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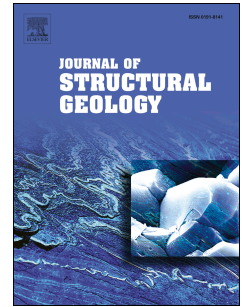
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**Strain in Sulphide Filled Foliation Boudinage Structures at the Mount Isa Cu Deposit,
Australia**

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Keywords:

**Strain, Shape preferred orientation, Crystallographic preferred orientation, Mount Isa,
Pyrrhotite, EBSD**

Highlights:

- Electron backscatter diffraction analysis of pyrrhotite filled foliation boudinage structures at Mount Isa
- The pyrrhotite has shape and crystallographic preferred orientations
- Dislocation creep and dynamic recrystallisation by subgrain rotation were facilitated by basal slip in the pyrrhotite
- Plane strain changed to flattening during deformation of the pyrrhotite infill as the foliation boudinage structures evolved

21 Abstract

22 Foliation boudinage structures are vein-like boudins forming in homogeneous, anisotropic
23 rocks that can be filled with significant volumes of mineral infilling. At Mount Isa, foliation
24 boudinage structures are associated with and filled by ore-related sulphides, predominantly
25 pyrrhotite. Deformation of syn-tectonic, ore-related sulphides at Mount Isa has generally
26 not been documented, fuelling controversy about ore timing. EBSD techniques, combined
27 with strain analyses show that the ore-related sulphide infill of foliation boudinage
28 structures underwent significant intracrystalline deformation. Pyrrhotite grains show well-
29 developed grain shape and crystallographic preferred orientations, indicating that the
30 foliation boudinage structures have undergone layer normal shortening at a relatively late
31 stage in the deformation history. Deformation of pyrrhotite grains by dislocation creep was
32 dominated by basal slip leading to dynamic recrystallisation of pyrrhotite by subgrain
33 rotation. A change from plane to flattening strain maintained the same shortening direction,
34 and may therefore have occurred continuously, rather than as two separate deformation
35 events. Results of this study are consistent with a current kinematic model for the Mount Isa
36 system and may have implications for future exploration in the area.

1. Introduction

Foliation boudinage structures are vein-like boudin structures that form by the layer normal shortening and layer parallel extension of fractures in homogeneous anisotropic rocks (Arslan et al., 2008; Platt & Vissers, 1980). Foliation boudinage structures are considered to maintain an open cavity throughout their evolution, over time periods long enough to produce fold-like structures in the adjacent wall rock, even though they have formed at greenschist facies or above (Arslan et al., 2008). Previous studies have detected little to no deformation within the mineral infill of foliation boudinage structures (Arslan et al., 2008; Hambrey & Milnes, 1975; Platt & Vissers, 1980).

Foliation boudinage structures have recently been identified at the Mount Isa copper deposit (Williams et al., 2023), where they are predominantly filled with quartz, dolomite and later ore-related sulphides (pyrrhotite $\pm$ chalcopyrite) that are associated with the Cu mineralisation event. Foliation boudinage structures are known to be important controls on mineralisation at other ore deposits, such as the Rosebery Pb-Zn deposit in Tasmania (Aerden, 1991).

Mount Isa mine is in the late Paleo- to early Meso-Proterozoic Mount Isa Inlier of northwest Queensland [Figure 1]. The mine is a world-class copper (Cu) and lead-zinc (Pb-Zn) deposit as well as being the second largest copper producer in Australia (Glencore, 2022). Recent studies (Cave et al., 2020; Davis, 2004; Miller, 2007; Williams et al., 2023 etc.) highlight the importance of geological structures in the localisation of the Cu deposit. However, the timing and the detailed controls on sulphide deposition are still controversial. A widely accepted view is that the Cu and possibly Pb-Zn orebodies were syn-tectonic and hydrothermally precipitated at a late stage during a protracted deformation history (Cave et

al., 2020; Perkins, 1984). However, deformation of ore-related sulphides, which might be expected in this scenario, has seldom been demonstrated.

Examination of the mineral infill of foliation boudinage structures using electron backscatter diffraction (EBSD) analysis therefore offers an opportunity to test both the mechanism of formation of foliation boudinage structures, and details of the ore-related sulphide history at Mount Isa. We used EBSD analysis to evaluate the relationship between the finite strain geometry, crystallographic orientations, and deformation mechanisms of pyrrhotite. If a relationship exists between the shape preferred orientation (SPO), crystallographic preferred orientation (CPO) and grain finite strain geometry, then it can be used to determine the relative timing of formation and deformation of the foliation boudinage structure infill.

2. Background

2.1 Regional Geology

Mount Isa is situated within the Proterozoic Mount Isa Inlier, a significant base metal province in northwest Queensland, Australia [Figure 1]. Structurally, the Mount Isa Inlier is subdivided into the Western, Eastern and Kalkadoon-Leichardt fold belts, with Mount Isa located in the Leichardt River Fault Trough of the Western Fold Belt (Blake, 1987; Day et al., 1983). The Kalkadoon-Leichardt Fold Belt separates the less deformed Western Fold Belt from the typically more deformed and metamorphosed Eastern Fold Belt (O'Dea, Lister, Betts, et al., 1997; O'Dea, Lister, MacCready, et al., 1997). The basement rocks of the Kalkadoon-Leichardt Fold Belt were deformed and metamorphosed during the 1900-1870 Ma Barramundi Orogeny (O'Dea, Lister, MacCready, et al., 1997; Page & Williams, 1988).

The post-basement stratigraphy is characterised by episodic intracontinental rifting and is commonly separated into the Eastern and Western Successions (Blake, 1987; Blake & Stewart, 1992). The Western Succession broadly overlies the Western and Kalkadoon-Leichhardt Fold Belts. Three stacked superbasins, the Leichhardt (1800-1750 Ma), Calvert (1735-1690 Ma) and Isa (1670-1575 Ma) superbasins, overlay the basement metamorphics in the Western Succession (Jackson et al., 2000; Page et al., 2000; Scott et al., 2000; Southgate et al., 2000). The younger Isa Superbasin is host to multiple mineral deposits in the region including at Mount Isa, Hilton and George Fisher (e.g. Cave et al., 2020). Emplacement of the 1670 ± 3 Ma Sybella Granite marks the separation of the Calvert from the Isa Superbasin (Neumann et al., 2006). The Isa Superbasin is divided into the Gun, Loretta, River, Term, Lawn, Wide, and Doom Supersequences (Domagala et al., 2000; Page et al., 2000). The Mount Isa deposit is in the Mount Isa Group which encompasses the basal Gun and Loretta Supersequences. Lithologies of the Mount Isa group are dominated by siltstone, turbiditic sandstone, carbonaceous shale and stromatolitic dolostone (Domagala et al., 2000; Page et al., 2000; Southgate et al., 2000). The age of the Mount Isa Group spans from ca. 1668 to 1648 Ma and includes the Moondarra Siltstone, Breakaway Shale, Native Bee Siltstone, Urquhart Shale, Spear Siltstone, Kennedy Siltstone and Magazine Shale (Page et al., 2000; Page & Sweet, 1998; Southgate et al., 2000).

2.2 Mount Isa Deformation

The host rocks at Mount Isa have undergone at least five deformation events [Figure 2], predominantly during the ~1600-1500 Ma Isan orogeny. The deformation events are outlined briefly below.

D1 – North-south shortening of previous syn-rift normal faults producing east-west oriented folds and reverse faults (O’Dea, Lister, Betts, et al., 1997; O’Dea, Lister, MacCready, et al., 1997; O’Dea & Lister, 1995). D1 is dated to 1610 ± 13 Ma (Page & Bell, 1986).

D2 – East-west shortening forming north-south striking upright folds and shallowly plunging regional anticlines and synclines (Bell, 1983; Bell et al., 1988; Bell & Hickey, 1998; Blake, 1987; Davis, 2004; Page & Bell, 1986; C. Perkins et al., 1999; W. G. Perkins, 1984; Swager, 1985; Wilde, 2011). A subvertical S2 axial planar fabric is generally bedding parallel at Mount Isa (Swager, 1985; Wilde, 2011). Dolomite filled extension veins at high angles to bedding are truncated against bedding planes (Swager, 1985; Wilde, 2011). D2 is dated to ca. 1575 Ma during peak metamorphism (Duncan et al., 2006).

D3 – Top to the east shear resulting in reactivation and intensification of S2, as well as rotation of S2 into shallower orientations (Bell & Hickey, 1998). This deformation is localised, being poorly developed at Mount Isa Mine (Bell & Hickey, 1998; Perkins, 1997).

D4a – ENE-WSW shortening forming localised NNW-SSE striking fold zones that are typically 10-20 m wide in the Urquhart Shale, but locally up to 100 m (Davis, 2004). This deformation event was termed D4a by Williams et al. (2023) and is the D4 event of Davis (2004) (and previously D3). Most folds in the mine belong to this generation, the largest being the Mount Isa Fold that runs through the north of the mine (Davis, 2004). An S4 fabric that is sub-parallel to S2 is defined by a slaty cleavage in dolomitic shale and crenulation cleavage in black shale lithologies. D4a is dated at 1510 ± 13 Ma using whole-rock Rb-Sr isotopes (Page & Bell, 1986).

D4b – NW-SE shortening of the approximately north-south striking Urquhart Shale resulting in sinistral-reverse shear along bedding (Miller, 2007). Miller (2007) identified this

deformation stage as the main Cu mineralisation event with a post-D4a and pre-D5a age and a stress field distinct from D4a. This event was termed D4b by Williams et al. (2023).

D5a – East-west shortening resulting in steeply west-dipping reverse faults that are parallel to the Mount Isa Fault Zone and overprint the Cu orebodies (Miller, 2007). Most D5a faults show slip along bedding with a top to the east transport direction (Miller, 2007).

D5b – East-west shortening resulting in conjugate NW trending sinistral-reverse faults that have top to the west transport directions and NE trending dextral-reverse faults with top to the east transport directions (Miller, 2007).

2.3 Deposit Geology

The Cu orebodies at Mount Isa are located on the western limb of a regional D2 anticline within the Urquhart Shale unit of the Mount Isa Group [Figure 3] (Bell et al., 1988). The Urquhart Shale is composed of finely laminated interbedded siltstones, mudstones and dolomitic shales that have a strong anisotropy (Davis, 2004; Neudert, 1983). The orebodies are located proximal to the sigmoidal-shaped Paroo Fault, which is locally termed the basement contact (Bell et al., 1988; Davis, 2004; Long, 2010). The largest Cu orebodies (known in the mine as the 1100, 1900, 3000, 3500 orebodies) are typically located adjacent to the Paroo Fault and the smaller orebodies (200, 500, 650) are at greater distances (Davis, 2004). Previous studies have associated the Cu orebodies with NNW plunging F4 fold hinges (Bell et al., 1988; Davis, 2004; Perkins, 1984). However, new evidence by Miller (2007) identified the sinistral-reverse structures of the D4b event to be the controlling feature based on orebody geometries and kinematics of Cu extension veins.

The Cu orebodies occur as chalcopyrite replacement or infill in breccias that are hosted within silica-dolomite alteration halos (Perkins, 1984). Silica-dolomite produces four main rock types depending on variability of alteration and brecciation that reflect silicification ('siliceous shale' and 'brecciated and fractured siliceous shale') and dolomitisation ('recrystallised shale' and 'irregularly brecciated and recrystallised shale') (Clark, 1968; Knights, 1976; Mathias & Clark, 1975; Miller, 2007; Perkins, 1984).

Quartz is overprinted by dolomite, which both undergo dissolution during chalcopyrite mineralisation (Perkins, 1984; Wilde, 2011; Wilde et al., 2006; Williams et al., 2023). Fine-grained pyrite is considered to be diagenetic or early in the alteration sequence, and is followed by coarse-grained pyrite, calcite, quartz and silica-dolomite alteration, all pre-dating pyrrhotite and chalcopyrite (Cave et al., 2020; Gulson et al., 1983; Perkins, 1984; Williams et al., 2023). Pyrrhotite is associated with chalcopyrite and their timing is often coeval (Cave et al., 2020; Williams et al., 2023). Chalcopyrite forms replacive growths across all generations of veins and microstructures (Miller, 2007; Perkins, 1984) and the timing of Cu mineralisation is post-peak metamorphism during D4a to D4b (Miller, 2007; Perkins, 1984; Smith et al., 1978; Swager, 1985).

McClay and Ellis (1983) documented evidence of deformation within the early pyrite at Mount Isa, whilst McClay (1978) also described deformation of sulphides in the ore assemblage, attributed to deformation during remobilisation and reprecipitation of syngenetic sulphides.

2.4 Foliation Boudinage Structures

Modelling by Arslan et al. (2008), Arslan et al. (2012) and Mandal and Karmakar (1989) showed that foliation boudinage structures form by the ductile (low strain rate)

deformation of foliation or layering adjacent to a brittle (high strain rate) fracture, where they can form in both rocks (e.g. Platt & Vissers, 1980) and glacier ice (Hambrey & Milnes, 1975). Foliation boudinage structures can be either symmetrical or asymmetrical in geometry depending on initial fracture orientation and whether the deformation is coaxial or non-coaxial [Figure 4] (Arslan et al., 2008; Hambrey & Milnes, 1975; Mandal & Karmakar, 1989). Arslan et al. (2008) showed that coaxial deformation of an extensional fracture results in the symmetrical opening of a central void that is subsequently filled to form a lozenge-shaped foliation boudinage structure. Coaxial deformation of a fracture with an initially curved geometry may result in asymmetrical x-type, crescent, or double-crescent foliation boudinage structures (Arslan et al., 2008). Alternatively, non-coaxial deformation of a shear fracture or fracture that is oblique to the layering results in asymmetrical opening of the fracture and produces x-type foliation boudinage structures (Arslan et al., 2008; Mandal & Karmakar, 1989; Platt & Vissers, 1980). Asymmetrical foliation boudinage structures can form as conjugate sets as well as single structures (Arslan et al., 2012; Sawyer, 1983; Williams et al., 2023).

Bending of the layering or foliation into the fracture defines the neck of the foliation boudinage structure, which can be easily identified in cross-section normal to the long axis. As with boudins, the long axes necks of a foliation boudinage structures are sub-parallel to the intermediate principal axes of the finite strain ellipsoids (Arslan et al., 2008; Goscombe et al., 2004). The direction of extension along layering is the maximum principal strain axis and the normal to the layering is the minimum principal strain axis.

The interior of foliation boudinage structures can contain millimetres to 100s of cubic metres of mineral infill in the largest structures (Aerden, 1991; Arslan et al., 2008; Williams

et al., 2023). The most commonly observed mineralisation is quartz but also biotite, feldspar, chlorite, tourmaline, dolomite, pyrrhotite and chalcopyrite (Arslan et al., 2008; Platt & Vissers, 1980; Swanson, 1992; Williams et al., 2023). The mineral fills are often blocky or massive quartz indicative of growth into open fluid filled space (Arslan et al., 2008). Arslan et al. (2008) infer that fluid-filled foliation boudinage structures could remain open for a significant period before mineral precipitation, providing the fluid pressure is above lithostatic to keep the fractures open. The absence of deformation within the mineral infill has been used as evidence for a protracted period of time between the initial formation of the structure and the final infill stage (Arslan et al., 2008; Hambrey & Milnes, 1975; Platt & Vissers, 1980).

Foliation boudinage structures have been described at Mount Isa occurring adjacent to the Cu breccia-style mineralisation (Williams et al., 2023). All of the structures have an initial rim of quartz and dolomite, followed by later sulphides both replacing and infilling around earlier grains (Williams et al., 2023). The predominant sulphide infill is pyrrhotite with minor chalcopyrite in some samples (Williams et al., 2023). The foliation boudinage structures at Mount Isa are mainly located in the less deformed, brecciated and altered lithologies where the anisotropy provided by the finely laminated Urquhart Shale or layer parallel foliations is still intact (Williams et al., 2023). More altered and brecciated lithologies are associated with greater proportions of high grade Cu (>3%), but few small-scale foliation boudinage structures are found in these lithologies (Williams et al., 2023). However, Williams et al. (2023) suggest that this does not exclude a larger-scale role of foliation boudinage structures in controlling Cu mineralisation at Mount Isa. The dominant planar anisotropy at Mount Isa dips steeply (~65°) to the west with the foliation boudinage structures lying in this plane. Williams et al. (2023) show that the necks of foliation boudinage structures at

Mount Isa plunge gently to both the north and south because of progressive development during dextral-reverse shortening (D4a) through to sinistral-reverse shortening (D4b).

2.5 Deformation of Pyrrhotite

Under most conditions of mechanical deformation in the crust, pyrrhotite behaves as a ductile material (Graf & Skinner, 1970; Handin, 1966). Brittle failure of pyrrhotite predominates at room temperature (Graf & Skinner, 1970). An increase in temperature reduces the strength of pyrrhotite and increases the intracrystalline deformation (Clark & Kelly, 1973; Graf & Skinner, 1970; Kelly & Clark, 1975). Pyrrhotite is one of the strongest sulphides at room temperature but becomes significantly weaker at higher temperatures [Figure 5] (Clark & Kelly, 1973; Kelly & Clark, 1975). The same workers showed that pyrrhotite aggregates are weaker than dolomite rock at temperatures greater than 125 °C.

The pyrrhotite basal plane serves as the dominant shear plane during plastic deformation (Kübler, 1985). Pyrrhotite is strongest perpendicular to the (0001) plane and weakest at a 55° angle (Graf & Skinner, 1970). The pyrrhotite critical resolved shear stress (CRSS) for basal slip, at a strain rate of 10^{-5} (s⁻¹) and confining pressure of 300 MPa, changes significantly with temperature (Kübler, 1985; Niederschlag & Siemes, 1996). At temperatures of 200 °C, 300 °C and 400 °C, the CRSS for basal slip is 60 MPa, 8 MPa and 4.7 MPa, respectively.

3. Samples and Methods

3.1 Samples

Unoriented Mount Isa Copper Operations (MICO) drillholes that are proximal to the Mount Isa Cu orebodies and pyrrhotite mineralisation (Williams et al., 2023) were logged for

structures. Foliation boudinage structure samples were collected from several drill cores ('201903052', '201903092' and '201904012') that represented various locations in the deposit [Figure 3]. Representative samples were cut, and polished thin sections were made in the XZ-, YZ- and XY-planes in Figure 6. FBS3 includes two adjacent foliation boudinage structures in the same polished section and is further divided into FBS3 (left) and FBS4 (right) in Figure 7g. The sample containing FBS3 and FBS4 was cut in two orthogonal directions (XZ and YZ; Figure 7) and two samples (FBS1 and FBS2) were cut in three orthogonal directions (XZ, YZ, XY; Figure 7). Results for the YZ-plane shown in Figure 7h are used for both FBS3 and FBS4 throughout this study.

3.2 Electron Backscatter Diffraction (EBSD) Preparation

EBSD analysis was performed using the Zeiss Sigma HD scanning electron microscope in the School of Earth and Environmental Sciences, Cardiff University. The eight representative polished thin sections were initially polished with colloidal silica for 10 minutes and then washed with de-ionised water before being checked under the microscope. A second 10 minute polish was performed on the samples, which were then placed in an ultrasonic bath for 5 minutes in de-ionised water. A thorough clean with de-ionised water is essential as colloidal silica can crystallise onto the slide if left uncleaned. A tissue was used to dry all the samples prior to them being given a thin carbon coat of 4-8 nm using an Agar Turbo carbon coating vacuum chamber. The carbon coat minimises electron charge across the sample yet maintains a strong crystallographic signal. All samples were analysed within 48 hours of polish to minimise tarnishing (oxidation) of exposed sulphide minerals.

3.3 Electron Backscatter Diffraction (EBSD) Analysis Set-up

Phases were indexed as consistently as possible between samples [Supplementary 1]. X-ray diffraction (XRD) analysis was used to determine that the pyrrhotite at Mount Isa is monoclinic (Fe_{1-x}S , where $x=0$ to 0.125) [Supplementary 2]. Monoclinic pyrrhotite is absent from the EBSD phase databases. However, the properties of monoclinic pyrrhotite (Fe_7S_8) relevant for EBSD analysis are almost identical to the hexagonal FeS phase, so the hexagonal phase Troilite (FeS) was used to index all pyrrhotite in samples in this study.

EBSD data were collected using a 20 keV beam with high-current turned on. Raw data were collected at step-sizes between 6 and $14.1\text{ }\mu\text{m}$, with an aperture size of $120\text{ }\mu\text{m}$ and EBSD detector insertion distance of $\sim 190\text{ mm}$ for all samples [Supplementary 3]. Mean angular deviation (MAD) was less than 1° for all samples.

3.4 Data Processing

The EBSD data were processed using the open-source MTEX toolbox for MATLAB (Bachmann et al., 2010, 2011; Mainprice et al., 2015). The EBSD data clean-up procedure is described below:

- Grains were determined using 10° bins
- Grains ≤ 5 pixels were removed
- Grains were redetermined using 10° bins
- Grains that had an area of non-indexed pixels $>50\%$ of the total grain area were removed, leaving only the well-constrained grains
- The remaining grains were extrapolated to fill the missing pixels and smoothed
- If required, the EBSD maps were rotated so the shale layering was horizontal in all maps

- Boundary grains that intersected the EBSD map edge were removed, leaving only those grains whose entire area was within the map

3.5 Relict and Recrystallised Grain Identification - Grain Orientation Spread

The grain orientation spread (GOS) can be used to define the threshold between relict (high strain) and recrystallised (low strain) grains **[Figure 8]** (Cross et al., 2017). Intragranular lattice distortions are represented by the misorientation angle between each pixel and the mean orientation of the grain **[Figure 9]**. The GOS value is then equivalent to the average misorientation relative to the mean grain orientation for each grain (Wright et al., 2011). A trade-off curve of the number of grains in the sample against their GOS value is used to calculate the threshold between relict and recrystallised grains **[Figure 8]**. Relict grains are defined as grains with GOS values greater than the knee of the GOS curve and recrystallised grains as those with GOS values lower than the knee (Cross et al., 2017). Separation of relict and recrystallised grains using the GOS values is used for the pyrrhotite infill of the foliation boudinage structures. Only relict grains are used for strain analyses as they represent the earliest pyrrhotite grains to form and should have accumulated the greatest amount of strain within the pyrrhotite infill.

3.6 Grain Analysis

Mulchrone and Choudhury (2004) showed that ellipses can be fitted to arbitrary shapes that are not necessarily elliptical, such as grains. Best fit ellipses were calculated for individual grains in each EBSD map using the ellipse fitting calculation in the MTEX toolbox (Bachmann et al., 2010, 2011; Mainprice et al., 2015; Mulchrone & Choudhury, 2004). Data for individual grains were extracted as grain centre co-ordinates (X and Y), grain aspect ratios (Rf) and grain long axis orientations (ϕ).

The above results were used to perform 2-dimensional grain analyses using the open-source EllipseFit program (v.3.8.2) (Vollmer, 2018) to create R_f/ϕ plots (Dunnet, 1969; Ramsay, 1967) in order to obtain the mean ellipse ratio (R_s) for the combined relict grains and the orientation of the longest axis of the best-fitted ellipses. R_f/ϕ plots are widely used in strain analysis (Lisle, 1985; Ramsay & Huber, 1983) where the R_f parameter is most commonly plotted in the logarithmic $\ln(R_f)$ form (Vollmer, 2018).

3-dimensional strain is represented on Flinn plots (Flinn, 1965), strain ellipsoids and lower hemisphere equal area stereographic projections for each foliation boudin sample in order to show the orientations of the principal strains within the pyrrhotite infill. Flinn plots were created by combining two orthogonal orientations of 2-dimensional finite strain ellipses.

3.7 Drill Core Reorientation

Drill core was reoriented in the structural framework based on a modified method of Holcombe (2016) for partially oriented core which is outlined in **Supplementary 4**. Measurements of shale layering from the oriented Resource Development (RD) drillhole 'T667ED1' were used to calculate the mean dip and dip direction of the bedding (61° towards 263°). Measurements of shale layering orientations near the underground drillhole locations are, in general, in close agreement with the calculated mean.

3.8 Section Reorientation

Polished sections that contain the drillhole edge can be reoriented using the drillhole direction [**Supplementary 5**]. The straight edge of the drillhole in the section must be parallel to the direction of the drillhole at the point where the sample was taken. The sample can be reoriented by rotating the section about this known direction until the

bedding is in the correct orientation, based on oriented samples proximal to the sample location, reoriented drill core bedding measurements (section 3.7), or an assumed bedding dip and dip direction.

4. Results

4.1 Strain in Grains

4.1.1 Misorientations

The misorientations to the mean grain orientation within each grain are shown in **Figure 9a** and **Figure 9d**. In general, larger grains show greater misorientations and the development of subgrains are apparent. Checkerboard style subgrains are visible in some samples.

4.1.2 GOS Analysis

Grain orientation spread (GOS) analysis has been used to differentiate relict from recrystallised grains [**Figure 8 and Figure 9c,f**]. The GOS analysis also identifies the significant intragranular lattice distortions of up to 20° in some grains [**Figure 9b,e**]. Relict grains are generally elongate, or ribbon shaped. Recrystallised grains are smaller, slightly elongate and form networks surrounding relict grains.

4.1.3 Grain Shape Preferred Orientations (SPO)

Orientations of the pyrrhotite grain long axes from the infill of foliation boudinage structures are shown in **Figure 10**. Both the XZ- and YZ-planes for the four foliation boudinage structures are plotted as equal area moving average rose diagrams in MATLAB (Munro & Blenkinsop, 2012). A weighting factor of 0.9 and aperture of 9° was used during plotting as recommended by Munro and Blenkinsop (2012). Grain shape preferred orientations (SPO) that are approximately horizontal can be observed in all samples for both

the relict and recrystallised grains. The horizontal SPO is approximately parallel to the far-field shale layering in all samples.

4.1.4 Crystallographic Preferred Orientations (CPO)

Equal area, lower hemisphere pole figures of pyrrhotite grains within foliation boudinage structures are shown in **Figure 11**. Pyrrhotite *c*-axes show a well-defined crystallographic preferred orientation (CPO) in all samples [**Supplementary 6**]. The CPO patterns show the normal to the *c*-plane to be sub-perpendicular to the shale layering. Normals to the *m*- and *a*-planes are within the plane of shale layering and lie in a girdle about the perpendicular to the shale layering (*c*-axis).

Measures of fabric strength are shown by the M-index of Skemer et al. (2005) (0.11 in the XZ-plane and 0.13 in the YZ-plane) and J-index (1.76 in the XZ-plane and 1.80 in the YZ-plane), indicating a weak to moderate fabric strength of the CPO in FBS1 [**Figure 11**]. There is little difference between the CPO produced using all grain data and using only relict grains [**Supplementary 6**]; as a result, the whole dataset is used.

4.2 2D Strain Analysis

Strain analyses of relict pyrrhotite grains are illustrated in 2-dimensions by $\ln(R_f)/\phi$ plots [**Figure 12; Supplementary 7**]. The $\ln(R_f)/\phi$ plots for the four foliation boudinage structures show a dominant shale layer parallel orientation of the pyrrhotite grains in the XZ- and YZ-planes of **Figure 7**, supporting the evidence for the SPO fabric in **Figure 10**. The 2-dimensional finite strain (R_s) within the infill of the foliation boudinage structures is estimated from the mean ellipse of the R_f/ϕ data. R_s -values vary from 1.43 to 1.62 in the XZ-plane, 1.36 to 1.60 in the YZ-plane and 1.04 to 1.28 in the XY-plane. For the two foliation

boudinage structures with R_s -values in the XY-plane, these are between the R_s -values in the XZ- and YZ-planes.

4.3 3D Strain Analysis

4.3.1 Flinn Plots

The shape of the 3-dimensional finite strain ellipsoid is shown in **Figure 13** as a Flinn plot of the four foliation boudinage structures. The shapes of the finite strain ellipsoids are calculated using the combined 2-dimensional strain values (R_s) in **Figure 12** and **Supplementary 7** for the two orthogonal XZ- and YZ-planes. All foliation boudinage structures analysed in this study plot in the flattening strain region of the diagram. FBS2 and FBS4 have maximum principal strain directions in the X-axis of the foliation boudinage structure, whilst FBS1 and FBS3 have maximum principal strain directions in the Y-axis of the foliation boudinage structure.

4.3.2 Orientation of Principal Strain Axes

Shortening directions for the relict pyrrhotite grain infill of the four foliation boudinage structures are plotted in **Figure 14**. Shortening directions are calculated as normals to the plane passing through the directions of the longest axes for the mean relict grain ellipses in the XY- and YZ-planes. Neck lines of foliation boudinage structures are estimated from the normal to the XZ-plane and are plotted for reference. Foliation boudinage structure neck lines plot close to the mean shale layering of Williams et al. (2023). The shortening direction within the relict pyrrhotite grain infill is the local minimum principal strain direction. The maximum principal strain direction is assumed to be up dip, based on the typically north-south neck line orientations of oriented foliation boudinage structures of Williams et al.

(2023). Shortening directions for the northerly plunging foliation boudinage structures are ENE-WSW and for the southerly plunging structures, they are WNW-ESE. Errors between $\pm 1^\circ$ and $\pm 9^\circ$ for the plunge and $\pm 3^\circ$ and $\pm 12^\circ$ for the azimuth of the shortening direction were calculated during the reorientation of polished thin sections [Supplementary 8].

5. Discussion

5.1 Pyrrhotite Indexing

Few studies have investigated pyrrhotite using EBSD analysis (Qian et al., 2011; Uwanyuze et al., 2022; Vukmanovic et al., 2014, 2019) and as far as we know, only one recent study has specified the mineral system used during pyrrhotite indexing (Uwanyuze et al., 2022). The pyrrhotite at Mount Isa is weakly magnetic, characteristic of monoclinic Fe_7S_8 . XRD analysis also determined Mount Isa pyrrhotite as type 29-0723 monoclinic phase [Supplementary 2]. There is no monoclinic pyrrhotite in the available EBSD indexing databases. As there is essentially no difference in lattice parameters between the hexagonal FeS Troilite phase and the monoclinic Fe_7S_8 pyrrhotite phase, indexing using the hexagonal phase has a good efficiency (MAD of $<1^\circ$, $>90\%$ indexing for the total EBSD map and approximately $>98\%$ of pyrrhotite pixels indexed). These factors justify using the hexagonal FeS phase in this study for pyrrhotite indexing. Future EBSD studies of deformed pyrrhotite may consider using the available hexagonal indexing for pyrrhotite.

5.2 Deformation Mechanism of Pyrrhotite

The well-defined CPO patterns [Figure 11] with *c*-planes sub-perpendicular to the shale layering and large local misorientations (average relict grain misorientation $\sim 9^\circ$; Figure 9) for the pyrrhotite show that dislocation creep was the dominant deformation mechanism

within the infill of the foliation boudinage structures. Although we acknowledge that CPOs can also form due to solution-precipitation creep, the presence of large misorientation angles is indicative of dislocation creep and there is no evidence for solution surfaces that truncate grain boundaries. Cox (1987) showed that pyrrhotite commonly exhibits dislocation creep at the greenschist facies conditions, as at Mount Isa during the formation of foliation boudinage structures (Bell & Hickey, 1998; Perkins, 1984, 1997).

The CPO shows that basal slip sub-parallel to the shale layering dominated in the pyrrhotite samples. Pyrrhotite is weakest along the basal plane, where only a small angle between the compression axis and the *c*-axis is sufficient to induce basal slip (Kübler, 1985). Kübler (1985) also identified that prism slip, twinning, and kinking of pyrrhotite occur at higher temperatures.

It is likely the pyrrhotite deformation was dominated by coaxial flow (pure shear), with an element of non-coaxial flow (simple shear) owing to the pattern shown by the CPO. However, it is difficult to ascertain the degree of coaxial vs non-coaxial deformation. Determining the vorticity requires additional analysis that is beyond the scope of this study.

The clear *c*-axis texture shown in **[Figure 11]** is similar to the experimental results of Niederschlag and Siemes (1996), which show a strengthening of the pyrrhotite *c*-axis texture in pole figures where the angle between the compression axis and the initial *c*-axis is less than 35°. At these angles, the *c*-axes show a progressive tendency to orient parallel to the compression axis (Niederschlag & Siemes, 1996). Pyrrhotite pole figures in **Figure 11** show *c*-axes approximately normal to the horizontal shale layering and parallel to the approximate shortening direction. The experiments of Niederschlag and Siemes (1996) also suggest that at greater compression angles, the (0001) planes reorient perpendicular to the compression

axis, and the resulting pyrrhotite pole figures show this progressive reorientation. A similar behaviour may have occurred in some samples where the *c*-axes form a weak girdle [Supplementary 6].

5.3 Dynamic Recrystallisation of Pyrrhotite

The populations of relict and recrystallised grains are differentiated using the grain orientation spread (GOS) value. The GOS method is an EBSD based method for measuring intragranular distortions and hence determines recrystallised populations from relict grains independently of grain geometry. Unlike other methods, this method does not make assumptions that relict grains are larger and less spherical than recrystallised grains. This method has been used with success in dynamically recrystallised quartz grains (Cross et al., 2017; Tulley, 2021). Pyrrhotite grain geometry characteristics in **Figure 9** show evidence of dynamic recrystallisation by subgrain rotation (SGR). SGR is a response to the migration of dislocations into subgrain walls during progressive deformation, eventually forming recrystallised grains (Hirth & Tullis, 1992). Recrystallised grains are defined by the creation of subgrain boundaries at high angles ($>10^\circ$) to the relict grains.

Studies on quartz have shown that relict grains tend to be penetratively deformed and elongate or ribbon-shaped and contain numerous subgrains, whilst recrystallised grains usually have a minor elongation (Hirth & Tullis, 1992; Hobbs, 1968; Stipp et al., 2002a, 2002b, 2010; Stipp & Tullis, 2003). These characteristics are observed in the pyrrhotite infills of foliation boudinage structures [**Figure 9a,d**] and may explain why SPOs are found in both the relict and recrystallised grain populations **Figure 10**. Newly recrystallised pyrrhotite grains occur in sheets between old grain relicts forming a network of smaller recrystallised grains across the sample [**Figure 9**], similar to the quartz fabric observed by Hirth and Tullis

(1992) and Stipp et al. (2002b). CPO patterns can also develop in recrystallised grain aggregates and may originate from large relict grains that have been partially recrystallised (Passchier & Trouw, 2005).

5.4 Shape Fabric

The XZ- and YZ-planes of the foliation boudinage structures show grain shape preferred orientations (SPO) of the pyrrhotite grains [Figure 10]. Both the XZ- and YZ-planes have a dominant horizontal SPO, that is parallel or sub-parallel to the shale layering seen in Figure 7. The SPO is perpendicular to the c-axes of pyrrhotite grains as observed in other studies (Bayer & Siemes, 1971; McQueen, 1987; Ostwald & Lusk, 1978). Despite the relict grains typically having greater Rf-values (aspect ratios) than the recrystallised grains, the orientation of the SPO for both populations are remarkably consistent. It is difficult to make the distinction between the relict and recrystallised grains from looking solely at the rose plots of the SPO. The similarity in the orientations of relict and recrystallised pyrrhotite grain axes is probably caused by the recrystallisation process which forms slightly elongate recrystallised grains (Hirth & Tullis, 1992; Hobbs, 1968; Stipp et al., 2002a, 2002b, 2010; Stipp & Tullis, 2003). In all foliation boudinage structures analysed in this study, recrystallised pyrrhotite grains typically have a smaller area and are more numerous than the relict grains.

SPO can develop through dislocation creep (Passchier & Trouw, 2005), consistent with the results observed in the CPO. The deformation of the pyrrhotite resulted from dislocation creep along the basal planes. Pyrrhotite grains oriented favourably for basal slip will deform more readily and this could explain the relative lack of strain observed in some larger grains.

If the SPO is produced by coaxial flow, the SPO seen in **Figure 10** will be approximately parallel to the finite strain axes. The non-coaxial component has not been determined in this study but is thought to be relatively small within the foliation boudinage structures from the fabric observed in the CPO.

5.5 Strain in Pyrrhotite Filled Foliation Boudinage Structures at Mount Isa

The R_f/ϕ method has been used to calculate the aspect ratio and long axis orientation of the mean ellipse (R_s -value) for the relict pyrrhotite grain ellipses [**Figure 12**]. Fry plots were also created and showed similar results for the strain ellipses. The long axis orientation calculated in the R_f/ϕ analysis is consistent with that calculated by the SPO. A benefit of the R_f/ϕ method is the ability to consider the probable non-sphericity of the pre-deformation pyrrhotite grains. Recrystallised pyrrhotite grains were removed for the R_f/ϕ analysis to reduce the skew error in the resultant strain.

The directions of shortening during the pyrrhotite grain finite strain plunge gently towards either the ENE or ESE [**Figure 14**]. These shortening directions approximate the normal to the steeply west-dipping Urquhart Shale layering and can be explained as a strain response to the shortening of the steep anisotropy.

5.6 Timing of Pyrrhotite Deformation

Previous studies have shown that foliation boudinage structures form by plane strain deformation through layer normal shortening and layer parallel extension of a foliation or shale fabric (Arslan et al., 2008, 2012; Mandal & Karmakar, 1989; Platt & Vissers, 1980). This plane strain deformation results in the long axis necks of foliation boudinage structures in a single orientation parallel to the intermediate principal strain (Arslan et al., 2008, 2012;

Mandal & Karmakar, 1989; Platt & Vissers, 1980). Aerden (1991) showed evidence of 'chocolate tablet' foliation boudinage as a result of two boudinage axes parallel to the principal directions of the strain ellipsoid. This combination of strain directions is not observed at Mount Isa. Williams et al. (2023) showed that oriented foliation boudinage structures at Mount Isa have long axes that plunge gently to both the north and south. These orientations have been used as evidence for a progressive development from D4a ENE-WSW dextral-reverse shortening through to the D4b WNW-ESE sinistral-reverse shortening event of Miller (2007).

The maximum principal strain direction of FBS2 and FBS4 are in the X-direction of the foliation boudinage structures. This is to be expected as the foliation boudinage structures develop by the extension of layering in the X-axis. However, the maximum principal strain axis of FBS1 and FBS3 are in the Y-direction of the foliation boudinage structures. This is not expected based on the assumed foliation boudin formation mechanism and may be evidence for flattening post-pyrrhotite infill. The evidence of one dominant linear orientation of the necks of foliation boudinage structures (north-south) (Williams et al., 2023), implies the plane strain was dominant during their initial formation. Flattening strain during their initial formation would have resulted in the presence of two orthogonal orientations of foliation boudinage structures, similar to Rosebery Mine (Aerden, 1991).

For this reason, the flattening strain recorded in the pyrrhotite infills is likely to have post-dated the initial development of the foliation boudinage structures. The change from plane strain during the initial formation to a late stage flattening strain in the pyrrhotite infills is interpreted as an evolving strain path over the entire time of foliation boudin development at Mount Isa. The flattening strain could also have been a result of strain partitioning in the

weaker pyrrhotite infills of the foliation boudinage structures, but this would not account for the plane strain required for the initial formation of the structures. Some post-mineralisation flattening could have occurred during the later D5 events.

The single steeply SW-plunging structure in **Figure 14** is not compatible with the orientations of the necks of foliation boudinage structures in Williams et al. (2023) and may be a result of rotation during the later D5 events. However, the shortening directions of the pyrrhotite infills [**Figure 14**] are the same as the shortening of the foliation boudinage structures in Williams et al. (2023). North-plunging foliation boudinage structure lineations have a shortening direction that is to the ENE, whilst the south-plunging structure has a shortening direction to the ESE. This agrees with the evidence presented by Williams et al. (2023) that north plunging foliation boudinage structures are formed by dextral-reverse slip during D4a ENE-WSW shortening and south plunging structures resulting from the sinistral-reverse slip during the D4b WNW-ESE shortening event.

6. Conclusions

This study analysed foliation boudinage structures from the Mount Isa copper deposit using electron backscatter diffraction. The electron backscatter diffraction patterns show that pyrrhotite grains within foliation boudinage structures have significant intracrystalline deformation, resulting in large local misorientations within grains. The deformation observed within the pyrrhotite filled foliation boudinage structures have allowed conclusions to be made about the deformation mechanism, grain fabric, finite strain geometry and timing of deformation.

- Deformation of pyrrhotite in foliation boudinage structures was by dislocation creep, dominated by basal slip. Deformation was mainly coaxial, with a minor component

of non-coaxial deformation. Pyrrhotite grain c-axes are normal to the shale layering at Mount Isa, and parallel to the shortening direction.

- Dynamic recrystallisation of pyrrhotite occurred by subgrain rotation.
- Both relict and recrystallised grains show a grain shape preferred orientation due to deformation. Recrystallised grains form a network of typically smaller grains between relict grains.
- The shape preferred orientation of the relict grains is sub-parallel to the shale layering and mimics the geometry of the 3-dimensional strain ellipsoid, as shown by combining 2-dimensional strain analyses from the R_f/ϕ method.
- Shortening directions of the pyrrhotite grains are approximately east-west and are similar to the shortening directions of the foliation boudinage structures. This evidence for the shortening directions agrees with independent inferences from other foliation boudinage structures at Mount Isa (Williams et al., 2023).
- Foliation boudinage structures at Mount Isa developed during plane strain deformation in D4a through to D4b. Deformation of the pyrrhotite infill shows flattening strain that post-dates the initial formation of the foliation boudinage structures. The change from plane to flattening strain may reflect progressive deformation during foliation boudinage, because both strains have approximately the same common shortening direction.
- Strain in the sulphide infills of foliation boudinage structures at Mount Isa shows that foliation boudinage structures do not necessarily remain as open voids throughout their formation.
- The clear evidence for deformation of ore-related, hydrothermal sulphides confirms the syn-tectonic nature of copper mineralisation at Mount Isa.

CRedit authorship contribution statement

Benjamin J. Williams: Conceptualisation, Methodology, Software, Formal Analysis, Investigation, Data Curation, Visualisation, Writing - Original Draft, Writing – Review and Editing. **Thomas G. Blenkinsop:** Conceptualisation, Methodology, Writing – Review and Editing, Supervision.

Data Availability

Data will be made available on request.

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FIGURE CAPTIONS

Figure 1: (a) The location of Mount Isa and the main divisions of the Proterozoic Mount Isa Inlier, northwest Queensland, Australia. After Williams et al. (2023), Blake and Stewart (1992) and Davis (2004). (b) A geological map of the Mount Isa region with local mine grid, after Williams et al. (2023) and Spatial and Graphic Services Statewide Operations Department of Natural Resources Mines and Energy (2020). A-A' section line shown in **Figure 3a**. B-B' section line shown in **Figure 3b**.

Figure 2: Deformation events at the Mount Isa deposit based on previous deformation schemes (Bell et al., 1988; Davis, 2004; Miller, 2007; O'Dea, Lister, Betts, et al., 1997; O'Dea, Lister, MacCready, et al., 1997; O'Dea & Lister, 1995; W. G. Perkins, 1984; Swager, 1985; Wilde, 2011; Williams et al., 2023). **D1** – localised reverse reactivation of normal faults as a result of north-south shortening; **D2** – east-west shortening and peak metamorphism; **D3** – top to the east shear, weakly developed at Mount Isa Mine; **D4a** – ENE-WSW shortening and mineralisation at Mount Isa; **D4b** – WNW-ESE shortening and mineralisation at Mount Isa; **D5a** – top to the east transport direction on west-dipping reverse faults; **D5b** – conjugate sinistral-reverse and dextral-reverse faults with top to the east or west transport directions consistent with continued east-west shortening.

Figure 3: East-west cross-sections through the Mount Isa deposit, showing the relative positions of the Pb-Zn and Cu orebodies, pyrrhotite and silica-dolomite after Williams et al. (2023). Sample locations are shown for each foliation boudinage structure analysed in this study and correspond to their relative positions in relation to the mineralisation. (a) Cross section in the south of the mine at 4350mN. B-B' in **Figure 1**. (b) Cross-section in the north of the mine at 7000mN. A-A' in **Figure 1**.

Figure 4: The formation mechanisms of the common symmetrical and asymmetrical foliation boudinage structures by approximate layer normal shortening and layer parallel extension of fractures, based on Arslan et al. (2008, 2012), Hambrey and Milnes (1975), Mandal and Karmakar (1989) and Platt and Vissers (1980). Different geometries of foliation boudinage structures can form depending on fracture orientation and flow type. Figure after Williams et al. (2023).

Figure 5: Experimental deformation showing the strengths (differential stress) of mineral aggregates and carbonate rocks as a function of temperature at a strain rate of $\sim 7.5 \times 10^{-5} \text{ s}^{-1}$ and 100 MPa confining pressure. Modified from Clark and Kelly (1973) and Kelly and Clark (1975).

Figure 6: Oriented reference axes of foliation boudinage structures at Mount Isa.

Figure 7: Samples of foliation boudinage structures cut in a minimum of two orthogonal directions, represented by the XZ-, YZ- and XY-planes.

Figure 8: Cumulative plot of the number of pyrrhotite grains versus the grain orientation spread (GOS). The GOS threshold (red circles) defines the difference between relict (above threshold) and recrystallised grains (below threshold). (a) GOS graph of the XZ-plane for FBS1. (b) GOS graph of the YZ-plane for FBS1.

Figure 9: EBSD maps of pyrrhotite infills of FBS1 in cross sections XZ-plane (left) and YZ-plane (right). (a) Map of all pyrrhotite grains showing the local misorientations within each grain. (b) Grain orientation spread (GOS) value for each grain. (c) Relict and recrystallised grains as determined by the GOS threshold in **Figure 3a**. (d) Map of all pyrrhotite grains

showing the local misorientations within each grain. (e) GOS value for each grain. (f) Relict and recrystallised grains as determined by the GOS threshold in **Figure 3b**.

Figure 10: Rose plots of relict and recrystallised grains in two perpendicular planes (sample XZ-plane and YZ-plane). (a) FBS1. (b) FBS2. (c) FBS3. (d) FBS4.

Figure 11: Equal area, lower hemisphere pole figures for pyrrhotite grain orientations within FBS1, with one point per grain. Contours in units of multiples of uniform distribution (MUD). Pyrrhotite $(11\bar{2}0)$ and $(10\bar{1}0)$ planes form a girdle parallel to the shale layering. Pyrrhotite (0001) planes show a defined crystallographic preferred orientation (CPO), with c-axes normal to the shale layering. N is equal to the number of grains. Fabric strengths are presented using the J-index and M-index for both (a) XZ-plane of FBS1 and (b) YZ-plane of FBS1.

Figure 12: Logarithmic $\ln(R_f)/\phi$ plots of relict pyrrhotite grains (blue points) within one foliation boudinage structure (FBS1). Grain long axis orientations relative to the XY-plane of the foliation boudinage structure are plotted on the X-axis and R-values are plotted on the Y-axis. R_f/ϕ plots of XZ- and YZ-planes are shown with the R_s -value and grain long axis orientation of the mean ellipse (red point). R_s -values of the pyrrhotite approximate the 2-dimensional finite strain within the foliation boudinage structure infill. (a) XZ-plane and (b) YZ-plane of FBS1.

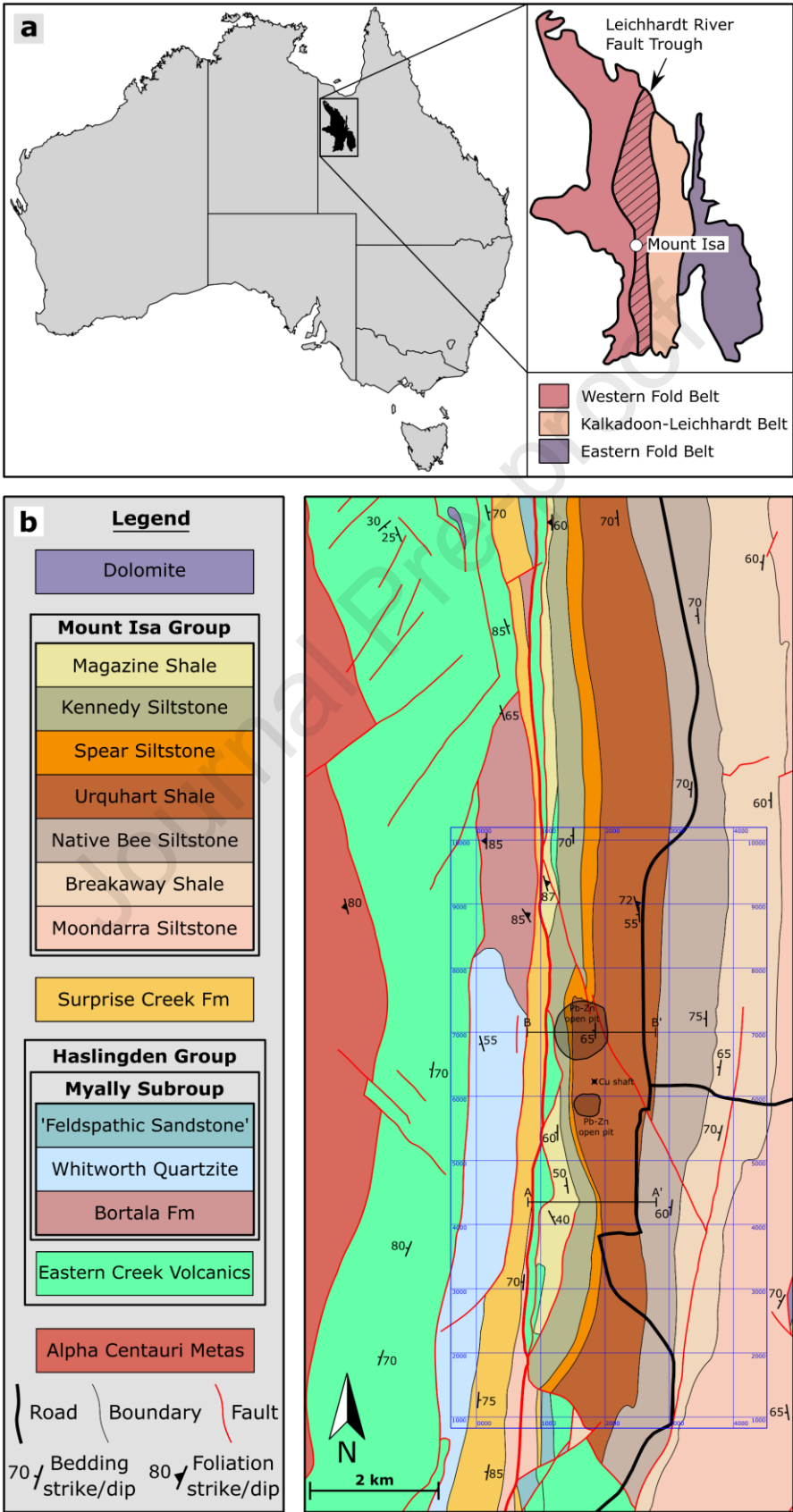
Figure 13: Flinn plot showing the 3-dimensional shape of the finite strain ellipsoid in the pyrrhotite infill of four foliation boudinage structures. The strain ellipsoid is calculated using the 2-dimensional strain values in **Figure 6**. The foliation boudinage structures plot in the flattening strain region of the diagram. Square: X-axis of foliation boudinage structure =

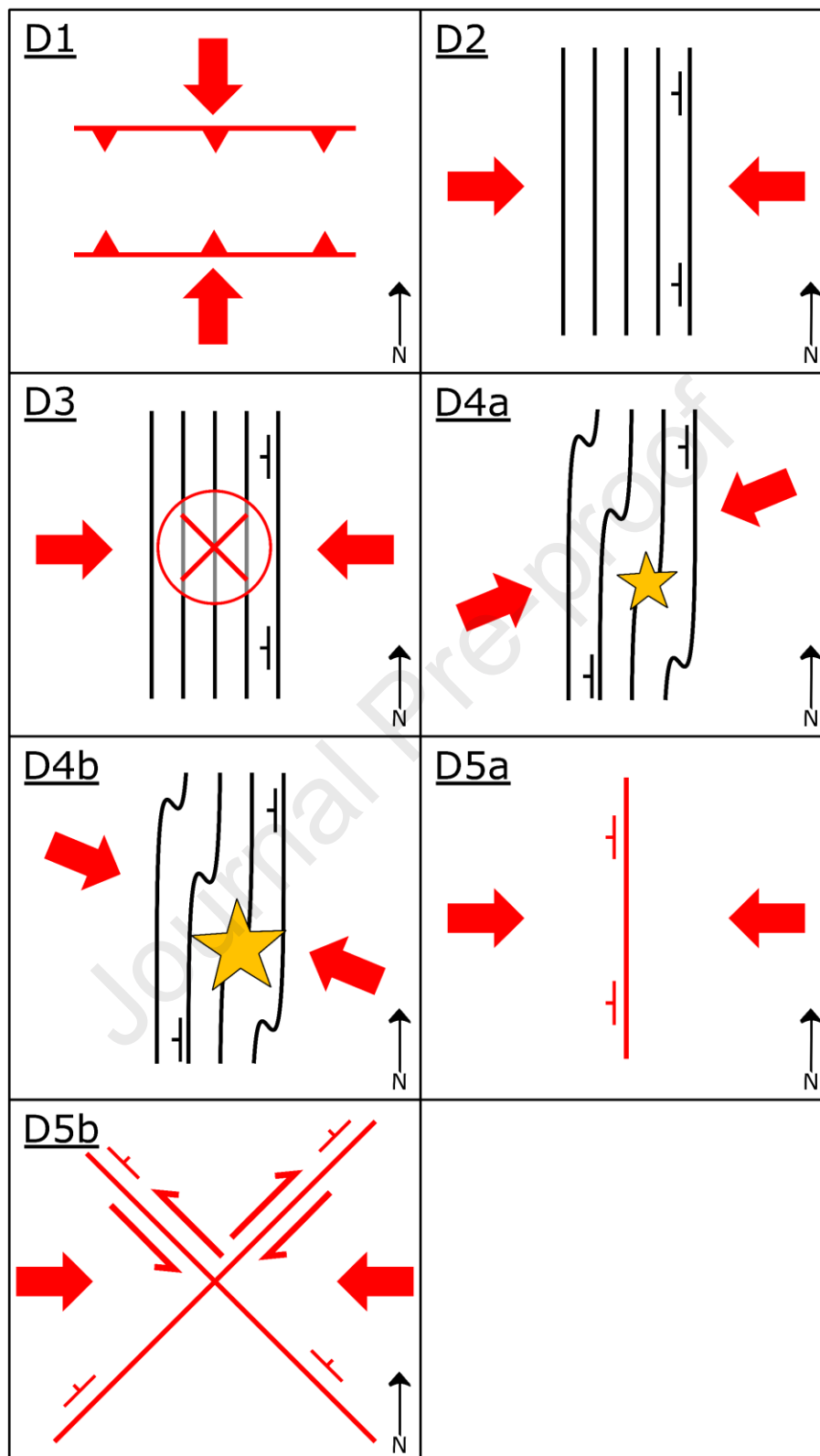
912 direction of maximum principal strain; Circle: X-axis of foliation boudinage structure =
913 intermediate strain direction.

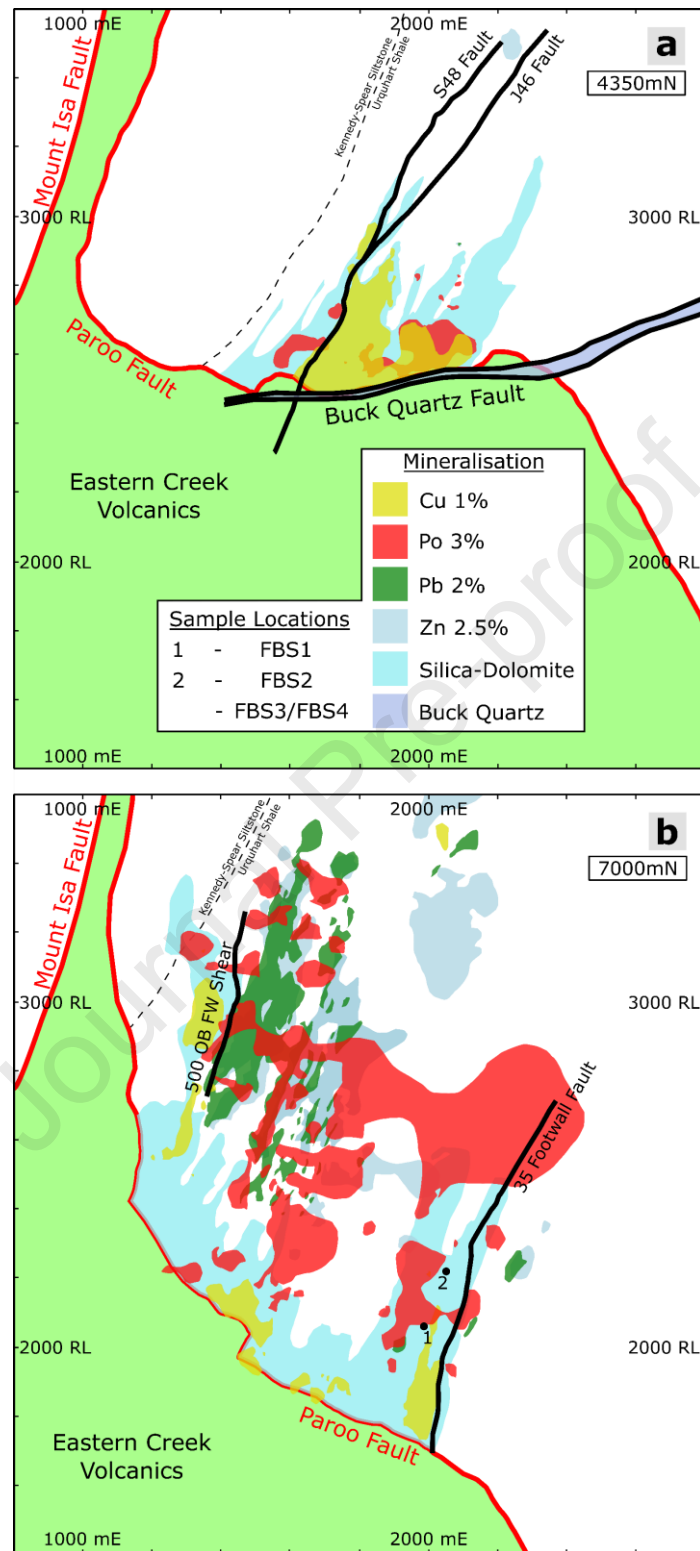
914 **Figure 14:** Shortening directions for the relict pyrrhotite grains within the four foliation
915 boudinage structures. Shortening directions, neck lines of foliation boudinage structures
916 and the mean shale layering orientation of Williams et al. (2023) are plotted.

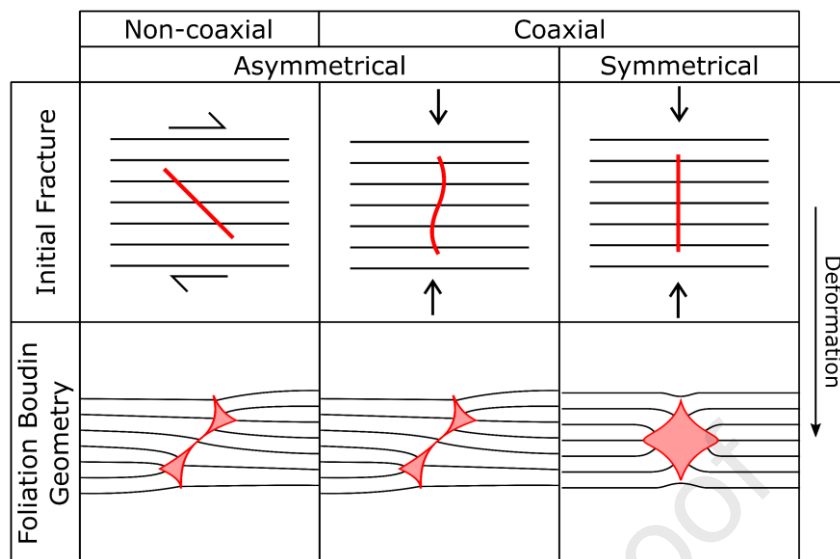
917 **FIGURES**

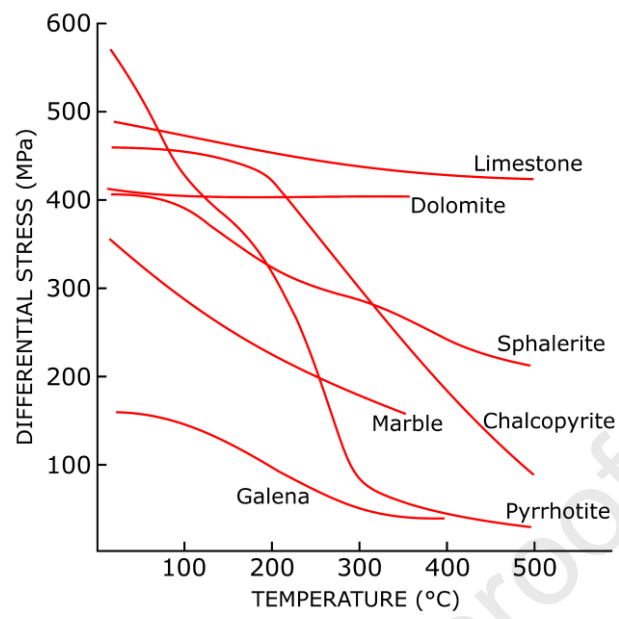
918 **Figure 1**

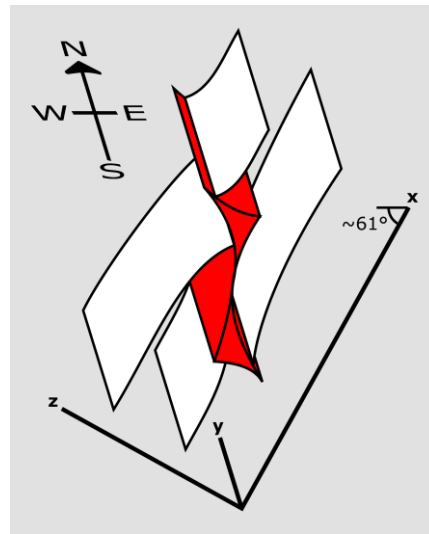


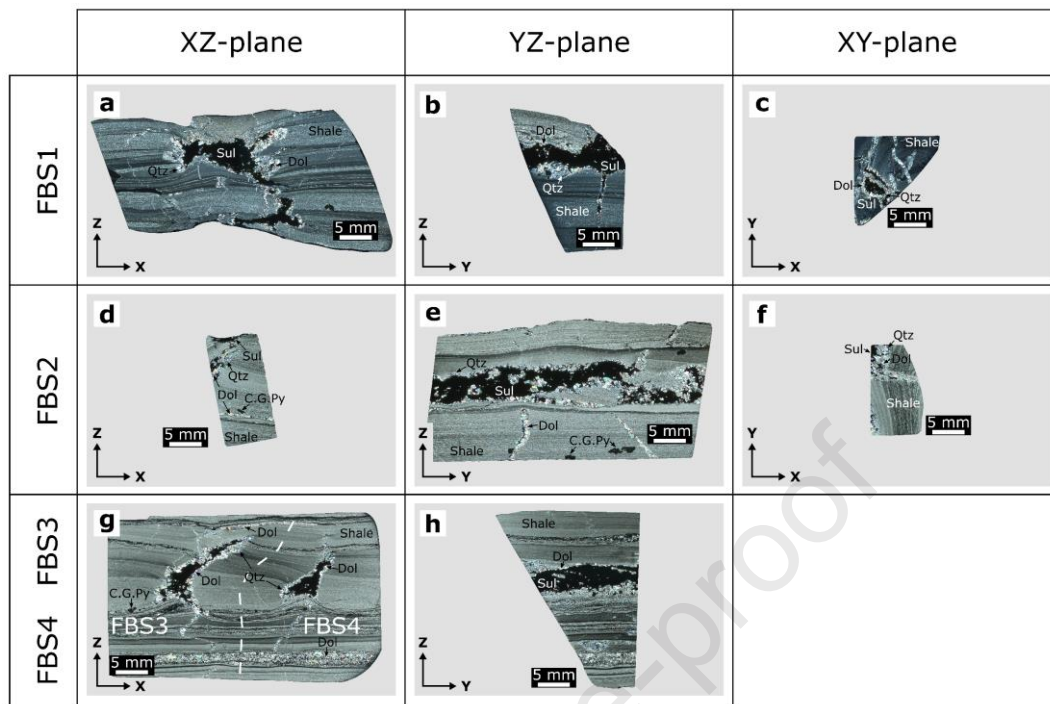
919 **Figure 2**

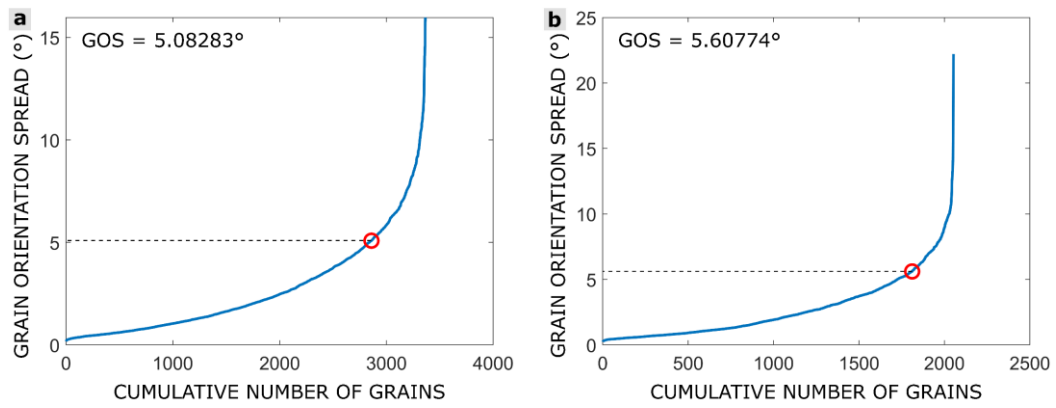
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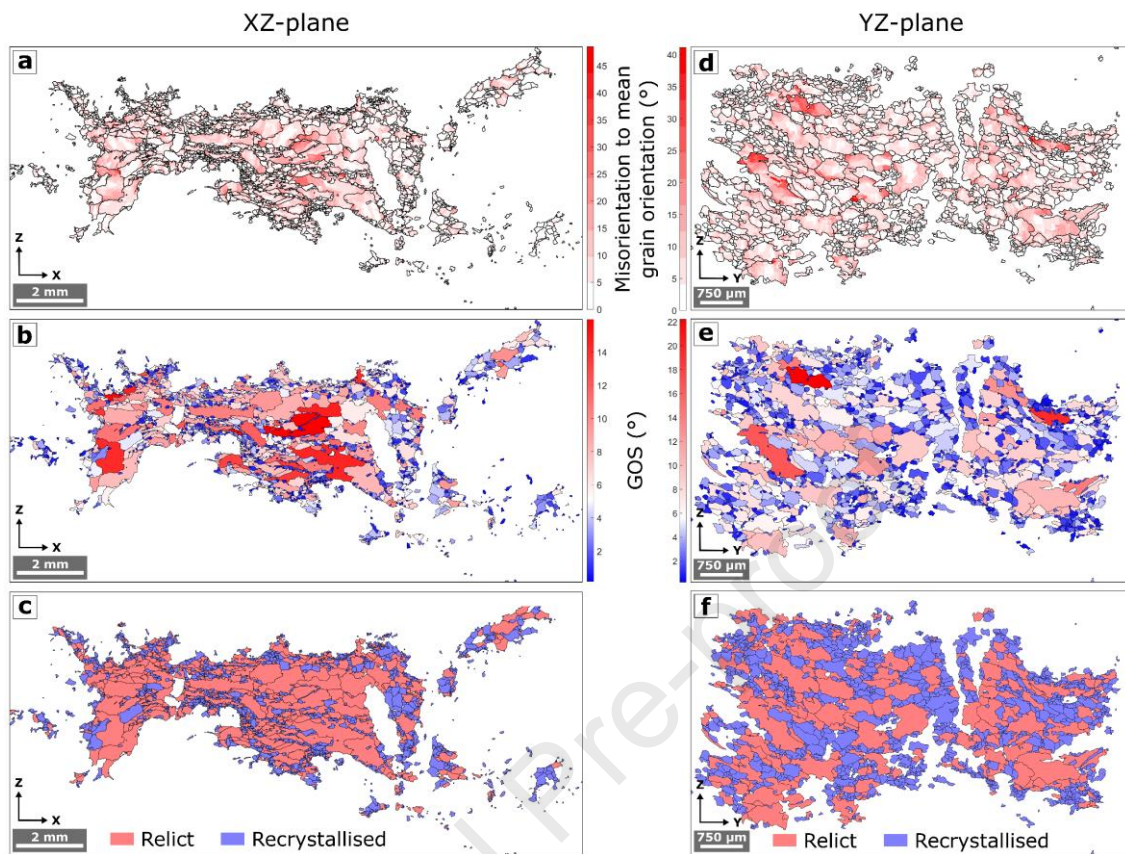
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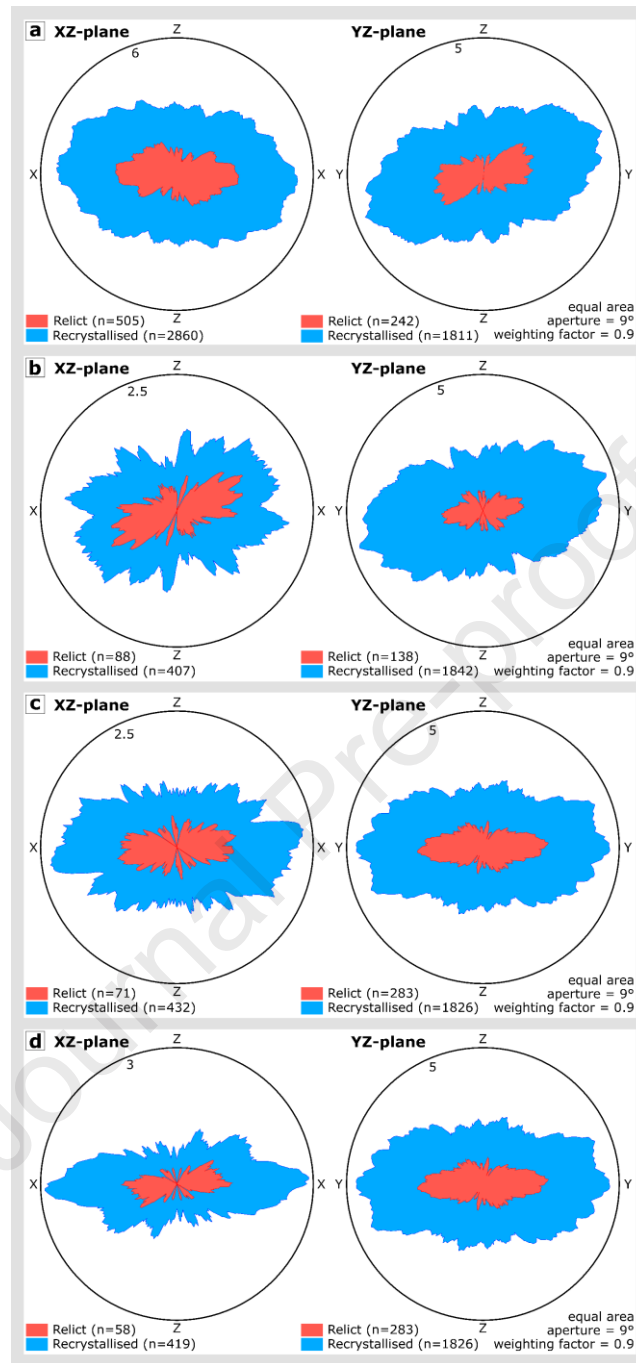
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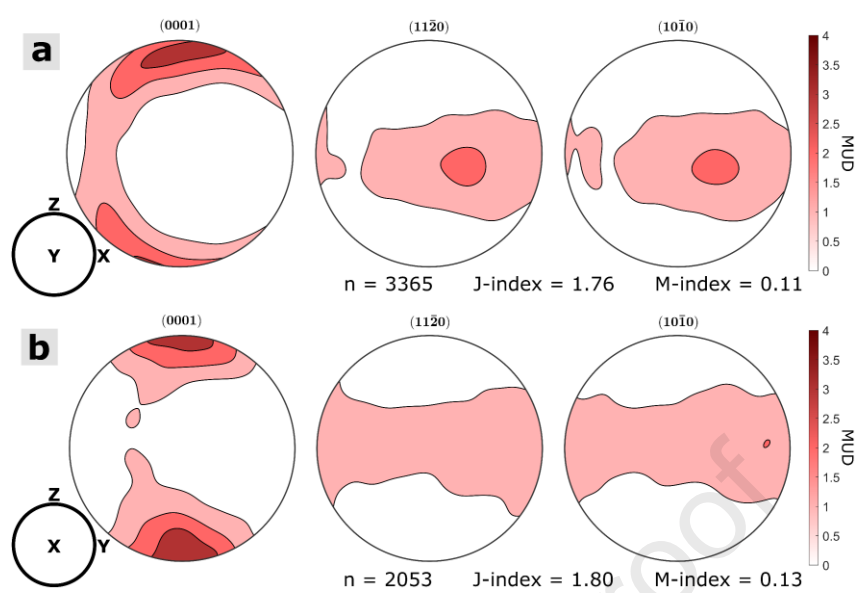
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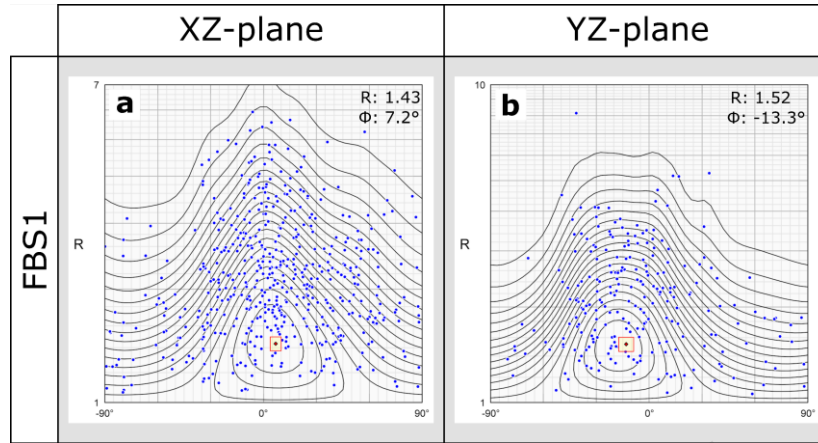
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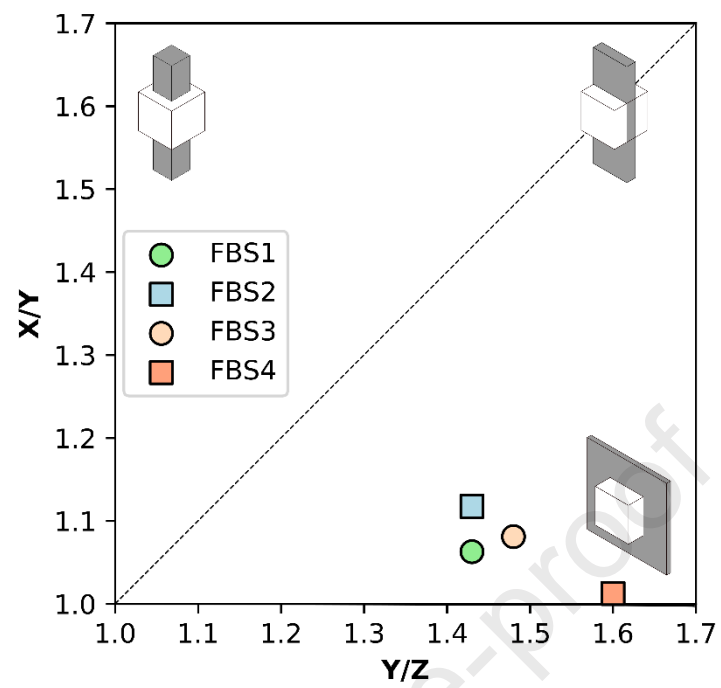
925 **Figure 8**

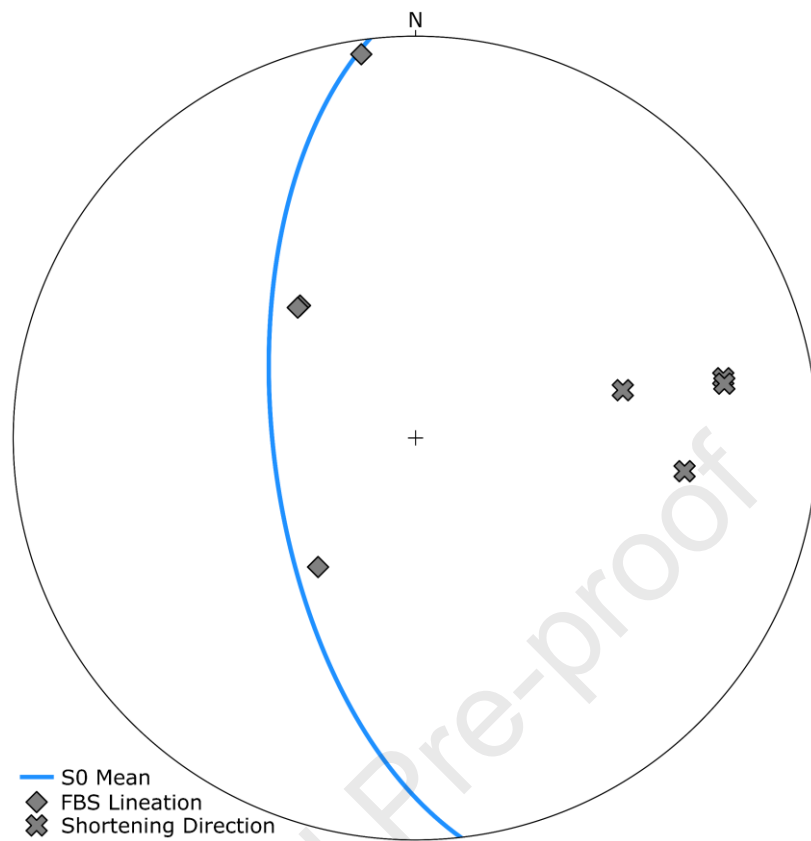
926 **Figure 9**

927 **Figure 10**

928 **Figure 11**

929 **Figure 12**

930 **Figure 13**

931 **Figure 14**

Highlights:

- Electron backscatter diffraction analysis of pyrrhotite filled foliation boudinage structures at Mount Isa
- The pyrrhotite has shape and crystallographic preferred orientations
- Dislocation creep and dynamic recrystallisation by subgrain rotation were facilitated by basal slip in the pyrrhotite
- Plane strain changed to flattening during deformation of the pyrrhotite infill as the foliation boudinage structures evolved

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: