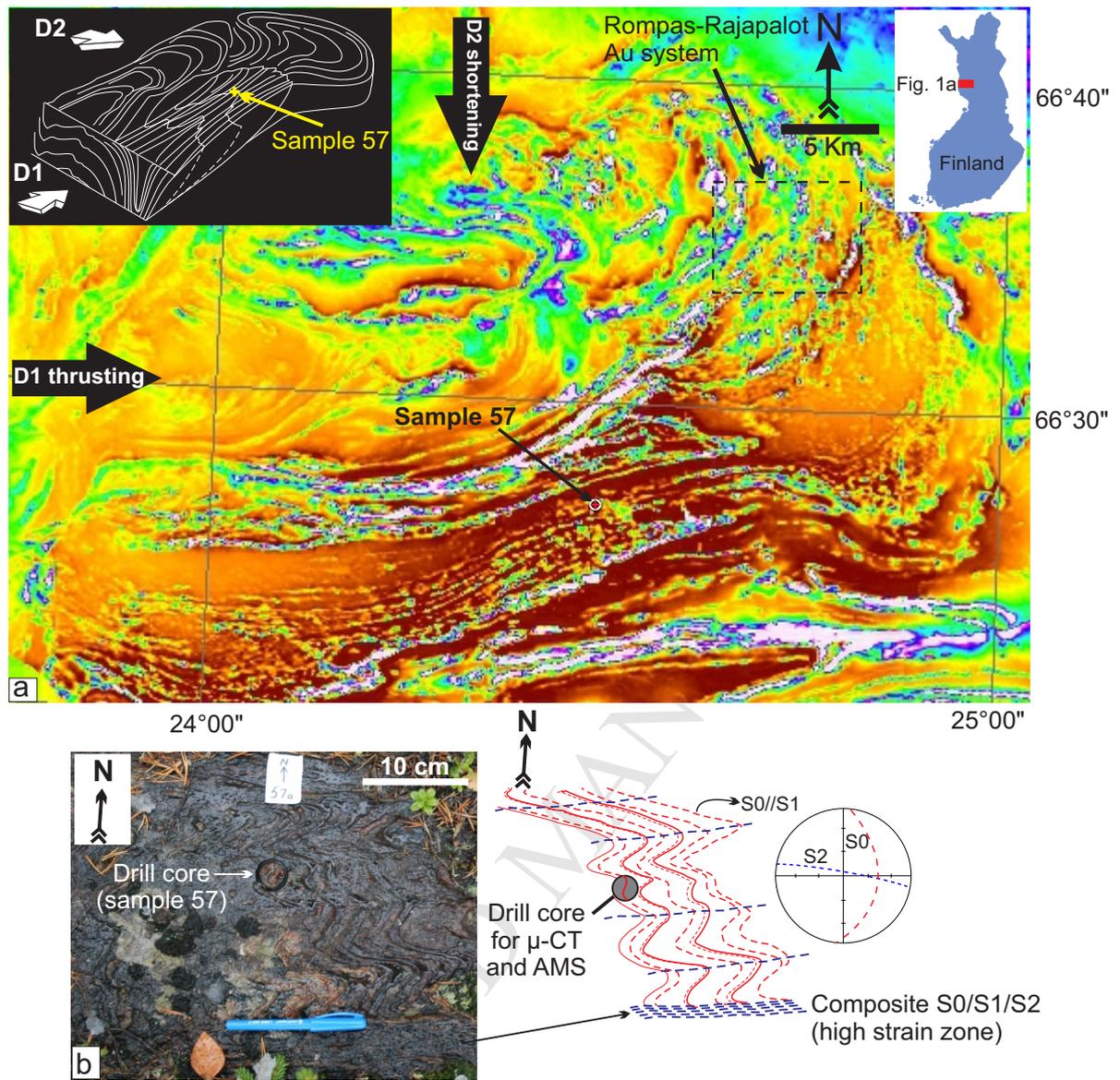


Fig. 1

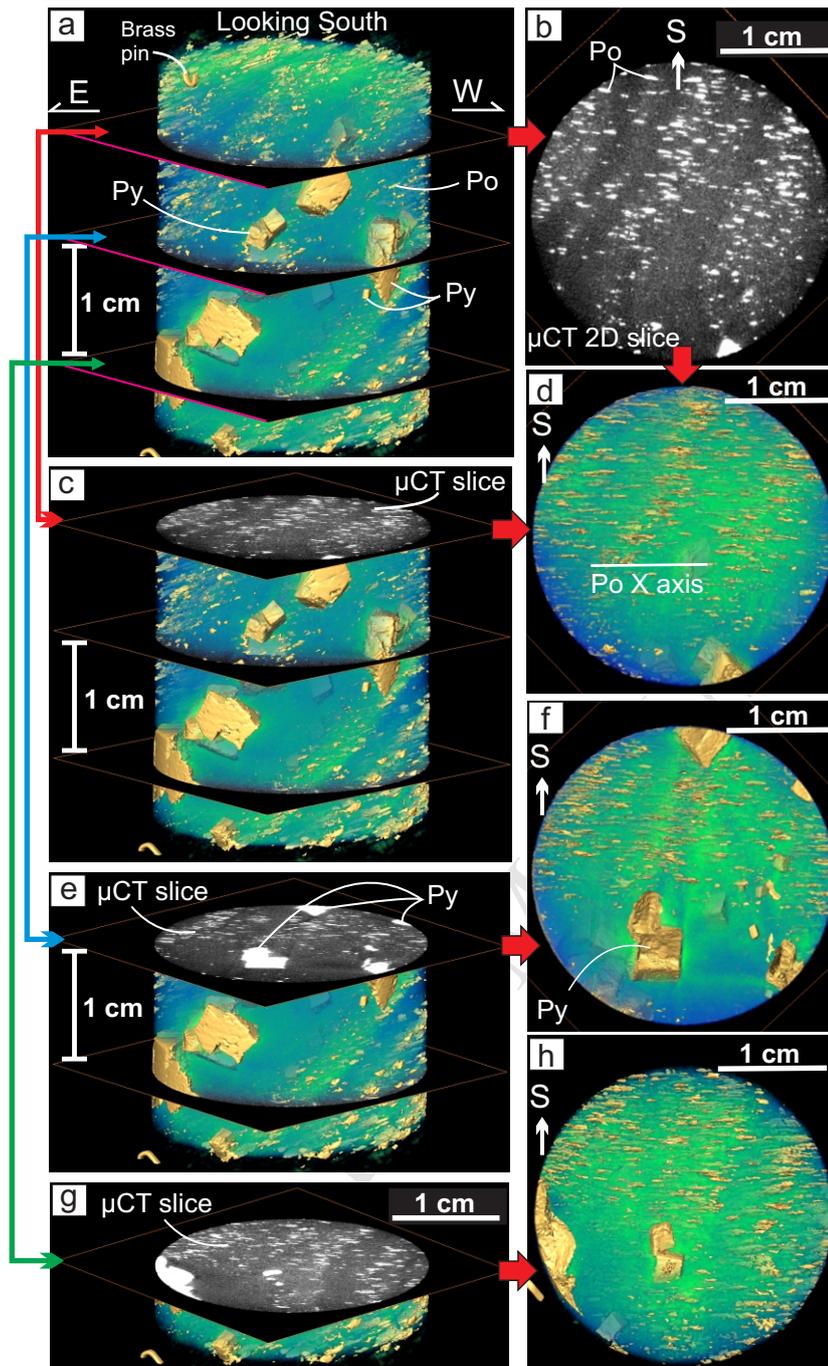


Fig. 2

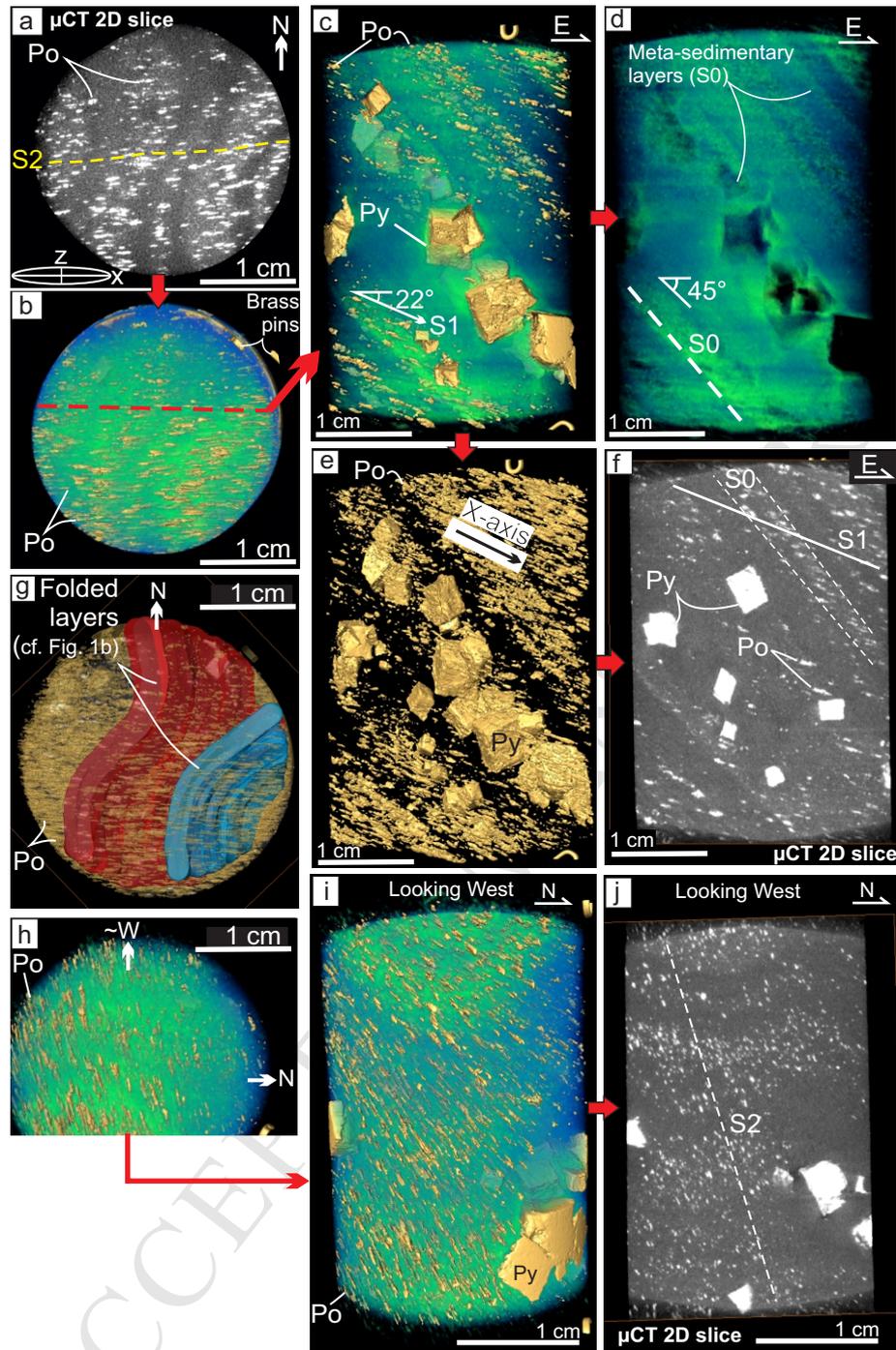


Fig. 3

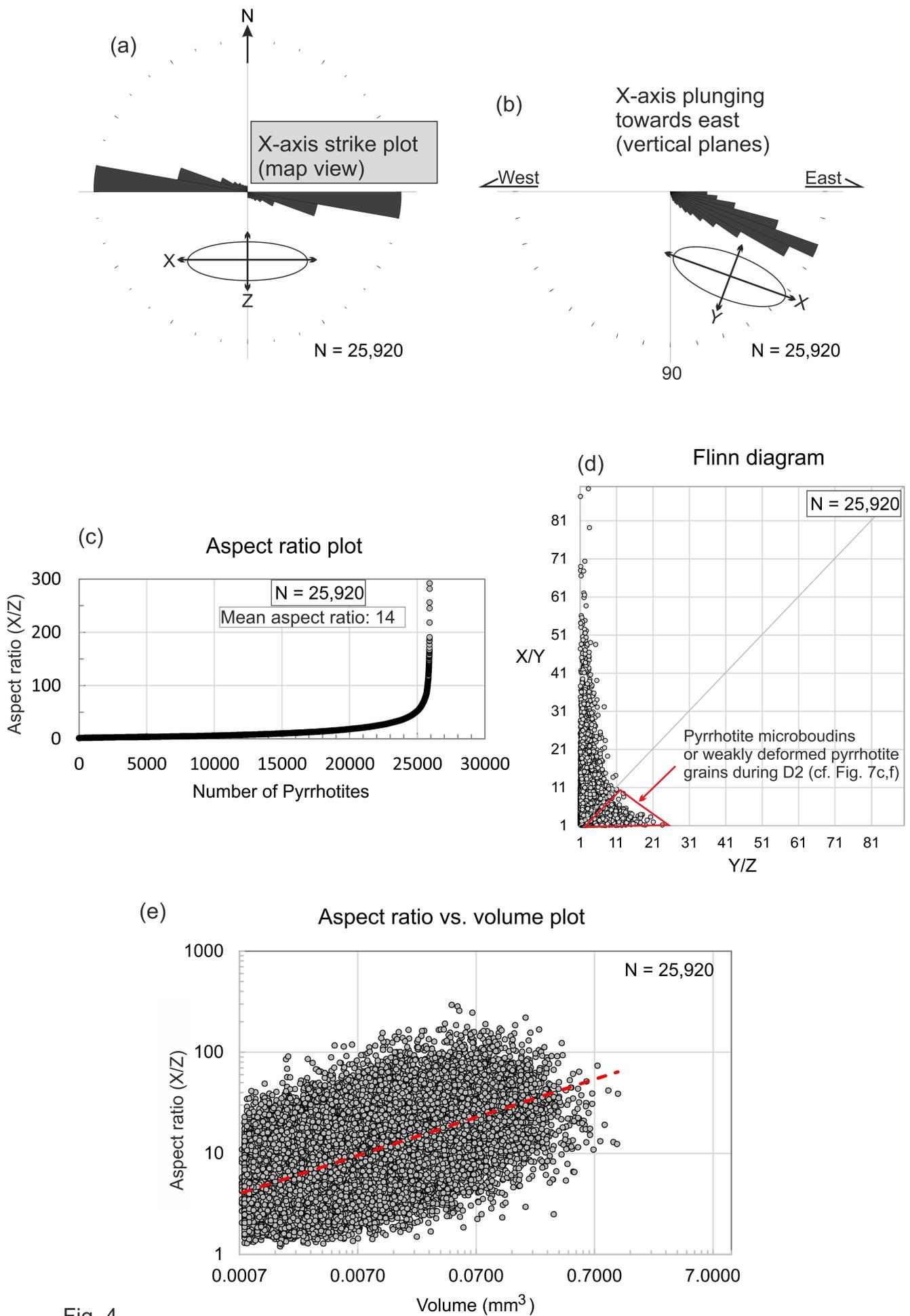


Fig. 4

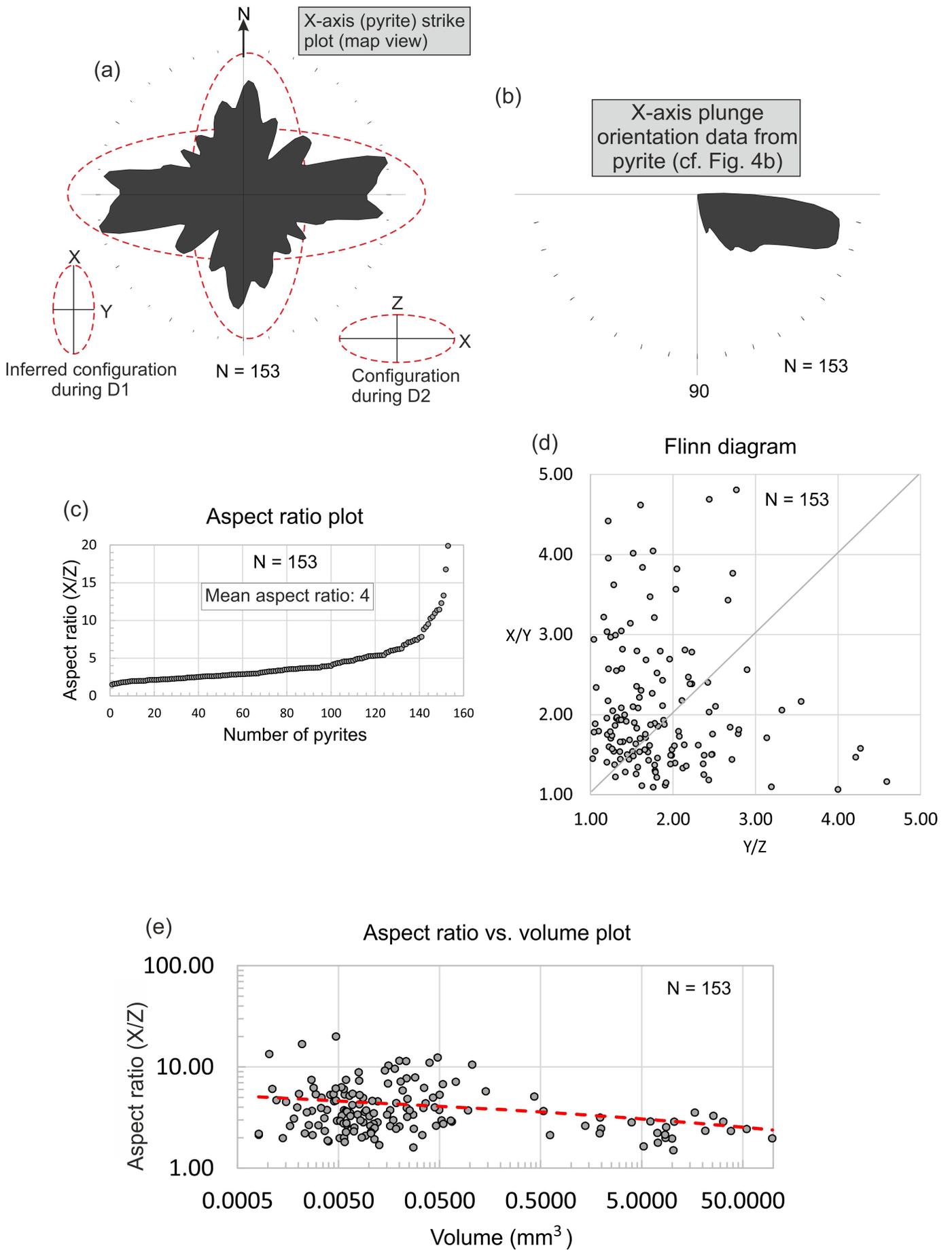


Fig. 5

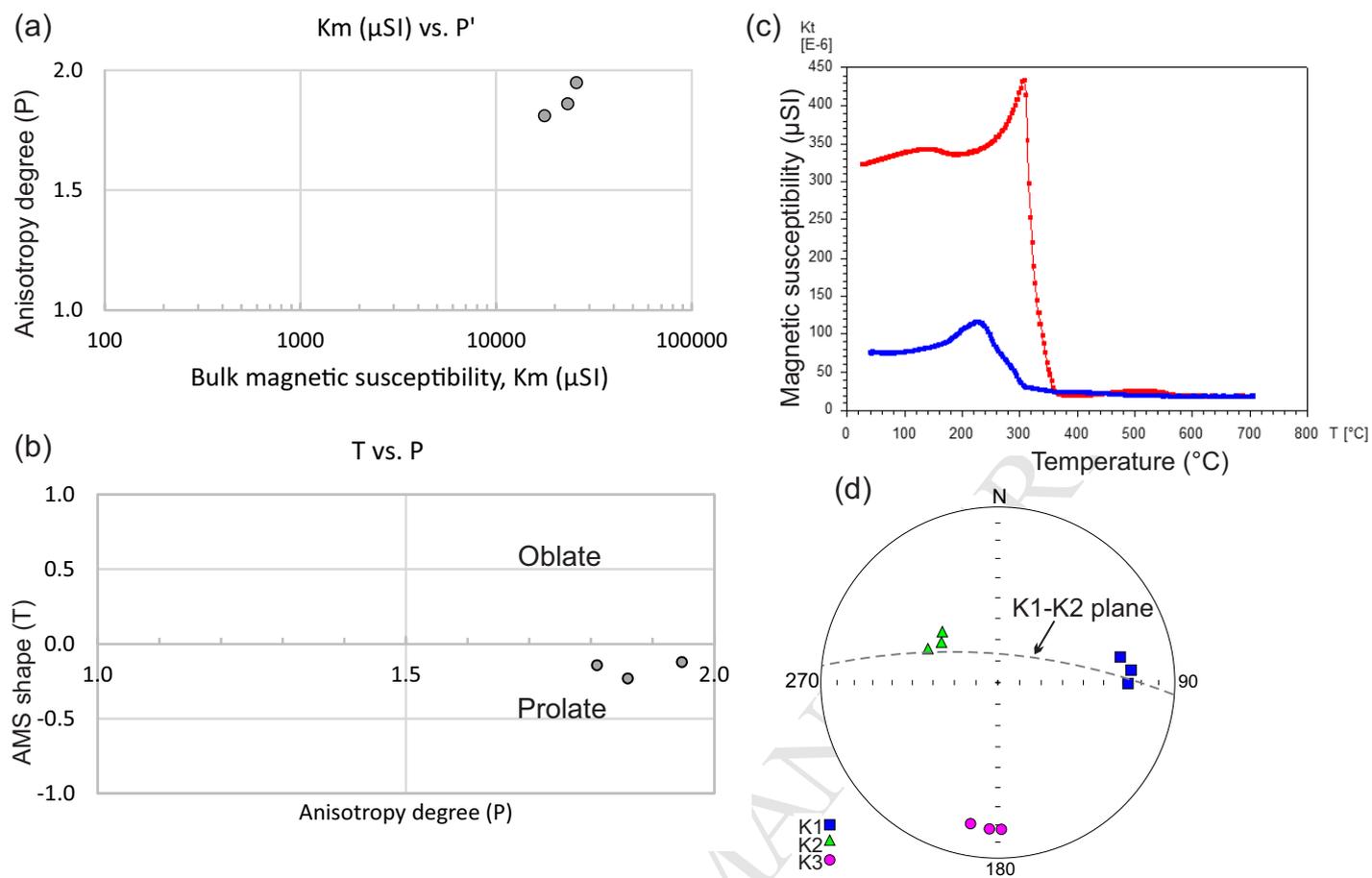


Fig. 6

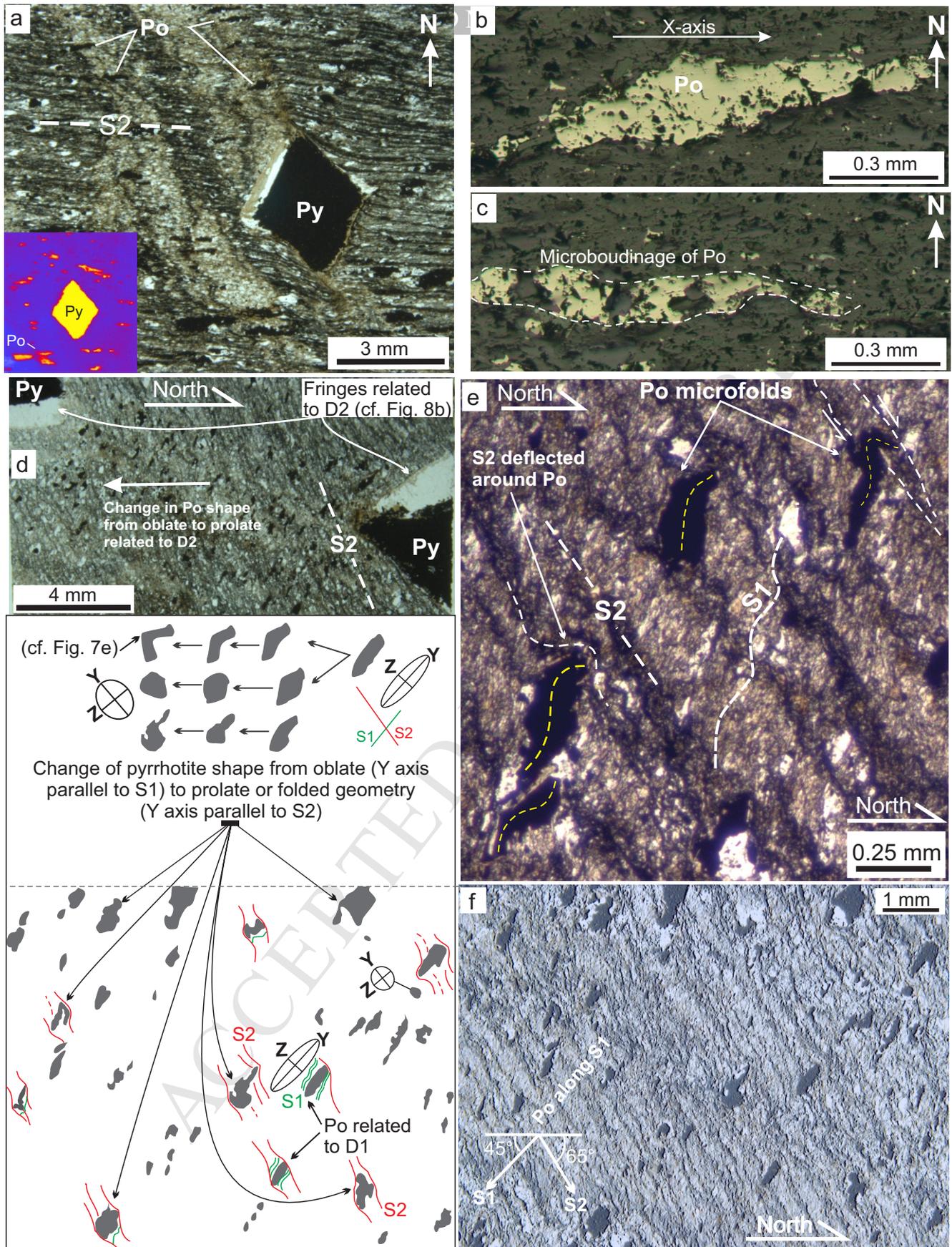


Fig. 7

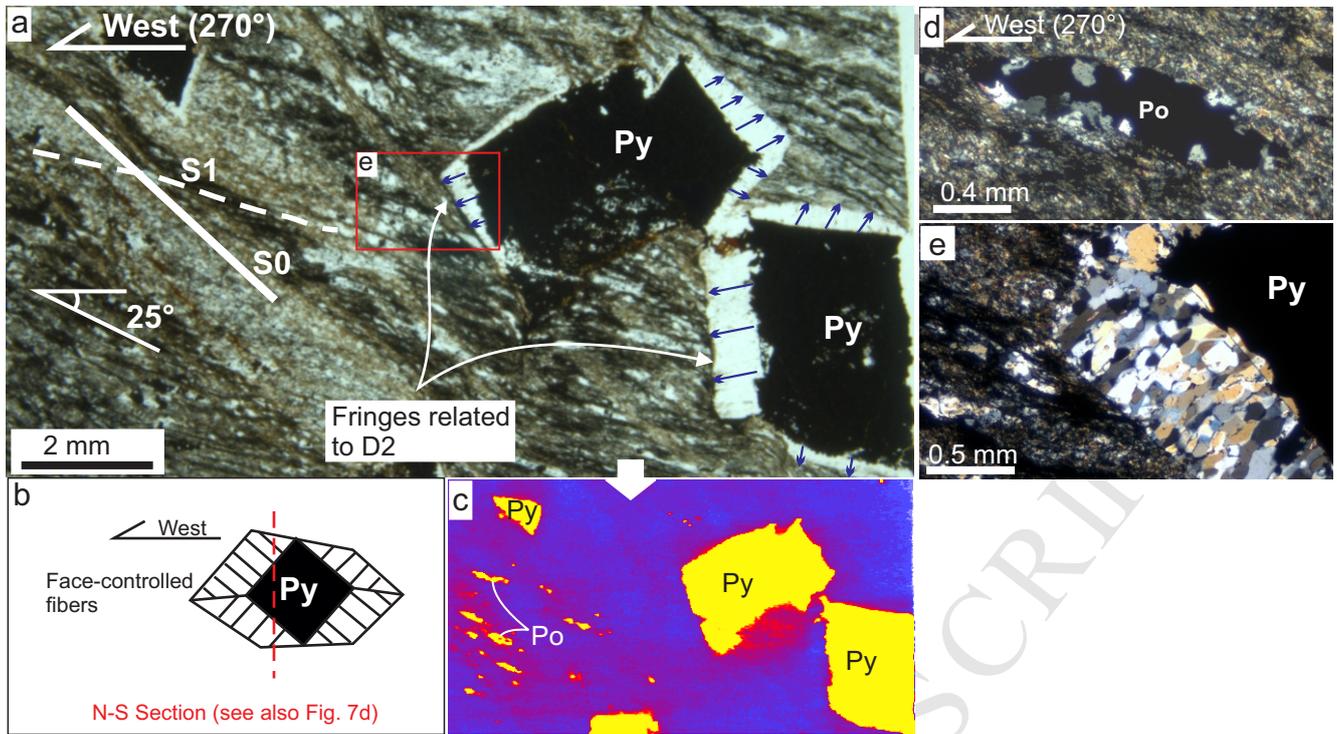


Fig. 8

ACCEPTED MANUSCRIPT

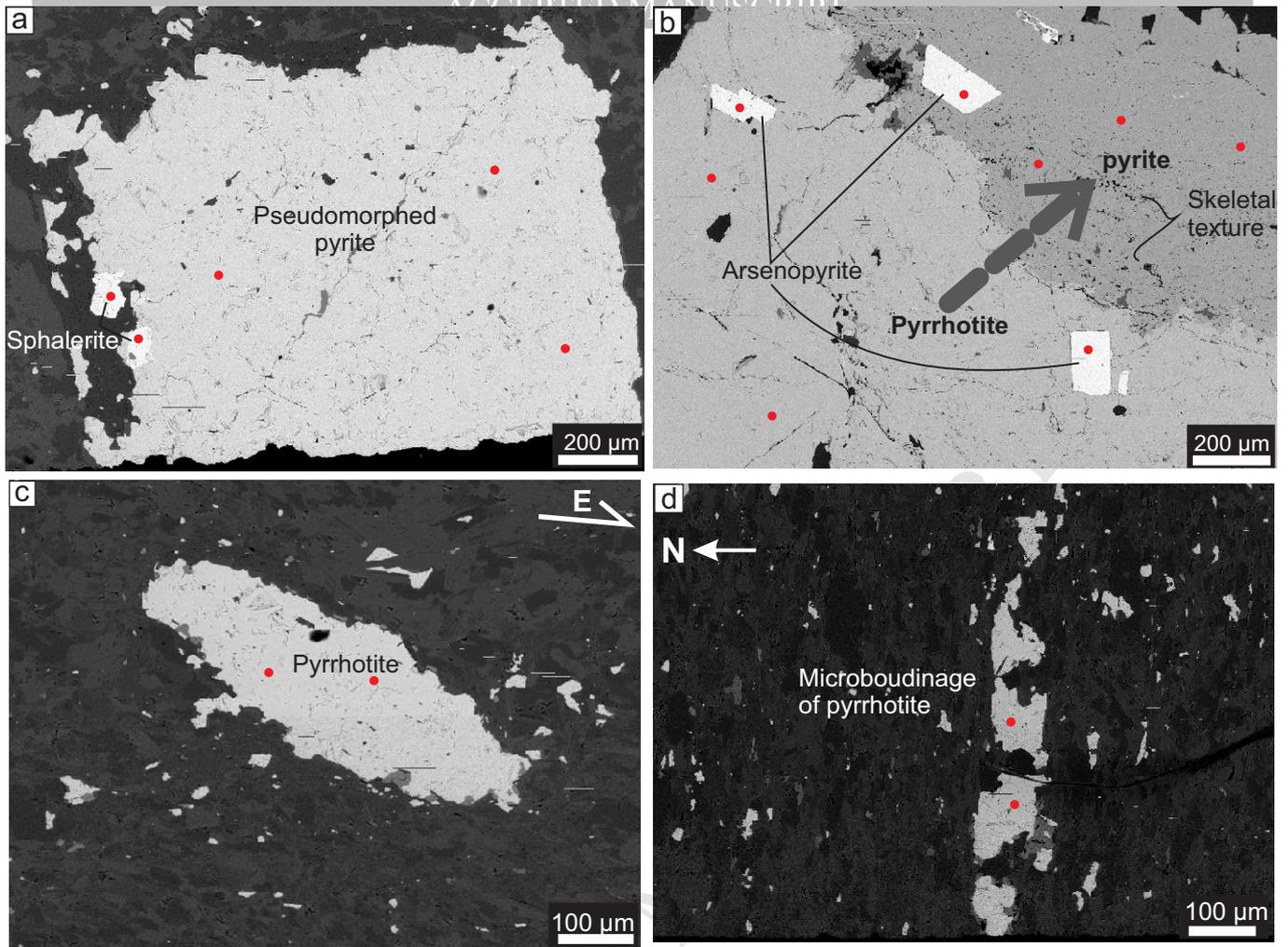


Fig. 9

ACCEPTED

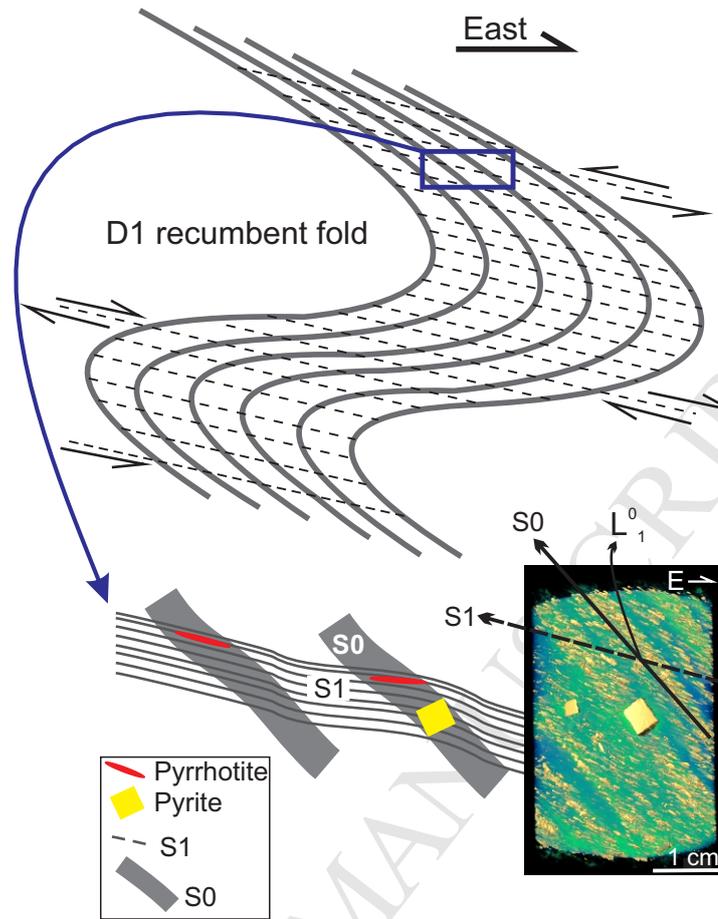
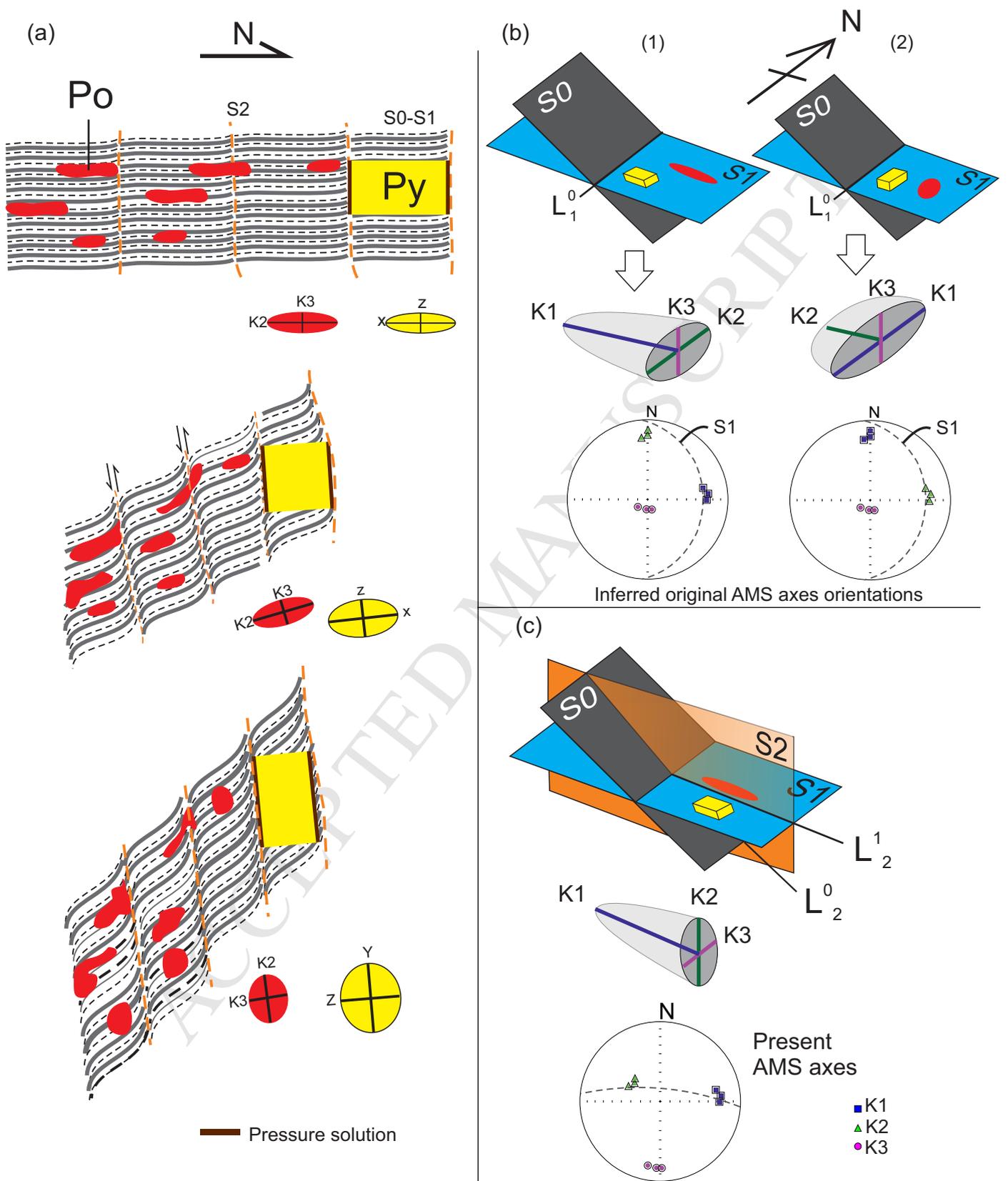


Fig. 10



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

ACCEPTED MANUSCRIPT