Reconstructing the pyrite deformation mechanism map

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ABSTRACT

Deformation mechanism maps are important tools to understand how minerals deform across a range of pressure, temperature and strain rate conditions. Recent studies have suggested that the current deformation mechanism map for pyrite is in need of revision, with plastic deformation evident over a much wider range of conditions than it proposes. This study uses stress-strain data from experimentally deformed pyrite, combined with electron backscatter diffraction (EBSD) results from a range of experimental and natural samples to construct a revised pyrite deformation mechanism map. This map corresponds to polycrystalline pyrite with a grain size of ~35μm and suggests the onset of crystal plasticity at geological strain rates ($\sim 10^{-12}$-10^{-16} s^{-1}) occurs at temperatures as low as ~260 ºC. The revised mechanism map is based upon all available data, experimental data for pyrite are limited and acquired at geologically unreasonable, fast strain rates (10^{-4}-

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10^{-5} s^{-1}), and strain rates for the naturally deformed samples can only be estimated. Therefore, considerable extrapolation is required and while this revised map is an improvement upon the existing map, it should still only be considered a 'guide' to the potential deformation mechanisms, which may operate across a range of P-T conditions in polycrystalline pyrite-rich ores.

Keywords: pyrite, deformation mechanism map, experimental deformation, natural deformation, EBSD

1. Introduction

Pyrite (FeS_2) is the most abundant sulphide mineral in the Earth's crust and occurs in a myriad of environments, but is ubiquitous in sulphide ore deposits (Mann et al. 1990; Gu and McClay 1992; Brown and McClay 1993; Craig and Vokes 1993; Cook 1996; Craig et al. 1998). Many ore deposits form on or close to the seafloor at rifted margins and, therefore, at some stage in their history become involved in orogenic events, experiencing deformation and/or metamorphism (Vokes, 1980; Allen et al., 2002). Pyrite itself is not of economic importance, but its refractory nature allows it to resist the remobilisation and recrystallisation processes that commonly affect the softer, economic sulphides such as sphalerite, galena and chalcopyrite during retrogression, enabling it to preserve evidence of preceding high temperature events (McClay and Ellis 1983; Marshall and Gilligan 1987; Plimer 1987; Marshall and Gilligan 1993; Bailie and Reid 2005). Understanding the deformation mechanisms that operate in pyrite across different P-T conditions is, therefore, essential in reconstructing the post-depositional history of ore deposits and maximising their exploitation (Freitag et al., 2004).

Pyrite deformation mechanisms have been the focus of a number of studies since the early 1960s, initially using experimental deformation, reflected light analysis and etching to reveal dislocation microstructures (Gill 1969; Ramdohr 1969; Graf and Skinner 1970; Atkinson 1975). All of this work suggested pyrite was a hard mineral, which deformed by brittle mechanisms at all levels in the Earth's crust. Subsequent high resolution transmission electron microscopy (HR-TEM) of experimentally deformed pyrite revealed the presence of dislocations within pyrite grains suggesting crystal-plastic deformation could operate at temperatures as low as ~425 ºC (Couderc et al., 1980; Cox et al., 1981; Graf et al., 1981; Levade et al., 1982). These experimental results were combined with analysis of naturally deformed pyrite to construct the current pyrite deformation mechanism map (McClay and Ellis, 1983) (Fig. 1).

FIG. 1 HERE

Deformation mechanism maps are graphical models that show the dominant deformation mechanism for a given mineral at different conditions (Ashby, 1972; Frost and Ashby, 1982). These maps assist understanding of the general conditions under which deformation mechanisms operate and are available for a number of minerals (quartz, calcite, galena, etc) (e.g. White 1976; Atkinson 1977; Frost and Ashby 1982; Ranalli 1982; Tsenn and Carter 1987; Drury and Humphreys 1989). Whilst the field name denotes the dominant mechanism, this mechanism is not exclusive and other subordinate mechanisms may also be active (Mohamed and Langdon, 1974; Pearce and

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Wheeler, In Press). Deformation mechanism maps need to be used with caution, partly because of the limited data on the high temperature operation of various mechanisms and also because construction requires significant extrapolation from experimental to geological strain rates (Rutter, 1976; White, 1976; Cox, 1987; Siemes, 1991).

Recent studies of deformed pyrite using electron backscatter diffraction (EBSD) have indicated the pyrite deformation mechanism map of McClay and Ellis (1983) (Fig. 1) needs revision. Results from these studies reveal crystal-plastic deformation operates in pyrite over a much wider range of P-T, grain size and strain rate conditions than previously suggested and potentially at temperatures as low as ~200 ºC (Barrie et al., 2007; Barrie et al., 2009; Barrie et al., 2010a). Combining the results of these previous findings with assessment of further experimental samples this study has three principal aims:

1. To determine how deformation mechanisms in pyrite vary across a range of well-constrained temperature, stress and strain rate conditions from experimentally deformed samples and how they compare with results from naturally deformed samples.

2. To understand how cataclastic textures and recovery/recrystallisation processes affect the ability to detect crystal-plastic deformation in pyrite.

3. To use the results gathered from this, and earlier studies of both natural and experimentally deformed pyrite to construct a new pyrite

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deformation mechanism map, outlining the problems and opportunities of this revised map.

2. Methodology

The samples discussed in this study have all been prepared and analysed using the same techniques as those of Barrie et al. (2007). Therefore, only a brief description of sample preparation and data analysis is given below.

TABLE 1 HERE

2.1. Samples

All of the experimentally deformed samples discussed in this study (Table 1) come from an original block of fine grained polycrystalline pyrite-rich ore from the Blow ore body of the Mt Lyell Mining and Railway Company near Queenstown, Tasmania (Cox et al., 1981; Barrie et al., 2007). Suites of the cored samples were deformed in a gas apparatus by triaxial shortening (Cox et al., 1981) at strain rates between 1.2×10^{-1} s^{-1} and 2×10^{-5} s^{-1}, confining pressure of 300 MPa and temperatures between 450-700 ºC (Fig. 2). With the exception of Runs 026 and 063, which represent unpublished experimental data, all of the samples in this study were initially discussed in Cox et al. (1981). After deformation, and prior to any analysis, the samples were cut parallel to their core length, impregnated in resin blocks and mechanically polished (Cox et al., 1981; Siemes et al., 1993; Barrie et al., 2007). All of the naturally deformed samples included in this study are from polymetallic,

pyrite-rich ore deposits and were previously discussed by Barrie et al. (2009; 2010a).

FIG. 2 HERE

2.2. Electron backscatter diffraction (EBSD)

All pyrite crystallographic orientation data were collected by EBSD at the University of Liverpool using a CamScan X500 crystal probe scanning electron microscopes (SEM). The CamScan X500 is equipped with a thermionic field emission gun and was used with an accelerating voltage of 20kV and a beam current of ~5nA during analysis. Electron backscatter patterns were collected on a phosphor screen and processed using the software package CHANNEL 5 service pack 6 (*Oxford Instruments Ltd).* The angular resolution of this technique is typically better than ~1º with a spatial resolution of ~0.05µm (Prior et al., 1999; Wheeler et al., 2001; Ohfuji et al., 2005). All of the samples, prior to analysis, were polished on a polyurethane lap for ~2h using a suspension of 0.05μm colloidal silicon (SYTON^{TM}) to remove any surface damage. Samples were also given a thin carbon coat (~4-6nm), prior to EBSD, in order to reduce charging effects and maintain a strong crystallographic signal (Prior et al., 1996; Prior et al., 1999). Following acquisition the data were post-processed to remove noise and interpolate a small number of missing points using routines outlined by Barrie et al. (2007).

2.3. Orientation Contrast Imaging and Band Contrast Mapping

Forescatter orientation contrast (OC) images were utilised throughout

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this study and are generated by detectors in an SEM positioned in front of samples (Prior *et al.*, 1999). These images reveal misorientation of the crystal lattice of individual grains through grey scale variation. Where grains are undeformed they will appear homogeneous in OC images. If, however, deformation has occurred then the grain will contain grey scale variation as a result of lattice misorientation (for further details see Prior *et al.*, 1996, 1999).

Band contrast maps are generated during EBSD and are indicative of diffraction pattern quality, with grey-scale differences representing the quality of the diffraction pattern at individual points (Bestmann and Prior, 2003; Prior et al., 2002). The quality of the diffraction patterns is a function of a number of factors but largely represents the quality of the lattice so that pristine crystal lattice is bright whilst distorted lattice with abundant dislocations is darker, Grain boundaries and fractures do not return any patterns so appear black meaning that band contrast images give a good picture of the rock microstructure. Band contrast maps are used throughout this study to highlight changes in pyrite microtextures, with increasing temperature of deformation (Barrie et al., 2007), which are beyond the resolution of SEM imaging.

To quantify the amount of lattice distortion within a sample, the mean misorientation was calculated for each grain. This is the sum of the misorientation between each pixel of a grain and the mean orientation of that grain divided by the number of pixels and therefore is analogous to the variance of the orientations within a grain. These mean misorientations are then averaged over the whole sample to give an average mean misorientation for each sample.

3. Results

Detailed results for the naturally deformed samples discussed in this study are presented elsewhere (Barrie et al., 2009; Barrie et al., 2010a) as are those for one suite of the experimental samples (Barrie et al., 2007). Therefore, only the characteristics of the experimental samples new to this study will be outlined. Siemes et al. (1993) and Barrie et al. (2007) described the starting material, from which all of the experimental samples were deformed, and their results are included in this study to assist with textural, grain size and grain-distortion comparisons (Fig. 3, 4A).

FIG. 3 HERE

FIG. 4 HERE

3.1. Low-Temperature Experimental Runs (450-500 °C)

Three samples were deformed at temperatures between ~450-500 °C (Table 1; Fig. 2) and strain rates of 2×10^{-4} s^{-1} (run 049) and 2×10^{-5} s^{-1} (runs 032 and 035). All of these runs failed prior to completion and preserve evidence for brittle textures (Fig. 5). Fracturing is widespread in run 032 (450 °C) where conjugate shear zones have developed at ~45° to shortening (Fig. 5A, B). Pyrite grains within these zones contain sharp, irregular boundaries and grain size is ~10-20μm (Fig. 5C), while pyrite grains outside these zones are equant and ~35-40μm in size (Fig. 3, 5D). Runs 049 (470 °C) and 035 (500 °C) have less extensive fracturing, and are characterised by equant pyrite grains with relatively straight, often bulged grain boundaries (Fig. 4B, C). While all of the samples contain a similar, mean grain size of ~35-40μm

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(Fig. 3A), Runs 049 and 035 do contain suites of <10μm, rounded pyrite grains surrounding larger pyrite grains (Fig. 3B, C, 4B, C).

Orientation contrast (OC) images show lattice orientation varying within pyrite grains in all three of the low temperature runs. EBSD results indicate cumulative lattice distortion (Fig. 6A, B) within pyrite grains and the development of low-angle (~2º) sub-grain boundaries, although this is rare at 450 °C (032). The average mean misorientation within pyrite grains at 450 ºC is ~2º (Fig. 6B), while results for the 470-500 ºC samples are larger ~4-5º (Fig. 6A, B). Neighbour-pair misorientation histograms (Wheeler et al., 2001) reveal low-angle (~2º) peaks within all of these runs with the peak height increasing with temperature (Fig. 7).

FIG. 5 HERE

3.2. Mid-Temperature Experimental Runs (550-600°C)

Six samples were deformed at temperatures between 550-600 ºC and strain rates of 2×10^{-4} s^{-1} (runs 048, 063, 092), 2×10^{-5} s^{-1} (runs 037, 054) 1.2×10^{-1} s^{-1} (run 026) (Table 1; Fig. 2). All of the 550-600 ºC samples show similar textural characteristics. Although four of the runs terminated with specimen failure (Fig. 2), fracturing appears minimal. Mean grain size within all of the runs is ~35-40μm (Fig 3A) with only 092 having a larger grain size, possibly due to the starting material being cored from a coarser layer of the original ore (Barrie et al., 2007; Cox et al., 1981). Pyrite grain boundaries tend to be sutured and decorated by small (<10μm), anhedral pyrite grains (Fig. 4D, E).

OC images indicate widespread internal grey-scale variation within pyrite grains in all of the samples (Fig. 8A, C). EBSD results reveal widespread and abundant low-angle (~2⁰) low-angle sub-grain boundaries within most if not all pyrite grains (Fig. 8B, D) with a maximum mean misorientation of ~12⁰ and an average across the samples of ~4-5⁰ (Fig. 6). Neighbour-pair misorientation histograms reveal a low-angle (~2⁰) peak for the 2×10^{-5} s^{-1} 550 ⁰C run (037), which decreases in intensity at higher temperature (for the same strain rate, sample 054). (Fig. 7).

FIG. 6 HERE

FIG. 7 HERE

3.3. High-Temperature Experimental Runs (650-700°C)

Six samples were deformed at temperatures between 650-700 ⁰C and strain rates of 2×10^{-4} s^{-1} and 2×10^{-5} s^{-1} (Table 1; Fig. 2). All of the runs have a mean grain size of ~35-40µm (Fig. 3A) with the exception of run 020, which has notably larger (~45µm) grain size, most probably reflecting a coarser layer of the original starting material. None of the runs within this temperature regime failed during deformation and there is minimal evidence for fracture development. Pyrite grain boundaries in the 650 ⁰C samples at 2×10^{-4} s^{-1} are sutured and many preserve elongate or 'ribbon' like textures, often decorated by suites of smaller anhedral pyrite grains (<20 µm). Pyrite grains deformed at 650 ⁰C, and 700 ⁰C in the 2×10^{-5} s^{-1} samples, have long, curved pyrite-pyrite grain boundaries often lobate (Fig. 4F) but not sutured.

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OC images indicate internal grey-scale variation is present within some pyrite grains, but the majority appear homogeneous (Fig. 8E, G). Low-angle (~2⁰) sub-grain boundaries are present within some pyrite grains, but are not widespread. They are generally restricted to grain boundaries and rarely within grain cores (Fig. 8F, H). Mean misorientation within pyrite grains is relatively low with a maximum of ~6⁰ at 650 ⁰C and ~4⁰ at 700 ⁰C (Fig. 6). The low-angle (~2⁰) boundary peak in the neighbour-pair misorientation histograms for the 2×10^{-5} s^{-1} runs at both 650 ⁰C (055) and 700 ⁰C (020) are of a similar height to the low-angle peak in the starting material (B-1) (Fig. 7).

FIG. 8 HERE

4. Discussion

4.1. Brittle Vs Crystal-Plastic Deformation

Brittle deformation textures are only evident in the lowest temperature runs (<500 ⁰C), with conjugate shear zones developed at 450 ⁰C (032) (Fig. 5A, B, C). However, while brittle mechanisms are clearly evident at 450 °C (032), OC imaging and EBSD analysis indicates lattice distortion is present in the pyrite grains surrounding the shear zones and unaffected by cataclasis (Fig. 5A, B). This distortion implies at least some crystal-plastic deformation has operated in these samples, although subordinate to the dominantly brittle behaviour. These results suggest that temperatures of ~450 ⁰C may represent the transition zone between cataclastic and crystal-plastic mechanisms of deformation at fast strain rates of 10^{-4} s^{-1}-10^{-5} s^{-1} (Fig. 2, 5). Brittle behaviour is also apparent in the runs deformed at >500 ⁰C, however, it is minimal and

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most, if not all, pyrite grains are internally dominated by dislocation-wall formation and thus, crystal-plastic deformation (Fig. 8).

Similarly, naturally deformed pyrite-rich ores that have experienced metamorphism and/or deformation are often characterised by brittle textures of deformation (Marshall and Gilligan, 1987; Cook, 1993; Cook et al., 1993; Craig and Vokes, 1993; Cook, 1995; Craig, 2001; Kuscu and Erler, 2002). Most of these studies consider such textures as evidence that pyrite has behaved in a brittle manner at peak metamorphic conditions. However, these assumptions, based primarily upon reflected light analysis and BSE imaging, can be misleading as the techniques reveal nothing about internal distortion in pyrite grains. Instead, the eye is drawn to the most prevalent visible deformation texture, which did not necessarily operate at peak conditions.

Pyrite from the Bleikvassli (Cook, 1993) and Rødhammeren (Nilsen, 1971) sulphide ore deposits, in the Scandinavian Caledonides, are characterised by cataclasis and 'pulverisation'. However, when investigated by OC imaging and EBSD, regardless of the prevalence of brittle textures, the pyrite preserves evidence for widespread internal crystal-plastic distortion (Barrie et al., 2010a). These results, coupled with those from similar studies (Boyle et al., 1998; Freitag et al., 2004; Barrie et al., 2009; Barrie et al., 2010b), imply brittle textures in naturally deformed pyrite are generally not indicative of peak metamorphism, but most likely represent textures formed during later overprinting retrograde conditions of metamorphism.

Recent study of low temperature deformed ore deposits (Barrie et al., 2009) indicate that crystal-plastic deformation in naturally deformed pyrite ($10^{-12} - 10^{-16}$ s^{-1}) potentially occurs at temperatures as low as ~200-260 °C, far

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lower than suggested by the current pyrite deformation mechanism map (Fig. 1) (McClay and Ellis, 1983).

4.2. Recovery and Recrystallisation Processes

Plastic mechanisms of deformation are often associated with recovery and/or recrystallisation processes (McClay and Ellis, 1983; Urai et al., 1986; Leiss and Barber, 1999). These processes either encourage ordering of dislocations within grains to form low-angle (~2º) dislocation walls/sub-grain boundaries (recovery) or remove dislocations from grains (recrystallisation). Textural evidence for recrystallisation within naturally deformed pyrite is uncommon (Barrie et al., 2010a; Barrie et al., 2010b), potentially due to a combination of 'softer' matrix sulphide effects favouring grain boundary sliding (GBS) processes over recrystallisation, slow strain rates (10^{-12} – 10^{-16} s^{-1}) and 'relatively' low temperatures of deformation.

In contrast to naturally deformed samples, the majority of experimental runs discussed in this study and by Barrie et al. (2007) preserve evidence for widespread recrystallisation (Fig. 4). The runs deformed at a strain rate of 2 x 10^{-4} s^{-1} and 550-700 ºC (Fig. 2B) preserve evidence for recrystallisation processes that systematically alter from bulging (BLG) recrystallisation to sub-grain rotation (SGR) and grain boundary migration (GBM) with increasing temperature (Hirth and Tullis, 1992; Barrie et al., 2007). The experimental samples described in this study cover a greater range of temperature and strain rate conditions than Barrie et al. (2007), thus allowing further assessment of recrystallisation processes at fast strain rates (Table 1; Fig 2). The lowest temperature run, 032 (450 ºC), contains no evidence for formation

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of new, recrystallised grains, and retains pyrite grain size and shape, outside shear zones (Fig 5), similar to the starting material (B-1) (Fig. 4). However, the 470 ºC and 500 ºC runs (Fig. 4) preserve microstructures similar to those in samples deformed at 550 ºC in Barrie et al. (2007), with suites of recrystallised, anhedral pyrite grains (<5μm) decorating the boundaries of remnant 'porphyroclastic' pyrite grains (Fig, 4). These textures, as well as apparent suturing along 'porphyroclastic' grain boundaries (Fig. 4B, C) suggest the onset of recrystallisation processes in pyrite, at fast strain rates (10^{-4} s^{-1}-10^{-5} s^{-1}), occurs at ~470-500 ºC and is initially dominated by BLG recrystallisation.

At a strain rate of 2 x 10^{-5} s^{-1} and temperatures between 550-600 ºC, the runs preserve a recrystallisation trend similar to that of Barrie et al., (2007), with BLG becoming replaced by SGR recrystallisation (Fig. 4). However, at 650 ºC and 2 x 10^{-5} s^{-1} (055) (Table 1, Fig. 4) GBM recrystallisation processes appear to dominate rather than SGR (Barrie et al., 2007). However, 055 not only experienced a different strain rate to the 650 ºC sample (059) discussed in Barrie et al, (2007) but also a much lower total strain ~15% compared to ~40% (Table 1; Fig. 2). This suggests relatively high deformation temperatures and low total strains potentially favour GBM over SGR recrystallisation. Despite these differences, the results imply recrystallisation processes are important at fast strain rates between ~450-700 ºC, and show a systematic trend with increasing temperature of recrystallisation altering from BLG to SGR and finally GBM, although the onset temperature of each process will be affected by other variables such as strain rate and percent shortening.

4.3. Recrystallisation and Crystal-Plastic Deformation

All of the experimental runs, with the exception of 032 (450 °C), contain evidence for crystal-plastic deformation by dislocation creep evident through the development of low-angle (~2°) sub-grain boundaries (Fig. 8) (Barrie et al., 2007). As temperature of experimental deformation increases from 450 °C to 700 °C, there is a systematic change in abundance and distribution of low-angle dislocation walls (Fig. 7, 8). The undeformed starting pyrite ore material (B-1) contains few low-angle dislocation walls and the low-angle (~2°) peak in the neighbour-pair (correlated) misorientation histogram is minimal (Fig. 7). The lowest temperature run, 032 (450 °C), also contains few low-angle (~2°) dislocation walls, but does show an increased low-angle, neighbour-pair peak compared to the starting material (Fig. 7).

Between 470 °C and 550 °C, low-angle (~2°) sub-grain boundaries increase systematically (Fig 7, 8). However, low-angle boundaries start to decrease in abundance at 650 °C and by 700 °C they are generally restricted to pyrite-pyrite grain boundaries and are minimal (Fig. 7, 8). This trend is mimicked by the graphs for mean cumulative lattice misorientation (distortion) within the experimental runs (Table 1; Fig. 9), with internal distortion increasing between 450 °C to 550 °C before decreasing at 600 °C (2 x 10^{-5} s^{-1}) and ultimately dropping to levels similar to the starting material at 700 °C (Fig. 9).

The wide distribution in some of the plots (Fig. 9C, D) reflects a lower mean misorientation within the 600-650 °C samples deformed at 2 x 10^{-5} s^{-1} compared with those deformed at 2 x 10^{-4} s^{-1} (Fig. 6, 9). This general trend

was reported by Barrie et al. (2007) and was attributed to changing recrystallisation and recovery processes with increasing temperature. A similar trend is preserved in the samples new to this study with reduction in low-angle dislocation walls at temperatures >600 ºC indicative of the removal of dislocations from pyrite grains by GBM at a pace faster than they can be generated. This removal process, with increasing temperature, also explains why low-angle boundary distribution is not uniform within individual pyrite grains but instead tend to concentrate at grain boundaries in the higher temperature samples (Fig. 8). In contrast the reduced dislocation-wall density at 450 ºC does not result from recovery processes, as with the other samples, but reflects sample failure during loading, prior to onset of significant steady-state flow and therefore, low-angle sub-grain boundary generation (Fig. 2).

FIG. 9 HERE

4.4. The pyrite deformation mechanism map

Recent pyrite studies (Freitag et al., 2004; Barrie et al., 2007; Barrie et al., 2009; Barrie et al., 2010a) have suggested a number of potential issues with the layout, construction and coverage of the strain rate contours and the deformation mechanism fields in the current proposed pyrite deformation mechanism map of McClay and Ellis (1983).

4.4.1. Problems with the current pyrite deformation mechanism map

The range of conditions at which crystal-plastic deformation operates in pyrite in the current deformation mechanism map (Fig. 1) was initially defined

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using the experimental results from Cox et al (1981). TEM analysis of these experimental runs suggested dislocation glide dominated at fast strain rates (10^{-4} s^{-1}-10^{-5} s^{-1}) between 450-600 ℃ but was replaced by dislocation creep at >650 ℃. However, the same runs investigated using EBSD in this study and Barrie et al. (2007), suggest dislocation creep is dominant between 500-700 ℃ rather than just above 650 ℃ (Fig. 7, 8, 9). EBSD studies of natural samples also indicate crystal-plastic deformation, specifically dislocation creep, is common in pyrite deformed at geological strain rates, potentially at temperatures as low as ~200-260 ℃ (Freitag et al., 2004; Barrie et al., 2009).

Dislocation glide and creep are both crystal-plastic mechanisms of deformation, whereby the lattice of minerals is deformed. Dislocation glide operates freely within deforming crystals when dislocations can glide along the lattice plane without becoming locked, due to 'obstacles' or 'opposite' dislocations along the glide plane (Cox, 1987; Barrie et al., 2010a). Where dislocation 'locking' occurs dislocation glide becomes either inhibited or impossible. In order for deformation to continue dislocation climb must occur, whereby the dislocation bypasses the blockage by moving into an adjacent lattice place and forming a new, low-angle (~2) dislocation wall or sub-grain boundary. The combination of dislocation glide and climb is what is referred to as the process of dislocation creep (Paschier and Trouw, 2008; Cox, 1987)

All of the work in this study indicates crystal-plastic deformation in pyrite, principally via dislocation creep, is prevalent over a much wider range of temperature conditions than suggested by the existing mechanism map of McClay and Ellis (1983) (Fig. 1).

As well as this difference in the range of deformation fields, grain size analysis of the experimentally deformed samples reveal a mean of ~35μm (Fig. 3A). This is in contrast to the ~100μm proposed by McClay and Ellis (1983) for their map, at least partially based upon the same samples (Cox et al., 1981). Grain size is an important constraint on the range of conditions at which diffusion and dislocation creep mechanisms operate (Atkinson, 1977; Frost and Ashby, 1982; Cox, 1987). Diffusion creep in particular is grain size sensitive and smaller grain size promotes this mechanism. Therefore, larger grain sizes have a much more restricted range of conditions at which diffusive mechanisms operate, thereby, favouring and expanding the range of conditions for dislocation creep (Atkinson, 1977; Rybacki and Dresen, 2004). Pyrite samples with a mean grain size of ~35 μm (this study) should have a smaller dislocation creep field on a deformation mechanisms map than samples with 100 μm as in the current pyrite deformation mechanisms map of McClay and Ellis (1983).

Deformation mechanisms are described by flow laws, which are controlled by variables including: stress, activation energy, temperature and grain size (Atkinson, 1976; Cox, 1987). These flow laws affect the slope of strain rate contours in stress-temperature plots, for a defined grain size, varying from horizontal (dislocation glide), to inclined (dislocation creep) and almost vertical (diffusion creep) relative to the graph axes. In contrast, the strain rate contours proposed by McClay and Ellis (1983) (Fig. 1) have a variable slope not consistent within any of the defined deformation fields. This unusual, convex and occasionally irregular shape of the strain rate contours, at geological strain rates (Fig. 1) are, instead, governed by a pressure-

temperature-time pathway proposed from an earlier study (Rutter, 1976). However, while this P-T-t pathway could result in strain rate contours with the orientation suggested (McClay and Ellis, 1983) the specific effects are difficult, if not impossible, to constrain, especially based upon the available data. The slope of the strain rate contours in the diffusion-creep field of the map are also inclined at ~45⁰ similar to the slope for both dislocation fields (Fig. 1). Diffusion creep is generally less sensitive to temperature than dislocation creep so strain rate contour slope for diffusion creep should be closer to vertical (Atkinson, 1977; Frost and Ashby, 1982). Clearly, significant revision of this map is essential if it is to be used as a guide to pyrite deformation.

4.4.2. A revised pyrite deformation mechanism map

Using the results presented and discussed above, we have generated a revised pyrite deformation mechanism map (Fig. 10), rectifying the issues with the existing map (Fig. 1) of McClay and Ellis (1983). As with the previous map of McClay and Ellis (1983), this revised map is based primarily upon experimental data at geologically unreasonable fast strain rates (10^{-4} – 10^{-5} s^{-1}) and has required considerable extrapolation.

FIG. 10 HERE

The strain rate contours, although generated from limited experimental data, no longer have an irregular appearance (compare Fig. 10 and Fig. 1) and have been calculated using the dislocation creep flow law (Fig. 11). Generating strain rate contours for this revised map was, nevertheless,

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problematic, due to both scarcity of available experimental data (Cox et al., 1981) and variability in stresses defining steady-state flow in the experimental runs due to strain-softening (Fig. 2). This issue is insurmountable at the present time, as no new, successful, pyrite deformation experiments have been carried out and results published since the 1980s (Cox et al., 1981; Graf et al., 1981; Cox, 1987).

A tentative flow law is proposed which uses the available mechanical data, and this was used to draw the strain-rate contours for the revised deformation mechanism map. The deformation fields in the map, however, have been constrained using EBSD results from a spread of natural and experimentally deformed pyrite-rich samples allowing greater control of the conditions at which different deformation mechanisms operate, particularly dislocation creep.

FIG. 11 HERE

4.4.3. Strain rate contours

Strain-rate contours on a deformation mechanism map are drawn by calculating the strain-rate as a function of the two variables plotted on the ordinate and abscissa. To calculate these values and extrapolate beyond the experimental conditions, a flow law of the form:

$$\dot{\varepsilon} = A\sigma^n \exp\left(\frac{-Q}{RT}\right) \tag{1}$$

where $\dot{\varepsilon}$ is the strain-rate, A is the pre-exponent, σ is the stress, Q is the activation energy, R is the gas constant, T is the absolute temperature,

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must be fitted to the data. The stress exponent, n, is calculated by plotting steady state flow stress against the strain rate for constant temperature (Fig. 11A). However, many of the experiments considered here failed to achieve steady-state due to continued strain softening right up to sample failure. The most reliable data are from higher temperature experiments (650-700 °C) where temperature dependent recovery can keep up with deformation rate (Fig. 4, 8).

In spite of not being able to calculate a precise value for the stress exponent (due to lack of steady-state data), it is possible to constrain the range of possible values of this parameter by using maximum (peak) and minimum (yield) values of stresses for the different experiments. These values are not meaningful in terms of steady-state flow, but provide bounds on the flow stress. Rearranging the conventional form of the creep law (equation 1) gives a form where n is the gradient of a straight line when strain-rate is plotted against stress:

$$\log_{10} \dot{\varepsilon} = \log_{10} A + n \log_{10} \sigma - \left(\frac{Q}{2.3RT} \right) \tag{2}$$

The data have been plotted in order to calculate the maximum and minimum values of n. These bounds are found by using different combinations of the maximum and minimum values of stress at for the two different strain-rates such that:

$$n_{\max} = \frac{1}{\log_{10} \sigma_{10^{-4} \min} - \log_{10} \sigma_{10^{-5} \max}} = \frac{1}{\log_{10} \left(\dfrac{\sigma_{10^{-4} \min}}{\sigma_{10^{-5} \max}} \right)} \tag{3}$$

and

$$n_{max} = \frac{1}{\log_{10}\sigma_{10^{-4}\,max} - \log_{10}\sigma_{10^{-5}\,min}} = \frac{1}{\log_{10}\left(\dfrac{\sigma_{10^{-4}\,max}}{\sigma_{10^{-5}\,min}}\right)} \tag{4}$$

where $\sigma_{\dot\varepsilon\,max}$ is the maximum stress at a given strain-rate.

Once n is calculated, the other parameters may be found by calculating the strain-rates for σ=1 and plotting the recalculated data on an Arrhenius plot ($\dot\varepsilon$ against $\frac{1}{T}$). The intercept of a line fitted to these data gives the value of A, with the gradient equal to $\frac{-Q}{2.3R}$ (Fig. 11B).

Since the recalculation of strain-rates for σ=1 involves using n, the values of A and Q subsequently calculated are a function of n. The range of n values calculated leads to a range of corresponding A and Q values. The dependence of the A and Q values on n means that one cannot be varied independently of the others. The deviation from the usual method of determining the flow law parameters based upon steady-state stresses is due to the limited availability of experimental data. Whilst we realise that the resulting flow law is imprecise, this work does constrain the parameter space potentially occupied by the flow law parameters.

The n_{MIN} values for all of the stress-strain curves (550-700 °C) are between 1.6 and 4.27 while the n_{MAX} values are more variable between 9.45 and -111. The latter values however, are not unexpected as due to the nature of log curves all negative values will extend along a curve to infinity. The n value calculated for 700 °C (n=3.9) is likely to be robust because both of these experiments achieved steady state (Fig. 2). The same is potentially true of the experiments at 650 °C but these results give an n value of 12.7 which is

outside the range of commonly expected stress exponents (Walker et al. 1990) suggesting these results are not as robust as they first appear. Using the n_{max}, n_{min} and steady state flow value for 700°C n values of 3.9 and 9.45 have been used to construct sets of strain rate contours (Fig. 11C).

Since the 700°C experiments reach steady state, the n-value used for the contours in the revised deformation mechanism map for pyrite is 3.9 (Fig. 10). The experiments, which do not reach steady state, are strain weakening so it is likely that the flow stresses are towards the minimum end of the estimates. Therefore, the minimum stress measurements have been used to calculate Q and A. Strain-rate contours were plotted for the minimum values of n, and using the corresponding values of Q and A, determined using the minimum stress estimates. Also plotted were contours using the maximum values of n, Q and A to show the potential variability in the date.

The strain rate contours generated from maximum values suggest pyrite would need deformation stresses of ~1000MPa in order for crystal-plastic deformation to proceed at ~300 °C and a strain rate of ~10^{-12} s^{-1}. In contrast, the strain rate contours generated from the minimum values suggest a much weaker pyrite with deformation stresses of less than 100MPa required for crystal-plastic deformation at ~300 °C and a strain rate of ~10^{-12} s^{-1} (Fig. 11C). The flow law and associated strain-rate contours allow extrapolation of the experimental data outside the range of conditions explored. Any extrapolations should be treated with caution due to the imprecise nature of the flow law. However, these results have an advantage over many other minerals because the deformation experiments were conducted at geologically reasonable temperatures for crustal deformation.

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The generic revised deformation mechanism map for pyrite presented in this study (Fig. 10), uses the minimum n value, and Q and A values calculated using the minimum stress estimates for construction of strain rate contours as these are thought to give the best estimation of the flow law for pyrite.

4.4.4. Deformation Fields

At temperatures between 500 $^\circ$C and 700 $^\circ$C and strain rates of 10^{-4} s^{-1} and 10^{-5} s^{-1} the stresses for the strain rate contours in the revised deformation mechanism map (Fig. 10) approximately correlate with those measured from the experimental stress-strain curves (Fig. 2). However, at temperatures between 450-470 $^\circ$C the stress-strain curves for runs 032 and 049 (Fig 2), record significantly lower stresses than those proposed from the revised strain rate contours (Fig. 10). This difference implies that either the slope of the strain rate contours changes at ~450 $^\circ$C, becoming virtually horizontal, indicative of a change in flow regime (Atkinson, 1976, 1977) or that the samples have experienced cataclastic failure prior to onset of steady-state flow. Cataclastic behaviour is evident in run 032 at 450 $^\circ$C (Fig. 3) and also present, but less apparent, in run 049 at 470 $^\circ$C. Therefore, while it is feasible that these samples represent a change from dislocation creep to glide mechanisms (Atkinson, 1976, 1977), the obvious failure of 032 (Fig. 5A, B) implies this zone represents the brittle-plastic transition zone at fast strain rates. The difference in stresses between the map (Fig. 10) and the stress-strain curves (Fig. 2) can, thus, be explained by early cataclastic failure, rather than achieving dominant steady-state flow.

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All of the samples analysed in this study and Barrie et al. (2007), at fast strain rates, preserve low-angle (~2º) dislocation walls at temperatures in the range 500-700 ºC, indicating these conditions are within the dislocation-creep field. Results from the naturally deformed samples (~30-80 μm grain size) also indicate dislocation creep is common at temperatures between ~260-540 ºC and geological strain rates (~10^{-8} s^{-1} to ~10^{-14} s^{-1}). These natural temperature conditions were previously considered to be dominated by diffusion creep mechanisms with crystal-plastic deformation at geological strain rates considered to be absent (Fig. 1). These results also suggest the brittle/diffusive-plastic transition zone at geological strain rates may be at temperatures as low as ~200-260 ºC. We therefore propose a wider range of temperature and strain rate conditions for the operation of dislocation creep in polycrystalline pyrite with a grain size of ~35μm (Fig. 10).

Diffusion creep was not recognised as a significant deformation mechanism in any of the EBSD-based studies of deformed pyrite (natural or experimental). However, identifying diffusion creep, as having been active during deformation, is notoriously difficult and problematic to assess. This difficulty arises from the fact that diffusion creep is only commonly reported as the principal deformation mechanism in minerals based upon lack of evidence for other deformation processes having operated. Similarly EBSD techniques themselves are inherently biased towards processes, such as dislocation creep, which result in crystal-lattice distortion. This may suggest, that the techniques utilised in this study, overemphasises the role of dislocation creep to the potential detriment of diffusive mechanisms. However, this study does not suggest that diffusion creep is not operating, simply that based upon the

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abundant evidence for crystal-plastic distortion, at all of the conditions investigated in this study, diffusion creep is subordinate to dislocation creep. Diffusion creep is however, potentially the dominant mechanism at low stress conditions beyond those investigated for pyrite deformation in this study (Fig. 10).

We therefore, propose a reduced range of conditions for diffusion creep than McClay and Ellis (1983), although the transitional boundary between diffusive and plastic mechanisms of deformation is at best highly speculative (Fig. 10). As diffusion creep is principally grain size rather than temperature dependent the slope of the strain rate contours within this field changes from inclined (dislocation creep) to nearly vertical (diffusion creep). The field defined as pressure solution in the previous deformation mechanism map (Fig. 1) has been removed and this region is simply considered to represent diffusive mechanisms due to the complexities of splitting this field when grain boundary fluid effects are considered (Atkinson, 1976; Siemes, 1991).

5. Conclusions

Detecting crystal-plastic deformation in pyrite and other cubic and opaque minerals is problematic, particularly using traditional techniques such as reflected light microscopy and SEM-based BSE imaging. These technqiues make it uncertain as to whether there really is no evidence for crystal-plastic deformation or if it has simply been missed due to the observational technique used, particularly where cataclastic textures are widespread. Using OC imaging and EBSD techniques on natural and experimental samples

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demonstrates that plastic deformation is much more widespread than at first apparent at both fast experimental (10^{-4} to 10^{-5} s^{-1}) and slower geological strain rates (10^{-8} to 10^{-14} s^{-1}).

Recovery and recrystallisation, while apparently rare in naturally deformed pyrite, is an important process in experimentally deformed pyrite at strain rates of 10^{-4} s^{-1} to 10^{-5} s^{-1} and temperatures >470 ºC. The experimental runs show a systematic trend in recrystallisation with increasing temperature from bulging (500-600 ºC) to sub-grain rotation (600-650 ºC) and finally grain boundary migration (700 ºC). The changing recrystallisation mechanisms become much more effective with increasing temperature, such that dislocation is virtually undetectable at >650 °C.

Studies of experimentally and naturally deformed pyrite samples across a range of P-T, strain rate and stress conditions indicate crystal-plastic deformation, via dislocation creep, is relatively common. These results combined with all of the available experimental stress-strain curves have been used to construct a revised deformation-mechanism map for polycrystalline pyrite with a grain size of ~35µm. The most notable differences of this map over the existing one are the wider range of conditions at which dislocation creep operates and the change in strain rate contour slopes, which now fit flow-law calculations.

Although the revised map (Fig. 10) better represents existing data, it should still be considered a guide to deformation in pyrite, just as McClay and Ellis (1983) indicated for their map. The reasons are the limited data available for its construction, the errors inherent with determination of the flow law, and the need for extrapolation from fast experimental strain rates to geologically

appropriate strain rates. Other factors, including: grain size distribution, recrystallisation processes, matrix composition, modal abundance, stress differences and strain heterogeneity can also affect pyrite behaviour and should always be considered in any study.

Figure Captions

Table 1 Summary table of the deformation conditions and strain experienced by all of the experimental runs investigated in this study and Barrie et al. (2007). All of the experimental data was gathered by S.F.Cox and is either initially presented in Cox et al. (1981) or to date unpublished.

Fig. 1 The deformation mechanism map for polycrystalline pyrite with a grain size of ~100 μm published by McClay and Ellis (1983). Strain rate contours are in 10^{-n} s^{-1} and were defined using the results from Cox et al. (1981).

Fig. 2 Stress-strain curves (Cox et al., 1981) for the experimentally deformed pyrite samples with a confining pressure of 300 MPa and a strain rate of 2 x

10^{-5} s^{-1} (**A**) and 2 x 10^{-4} s^{-1} (**B**). Run 026 (**A**) was deformed at a strain rate of 1.2 x 10^{-1} s^{-1}. Curves ending in a downward-pointing arrow terminated with specimen failure.

Fig. 3 A Box plots of grain size for the new experimental samples discussed in this study (all grains smaller than 20 μm removed): boxes represent 50% of the total data 'the body', limits of which are the interquartile range, the solid line within boxes is the median value. The 'whiskers' represent the extremes of the data (3/2s of the interquartile range). **B** Crystal size distribution (CSD) plot showing the population density versus grain size (>20 μm) for all the low temperature experimental samples. **C** Crystal size distribution (CSD) plot showing the population density versus grain size for pyrite grains between 0.25 μm and 15 μm in size for the low temperature experimental samples.

Fig. 4 High spatial resolution band contrast maps of a selection of samples, deformed at temperatures between 470-700 ºC with a strain rate of 2 x 10^{-5} s^{-1}, indicating textural changes.

Fig. 5 A/B Orientation contrast images of run 032 (450 ºC) highlighting the conjugate shear fractures running through the sample. **C**. Band contrast image highlighting the variable grain size and cataclastic nature of pyrite grains within the shear zones. **D**. Band contrast image of pyrite grains outside the conjugate shear zones highlighting their equant nature.

Fig. 6 Cumulative percentage histograms plotting lattice misorientation within individual grains against total percentage of grains within each sample. **A.** $2 \times 10^{-4} \ s^{-1}$ **B.** $2 \times 10^{-5} \ s^{-1}$

Fig. 7 Misorientation angle distribution histograms plotting neighbour-pair (correlated) and random-pair (uncorrelated) misorientations for a systematic suite of the samples deformed at a strain rate of $2 \times 10^{-5} \ s^{-1}$ and temperatures between 450 °C and 700 °C.

Fig. 8 Orientation contrast images (**A, C, E, G**) and band contrast maps with overlain low-angle (~2°) sub-grain (dislocation wall) boundaries (**B, D, F, H**) for the experimentally deformed pyrite samples at a strain rate of $2 \times 10^{-5} \ s^{-1}$ and temperatures between 550-700 °C. Pole figures are included for each map (*i, ii, iii, iv*) with lattice rotation axes (a, b) and rotation directions highlighted (arrows).

Fig. 9 Cumulative percentage histograms plotting total percentage of grains against mean lattice misorientation for all of the experimentally deformed samples discussed in this study. Each plot represents a particular temperature regime of samples **A.** 450-500 °C **B.** 550 °C **C.** 600 °C **D.** 650 °C **E.** 700 °C

Fig. 10 The revised deformation mechanism map for polycrystalline pyrite with a mean grain size of ~35 μm, constructed using EBSD data from this and previous studies (Barrie et al., 2007; Barrie et al., 2009; Barrie et al., 2010a).

The contours on the map are strain rate in units of 10^{-n} s^{-1} and were calculated using the original experimental stress-strain curves, some of which were presented in Cox et al. (1981).

Fig. 11 A. Stress-strain curves for four of the experimental samples, indicating how the maximum values for steady-state flow were measured. **B.** Graph plotting strain rate at 0 log stress against 1/Temperature (in degrees Kelvin) for all of the samples. The slopes of the best fit trend lines and the intercept of both the minimum and maximum end-members have been used to calculate Q (activation energy) and A (flow law constant). **C.** Strain rate contours plotted using the maximum, minimum and median values of Q, A and n with a constant grain size of 35µm.

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Table 1

Sample No.	Temperature	Confining P.	Strain Rate	Strain (%)
Run 032	450°C	300	$2 \times 10^{-5} \text{ s}^{-1}$	4.0%
Run 049	470°C	300	$2 \times 10^{-4} \text{ s}^{-1}$	20.0%
Run 035	500°C	300	$2 \times 10^{-5} \text{ s}^{-1}$	17.0%
Run 048	550°C	300	$2 \times 10^{-4} \text{ s}^{-1}$	34.0%
Run 026	550°C	300	$1.2 \times 10^{-1} \text{ s}^{-1}$	35.6%
Run 037	550°C	300	$2 \times 10^{-5} \text{ s}^{-1}$	29.0%
Run 092	600°C	300	$2 \times 10^{-4} \text{ s}^{-1}$	22.0%
Run 063	600°C	300	$2 \times 10^{-4} \text{ s}^{-1}$	40.5%
Run 054	600°C	300	$2 \times 10^{-5} \text{ s}^{-1}$	32.0%
Run 059	650°C	300	$2 \times 10^{-4} \text{ s}^{-1}$	40.0%
Run 055	650°C	300	$2 \times 10^{-5} \text{ s}^{-1}$	15.0%
Run 053	700°C	300	$2 \times 10^{-4} \text{ s}^{-1}$	32.0%
Run 020	700°C	300	$2 \times 10^{-5} \text{ s}^{-1}$	22.5%

Table 1

Figure 1
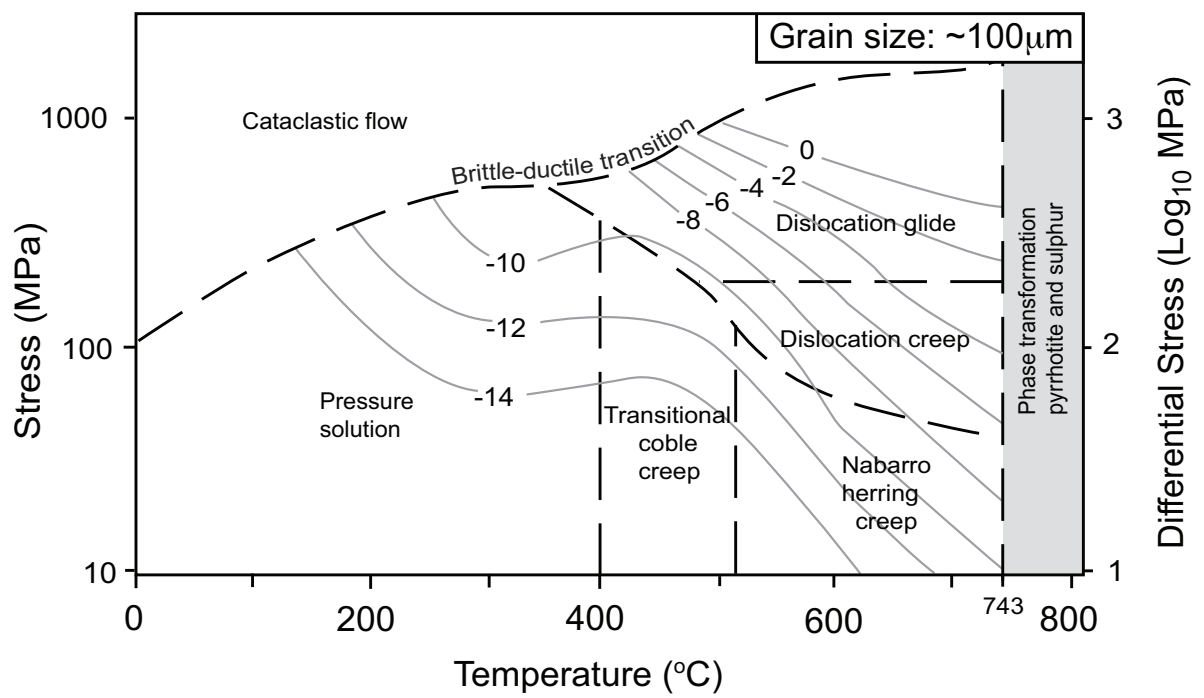
Fig. 1

Figure 2

Fig. 2

Figure 3
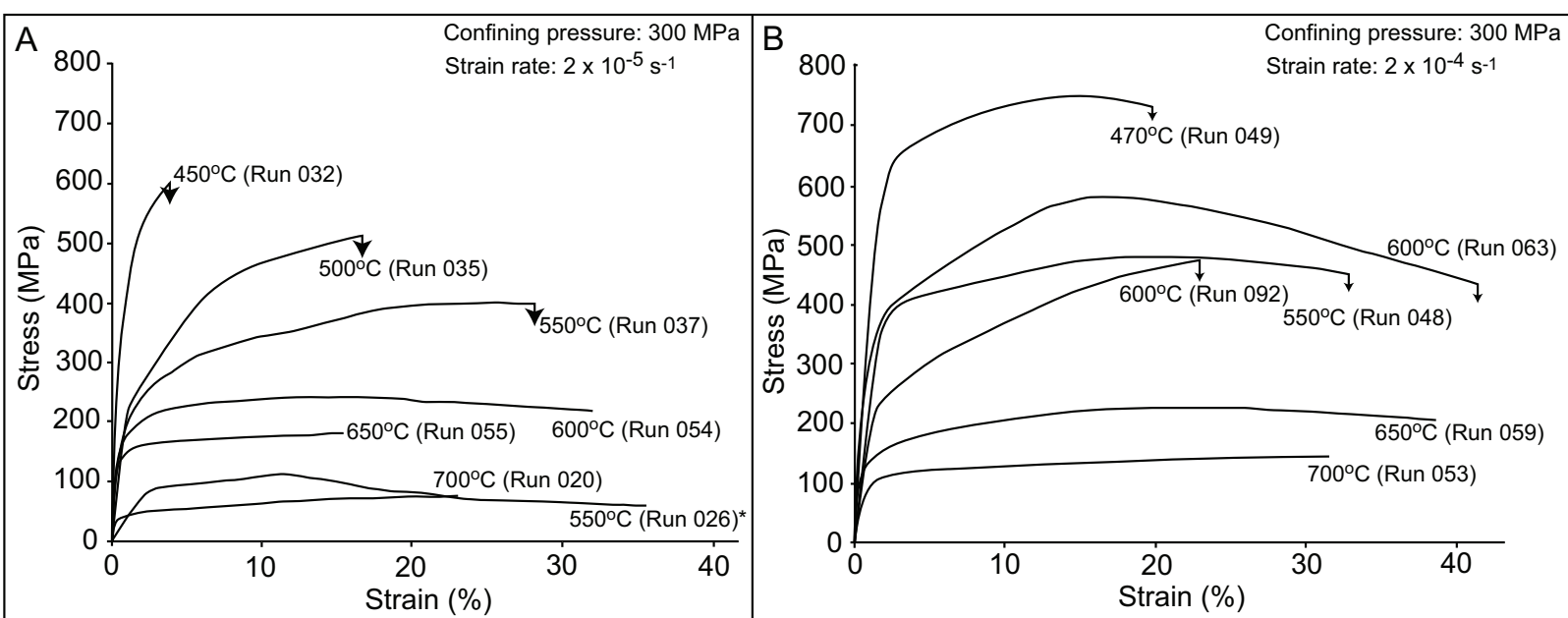
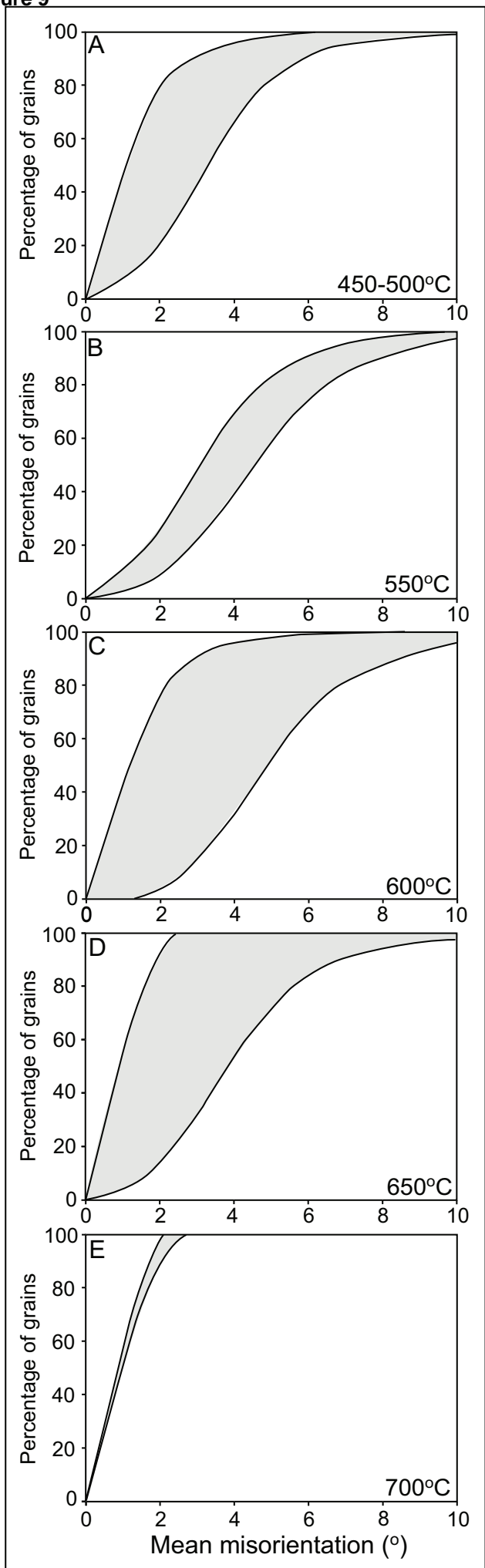| Sample	Temp.	Grain Size Plots	Mean	No.
Run 020	700°C		45μm	673
Run 053	700°C		38μm	4949
Run 055	650°C		32μm	728
Run 059	650°C		34μm	4783
Run 054	600°C		32μm	823
Run 063	600°C		30μm	563
Run 092	600°C		45μm	2096
Run 037	550°C		31μm	924
Run 026	550°C		36μm	648
Run 048	550°C		33μm	3134
Run 035	500°C		38μm	608
Run 049	470°C		37μm	627
Run 032	450°C		40μm	714
B-1	Undef.		38μm	2863
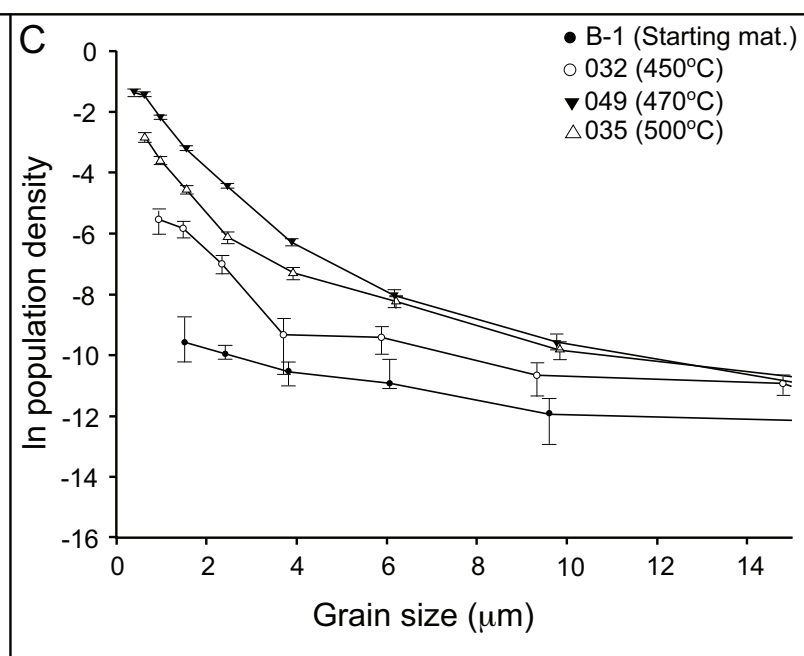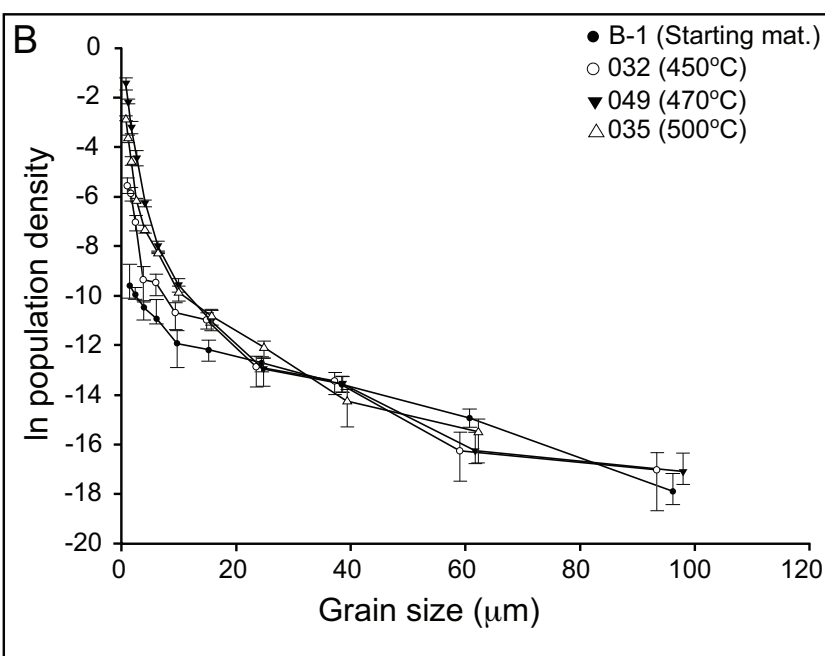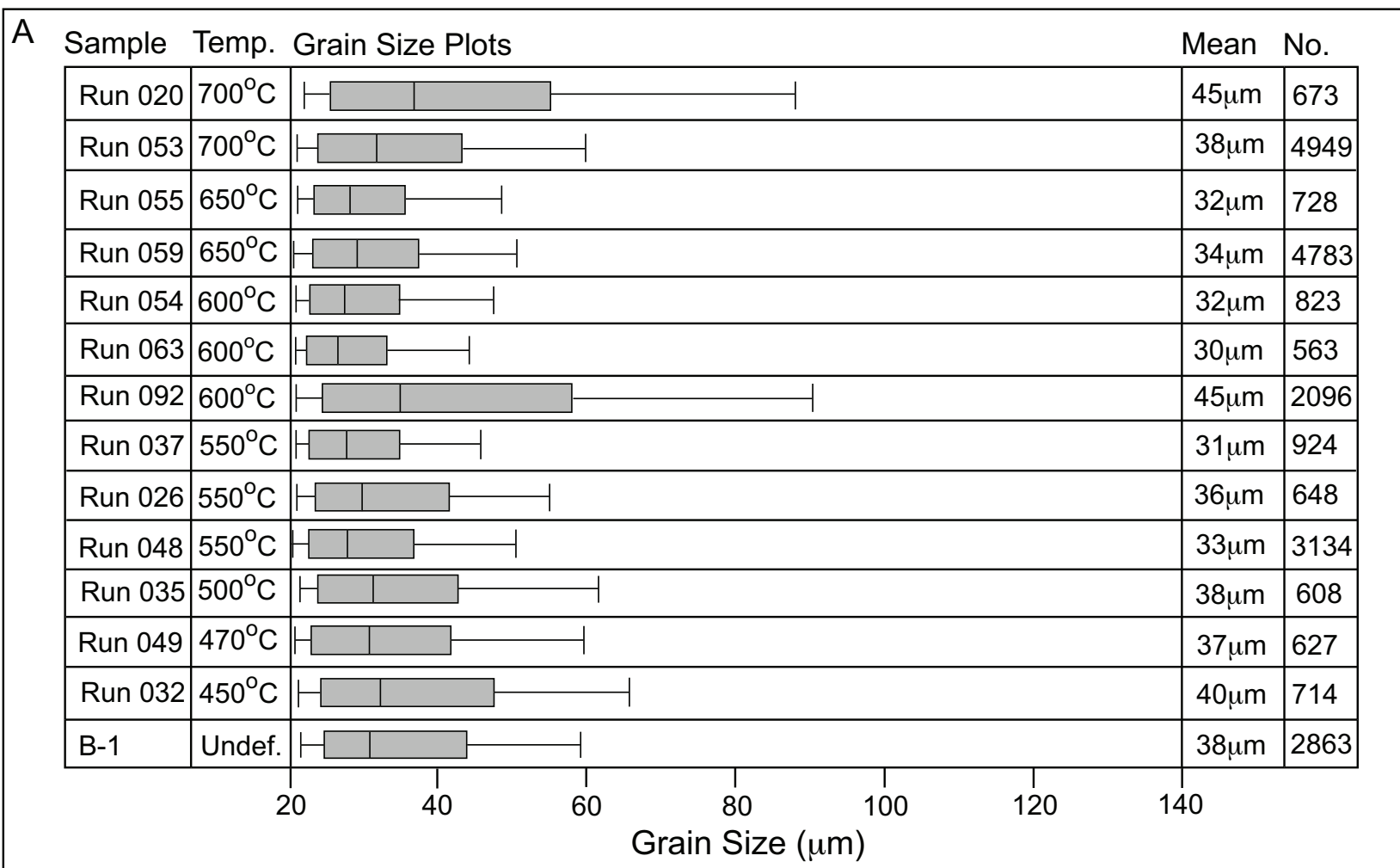
Fig. 3

Figure 4

Fig. 4

Figure 5

Fig. 5

A — 800µm — Orientation Contrast Image

B — 120µm — Orientation Contrast Image

C — 20µm — Band Contrast Image

D — 20µm — Band Contrast Image

Figure 6

A

Percentage of grains

2 x 10⁻⁴ s⁻¹; 300 MPa

B-1	
Run 049 (470)	
Run 048 (550)	
Run 092 (600)	
Run 059 (650)	
Run 053 (700)	

Mean misorientation (°)

B

Percentage of grains

2 x 10⁻⁵ s⁻¹; 300 MPa

B-1	
Run 032 (450)	
Run 035 (500)	
Run 037 (550)	
Run 054 (600)	
Run 055 (650)	
Run 020 (700)	

Mean misorientation (°)
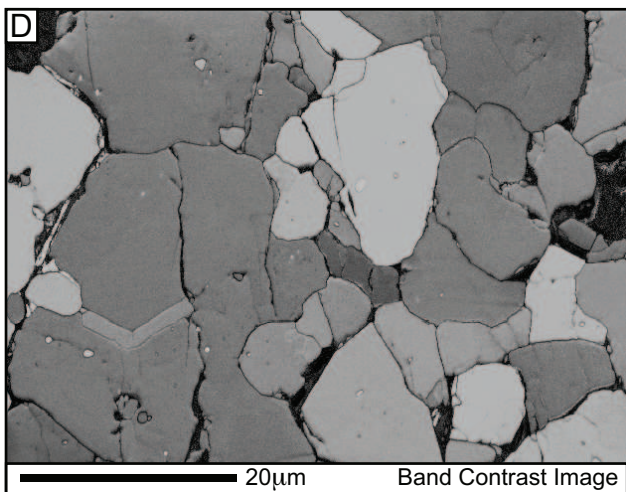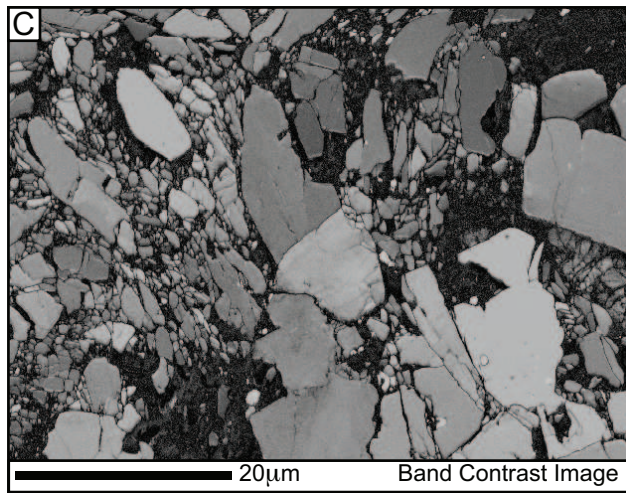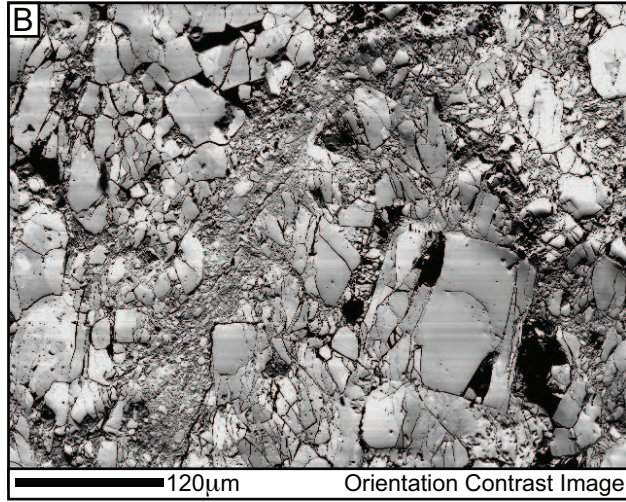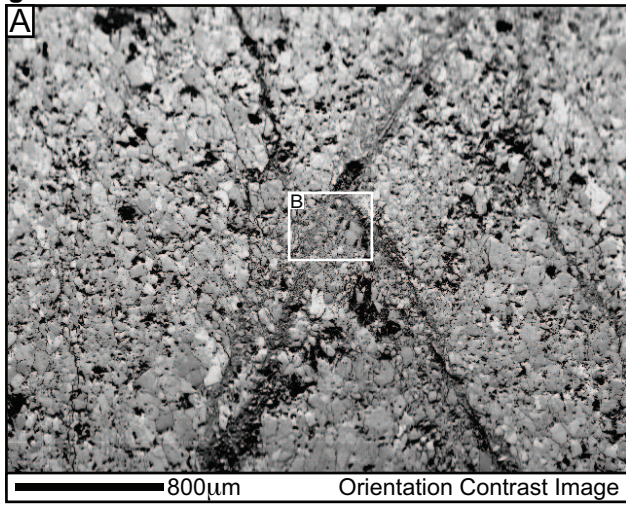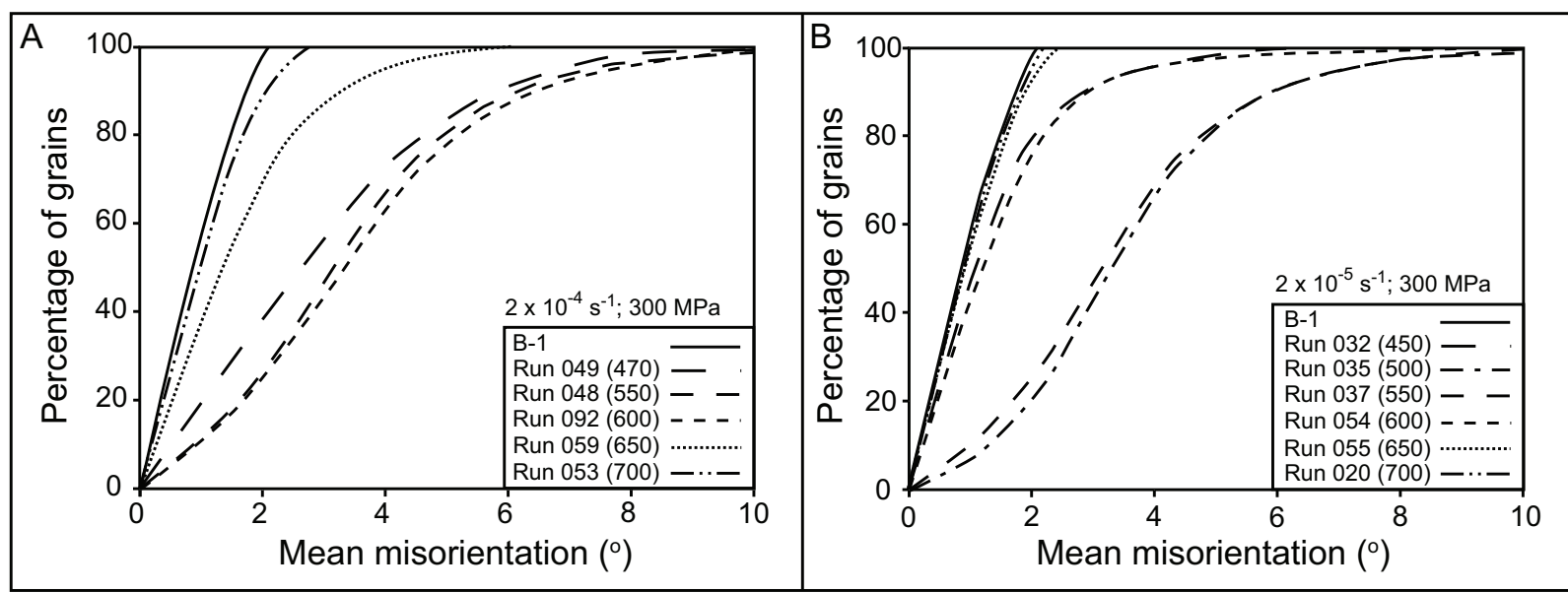
Fig. 6
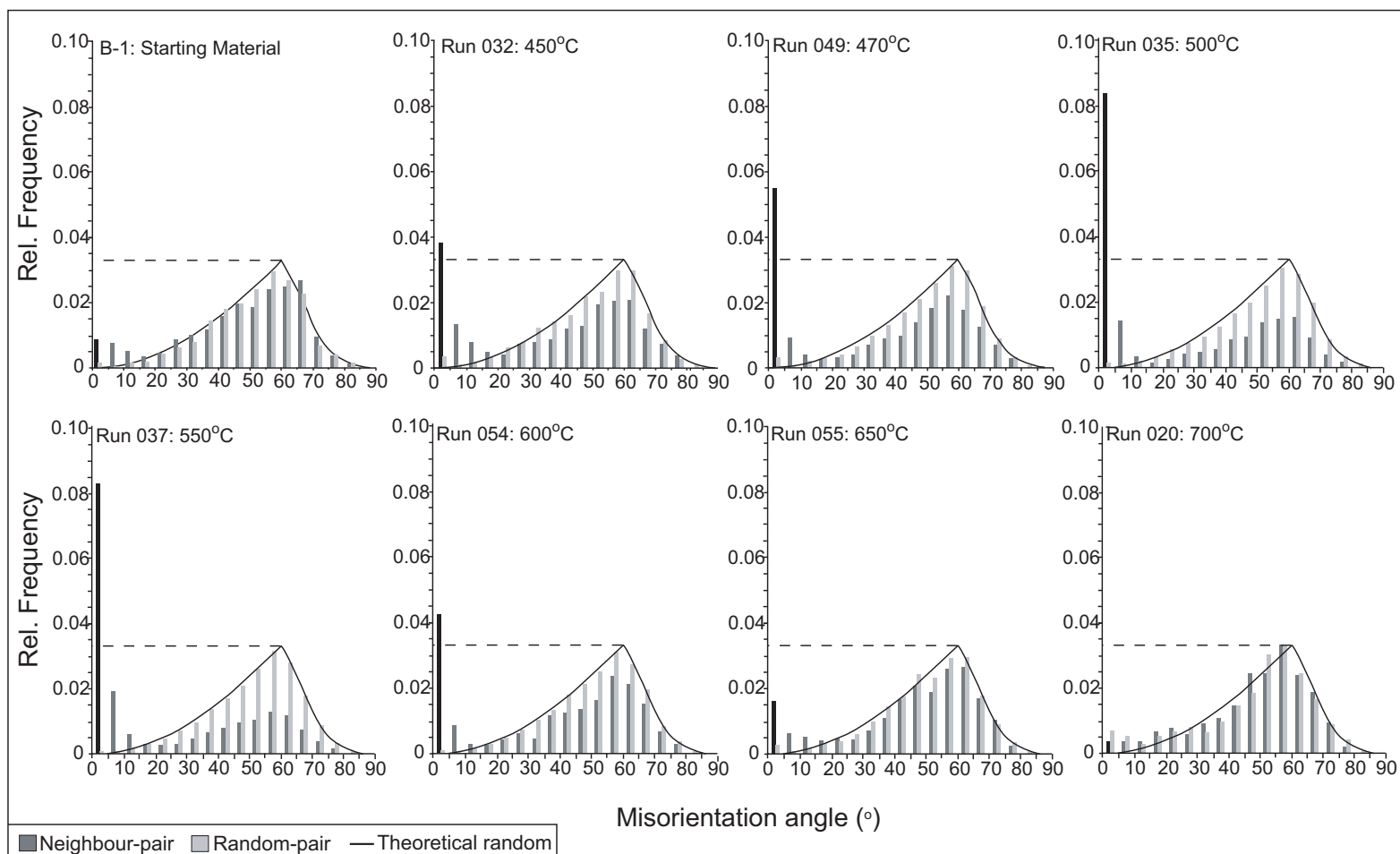
Figure 7

Fig. 7

Figure 8
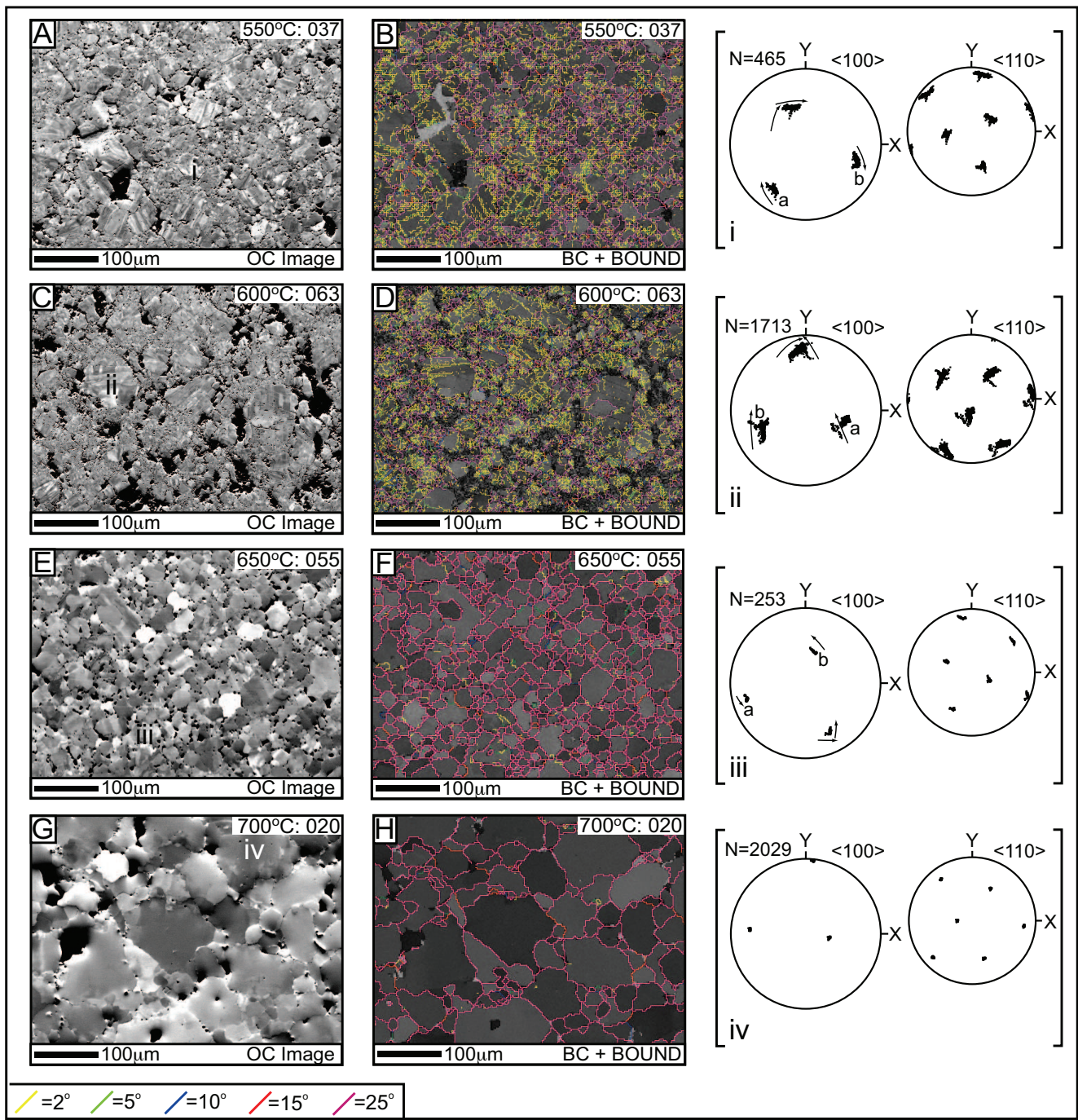
Fig. 8

Figure 9

Fig. 9

Figure 10

Fig. 10

Figure 11

Fig. 11