

1 **A study of secondary pyrite deformation and calcite veins in SAFOD damage zone with**
2 **implications for aseismic creep deformation mechanism at depths >3km**

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8

9 **Abstract**

10 Previous studies of the San Andreas Fault damage-zone samples from the San Andreas Fault
11 Observatory at Depth (SAFOD) have identified a variety of tectonic microstructures including
12 pressure solution cleavage, calcite-sealed fractures vein fabric, and pyrite and anhydrite
13 hydrothermal fracture sealing. Understanding the deformation provenance of the damage zone
14 rocks and operative deformation mechanism(s) based on preserved microstructures provide
15 insight into overall deformation behavior of the entire seismogenic zone in the creeping section
16 of this transform fault. We analyzed the deformation of hydrothermal secondary pyrite in
17 connection with network of calcite veins in a sample of foliated ultracataclasites bordering the
18 actively creeping Southwestern Deforming Zone (SDZ), using SEM, EBSD and CL microscopy.
19 The results show that calcite veins associated with the pressure solution cleavage are crosscut by
20 the secondary pyrite deformed under a range of P-T conditions. Relatively undeformed
21 secondary pyrite is found sealing implosion microbreccia. Our review of previously available
22 data indicates that the damage zone rocks may represent a collage of structural and
23 compositional domains from both locked and creeping sections of the SAF. This interpretation
24 together with results of this study suggest that weak-clay frictional deformation mechanism(s) is
25 likely to be the predominant aseismic creep mechanism at depths below the SAFOD.

26 **Keywords:** SAFOD; Deformation of pyrite; Calcite-vein Cathodoluminescence; Aseismic creep
27 in the SAF; Pressure solution.

28 **1. Introduction**

29 Plate-scale faults are active for extended periods of Earth history. They involve ductile
30 deformation mechanisms at depth and brittle mechanisms nearer the surface, and act as conduits
31 for typically bisulphide ion (HS⁻)-bearing low-salinity H₂O-CO₂ fluids escaping from
32 metamorphic and/or igneous activity at depth (Goldfarb and Groves 2015). These fluids are often

33 inferred to initiate deformation on faults via episodic over-pressuring leading to fault-valve or
34 seismic pumping mechanisms (Cox, 1995; Sibson, 1981, 1990, 1992, and 1994). Episodic
35 pumping of the fluids leads to decompression-related breakdown of the H₂O-CO₂ fluid as well as
36 the HS⁻ ligand complexes leading to carbonate precipitation associated with pyrite (McCuaig
37 and Kerrich, 1998). This behavior gives rise to the interesting possibility that episodic formation
38 of calcite and pyrite in an actively deforming fault could preserve significant parts of fault
39 histories. Recently, Holdsworth et al. (2011) and Bradbury et al. (2015) described the ubiquitous
40 development of calcite-filled veins and disseminated pyrite suggesting that the San Andreas
41 Fault Observatory at Depth (SAFOD) may be a suitable place to investigate the potential for
42 calcite and pyrite microstructures to record the fault deformation history.

43 Understanding of pyrite deformation mechanisms and microstructures over a wide range
44 of temperature conditions has expanded significantly over the last 20 years through using
45 electron backscatter diffraction (EBSD) methods (Barrie et al., 2007, 2008, 2010a; Boyle et al.,
46 1998; Reddy and Hough, 2013). Pyrite is a robust, refractory mineral well suited to preserving
47 evidence of any plastic or brittle deformation it has experienced below ~650°C and thus has
48 potential for preserving a memory of deformation over a range of temperature conditions. For
49 most of the 20th century, pyrite was thought to undergo a brittle-crystal plastic transition above
50 ~400°C, though grain growth and annealing were considered more likely above that temperature
51 (McClay and Ellis, 1983). Extensive EBSD-driven research has recently demonstrated that pyrite
52 behaves in a crystal-plastic manner to much lower temperatures of ~250°C (Barrie et al., 2009;
53 Freitag et al., 2004). The EBSD-based research has also developed a new deformation-
54 mechanism map for pyrite reflecting the much wider range of conditions for which it will deform
55 by dislocation creep or glide (Barrie et al., 2011).

56 This paper seeks to explore the hypothesis that segments of fault history can be
57 determined through study of episodically developed calcite and pyrite microstructures, which are
58 two minerals that are commonly developed in faults. More specifically, the study is focused on
59 understanding the microstructural and mechanical implications of deformation of the pyrite in
60 association with calcite veins for possible creep deformation mechanisms below the SAFOD
61 depths. It has been argued that deformation by pressure solution is a viable creep mechanism
62 throughout the entire seismogenic zone (Gratier et al., 2011; Richard et al., 2014). On the other
63 hand, several studies have suggested that the weak-clay frictional deformation mechanisms

64 similar to those observed in SDZ and CDZ are likely to operate at depths below the SAFOD and
65 probably over the entire seismogenic profile (e.g. Carpenter et al., 2012; French et al., 2015;
66 Carpenter et al., 2015; Moore et al., 2016). The latter model is supported by experimental work
67 (Moore and Lockner, 2011; Moore and Lockner, 2013) and studies of fault-rock outcrops in the
68 creeping segment of the SAF that suggest the creep behavior involves a continuous generation of
69 low-friction smectite clays due to entrainment of serpentinite bodies in this section of the fault
70 over the past 2.5 million years (Page et al. 1998; Titus et al. 2011). Experimental work by Bos
71 and Spires (2001 and 2002) indicates that a hybrid deformation mechanism model involving
72 frictional-viscous flow of phyllosilicates and pressure solution may be applicable to mature fault
73 zones. A firm understanding of the mechanism of aseismic creep and its evolution over the
74 entire seismogenic profile would provide insight into the question about the potential for
75 damaging seismic ruptures in the creeping section of the SAF.

76

77 **2. The SAFOD sample**

78

79 The SAFOD is sited on the Pacific plate 1.8 km southwest of the surface trace of the San
80 Andreas Fault, and 9 km northwest of Parkfield, California. The observatory's ~4km long drill
81 hole (~2.77km vertical) penetrates Salinian block in the Pacific plate and turns northeast across
82 the fault into the Great Valley/Franciscan block in the North American plate. The geologic
83 setting of the site indicates contractional, extensional and strike-slip deformation with the
84 resulting structural and material complexities. The plate motion in the central section of the SAF,
85 including at the SAFOD site, is accommodated via aseismic creep. For detailed lithological
86 descriptions and further information regarding the geology and seismotectonics in the vicinity of
87 the SAFOD, the reader is referred to other studies (e.g. McPhee et al. 2004, Thayer et al., 2004,
88 Arrowsmith et al. 2005, Titus et al. 2006, Bradbury et al. 2007 and 2011, and Holdsworth et al.
89 2012).

90

91 This study is based on two polished thin sections from a single sample with well-
92 developed calcite-filled veins in clasts and matrix of the cataclasites and the presence of
93 significant pyrite. The use of a single suitable sample is vindicated by the main aim of this paper
94 which is to investigate the hypothesis that segments of fault history can be determined through
study of episodically developed calcite and pyrite microstructures. We want to see if combined

95 study of calcite and pyrite microstructures can provide a tool to document otherwise unavailable
96 information about fault evolution through time. While other studies of SAFOD have studied
97 multiple samples (e.g. Moore and Rymer, 2007; Carpenter et al., 2009 and 2011; Bradbury et al.,
98 2011; Schleicher et al., 2009a, 2009b, 2010, and 2012; Janssen et al., 2012; Lockner et al., 2011;
99 Moore and Rymer, 2012; Hadizadeh et al., 2012; Moore and Lockner, 2013; Moore, 2014; Warr
100 et al., 2014; Morrow et al., 2014), none have addressed the potential combined use of calcite and
101 pyrite to elucidate fault history. Successful demonstration of using calcite and pyrite as tools for
102 determining SAFOD history should encourage their use in a wider range of samples.

103 Our sample was extracted as a billet measuring $\sim 75 \times 70 \times 50$ mm from interval 3193.91m
104 to 3193.98m Measured Depth (MD) of run 2, core section 4 of the lateral borehole G (Fig. 1a).
105 The core sample through the fault rock consists of a hard, dark-colored foliated siltstone-shale
106 ultracataclasite with visible veins running sub-parallel to foliation (Fig. 1b). Note that although
107 the ultracataclasite is a cohesive fault rock, we simply refer to it as the gouge. The sample billet
108 included a sharp border with a massive gray-black shale toward the core bottom (NE end of the
109 core section).

110 The lateral boreholes depicted in Fig. 1a were drilled in phase III of the SAFOD project
111 after actively creeping zones were discovered in the SAFOD Main Hole in phase II. The two
112 actively creeping strands of the SAF named the Southwest Deforming Zone (SDZ) and Central
113 Deforming Zone (CDZ) were identified within a ~ 200 m-wide low-velocity zone, which is
114 considered to be the SAF damage zone (Zoback et al., 2010, 2011). Our sample was located at
115 the margins of the damage zone marked in Fig. 1a, ~ 2.5 m SW of the actively creeping SDZ
116 strand at 3196.5m, MD. Notwithstanding the low-velocity extent of the damage zone, Bradbury
117 et al. (2011) estimated width of the SAF damage zone to be at least 350m including regions that
118 may not be currently active. Holdsworth et al. (2011) considered the gouge outside the creeping
119 strands to be inactive. Terminus of the SAFOD Main Hole, at true vertical depth of ~ 2.7 km, has
120 registered a temperature of $112 \pm 2^\circ\text{C}$.

121 Several different intervals from the G24 core section have been sampled and studied by
122 others (e.g. Gratier et al., 2011; Holdsworth et al., 2011, Bradbury et al., 2011, 2015; Moore
123 2014). This core section was characterized as black cataclasite and ultracataclasite with localized
124 mm to sub-mm pyrite and calcite veins running sub-parallel to cataclastic foliation. The gouge
125 was found to be uniquely rich in carbonaceous phases, suggesting hydrocarbon migration in the

126 gouge, and unusually enriched in disseminated secondary pyrite. Bradbury et al. (2011) showed
127 the disseminated pyrite was present in the SDZ wallrock on both sides, and on the NE side of the
128 CDZ. Bradbury et al. (2011) and Holdsworth et al. (2011) suggested that presence of
129 disseminated pyrite, magnetite, and organic carbon in the wallrocks adjoining SDZ and CDZ
130 may be related to the observed loss of Fe from the actively creeping zones.

131

132 **3. Methods**

133 The sample billet was impregnated with clear epoxy resin and cut for two parallel
134 petrographic thin sections ~10mm apart (G24a, and G24b) at right angles to foliation to enable
135 characterization of the microstructural change within foliation planes. The section surfaces were
136 machine-polished using 0.05 μ m silicon particle colloidal suspension and vapor-coated by thin
137 carbon deposit. However, most of the analytical work used section A as the 10mm depth
138 revealed little microstructural change. Deformation microstructures were studied using a Zeiss
139 Supra 35VP high resolution SEM with backscattered electron detector and EDX capabilities.
140 Selecting typical areas for electron microscopy was facilitated by creating reference image
141 mosaics using reflected-light optical microscope where pyrite appeared in sharp contrast against
142 the siliciclastic gouge.

143 The presence of both deformed and undeformed pyrite in the sample prompted a more
144 focused microstructural and crystallographic characterization of the pyrite phase via SEM-based
145 electron backscatter diffraction (EBSD), analysis. For EBSD the samples were polished on a
146 polyurethane lap for one hour using a suspension of 0.05 μ m colloidal silicon (SYTONTMTM) to
147 remove any surface damage. Samples were given a thin carbon coat prior to EBSD to reduce
148 charging effects and maintain a strong crystallographic signal (Prior et al., 1996). All
149 crystallographic data were collected at the University of Liverpool using a CamScan X500
150 crystal-probe scanning electron microscope (Bestmann et al., 2003) with a thermionic field
151 emission gun, an accelerating voltage of 20kV and a beam current of ~5nA. This technique has a
152 typical angular resolution of <1° and a spatial resolution of ~0.05 μ m. All data were processed
153 using the software package CHANNEL5 (Oxford Instruments Limited). Sets of rectilinear maps
154 were collected with a 0.5-1.5 μ m sampling spacing. Typically, around 70% of points in the grid
155 were successfully indexed. To improve the completeness of maps and thus obtain better grain
156 size, shape and misorientation statistics, the Tango utility (part of CHANNEL 5 software) was

157 used in a systematic way to interpolate data by growing existing mapped grains to fill the non-
158 indexed areas without introducing artefacts (Barrie et al., 2007; Bestmann et al., 2003). Using
159 this reconstruction method generally improved map coverage to ~ 90%. Tango was then used to
160 define grains based on lattice orientation; to investigate lattice misorientation data for
161 visualization of Euler-angle maps and grain-boundary misorientation; to produce band contrast
162 (BC) images from the contrast between diffraction bands and background; and to produce data
163 subsets for plotting misorientation histograms and plotting pole figures (using Mambo). A
164 discussion of the systematic errors in EBSD data collection process, directly applicable to this
165 study, is presented by Prior et al. (1996) and Barrie et al. (2007).

166 We used Cathodoluminescence (CL) images and spectral color variations of calcite vein
167 networks in the sample to better understand the origin and relative order of the calcite veining
168 and its correlation with pyrite deformation obtained from the EBSD data. The CL images were
169 acquired optically using a CITL-Mk5 cold CL stage system and exposure time between 0.5 and
170 10 seconds to maximise dynamic range in the acquired image. Since the calcite vein network
171 mostly consisted of vein-saturated clasts and narrow 50-150 μm wide individual veins in the
172 gouge matrix, the spectral analyser in the instrument could not provide uniformly reliable results.
173 Instead, we estimated the spectral values for CL colors of distinct vein generations by a digital
174 color-sampling method: A high-resolution strip image of standard visible-light spectrum (350-
175 750nm band measuring 3500×646 pixels currently available from NASA.gov, or
176 Wikimedia.org) was calibrated using the graduated 1964 CIE color space such that 8.75 pixels
177 on the spectrum band represented $\sim 1\text{nm}$ of wavelength on the color space. Image processing
178 software Sigma Scan Pro[©] v.5 was then used to acquire several multi-pixel color samples from
179 uniformly-colored regions of each distinct calcite vein on CL images. Upon zooming, the color
180 samples yielded small (100-400 square pixel) apparently mono-color areas, which was reshaped
181 into a slender bar measuring $\sim 8.75 \times 646$ pixels for use with the calibrated chart. The color
182 sample bar was moved against the graduated chart until it became indistinguishable from the
183 chart color within a very narrow range of wavelengths. At this sampling scale, the color samples
184 were mostly mono-color, but occasionally included pixels of a different color. In all
185 measurements, however, a color sample matched the chart colors if it was moved back and forth
186 within a 25 pixel band ($\sim 3\text{nm}$) in the calibrated chart, which gave a $\pm 3\text{nm}$ accuracy for the
187 wavelength estimates.

188

189 **4. Results**

190 4.1 Microstructures and general compositional observations

191 The gouge retains a well-defined shape-preferred orientation (SPO) foliation in a clast-in-
192 matrix microstructure (Fig. 2a-b), where large fragments in the gouge consist of extensively
193 fractured and veined elongate sandstone and arkosic siltstone clasts (long axis ~200 μ m to a few
194 mm). Some of the clasts are cemented aggregates of reworked and intricately veined quartz-rich
195 fragments (Fig. 2c). Similar microstructural features in this and other gouge samples from the
196 SAFOD damage zone cores have been noted and described (e.g. Holdsworth et al. 2011,
197 Bradbury et al. 2011, Richard et al. 2014). The clasts commonly host a network of calcite
198 fracture-sealing veins that generally run at large angles to the clast length (Fig. 2c). It has been
199 shown that in-clast calcite veins in samples from G24 core section, including our sample,
200 resulted from sealing of fractures that propagated from grain contact impingement points at high
201 angles to pressure solution seams (Gratier et al., 2011; Hadizadeh et al., 2012). The pervasive
202 presence of this vein fabric suggests that the gouge foliation (dashed line S in Fig. 2) is a
203 pressure solution cleavage. Evidence of pressure solution in the studied sample is in agreement
204 with previous studies of the damage zone rocks exposed in Holes E and G (Schleicher 2009a;
205 Holdsworth et al 2011; Hadizadeh et al. 2012; Richard et al. 2014; Bradbury et al. 2011, 2015).
206 The gouge matrix comprises shale and clay-rich alteration products of highly comminuted rock
207 fragments (mainly quartz and feldspars). In contrast to calcite veins in the clasts, the calcite veins
208 in the matrix generally run parallel to foliation (Fig. 2a-c). Pyrite in the sample appears as
209 framboids, polycrystalline blocks and stretched clusters.

210 Pyrite framboids appearing within and around the clasts in Fig. 3 are presumed to be the
211 primary diagenetic pyrite in the fault zone protolith (Sawlowicz 1993; Rickard 1970). The
212 disseminated pyrite similar to those shown in Fig. 3 was described by Holdsworth et al. (2011) as
213 relatively late-stage authigenic pyrite. We do not find direct evidence of relationship between
214 this primary pyrite and the polycrystalline pyrite associated with calcite veins in our sample. It is
215 likely that the polycrystalline pyrite in the gouge is a byproduct of secondary epigenetic
216 mineralization from hydrothermal fluid-rock interactions as HS⁻-bearing H₂O-CO₂ fluids were
217 focused in the SAF (Menzies et al. 2016). Mittempergher et al. (2011) reported the presence of
218 anhydrite (CaSO₄) veins in the SAFOD damage zone samples. Experimental work has shown

219 pyrite could form at $T > 200^{\circ}\text{C}$ via reduction of anhydrite in a hydrothermal environment
220 (Graham and Ohmoto 1994). Most relevant to this study, however, is the deformation of the
221 secondary pyrite (hereafter simply referred to as the pyrite, unless otherwise is stated) and its
222 implications for the deformation mechanisms of creep in the SAF at elevated temperatures. As
223 shown in Figs. 2a and 2b, the bulk of the polycrystalline pyrite in the sample appears in two
224 different microstructures: A sigmoidal-block (8×5 mm) and an elongate ~ 3.5 mm long cluster
225 varying in width from 100 to $500\mu\text{m}$ (Fe-map inset in Fig. 2b). These microstructures suggest a
226 range of formation and deformation conditions for the pyrite. The sigmoidal-block pyrite
227 consists almost entirely of xenomorphic polycrystalline pyrite with no other sulfide minerals
228 present (Fig. 4a). Clear evidence of deformation by brittle fracturing exists as well as brittle
229 attrition at contacts with the gouge matrix (Fig. 4b). The block is smeared out along the foliation
230 in a σ -type kinematic indicator, where the smear consists of comminuted pyrite crystals (Fig. 4c)
231 stretched out along the foliation. The elongate-cluster-pyrite and its margins (Fig. 5) record a
232 different type of deformation and growth relationship (Fig. 5) than the passive brittle attrition
233 observed in the sigmoid-block pyrite. Notable microstructural features at the margins of the
234 elongate-cluster-pyrite include pyrite crosscutting calcite veins (Fig. 5a-c) and implosion
235 microbreccia with pyrite-sealed jigsaw texture (Fig. 5d).

236

237 4.2 The Cathodoluminescence of calcite veins

238 Calcite vein networks in the sample targeted for the Cathodoluminescence (CL) study
239 were found in three categories of microstructures, namely: clasts of different size and vein
240 density (labelled C and SC), veins in clay-rich matrix surrounding the clasts (labelled M), and
241 the gouge regions where pyrite crosscuts the calcite veins (labelled P in Fig. 6a). The large
242 siltstone clast (SC) in the sample (Fig. 6a) is less densely pervaded by calcite veins than the,
243 smaller, quartz-rich clasts (C). Instrument-output CL images for the representative
244 microstructures are shown in labelled boxes in Fig. 6b. We note that CL imaging was carried out
245 at five location in one siltstone clast (SC1 through SC5), but only two representative images
246 (SC1 and SC2) are displayed in Fig. 6b. They are representative because the calcite-vein
247 luminescence characteristics showed little variability over the entire clast. The CL data for
248 calcite veins in the gouge matrix (M) were collected from the veins marked in Fig. 6b-C2 and C4
249 (solid white arrows). Except for fracture-sealing calcite in the pyrite block (Fig. 6b-P2), which

250 we regarded as a matrix vein, all other matrix veins were found to be sub-parallel to the trace of
251 the foliation.

252 In general, luminescence images of the vein-calcite tend to change from dark red
253 (679nm-e.g. SC1, SC2 in Fig. 6b) to a bright red-orange color (588nm-e.g. vein marked M, in
254 Fig. 6b-C2). The luminescence range, however, includes two distinct step-downs between the
255 maximum and minimum wavelength values as presented in a box-whisker plot of luminescence
256 wavelengths based on 78 spectral measurements (Fig. 7). The plot reveals a significant
257 difference in median wavelength values between the large siltstone clast (672nm) and a group of
258 microstructures consisting of smaller quartz-rich clasts and the veins crosscut by the pyrite
259 (622nm). The decrease in median wavelength value for the matrix veins relative to the smaller
260 clasts group appears to be transitional as indicated by the fitted curve in Fig. 7. The EDX spot
261 analysis showed combined Fe + Mn + Mg elemental concentrations of ~1 wt.%, ranging from
262 0.31-1.63 wt.% in the typical calcite vein composition. The results plotted in a ternary diagram
263 (Fig. 8) indicate that calcite veins with dull luminescence have high mean atomic wt.%
264 concentration of Fe (0.44) with lower concentrations for Mn (0.34), and Mg (0.23). The bright
265 veins, on the other hand, have high mean atomic wt.% concentration of Mn (0.67) with lower
266 concentrations for Fe (0.23) and Mg (0.11). The mean atomic wt.% concentrations for the
267 medium luminescence veins also have high mean atomic wt.% concentrations of Mn (0.44) with
268 lower Fe (0.3) and Mg (0.15). Correlation of the calcite luminescence color and ternary
269 distribution of these elements as indicated in Fig. 8 is consistent with previous findings that
270 suggest Fe acts as a quenching agent in calcite luminescence (Machel 2000).

271 272 4.3 EBSD ***

273 The results of Electron Backscattered Diffraction (EBSD) study of the sample
274 concentrate on pyrite and are presented as Euler-angle maps with grain boundaries classified by
275 misorientation angle, misorientation histograms and profiles, and pole figures. The sigmoidal-
276 block pyrite and elongate-cluster pyrite microstructures are of particular interest because they
277 texturally interact with the calcite vein networks and matrix of the gouge (Figs. 2-5).

278 Figure 9 contains two Euler-angle maps summarising EBSD-derived relationships in part
279 of the sigmoidal-block pyrite located in Fig. 4a. Fig. 9a is an 800×500 μm area analysed at 1 μm
280 step size, which shows the pyrite aggregate has undergone brittle deformation including a curved

281 thoroughgoing microfracture and a 100 μm wide zone of attrition breccia. Fig. 9c is a 180 \times 135
282 μm sub area of Fig. 9a analysed at a 0.2 μm step size. Grain sizes are typically $<5\ \mu\text{m}$ up to ~ 100
283 μm . The wide Euler-angle color variations between pyrite grains in Figures 9a and 9c reflect the
284 absence of any significant crystallographic preferred orientation (CPO) in the pyrite aggregate,
285 which is typical due to the cubic symmetry of pyrite (Barrie et al., 2007). The grain-boundary
286 colors indicate a significant number of low-angle misorientations: white ($1\text{-}2^\circ$) and yellow ($2\text{-}5^\circ$)
287 boundaries. The same excess of low-angle boundaries is summarised in the misorientation
288 histograms in Figures 9b and 9f. The random-pair (uncorrelated) histograms correspond closely
289 to the theoretical random distribution, which is common in many cases of massive sulphide-ore
290 pyrite deformation (Barrie et al., 2010a; Barrie et al., 2010b; Barrie et al., 2007; Boyle et al.,
291 1998). On the other hand, the neighbour-pair (correlated) misorientation distributions differ
292 significantly from the theoretical random distribution confirming the development of low-angle
293 grain boundaries. On the basis of grain boundaries being defined by a 2° misorientation, about
294 8% of the 8,714 pyrite grains identified in Figure 9a have a mean internal misorientation of 1° or
295 greater, with a maximum mean internal misorientation of 6.5° .

296 The higher resolution analysis shown in Fig. 9c illustrates a microstructure where some
297 larger grains of pyrite are mantled by smaller grains of pyrite. Two misorientation profiles across
298 one of the larger grains and the adjacent smaller grains in Fig. 9d indicate the development of a
299 core and mantle relationship (Urai et al., 1986), with the small grains having progressively
300 rotated lattice orientations relative to the large core pyrite grain. Pole figures in Fig. 9e
301 summarise lattice orientations in three of the larger grains in Fig. 9c. The large central pale-blue-
302 colored grain (i) shows a dispersion of $\langle 100 \rangle$ and $\langle 110 \rangle$ orientations with no systematic rotation
303 around any particular pyrite crystallographic axis. The upper large purple-colored grain (ii) in
304 Fig. 9c has developed a low-angle grain boundary to form two main sub-grains misoriented by a
305 rotation about the near-vertical $\langle 110 \rangle$ axis in Fig. 9e (ii). The area (iii) in Fig. 9c consists of two
306 grains sharing a high angle ($>25^\circ$) boundary. However, given the nature of deformation in the
307 neighbouring grains (i) and (ii), and lattice rotation about a common $\langle 100 \rangle$ axis (Fig. 9c, pole
308 figure for iii) we infer that the two grains in (iii) were part of a single large grain. The alternative
309 interpretation is that the common $\langle 100 \rangle$ direction is purely coincidental.

310 The EBSD results from a part of the sigmoidal-block pyrite strongly affected by calcite-
311 sealed brittle fracturing are presented in Fig. 10. The Euler-angle map (Fig. 10b) shows most

312 pyrite grains are $<10\mu\text{m}$ in size, ranging up to $\sim 100\mu\text{m}$, and low-angle sub-grains with
313 misorientation boundaries $\leq 5^\circ$ are abundant (boundaries mapped in white and yellow in Fig 10b).
314 The misorientation histogram of the map area in Fig. 10c reveals that uncorrelated grains have a
315 misorientation-angle distribution close to the expected theoretical distribution for randomly
316 oriented pyrite grains. In contrast, the correlated misorientation angle distribution (pairs of grains
317 with a mutual grain boundary) in Fig. 10c differs markedly from the expected theoretical
318 distribution, most notably by having an excess of low angle misorientation boundaries. This
319 distribution is simply a reflection of the abundant low-angle boundaries mapped in Fig. 10b
320 indicating the development of pyrite sub-grains.

321 Moving on to the elongate-cluster pyrite (referenced in Figs. 2a and 5c), the X-ray
322 element map in Fig. 11a, the CL image in Fig. 11c, and the BSE image in Fig. 11d clearly show
323 pyrite-growth microstructures continue out from margins of the elongate-cluster pyrite to
324 crosscut both quartz clasts and calcite veins. The Euler angle map (Fig. 11b) shows that the core
325 of the elongate cluster consists of fine-grained (mostly $<5\mu\text{m}$) polycrystalline pyrite with
326 abundant low-angle misorientation boundaries (mapped white and yellow), confirmed by the
327 misorientation histogram in Fig. 11g. In contrast, the microstructures at the elongate-cluster
328 margins comprise relatively large grains with locally idiomorphic outlines and little or no
329 internal microstructure. Figs. 11e and 11f show misorientation angle profiles for the 6 EBSD
330 transects in Fig. 11b. Profile 1 (Fig. 11e-1) demonstrates sub-grain development in one of the
331 larger elongate-cluster core grains, whereas Profile 2 (Fig. 11e-2) demonstrates no measurable
332 internal strain (total angle change of $<1^\circ$) in a marginal idiomorphic pyrite grain that cross-cuts
333 calcite veins in the adjacent quartz. Profile 3 (Fig. 11e-3) crosses a large ($<200\mu\text{m}$) pyrite grain
334 adjacent to the elongate cluster. The pyrite grain contains calcite inclusions with similar CL
335 colouring to the calcite veins that cut the adjacent quartz. The profile indicates the development
336 of a low-angle misorientation boundary and little or no other measurable microstructure. Profile
337 4 (Fig. 11e-4) is across another pyrite grain adjacent to the elongate cluster. This small grain cuts
338 calcite veins in the quartz and records evidence of significant internal misorientation. Profile 5
339 (Fig. 11f-5) illustrates progressive misorientation and the development of Dauphiné twins in a
340 large quartz clast bordered by calcite veins. Profile 6 (Fig. 11f-6) shows that quartz cataclasite
341 fragments, cemented by calcite veins, are separated by sub-grain boundaries with misorientation
342 angles mostly ranging from $2\text{-}10^\circ$. The central region of the pyrite-sealed implosion

343 microbreccia (see SEM image of Fig. 5d) was selected for the EBSD analysis in Fig. 12. An X-
344 ray element map shown in Fig. 12b reveals distribution of S, Si and K and resolves the
345 compositional make-up of the area in Fig. 12a. The 0.6 μ m step-size Euler map (Fig. 12c) and
346 misorientation profile (Fig. 12d) of the pyrite sealing the breccia indicate it is a single crystal
347 with a cumulative lattice misorientation of $\sim 3^\circ$ across $\sim 80\mu$ m profile length.

348

349 **5. Discussion and conclusions**

350

351 5.1 What do SAFOD damage zone rocks represent?

352 It is debatable whether the damage zone rocks bordering CDZ and SDZ are relicts of
353 locked section of the SAF to the south, or they represent uplifted deeper sections of the currently
354 creeping segment of the fault. The answer to this question seems to depend on the overall
355 trajectory of the N-NW-moving block of the SAF as well as interactions of the block with the
356 entrained serpentinite bodies on the E-NE block of the fault. The horizontal component of the
357 trajectory for the last 1-2 million years involves ~ 30 km strike-parallel displacement between the
358 locked-to-creeping transition boundary at Cholame, SE of Parkfield and position of SAFOD drill
359 site. Assuming E-NE block of the SAF stationary, at a displacement rate of ~ 30 mm/year, it
360 would take the locked section rocks ~ 1 million years to be placed at SAFOD site. Although the
361 creeping section of the SAF in central California has been the subject of numerous geophysical
362 and geotectonics studies (e.g. Wang et al. 1984; Montgomery 1993; Page et al. 1998; Townend
363 and Zoback 2004; Rolandone et al. 2008; Blythe et al. 2004; Titus et al. 2007 and 2011), the
364 uplift component of the trajectory remains uncertain. Based on fission-track data from the
365 SAFOD pilot hole, Blythe et al. (2004) estimated that the site had experienced ~ 1 km of uplift in
366 the past 4-8 million years. Schleicher et al. (2009) estimated a temperature of 200°C for the
367 formation of phyllosilicates (illite-smectite) from the bottom of the Main Hole. At $35^\circ\text{C}/\text{km}$
368 gradient, this would represent an uplift of ~ 2.5 km. It is not clear how much of this uplift could
369 predate faulting. Based on sedimentary basin temperature profiles, Moore (2014) estimated the
370 upper and lower temperature limits for saponite-to-corrensite transition respectively at 225°C
371 and 125°C and suggested that the lower limit requires ~ 0.5 km uplift of the SDZ rocks. She
372 suggested that the cooling might have resulted from up-dip movement of low-density material in
373 the active bands relative to the damage zone rocks. However, the net uplift estimates are further

374 complicated by the buoyancy-driven up-dip movement of low-density bodies of gouge within the
375 entire damage zone of the SAF (Wang 1984). With respect to the latter point, it is important to
376 note that mineral signatures of serpentinite alteration (e.g. chrysotile, lizardite, and smectite clays
377 as well as chromium oxide and magnetite) are present in the damage zone rocks (Fig. 3,
378 Bradbury et al. 2012). Thus, emerges a very gently uplifting N-NW-directed displacement
379 trajectory for the quartzofeldspathic rocks, which are likely to include strands/pods of the clay-
380 rich gouges progressively generated through displacement as well as hydrothermal reactions with
381 the entrained serpentinite bodies (Moore and Rymer 2012). We, therefore, propose that the
382 damage-zone rocks encountered in the SAFOD have an interlaced structure consisting of the
383 SAF locked-section relicts as well as clay-rich gouge that represents deeper sections of the
384 creeping segment of the fault.

385

386 5.2 Analysis of sample results

387 We identified two distinct generations of calcite veins. The veins in the
388 survivor/reworked clasts were generally perpendicular to enclosing foliation (Fig. 2), which
389 allowed us to define the foliation as pressure solution cleavage. We also noted calcite veins sub-
390 parallel to foliation (white arrows in Fig. 2c) and calcite-sealed late fractures in the block pyrite
391 (Fig. 4 and Fig. 6-P2). The CL results enabled us to resolve the two vein generations in terms of
392 relative depth of crystallization (P1 and P2 in Fig. 13). Studies of calcite from different
393 geological environments have attributed the CL sequence non-luminescent, bright, and dull
394 emission in calcite to a progressive decrease in the oxidation state of the pore water and,
395 therefore, deeper environments during crustal burial (Grover and Read 1983; Machel et al. 1985;
396 McManus and Wallace 1992; Machel 2000; Verhaert et al. 2004). We assume that, at the
397 minimum, the observed luminescence of calcite veins in both clasts and matrix of the gouge (Fig.
398 6b) suggests a reducing tectonic environment mostly isolated from direct interaction with
399 oxygen-rich meteoric water. The analysis of visible-light wavelengths in the CL images in Fig. 7
400 indicates that luminescence of the studied calcite veins varies with the type of microstructure
401 they pervade. The wavelength median values (curve fitted to Fig. 7 plot) suggest that veins in the
402 large arkosic siltstone clast (SC boxes in Fig. 6a-b) precipitated at a greater depth relative to the
403 vein-calcite elsewhere in the sample (Fig. 13). The calcite veins from the deepest environment
404 are part of this microstructural record simply because they happened to be in this particular large

405 survivor clast. Thus, in this instance, clast size difference can explain the large difference in CL
406 wavelength between SC veins and other calcite veins in Fig. 7. The lowest luminescence median
407 values in Fig. 7 are from the foliation-parallel matrix veins (marked M in images C2, C4 in Fig.
408 6b and in Fig. 13), which we consider unrelated to pressure solution cleavage. The CL
409 wavelength plot in Fig. 7 suggests that late fractures in the pyrite sigmoidal block (P2 in Fig. 6b
410 and 13) are sealed with calcite that precipitated at depths equivalent to the bottom of the SAFOD
411 borehole (2-4km). Luminescence of the veins in the highly reworked, quartz-rich clasts thus falls
412 within an intermediate range of depths relative to the end members. The wavelength median
413 value for calcite luminescence in the quartz-rich clasts is ~620nm, which is close to the observed
414 typical calcite CL (Cazenave et al. 2003). We also independently used the trace element
415 proportions in Fig. 8 to check the luminescence results. A highly debated factor affecting the
416 range of luminescence of calcite is the Fe/Mn ratio in the atomic structure of calcite (both
417 elements in ppm concentrations), which also correlates with pore-water chemistry (Barnaby and
418 Rimstidt 1989; Budd et al. 2000; Habermann et al. 2002; Cazenave et al. 2003). In general,
419 increasing Fe tends to suppress (quench) luminescence while Mn acts as an enhancer of the
420 luminescence. The latter effect is demonstrated in Fig. 8 as high wt.% concentrations of Mn in
421 the bright and medium luminescence calcite veins. The highest and lowest Fe/Mn ratios (1.30
422 and 0.35) occur in the dull and bright luminescence calcite veins, respectively. The change in
423 luminescence of the calcite veins suggests that earliest microstructures in the gouge were
424 deformed at a deeper, more reducing environment (Fig. 13), where more iron atoms were
425 available to be incorporated into the CaCO₃ unit cell. The trace element concentration data
426 together with the observed range of calcite vein luminescence, and a fabric defined by the vein
427 network suggest clear evidence of deformation by pressure solution in the gouge that was
428 transported and uplifted to the current position in the SAFOD borehole.

429 We use the revised pyrite deformation mechanism map published by Barrie et al. (2011)
430 to infer that pyrite should deform by brittle and/or Coble-creep mechanisms under the observed
431 SAFOD P-T-e• conditions (~3km, ~120°C, and ~10⁻¹⁰/s to ~10⁻¹²/s strain rates corresponding to
432 creep rates of ~25 mm/year). While the pyrite deformation involved brittle fracture, the pyrite
433 also deforms by other mechanisms. The sigmoidal block and the main part of the elongate cluster
434 are characterised by small grain-size, distributed misorientations, abundant low-angle boundary
435 misorientations, and subgrain development. Further, the core and mantle relationship in Fig. 9c is

436 consistent with progressive misorientation of the core pyrite and the development of new grains
437 by subgrain rotation recrystallization (SGR) that developed a mortar texture (Urai et al., 1986). A
438 more general occurrence of SGR would be consistent with the observed development of excess
439 low-angle misorientation boundaries in both the sigmoidal block and the elongate cluster
440 microstructures.

441 The only previously reported case of SGR deformation in pyrite is in Barrie et al. (2007,
442 Figure 15) in a study of the samples experimentally deformed by Cox et al. (1981) at 300 MPa, a
443 strain rate of $2 \times 10^{-4} \text{s}^{-1}$, and a range of temperatures from 550°C to 700°C. Subgrain rotation
444 deformation was identified in the samples deformed at 600-650°C, whereas the lower
445 temperature run at 550°C was characterised by bulging deformation, and the 700°C run by
446 complete recrystallization and loss of low-angle boundaries, which is analogous to dislocation
447 creep regimes 1, 2 and 3 of Hirth and Tullis (1992) for quartz aggregates. The lack of widespread
448 development of classic regime 1 bulging and regime 2 SGR microstructures in naturally
449 deformed pyritic ores is probably due to the effects of weaker co-existing minor sulphide
450 minerals such as chalcopyrite, sphalerite and galena that commonly occur in pyrite parageneses
451 (Barrie et al., 2011). Cox et al. (1981) selected a very pure polycrystalline pyrite rock for their
452 experiments, and the sampled SAFOD pyrite aggregates are also very pure without other
453 sulphide minerals. Barrie et al. (2010a, Figure 8) documented evidence of bulging deformation in
454 a pyritic ore from the Godthåp mine in the Røros area of Norway deformed in biotite-zone pelitic
455 rocks during the Caledonian orogeny. The biotite isograd in low to medium pressure
456 metamorphic terrains is generally considered to represent a temperature of at least ~400°C with
457 garnet commonly appearing at ~470-480°C (Ferry, 1984; Wang et al., 1986), bracketing
458 temperature conditions for bulging deformation in pyrite at typical orogenic strain rates. Thus,
459 the SGR deformation microstructure in pyrite aggregates in our sample represents a temperature
460 significantly above 400°C. We therefore argue that the bulk of these sigmoidal block and the
461 elongate cluster pyrite aggregates experienced SGR deformation at a temperature significantly
462 above 400°C (Fig. 13), representing the oldest recorded deformation in these pyrite aggregates.

463 The side of the elongate-cluster pyrite facing the large siltstone clast boundary (Figs. 2
464 and 5) is characterised by fringing growth of relatively large, often idiomorphic pyrite grains
465 with little or no internal deformation (Fig. 11). These pyrite grains commonly crosscut calcite
466 filled veins and contain inclusions of calcite with similar CL colour to the vein calcite. In places,

467 the large pyrite grains form the seal to breccia, here interpreted as an implosion breccia (Fig. 12).
468 The EBSD results (Figs. 9-12) thus indicate three contrasting styles of pyrite microstructure in
469 the two clusters: earlier grain cores characterised by SGR deformation and T conditions
470 significantly below the present SAFOD depth (Fig. 13), later fringing growth rims that cut calcite
471 veins and preserve implosion breccias, and finally brittle fractures consistent with present-day
472 SAFOD conditions (calcite veins designated P2 and M in Figs. 6, 7 and 13). This interpretation
473 implies episodic pyrite mineralization at different times over a range of depths prior to late brittle
474 fracturing, and is consistent with the CL results indicating that late brittle fractures in the
475 sigmoidal-block pyrite are sealed by the youngest calcite veins (Fig. 4b and Fig. 6b-P2). Since
476 the sample is only ~2.5m away from the SDZ, the late brittle fractures in the sigmoidal-block
477 pyrite (Fig. 4) could be the result of a local frictional stress transfer from this active shear zone.
478 In the elongate-cluster pyrite, deformation ranges from sub-grain rotation by dislocation creep in
479 the interior (Fig. 11b traverse 1) to almost undeformed idiomorphic crystals at the fringe (Fig.
480 11b, traverse 2). The calcite veins that formed at depths below the SAFOD (CL results in Fig.
481 6b-P1) as well as plastically deformed fragments of the quartz cataclasite (Fig. 11b, traverse 5)
482 are crosscut by successive epitaxial growths of the fringe pyrite crystals. The pyrite-sealed
483 implosion microbreccia (Fig. 12a) appears to be a late event in the pyrite crystallization at
484 hydrothermal temperatures (~200°C). The Euler angle map in Fig. 12c shows that the pyrite seal
485 in Fig. 12a is made of a relatively large single crystal with little to no crystal plastic deformation
486 (misorientation profiles in Fig. 12d). The microbreccia is further evidence of episodic nature of
487 hydrothermal events that involves pyrite mineralization in the SAFOD damage zone rocks. It has
488 been successfully argued that such microstructures indicate coseismic events in fault zones (e.g.
489 Sibson 1986; Holdsworth et al. 2011; Bradbury et al. 2011, 2015). Evidence of transient brittle
490 deformation due to local fluid overpressure in samples from the SAFOD damage zone gouge was
491 also reported by Mittempergher et al. (2011). The discussed results are synthesized in Fig. 13,
492 which plots calcite and pyrite microstructure development against depth and temperature.

493 As discussed in 5.1, the inactive damage zone of the SAF at SAFOD consist of discrete
494 and well-mixed structural and material domains representing both locked and creeping sections
495 of the fault. Our results provided evidence of pressure solution microstructures as well as
496 episodic hydrothermal pyrite mineralization and pyrite sealing in implosion microbreccia. While
497 the secondary pyrite (and anhydrite reported by Mittempergher et al. 2011), is likely to represent

498 microearthquake activity at a range of depths (Jolivet et al. 2015) in the creeping segment, it is
499 possible for the pressure solution cleavage in the damage zone rocks to develop in either
500 creeping or locked section of the SAF at depths >3km. The evidence of pressure solution in
501 SAFOD cores is taken by some previous studies to represent the dominant deformation
502 mechanism of the aseismic creep over the entire seismogenic zone. Gratier et al. (2011)
503 attributed the microseismic activity in a pressure solution regime to cyclical increase in local
504 diffusion distance required for mass transfer from dissolution sites. They suggested that in the
505 SAF creeping section, regularly recurring microearthquakes would be necessary for the pressure
506 solution to operate at displacement rates > 20mm/year and depths >3km. In this case, the
507 cleavage-related calcite veins generated at a range of depths should be found crosscut and
508 disrupted by co-seismic mineralization of that depth range as the fault rocks are uplifted to
509 SAFOD depth. In the view of this study, however, it is also possible that the observed pressure
510 solution microstructures represent interseismic deformation in the locked section of the SAF,
511 having developed prior to juxtaposition against entrained serpentinite bodies in the E-NE block
512 of the fault. Our EBSD results (Fig. 11) support the latter case since we show that multiple
513 episodes of pyrite crystallization and deformation do not crosscut a single generation of the
514 cleavage-related calcite veins as identified by CL. The latter observation implies that the weak-
515 clay frictional deformation mechanism(s) rather than pressure solution creep is likely to be the
516 predominant aseismic creep mechanism at depths below the SAFOD. It is also possible to infer
517 that the repeating microearthquakes in the creeping section may be due to strain accumulation
518 associated with transposed quartz-rich inclusions of the SAF locked section (also see Collettini et
519 al. 2011; Jolivet et al. 2015). It is important to note that similar studies on a larger sample set are
520 needed to confirm or rule out these implications for the deformation behaviour of the creeping
521 section of the SAF. However, this detailed study of a single sample does demonstrate the utility
522 of using combined calcite and pyrite microstructural analysis to elucidate fault history,
523 suggesting the approach should be more widely used.

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Figure Captions

Figure 1

The SAFOD core sample. (a): Section at $\sim 90^\circ$ to surface trace of the San Andreas Fault plane in central California, referenced to SAFOD drill site at Middle Mountain as zero distance on x-axis (not shown). Actively creeping strands of the SAF, Southwest Deforming Zone (SDZ) and Central Deforming Zone (CDZ), were cored with the lateral drilled holes (red lines). Small rectangular boxes (E, and G) represent phase III core intervals within the lateral holes. The sample for this study was extracted SW (up-hole) of SDZ between 3192m MD (Measured Depth) and 3196.5m MD. The section diagram is modified after Bradbury et al. (2011). (b): Outer surface of the SAFOD core section splits marked with metric Measured Depths. The white box marks exact location of the sample extracted for use in this study. Light-color discontinuous streaks in the core are strings of siltstone clasts of different sizes. A prominent calcite vein, (arrow in both halves of the core) separates relatively undeformed massive shale on the right from the foliated gouge. Minor tick marks are 3cm apart; core section feet-to-meter conversion difference/small division = -0.96mm.

Figure 2

Reference optical micrographs. (a-b): Sample G24 whole-section cross-polarized light images of thin sections a and b cut parallel to each other ~ 10 mm apart at right angles to foliation (S). Inset boxes labeled on both sections indicate location of images used in other figures. Section G24b (second down) includes inset Fe-map (box with an arrow attached), which highlights location of pyrite elongate clusters streaked against a large siltstone clast. Sigmoid-block pyrite microstructure (labelled pyr) appears in both sections. Note foliation-parallel calcite veins indicated by a white arrow in top section. (c): A close-up plane-polarized light image of the area shown as box c in whole-section G24a image is typical microstructure of the clay-rich gouge. Note large compound clast covering the top portion of the image that consists of heavily veined reworked quartz-rich fragments. White arrows indicate abundance of foliation-parallel calcite veins.

Figure 3

Framboidal pyrite preserved in a large siltstone clast of siltstone-shale ultracataclasite. The main SEM image is the enlarged area shown within dashed line box (see Fig. 2a). The framboids appear to be associated with pores and cavities within the clast but unrelated to the marginal microstructures of an elongate pyrite cluster that borders the clast. Backscattered SEM images.

838 **Figure 4**

839 Cataclasis of polycrystalline sigmoidal-block pyrite (located in Fig 2a). (a): Optical reflected-light image
840 marked with reference boxes for other images. The deformed block pyrite appears with a tail streaking to
841 lower right of the image smeared parallel to foliation S against a large compound clast. (b): BS-SEM
842 close-up image showing intense fracturing of the pyrite (PY) in contact with gouge matrix (cg) as well as
843 calcite-sealed transgranular fractures (cal). Note the extremely fine pyrite particles (white arrow) swirling
844 in the gouge matrix. (c): BS-SEM close-up image from smeared tail of the block pyrite clearly showing
845 cataclastic nature of the pyrite deformation.

846 **Figure 5**

847 Microstructures of polycrystalline elongate-cluster pyrite. (a): Image (located in Fig. 2b) showing part of
848 elongate-cluster pyrite mass and its marginal microstructures grown along preexisting fracture network
849 and clast boundaries, crosscutting calcite veins. Location of Fig. 5b also shown. (b): Close-up of crosscut
850 calcite-veins (cal). (c): Optical reflected-light image showing another example of elongate-cluster pyrite
851 with marginal microstructures in relation to calcite veins. The pyrite growth is in the gouge matrix along
852 boundaries of a large siltstone clast (whole- section reference in Fig 2a). (d): Close-up image showing
853 Jigsaw-puzzle texture in local implosion microbreccia sealed with pyrite (whole-section reference in Fig.
854 2b).

855 **Figure 6**

856 Cathodoluminescence images of calcite veins in clasts and matrix of the gouge. (a): Whole- section
857 reference image (plane-polarized light) showing areas selected for CL imaging. (b): Collage of the CL
858 images (unmodified microscope output) from common vein-bearing microstructures in the gouge,
859 namely: Large siltstone clast veins SC1 and SC2 representing all SC areas shown in a; Smaller quartz-
860 rich siltstone clasts C1 through C5; Calcite veins in gouge matrix (M, highlighted by white arrows),
861 orientated parallel to foliation (S) shown within C2 and C4; Fracture-seal veins P1 and P2 respectively in
862 fringe-growth and sigmoid-block pyrite microstructures. Note similarity of CL emission color between P2
863 and M veins. Reference boxes are provided in P1 and P2 for other images.

864 **Figure 7**

865 Spectral data from calcite CL images plotted vs. four different types of vein-bearing microstructures in
866 the gouge (see Fig. 6 caption for descriptions). Number of measurements per image is shown on each box.
867 Boxes indicate the interquartile range of the CL wavelengths for each microstructure type. Line across
868 each box is the median data value, and vertical lines through boxes span the maximum and minimum
869 wavelength values in each type.

870

871 **Figure 8**
872 A ternary plot of atomic wt.% Mg-Mn-Fe trace concentrations (<1%) for a selection of calcite veins in the
873 sample. A trend exists for lower Fe-Mg content with decreasing brighter luminescence.

874 **Figure 9**
875 EBSD analysis of polycrystalline bulk in sigmoidal-block pyrite. (a): 1.0 μm step-size, Euler-angle map
876 of pyrite aggregate in polished section G24a (see Fig, 4a for location). Different color areas indicate
877 different crystallographic orientations. Different color lines represent variations in lattice misorientation
878 across boundaries identified in the map. The box locates area (c). (b): Misorientation-angle distribution
879 histogram for data in (a). (c): 0.2 micron step-size, Euler-angle map of pyrite aggregate in polished
880 section (upper box in (a)). The lines terminated in circles or triangles represent transects for
881 misorientation profiles in (d). (d): Misorientation-angle profiles for transects located on (c). (e): Three sets
882 of pole figures (i, ii & iii) for large grains located in (c). Circles in (ii) and (iii) denote a lattice
883 deformation rotation axis. (f): Misorientation-angle distribution histogram for data in (c).

884 **Figure 10**
885 EBSD analysis of areas in sigmoidal-block-pyrite with calcite-sealed brittle fractures. (a): Optical
886 reflected-light view of pyrite cataclasis at its border with gouge matrix and calcite-sealed through going
887 fractures (reference box in Fig 4a). Inset box locates area selected for EBSD analysis. (b): Euler angle
888 map revealing sub-grain structure in polycrystalline block pyrite. (c): Misorientation histogram for the
889 entire Euler angle map. A significant tail of correlated boundary pairs indicates strong crystal-plastic
890 deformation by dislocation creep and sub-grain rotation in the fractured block pyrite.

891 **Figure 11**
892 EBSD analysis for elongate-cluster pyrite microstructures crosscutting quartz clasts and calcite veins. (a):
893 EDS X-ray composition map (red=Ca, green=Si, blue=Fe) showing cluster-pyrite mass with respect to
894 calcite veins and quartz cataclasites in gouge (see location for this image in Fig 5c). Inset box shows area
895 selected for EBSD analysis. (b): Euler angle map revealing sub-grain structure in polycrystalline cluster
896 pyrite and in fringing pyrite grains that crosscut gouge microstructures at the cluster margins. Individual
897 pyrite and quartz grains selected for subgrain misorientation transects from typical areas of the map are
898 labeled as 1 to 6. Letter L on transects indicates the left side of misorientation profiles shown in 11e and f.
899 (c): CL image of calcite veins cut by fringing pyrite structures (white lines mark limit of pyrite in (a)). (d):
900 Optical reflected-light image highlighting crosscutting of calcite veins in (a) and (c) by fringing pyrite.
901 (e): Misorientation profiles for elongate-cluster-pyrite mass (transect 1) and pyrite that crosscuts calcite
902 veins and quartz cataclasite (transects 2-4) and. (f): Misorientation profiles for quartz cataclasite (transects
903 5-6). (g): Misorientation histogram for the entire Euler angle map. A significant tail of correlated

904 boundary pairs indicates strong crystal plastic deformation by dislocation creep and sub-grain rotation in
905 the main part of the elongate-cluster pyrite.

906 **Figure 12**

907 EBSD analysis for the pyrite-sealed implosion microbreccia. (a): Optical reflected-light image showing
908 area of the microbreccia selected for the analysis (reference box in Fig 5d). (b): EDS X-ray map
909 summarizing distribution of S (green), Si (yellow) and K (magenta). The yellow area is quartz, the
910 magenta area is alkali feldspar and the green is pyrite. (c): Euler angle map showing the pyrite seal
911 consists of a single crystal. White line marks location of misorientation profile. (d): Misorientation profile
912 showing weak cumulative lattice misorientation.

913 **Figure 13**

914 Descriptive depth-temperature diagram correlating CL and EBSD results for calcite veins and secondary
915 pyrite microstructures in the studied sample. Depth order of calcite veins is based on relative depth
916 established via CL. Temperature range estimates for calcite veins are constrained by foliation-parallel (M)
917 veins, calcite-sealed fractures in the pyrite (P2), and P1 veins that were crosscut by fringe growth pyrite
918 (around 200°C isotherm). For further information on pyrite deformation see pyrite deformation
919 mechanism map in Barrie et al. (2011). Thermal gradient of 41°C was adopted based on SAFOD
920 temperature at ~2.7km depth.

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Fig. 1a

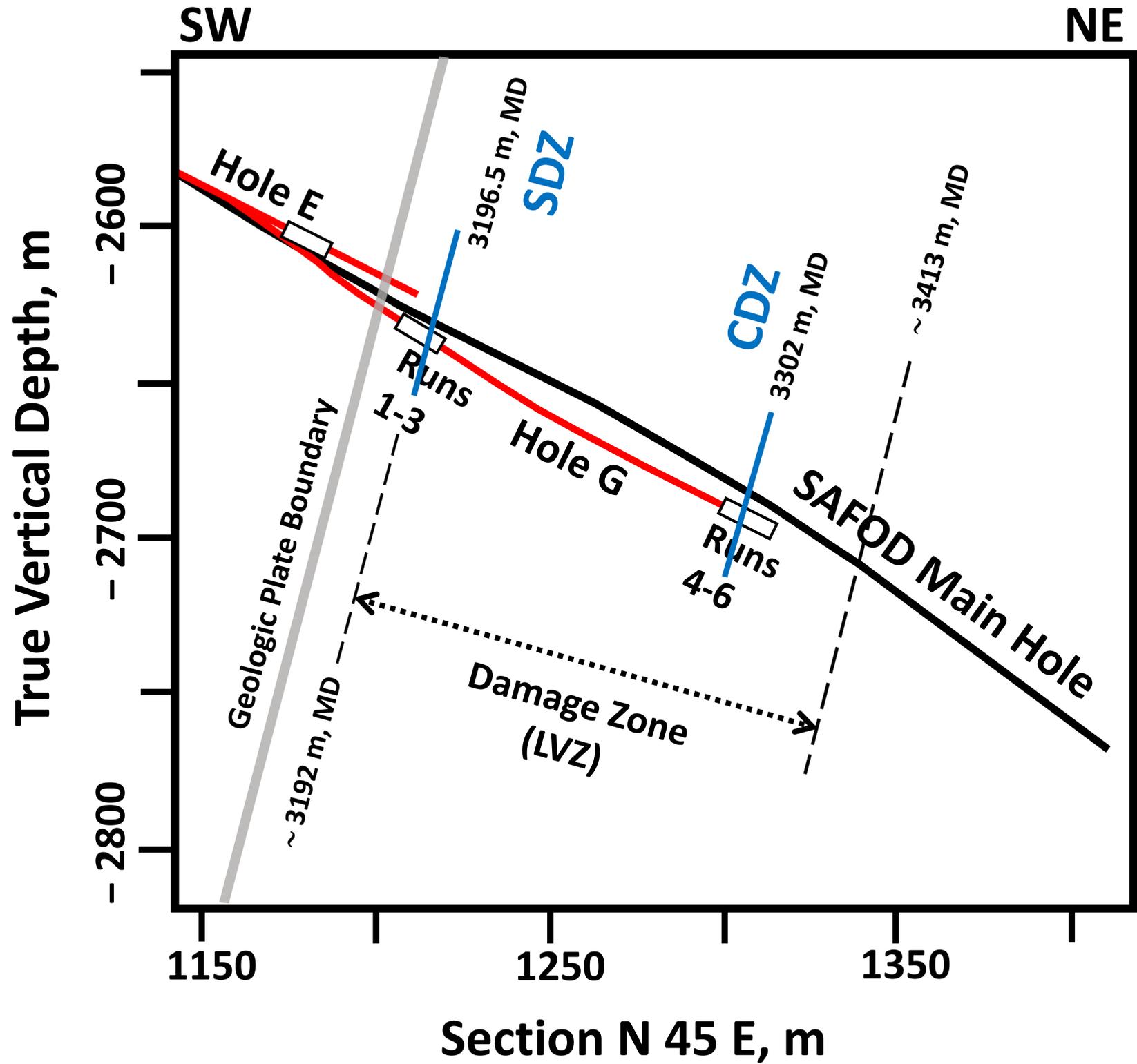


Fig. 1b

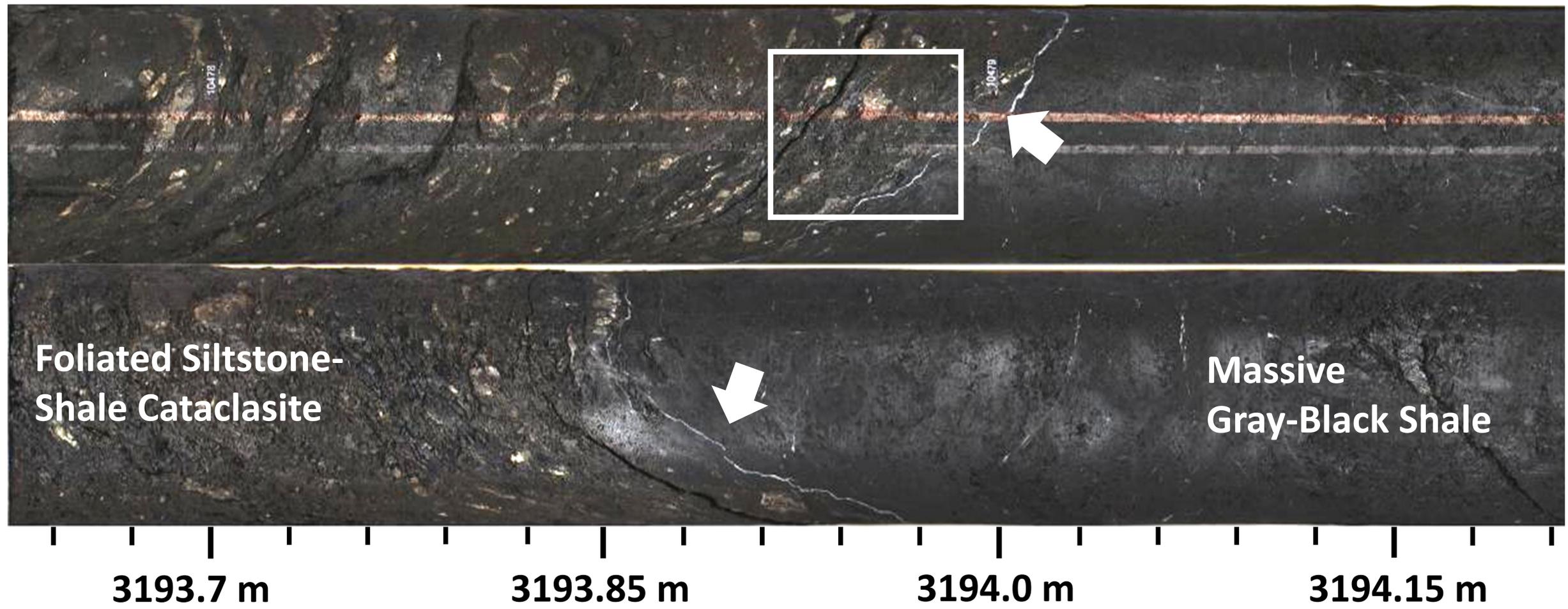


Fig. 2

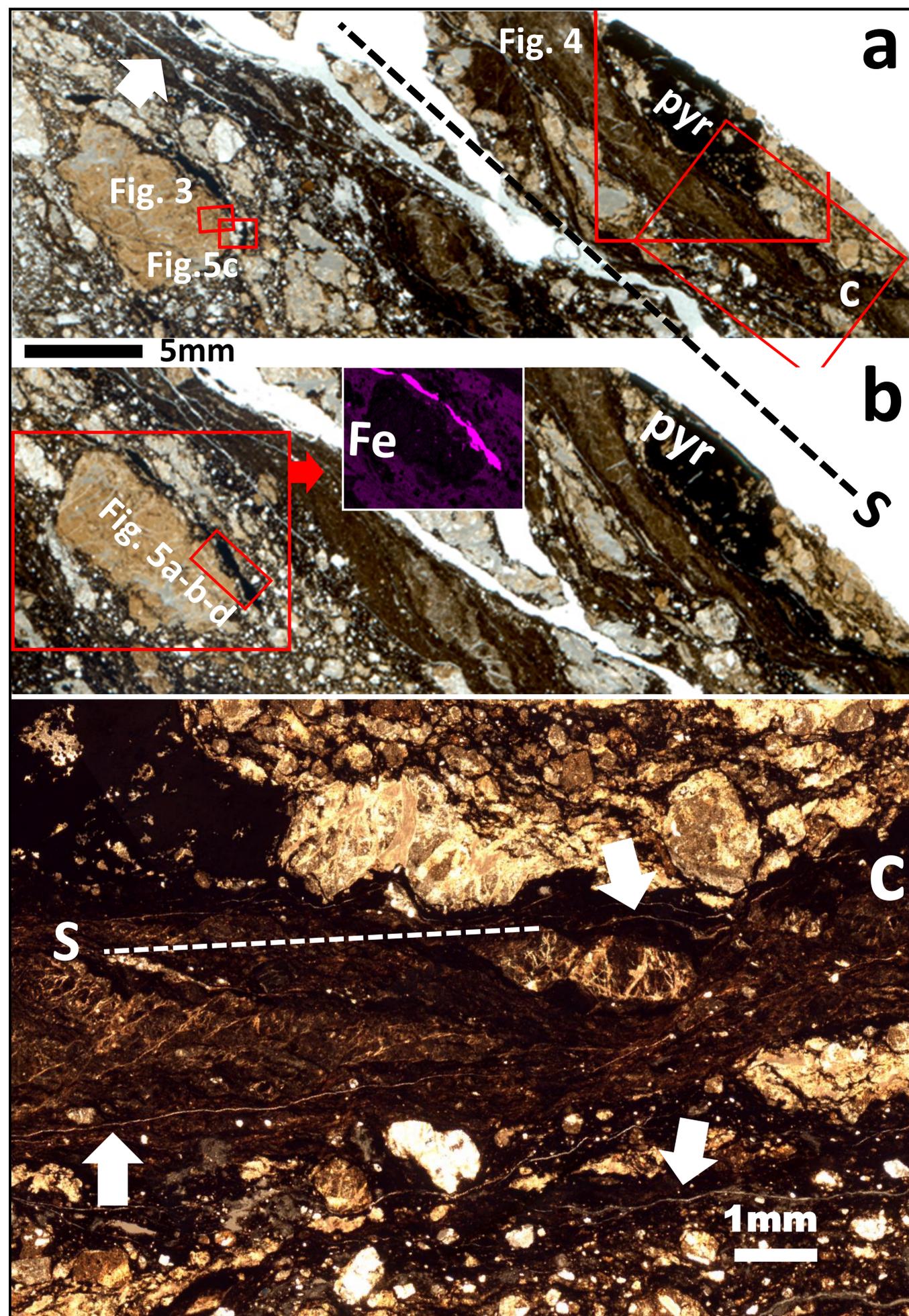


Fig. 3

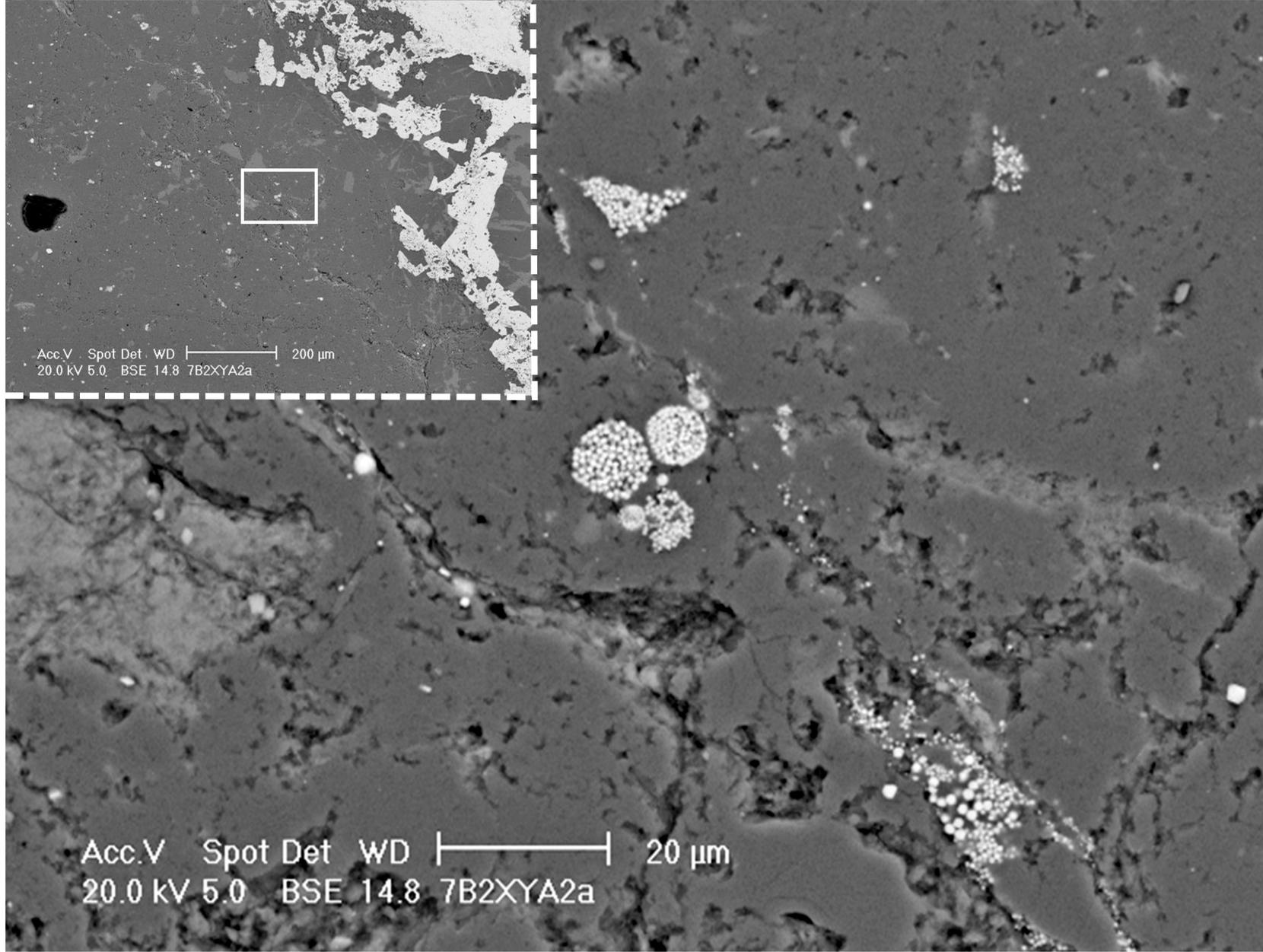


Fig. 4

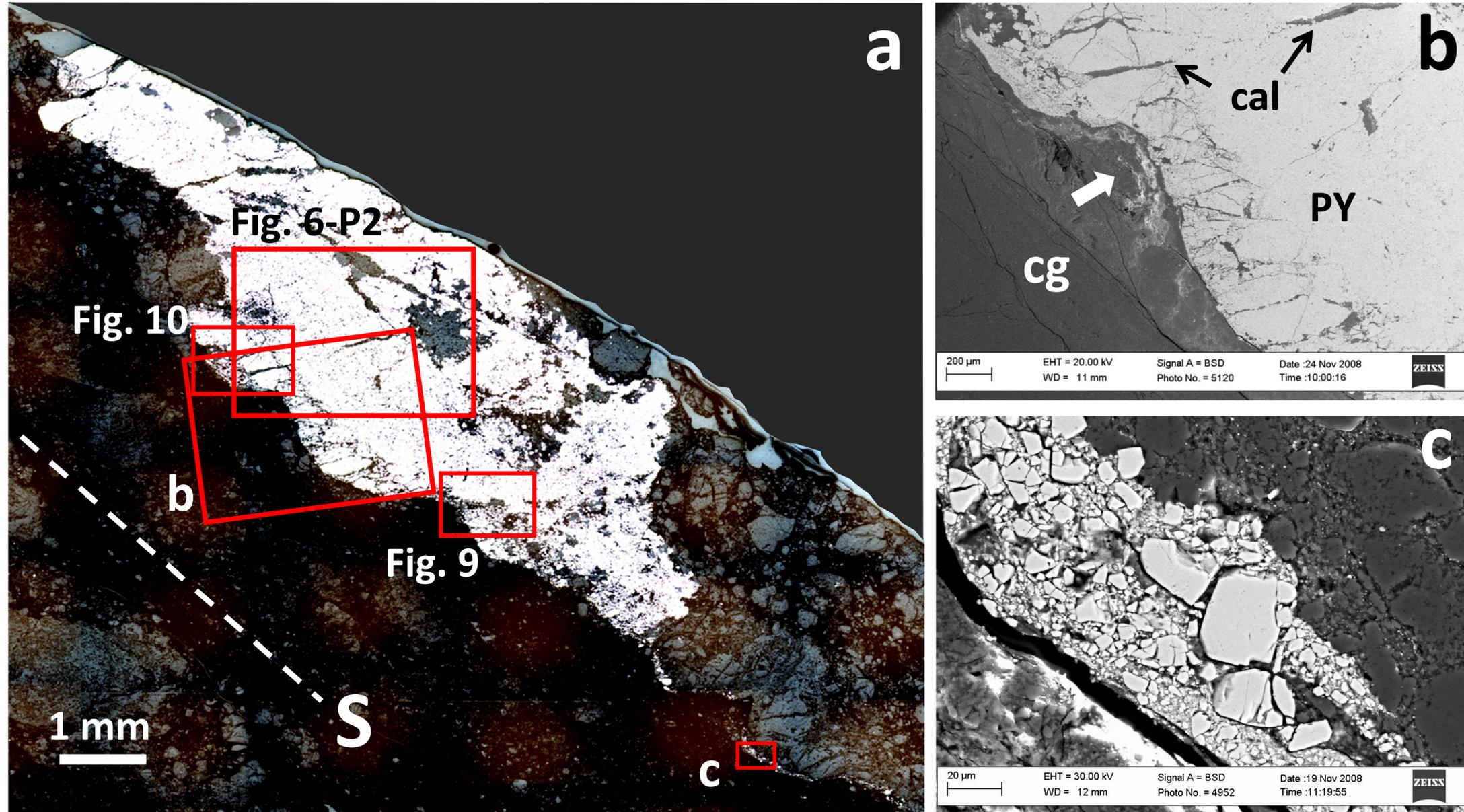


Fig. 5

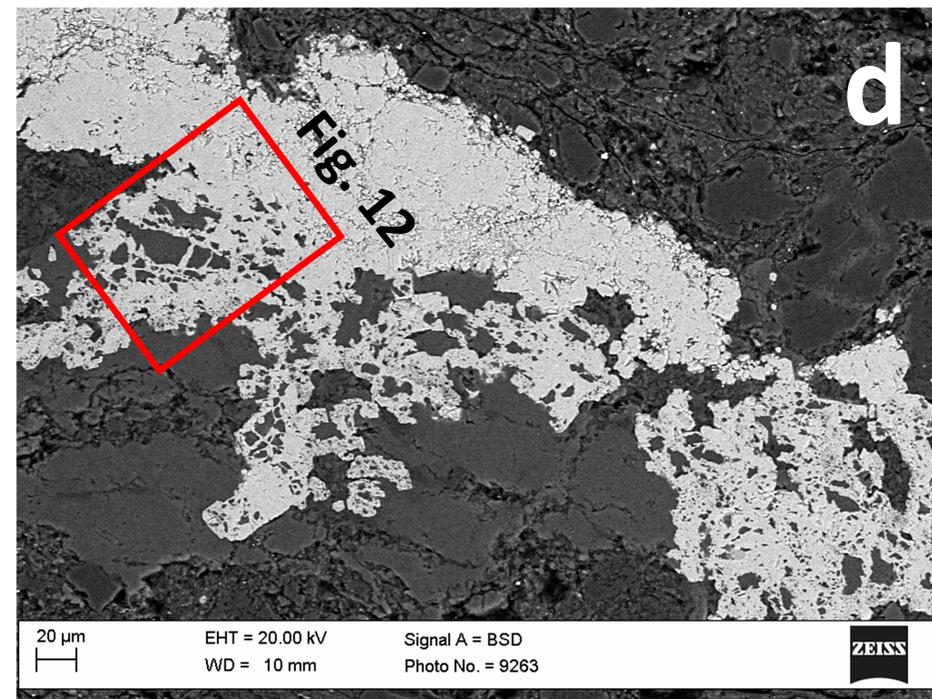
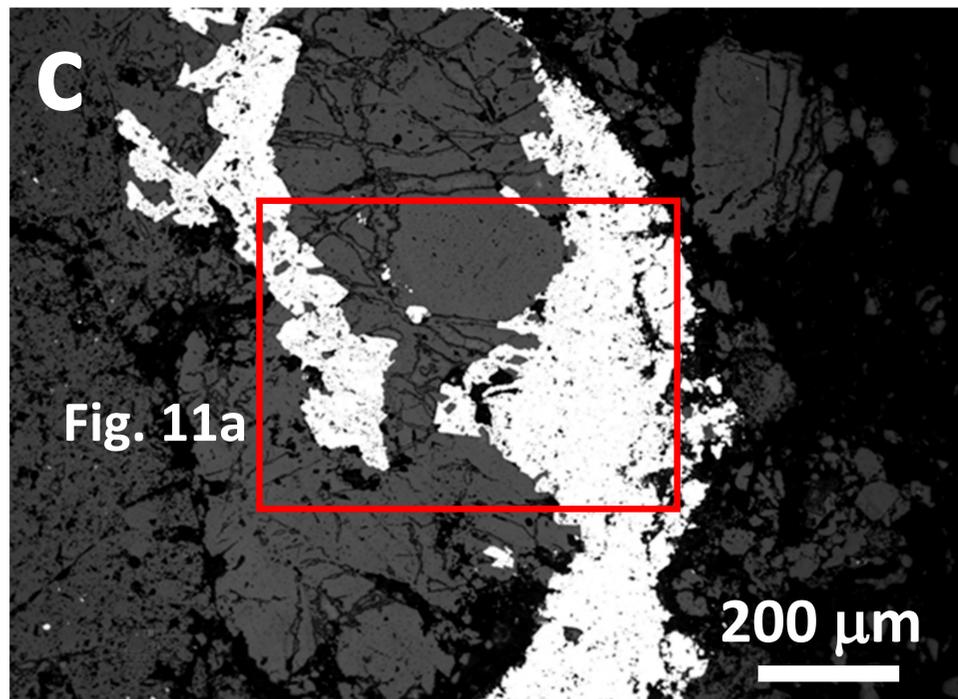
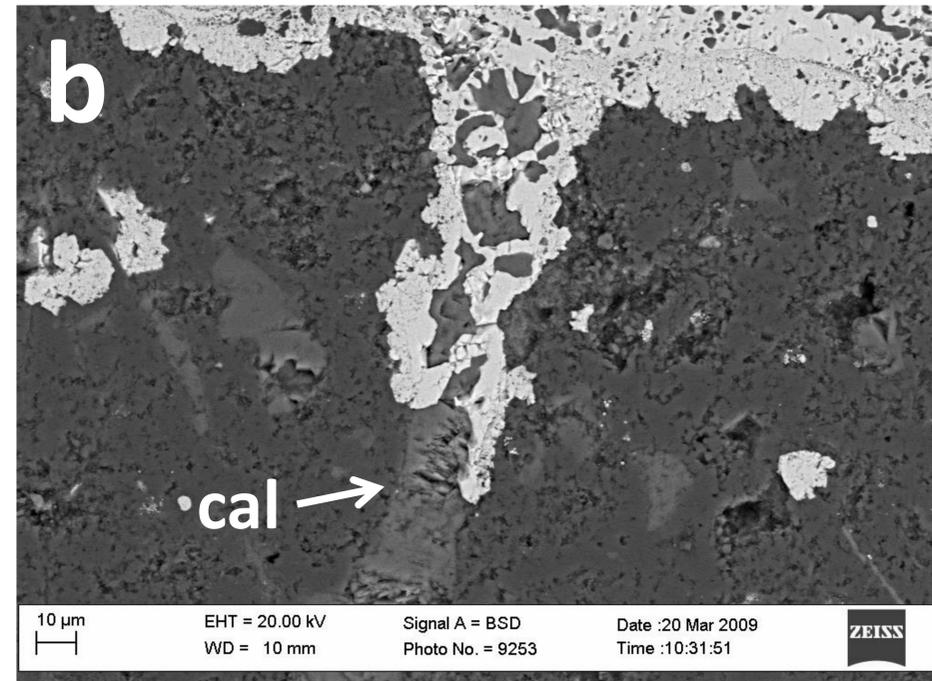
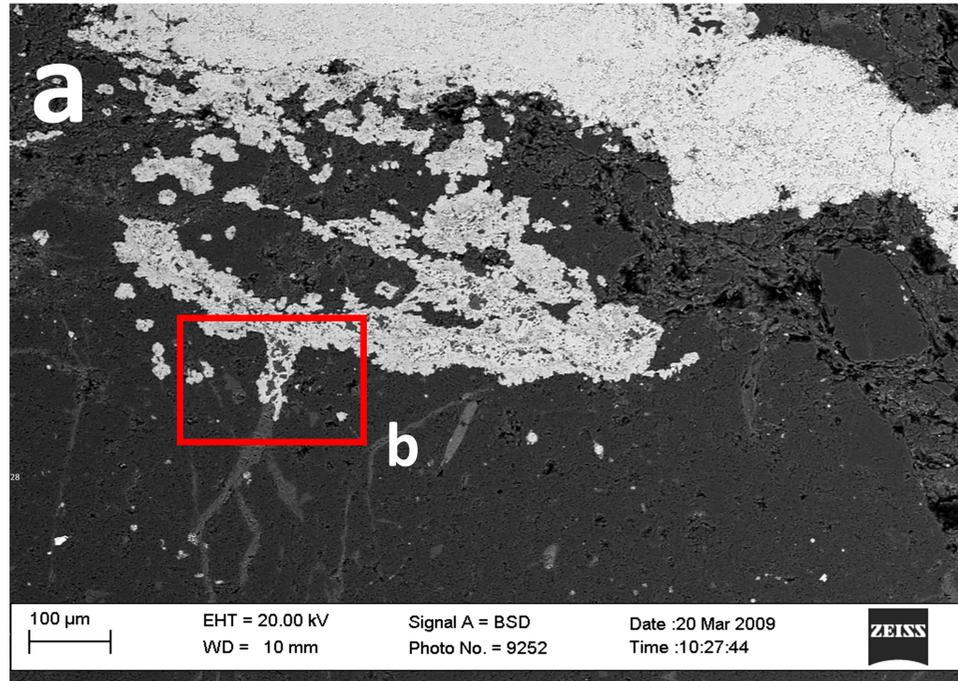


Fig. 6

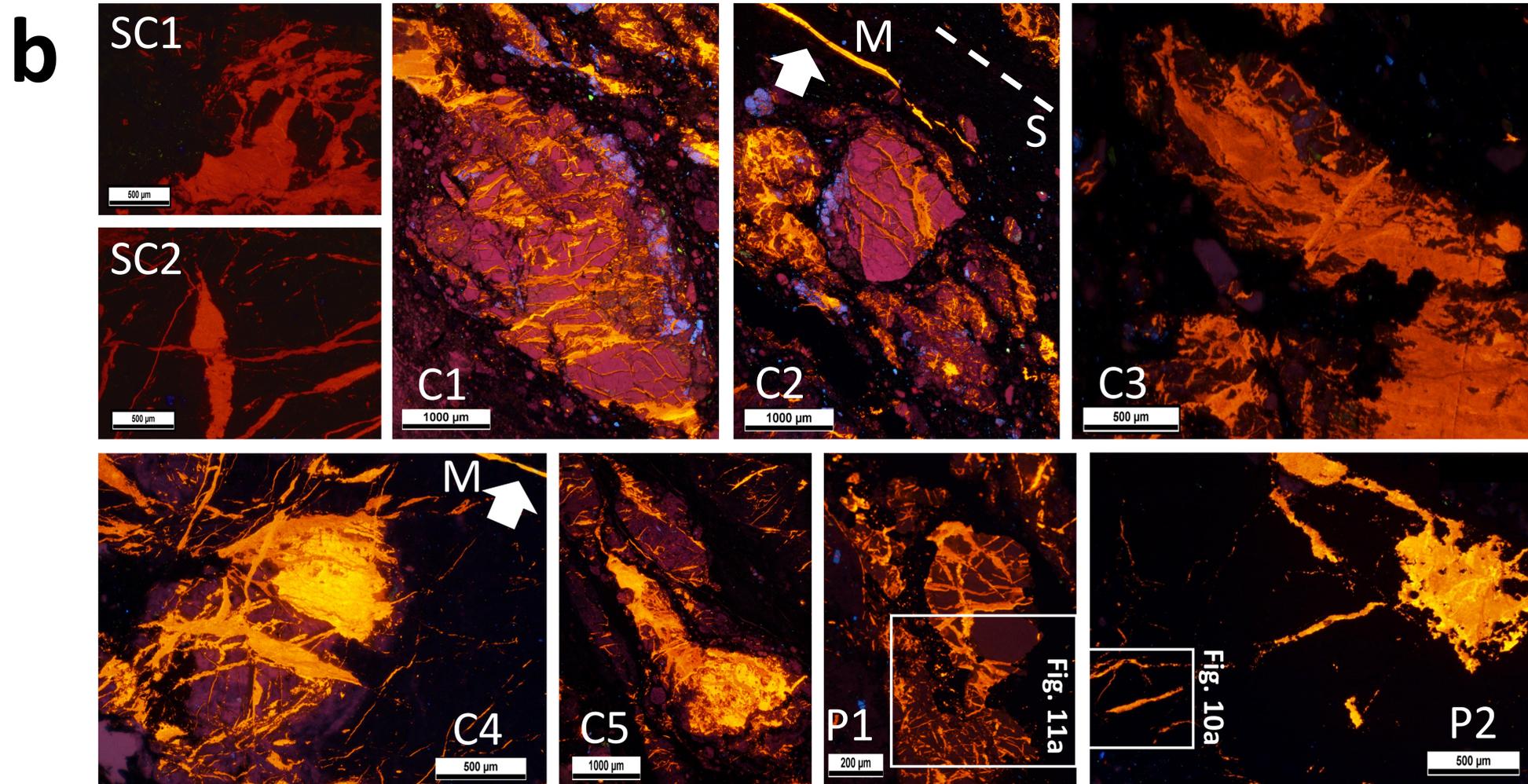
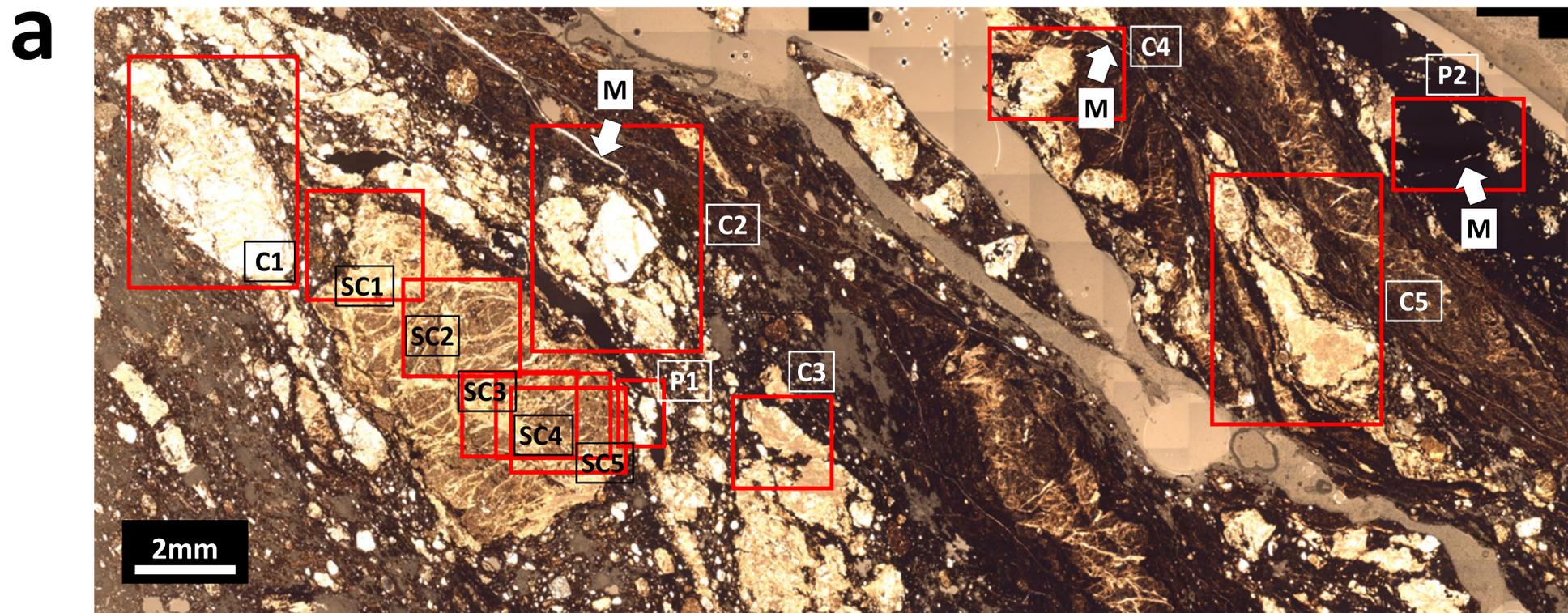


Fig. 7

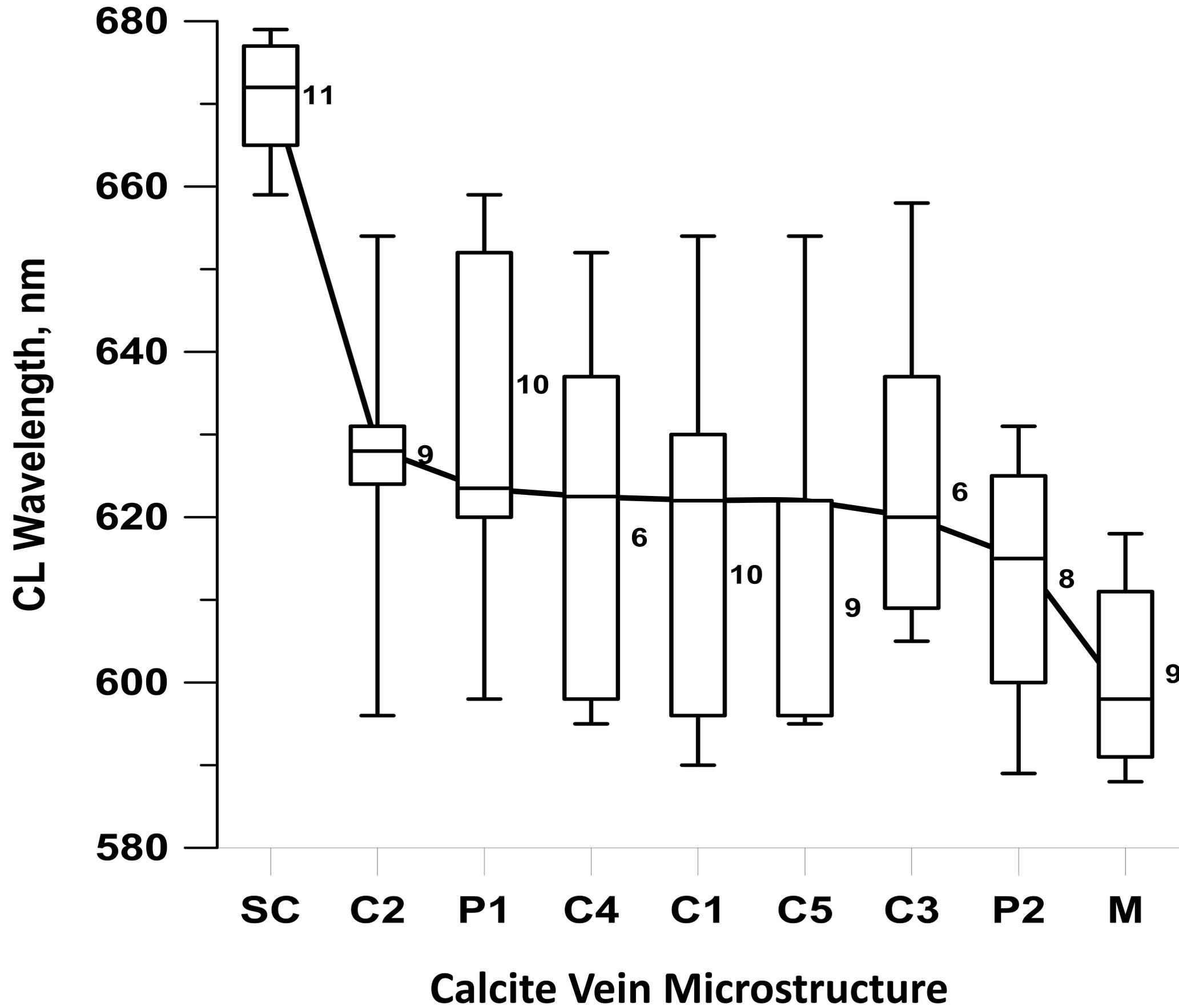


Fig. 8

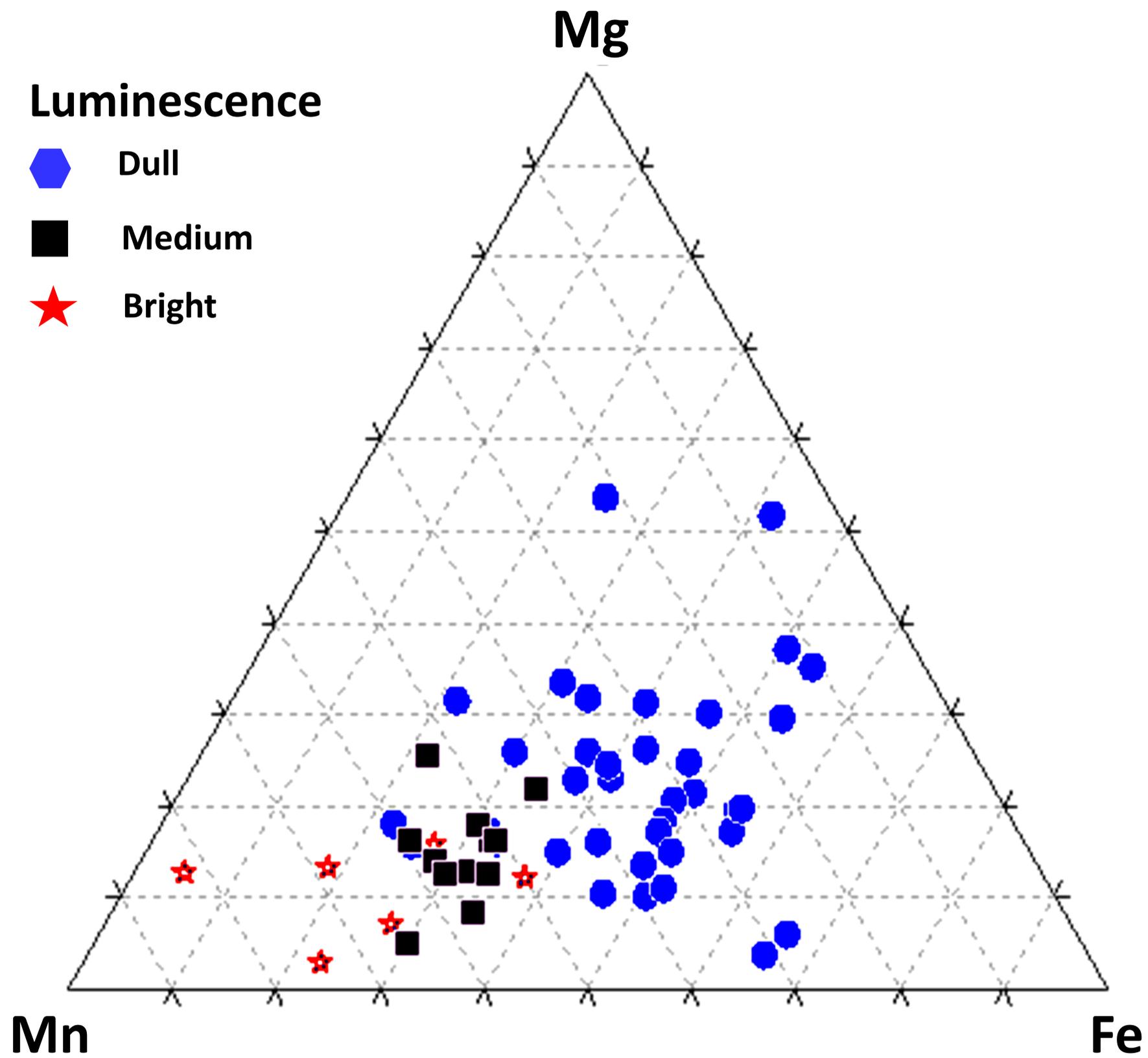
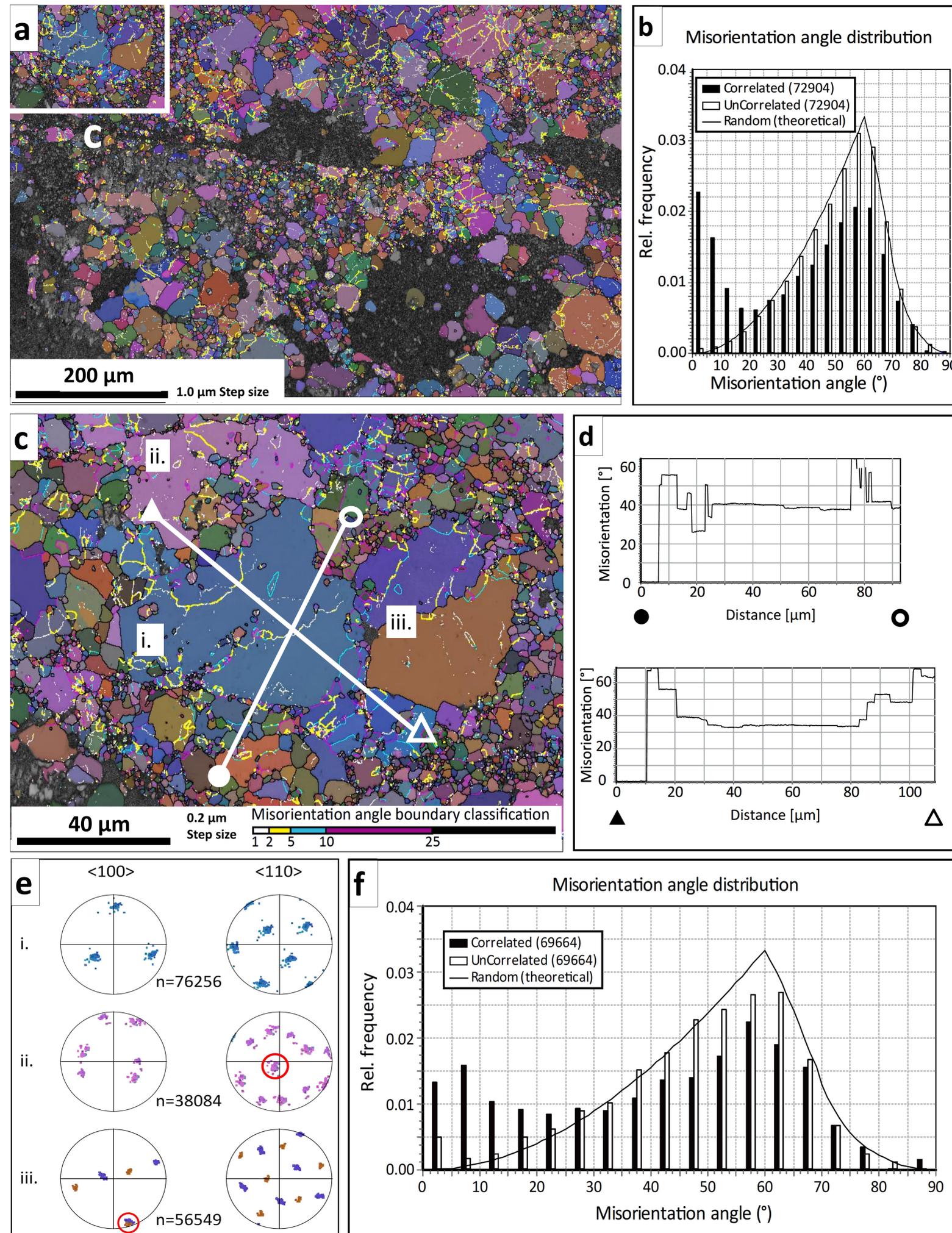
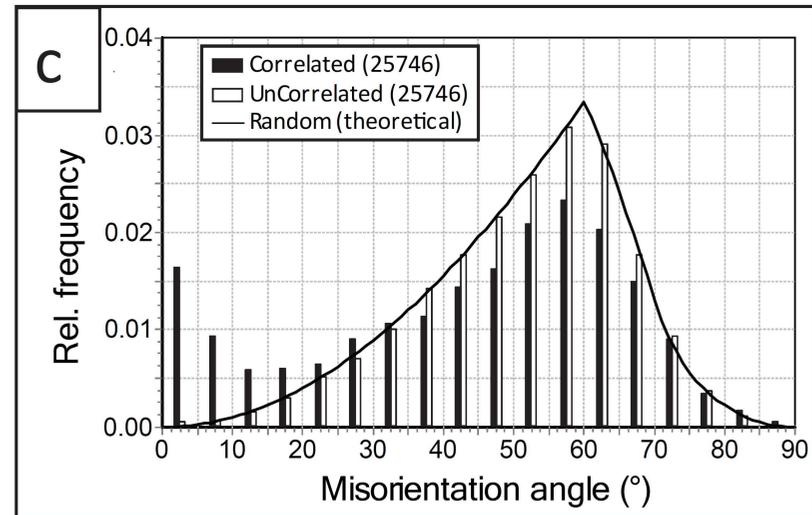
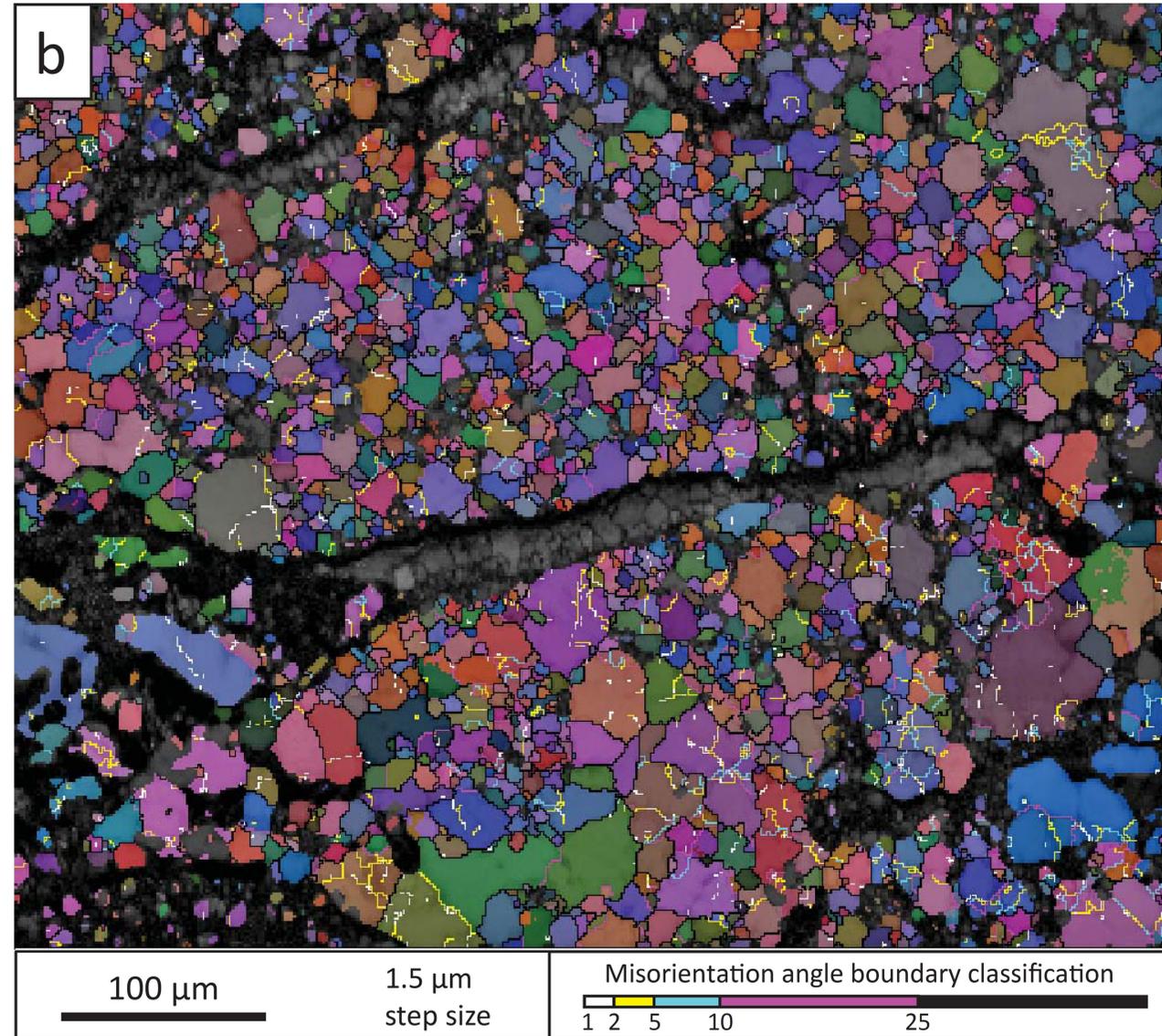
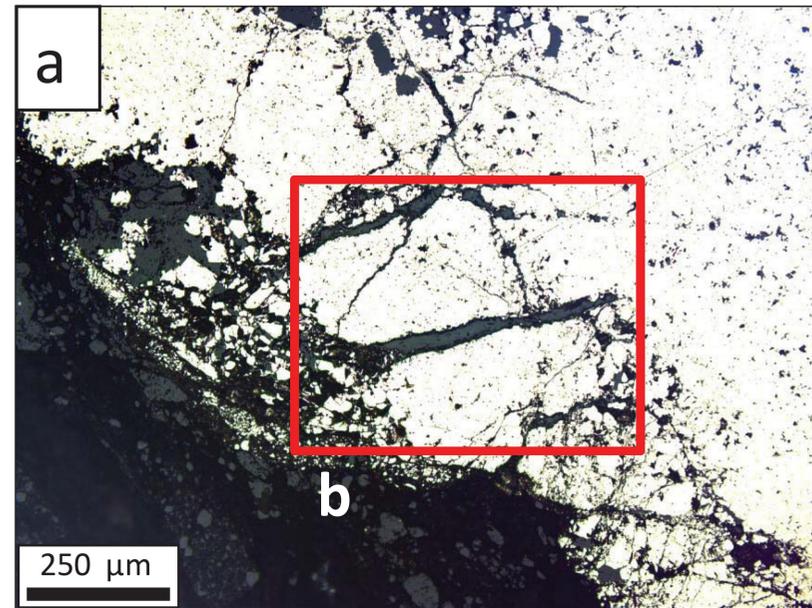
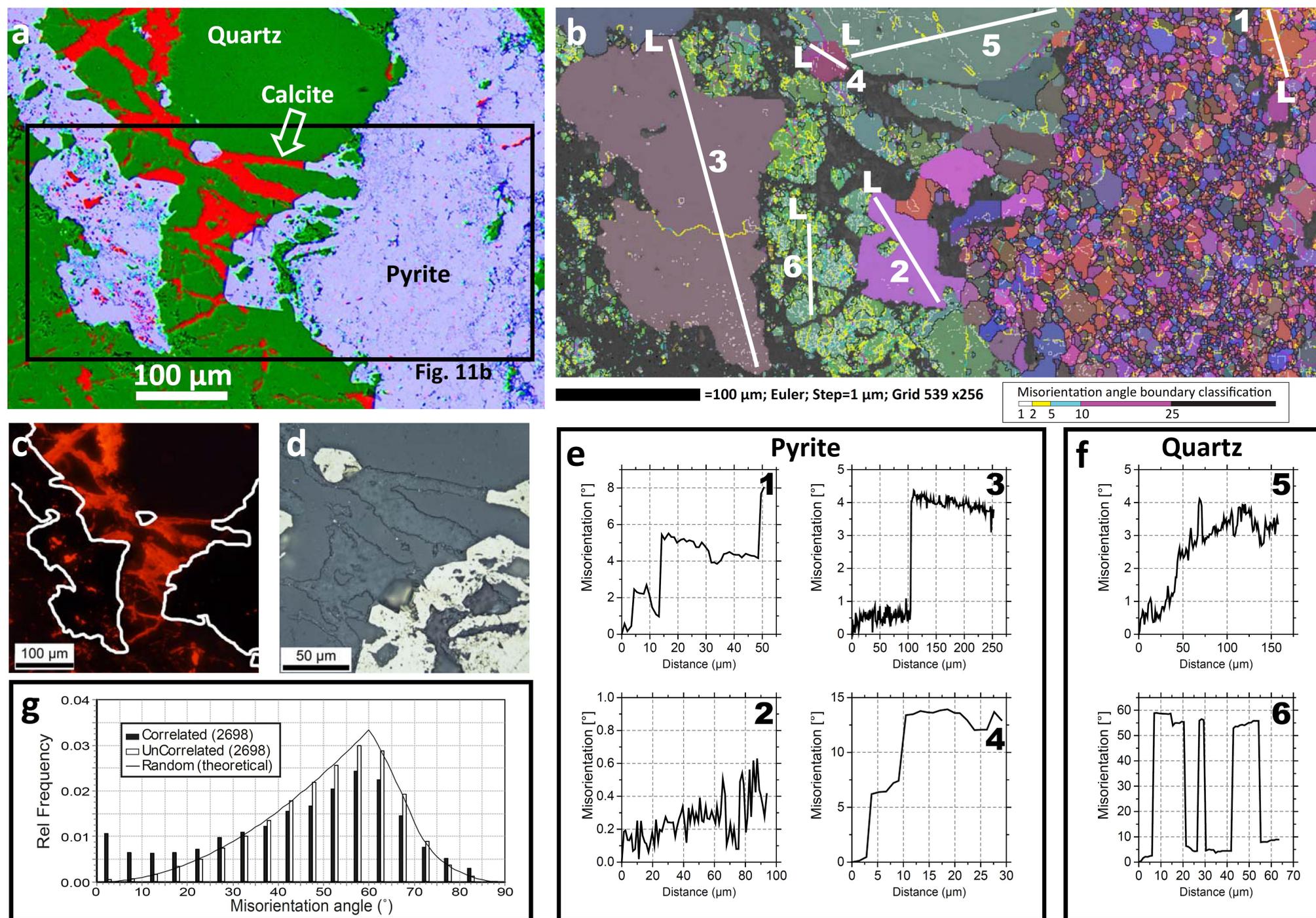


Fig. 9







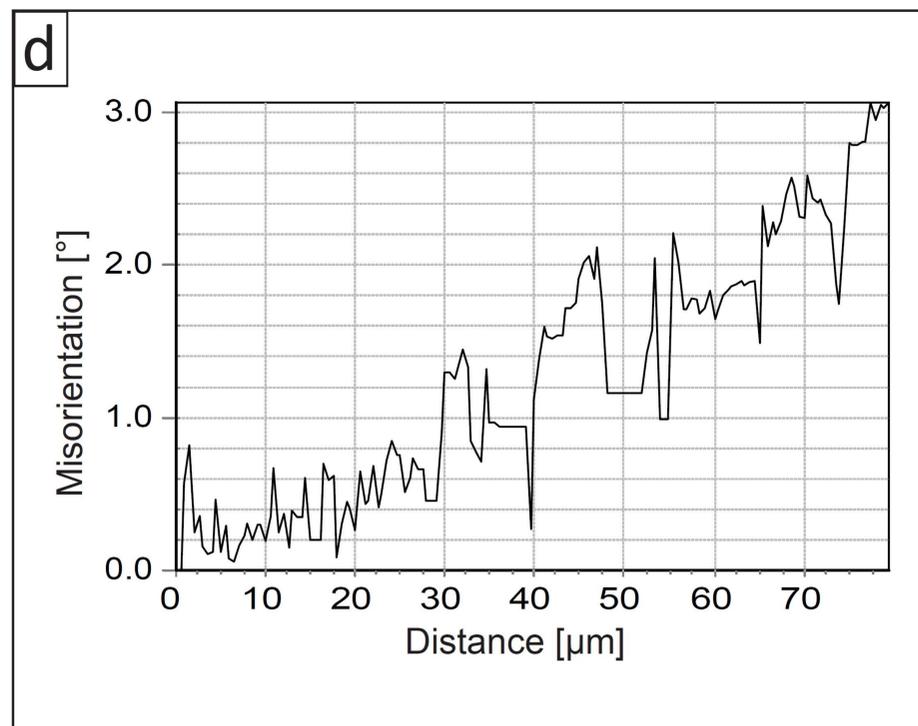
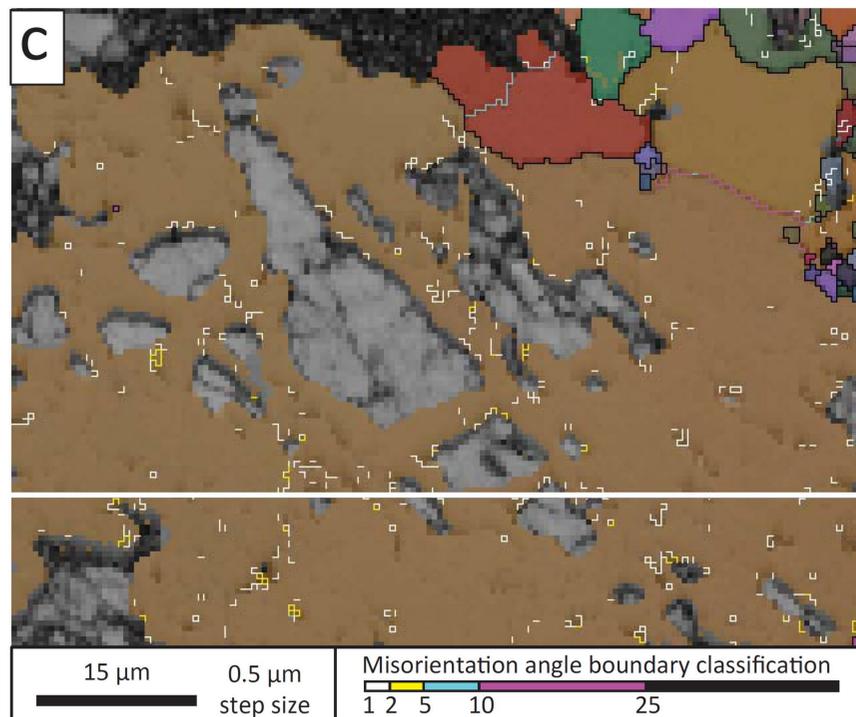
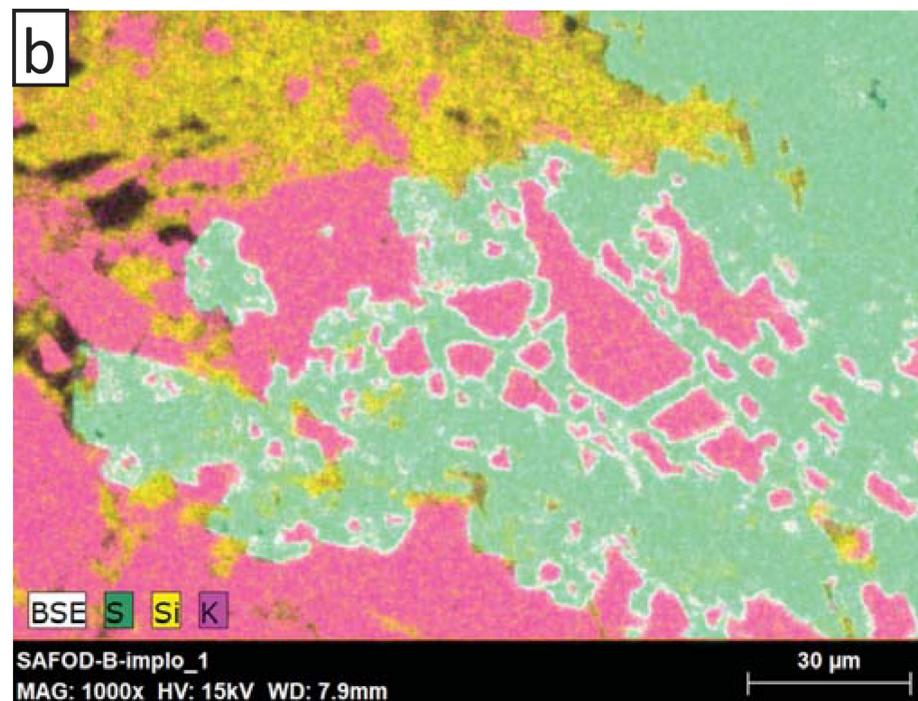
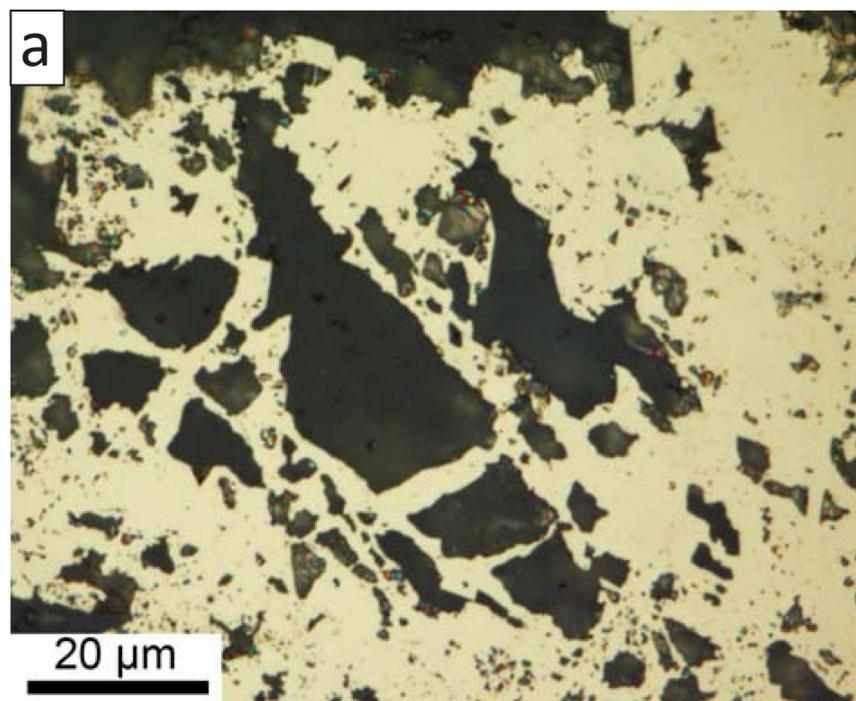


Fig. 13

