Analysis of dynamic recrystallization and nucleation in a quartzite mylonite

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Abstract

Electron backscatter diffraction (EBSD) has been used to analyse a natural quartz mylonite from the Stack of Glencoul, NW Scotland. This technique has been used to measure the misorientation between original protolith ‘parent’ grains and recrystallized ‘daughter’ grains. The angle of misorientation is important because it has implications for the controlling recrystallization mechanism. The sample exhibits approximately 65% a recrystallized microstructure. The average neighbor-daughter grain size is 12 μm and the average subgrain size of the parent grain is 16 μm. The parent grains do not show a systematic increase in misorientation from the centre of the grain to the edges. These data are inconsistent with the subgrain rotation recrystallization model. We propose that recrystallization was facilitated by bulging during strain-induced grain boundary migration. However, the majority of misorientations between the parents and their neighbouring daughter grains are in the range 10°–30° requiring that another process has operated to rotate grains to higher misorientations with respect to their parents. This process is likely to be grain boundary sliding.

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1. Introduction

Dynamically recrystallized rocks are of interest to geologists (Bell and Etheridge, 1976; Poirier and Guillopé, 1979; Urai et al., 1986; Lloyd and Freeman, 1994) as these microstructures are used to interpret the processes of deformation, the conditions of deformation and the kinematic strain path. Dynamic recrystallization is defined as being synchronous with deformation (Hobbs, 1968; Hobbs et al., 1982; Urai et al., 1986) and is important because creep deformation of rocks to high strains is facilitated by the processes of recovery and recrystallization (Urai et al., 1986; Passchier and Trouw, 1996). Without recovery and/or recrystallization, steady-state flow by dislocation movement would be impossible, as the dislocations would interfere with each other causing work hardening and embrittlement.

Recrystallization involves the formation/nucleation and growth of new grains. The individual mechanisms by which recrystallized grains nucleate and grow, and other processes that might operate in tandem, such as grain boundary sliding, have specific, predictable effects on the crystallographic relationships between host and recrystallized grains (Hobbs, 1968; Wheeler et al., 2001) or ‘parent’ and ‘daughter’ grains.
Partially and completely recrystallized microstructures can be distinguished. In partially recrystallized microstructures, a bimodal grain size distribution is characteristic, with aggregates of small grains of nearly uniform size between large grains exhibiting internal deformation features such as undulose extinction and subgrain boundaries. Fully recrystallized microstructures are similar to an equilibrium non-recrystallized microstructure or foam texture, exhibiting a uniform grain size and straight non-serrated grain boundaries.

There are several recrystallization mechanisms discussed in the literature (e.g., Urai et al., 1986). Mechanisms of specific interest are subgrain rotation (SGR) recrystallization and strain-induced grain boundary migration (SIGBM) recrystallization, both of which are recognised in quartz (White, 1982). Nucleation is the formation of a new strain-free volume of crystalline material surrounded by high-angle grain boundaries (HAGB). Low-angle grain boundaries (LAGB) often correspond to subgrain boundaries/walls, defined by an array of dislocations. In contrast, HAGBs do not generally have determinable structures. The minimum misorientation angle by which a HAGB is defined is mineral dependent; in this paper, we use 10°, a value determined from transmission electron microscopy research on the structure of boundaries in quartz by White (1977). A subgrain is a volume of crystalline material where at least a portion of the boundary is an LAGB whereas a grain is a volume of crystalline material completely surrounded by HAGBs. We now summarise separate mechanisms, which contribute to nucleation, recrystallization and consequent modifications of microstructures.

1.1. Subgrain rotation (SGR) recrystallization

SGR is an extension of the recovery process: the driving force for SGR is a reduction of the internal strain energy of the deforming aggregate. This is achieved by the movement and interaction of dislocations to form a subgrain wall (White, 1976; Urai et al., 1986). As more dislocations enter the subgrain wall, there is a progressive increase in misorientation between subgrains (Fig. 1a). This process is enhanced when dislocations are able to climb. The misorientation angle will continue to increase until the subgrain boundary becomes a HAGB and the

Fig. 1. Basic processes that can be involved in the development of recrystallized grains. (a) Progressive subgrain rotation leading to the development of high-angle grain boundaries and the formation of a new grain. (b) Bulge nucleation due to grain boundary migration. Bulge separated from parent grain due to the formation of a bridging subgrain wall.

Fig. 2. Terminology diagram. (a) Original protolith grains are termed parents. (b) Recrystallized grains that are in contact with a parent grain are termed neighbour–daughters. (c) All other recrystallized grains, which have come from the parent grains, are termed daughters. (d) When the data from only 1 μm either side of the parent to neighbour–daughters HAGB are studied, these are termed edges.
subgrain can no longer be classified as part of the original parent grain; this new daughter grain is often thought of as a nucleus and the process by which it is formed is known as SGR recrystallization (Poirier and Guillopé, 1979). As the recrystallized daughter grains which are formed are rotated portions of the original parent grain, they may exhibit an internal defect structure inherited from the parent grain (Drury and Urai, 1990). The primary line of evidence for SGR recrystallization is core and mantle structure, where the cores of original protolith grains pass out transitionally into the mantle with increasing subgrain development and misorientation. Around the core and mantle structure are aggregates of recrystallized grains with similar size and orientations to the nearby subgrains (Gifkins, 1976; White, 1976). Daughter grains developed by SGR recrystallization should be related to their parents by an orientation relationship that will reflect the dislocations involved in deformation, recovery and SGR (Lloyd et al., 1997; Prior et al., 2002).

1.2. Strain-induced grain boundary migration (SIGBM) recrystallization

SIGBM recrystallization process is driven by the reduction of the stored strain energy (the energy of dislocations, point defects, subgrain and grain boundaries) across an existing HAGB; it occurs principally when two neighbouring grains having significantly different dislocation densities (Fig. 1b). SIGBM rarely occurs by movement of all points along a boundary with equal velocity; hence, the microstructural signature of grain boundary migration is highly serrated grain boundaries (White, 1982).

1.3. Bulge nucleation

Grain boundary bulges form along moving boundaries during SIGBM, developing from HAGBs preferentially in areas of sharpest crystal bending (White, 1977) or where neighbouring grain boundary segments are pinned (Jessell, 1987). Full isolation of the bulge can be achieved by the development of a bridging subgrain boundary across the neck of the bulge and its conversion by progressive misorientation into a HAGB (Fig. 1b) (Urai et al., 1986). These bulges give rise to new strain-free grains (Bell and Etheridge, 1976). Bulge nucleated grains can be recognised by their polygonal structure and lack of internal deformation. The recrystallized grains will be related in orientation to the protolith parent grain, the angle depending on the magnitude and axis of rotation of the SGR of the bridging boundary.

1.4. Grain boundary sliding

The driving force for GBS is strain incompatibility. GBS is the movement of grains relative to one another. GBS accommodated by frictional processes (cataclasis, particulate flow) is common and requires dilatancy. Non-dilatant GBS is limited to fine-grained rocks and/or relatively high temperatures and is accompanied by a rate controlling accommodating process which can be diffusion, a solution or dislocation process or a combination of these (Evans and Langdon, 1976; Gifkins, 1976). The presence of voids or bubbles along a grain boundary will facilitate GBS (White, 1977). Evidence indicating that
GBS has become the dominant deformation mechanism includes polygonal grains, smooth grain boundaries, voids along grain boundaries especially located at triple junctions and alignment of groups of recrystallized grains to form planes of grain boundaries parallel to the mylonitic foliation (White, 1979). GBS is hypothesised to weaken or randomise crystallographic preferred orientations (CPOs), even when accompanied by continued dislocation creep (Zhang et al., 1994; Bestmann and Prior, 2003; Storey and Prior, 2005). It may also randomise grain boundary misorientation axes (Jiang et al., 2000).

1.5. Aim of the research

Our aim is to understand the mechanisms of nucleation and recrystallization. Key information is contained in the misorientation between parent and daughter grains. The misorientation relationships can be compared with the predicted effects of common recrystallization and nucleation mechanisms to determine the controlling mechanism(s). In this paper, we define a parent grain as an original protolith grain, which may have been deformed but is still recognisable. A daughter grain is any recrystallized grain that is deduced to have formed from a parent grain. Neighbour–daughter grains (Fig. 2) are those daughters which share a grain boundary with a parent grain (although not necessarily the parent grain from which they were formed).

We use electron backscatter diffraction (EBSD) (Venables and Harland, 1973; Dingley, 1984) to measure the full crystallographic orientations of individual areas.
<1 μm in diameter (Prior et al., 1999). Automated EBSD data collection enables construction of quantitative microstructural maps (Adams et al., 1993; Prior et al., 2002) from which misorientations within grains and between neighbouring grains can then be established. Using these data it is possible to analyse the evidence of recrystallization and to reconstruct the deformation and recrystallization processes.

Fig. 5. Equal area, lower hemisphere, looking NNE, stereonet of the (a) parent grains only, (b) neighbour–daughter grains only, and (c) edges of the neighbour–daughter grains only (data within 1 μm of the HAGB between parent and neighbour–daughters). Between (a) and (b) is a skeletonized diagram of the [c]-axis plot of (a). The diagram also shows the kinematic framework where foliation is E–W perpendicular to the page (Z=pole to foliation), lineation (X) is horizontal within the foliation. The shear direction of the Moine Thrust is top to the west.
2. Sample

The sample used in this study is a natural mylonitic quartzite from the Stack of Glencoul (Grid Ref. NC 28882876), in the northern part of the Assynt region, NW Scotland (Fig. 3). Callaway (1884) was one of the first to describe these rocks which are now classed as S>L and L–S tectonites. The protolith for these rocks was a Cambrian quartzite, which was deformed along the Moine Thrust at greenschist facies condition (Law et al., 1986).

Fig. 6. (a) Band contrast or pattern quality map for parent grain 1 and surrounding daughter grains (location shown in Fig. 5) with the grain boundaries marked on. $2^\circ$=yellow, $5^\circ$=lime green, $10^\circ$=blue, $20^\circ$=purple and $>30^\circ$=black. (b) Texture component map where every grain whose c-axis was within $30^\circ$ of the centre of the upper cluster in Fig. 5a (parent grain cluster orientation 1) was coloured red, whereas every grain whose c-axes was within $30^\circ$ of the centre of the lower cluster in Fig. 5a (parent grain orientation 2) was coloured blue. Some grains remain uncoloured due to the fact that they are dispersed more than $30^\circ$ away from the centre of either cluster. (c–e) show that neighbour–daughters of both cluster orientations are located around a single parent grain and that there has been some mixing of the daughters.
The Moine Thrust is a foliation parallel ductile contact between the mylonitic Cambrian quartzite and the similarly deformed overlying Moine metasediments (Christie, 1960; Christie, 1963; Weathers et al., 1979; Law et al., 1986; Law, 1987; Strine and Wojtal, 2004). The sample was collected 5 m below the Moine Thrust from the white Cambrian quartz mylonites and exhibits a partially recrystallized microstructure. The sample is equivalent to sample SG10 of a previous study (Law et al., 1986) and was chosen due to its clear dynamically recrystallized microstructure which can be easily compared with other mylonitic samples. The sample contains approximately 1% muscovite.

3. Methods

3.1. Sample preparation

Standard XZ (parallel to lineation and perpendicular to foliation), 30 μm thin sections were chemically–mechanically polished using SYTON fluid (Lloyd, 1987) and carbon coated to prevent charging.

3.2. Data acquisition

Sample analysis areas were selected using optical and electron microscopy utilising orientation contrast (OC) imaging (Prior et al., 1996). The OC images show where crystallographic orientations change. Full crystallographic orientation data were obtained from automatically indexed EBSD patterns collected in a CamScan X500 Crystal Probe SEM fitted with a field emission gun and a FASTRACK stage. The EBSD patterns were collected using a 20 kV acceleration voltage and a beam current of 30 nA. The working distance was 25 mm with a column tilt of 70°. Samples were mapped by the beam moving on a grid with a fixed step size of 2 μm, ensuring that the recrystallized daughter grains and the subgrains contained ample measurement points. The EBSD patterns were imaged on a phosphor screen, viewed by a low-light CCD camera and indexed using the HKL Technology manufacturer’s software package Channel 5 (Schmidt and Olesen, 1989). Average measuring time per point was 0.2 s. The raw data have 62% indexed as quartz and 38% non-indexed (EBSD patterns with no solution). The non-indexed points correspond to grain boundaries and secondary phases. Grain sizes and subgrain sizes are calculated as the diameter of a circle of equivalent area to the measured grain/subgrain area. The data shall be analysed using optical images (Fig. 4), stereonets (Fig. 5), EBSD maps (Fig. 6a and b) and misorientation data (Figs. 7–9).
4. Results

4.1. Microstructure characteristics

Parent grains were identified via their large size, internal deformation, elongation and highly serrated grain boundaries (Fig. 4). The parent grains defined for analytical purposes do not include all the parent material in the field of view. The daughter grains were identified by their small size, lack of internal deformation and polygonal appearance. The parent grains show an average aspect ratio of 1:9, calculated from the EBSD maps and exhibit internal distortion and subgrain boundaries. The average subgrain size within the parent is 16 μm whereas the average neighbour–daughter grain size is 12 μm (Fig. 6a). The internal substructure of the parent grains shows LAGBs with misorientations greater than 2°. These boundaries are not regularly arranged; the amount of misorientation on individual subgrain boundaries does not increase systematically away from the core of the parent grain to the edge (Fig. 6a). Subgrain boundary traces are preferentially oriented subparallel to the grain long axes. The misorientation between the parent and daughter grains is between 10° and 30° (Figs. 4 and 8a).

4.2. Orientation data

Parent grain [c]-axes form two fairly tight clusters, one at the top of the stereonet and one at the bottom (Fig. 5a). Parent grains 2, 4, 5 and 6 cluster at the top of the stereonet and will be referred to as parent grain cluster orientation 1, whereas parent grains 1, 3, 4, 7, 8, 9, 10 and 11 cluster at the bottom of the pole figure and shall be referred to as parent grain cluster orientation 2 (grain numbers marked in Fig. 5). The daughter grain [c]-axes form the same two clusters but they are more dispersed than the parent grain clusters (Fig. 5b and c). Neighbour–daughter grains (Fig. 5c) show the same orientation distribution as all daughter grains; there is no gradient in orientation dispersion with distance away from the parent grains. The [c]-axis fabric skeleton exhibits an asymmetric single girdle (Fig. 5). A texture component map (Fig. 6b) enables us to assess the distribution of grains that fall into orientation clusters 1 and 2. Most neighbour–daughter grains fall in the same orientation cluster as their neighbouring parent. Similarly, most daughter grains fall in the same orientation cluster as neighbouring daughters. However, there are areas where parents have neighbour–daughter grains from both orientation clusters and there are areas of recrystallized grains where daughter grains from both orientation clusters are intermixed (Fig. 6c–e).

4.3. Misorientation data

Misorientation profiles show that there are significant distortions (>10°) across and along the length of parent grains (Fig. 7a), in addition to the LAGBs. Large steps in misorientation occur at the boundaries between parents and neighbour–daughters (Fig. 7b) and between daughters (Fig. 7c). Statistically, the misorientations between the parents and their neighbour–daughter grains peak between 10° and 30° (Figs. 6a and 8a). This is similar to the range of misorientations between daughter grains (Figs. 6a and 8b) except that the boundaries between daughters include many more boundaries with misorientations greater than 30° and there is a stronger Dauphiné twin
signature (misorientation peak at 60°). In contrast parent grains contain only LAGBs and Dauphiné twins (Fig. 8c). Misorientation axes were plotted in a crystal reference frame (i.e., an inverse pole figure). This reference frame is suitable for testing hypotheses about crystallographically controlled deformation processes (i.e., slip systems) (Lloyd et al., 1997) and other processes, such as GBS, that are not dependent upon crystallography. Misorientation axes of LAGBs cluster about the \[c\]-axis (Fig. 9a). LAGBs are located mostly within parent grains and data from only those parent grains shown in Fig. 4g show the same pattern as all LAGBs. HAGB misorientation axes have been subdivided into 10° intervals (Fig. 9b–f). Although all show misorientation axes preferentially lying parallel to \[c\], the degree of preference is variable; it is weakest in boundaries with misorientation between 10° and 20° (Fig. 9h).

5. Discussion

We suggest that the daughter grains whose cluster contains parent grain cluster orientation 1 have recrystallized from those parents and, similarly, daughter grains whose cluster contains parent grain cluster
orientation 2 have recrystallized from those parents. An observation that needs explaining is the 10–30° misorientations of neighbour–daughter grains from the parent grain orientation; that is, there has been some rotation of the neighbour–daughters. Also, around a single parent grain, representatives of both neighbour–daughter orientation clusters are seen. If the neighbour–daughters have recrystallized from both parent orientation clusters, as suggested, then there has been some mixing of the neighbour–daughter grain populations.

5.1. Subgrain rotation recrystallization

The presence of subgrain boundaries within parent grains, and the range of misorientations these show, suggest that recovery and SGR have accompanied dislocation creep. LAGB misorientation axes are preferentially orientated parallel to [c]; if we assume a tilt boundary geometry, this would be interpreted as the geometrical consequence of an \{m\}〈a〉 slip (prism slip) system (Lloyd et al., 1997), (Lloyd, 2004). However, this implied slip system is incompatible with the overall CPO (Fig. 5) which would usually be interpreted as having formed via dominant \{h\}〈a〉 slip (basal slip) (Law, 1987). We therefore propose that the subgrain walls have a twist component. Lloyd (2004) discusses how combinations of different slips systems can give rise to twist boundaries. We do not concur with all aspects of his treatment, but do agree that the misorientation axis for a twist boundary will be perpendicular to all the dislocation line vectors in that boundary. Therefore, two or more non-parallel populations of screw dislocations formed by \{c\}〈a〉 slip can form a twist boundary with misorientation axis parallel to c. This is our provisional proposal to reconcile the CPO data (Fig. 5) with the misorientation axis data (Fig. 9).

Although we suggest that recovery processes including SGR were operative we still need to assess what role these processes played in recrystallization. When SGR recrystallization is responsible for the nucleation, the recrystallized daughter grains should be initially of a similar size to the internal subgrain size of the parent grain (Urai et al., 1986). Our data show that the average internal subgrain size is 4/3 times that of the neighbour–daughters. Therefore, SGR alone cannot be the nucleation method unless there has been a phase of subgrain growth within the parents after the recrystallization and nucleation of the neighbour–daughter grains. This is unlikely as subgrain boundaries have a much lower mobility than HAGBs (Humphreys and Hatherley, 1996); thus, one would expect the daughter grains to grow faster under similar conditions. There is likely to be an increased driving force for recrystallization (higher strain energy) within parent grains relative to daughters. However, we would expect the driving force to be decreased by migration of the parent–daughter boundaries into the parent grain, not by migration of the less mobile subgrain walls within the parent grain.

5.2. Strain-induced grain boundary migration recrystallization and bulge nucleation

The evidence for SIGBM is the highly serrated grain boundaries exhibited by the parent grains. In the bulge nucleation mechanism, the size of the bulge is not controlled by the subgrain size, so the size difference of subgrains and daughter grains is not a problem. A feature of bulge nucleation in dynamic recrystallization is that, while several boundaries of a new grain may be established by grain boundary migration, full isolation of the grain may commonly be achieved by the development and SGR of a bridging subgrain boundary. The neighbour–daughter grains are misorientated between 10° and 30° from the parent grain, with decreasing crystallographic control, as this misorientation increases. It is likely therefore that another deformation mechanism has increased the misorientation. If no bridging subgrain boundary developed then bulge nuclei would have the same orientation as parents. Any subsequent rotation without crystallographic control would eventually randomise misorientation axes (Jiang et al., 2000). Preferred alignment of misorientation axes parallel to [c]-axis persisting for all parent to neighbour–daughter boundaries (Fig. 9) may be evidence that subgrain boundary bridging has occurred.

Initial bulging would have been of boundaries between original parent grains. After the formation of neighbour–daughter grains between the two parents, subsequent bulging will be between these daughter grains and the parents. One might argue that the degree of dispersion observed could be generated by the progressive misorientation of each new ‘layer.’ However, there are no gradients in orientation dispersion and layers just a single grain wide between two parents are as dispersed as thicker daughter grain bands.

5.3. Grain boundary sliding

The data show that there has been some mixing of the neighbour–daughter orientations so that both daughter orientations are now located around many individual parents. Moreover, the misorientation axes of 20–30° boundaries are less well aligned (parallel to [c]) than the axes of lower angle boundaries or higher angle...
boundaries (Fig. 9). The higher angle boundary data is distorted by the Dauphiné twin signature and is difficult to interpret. We interpret the weakening of the misorientation axis alignment as resulting from grain rotations during GBS. GBS can slide grains past each other and cause neighbour switching (White and Mawer, 1988), but GBS requires straight segments or boundaries for the sliding to take place (White, 1977). The parent grains show quite serrated grain boundaries, which are not favourable for GBS, but the boundaries between the recrystallized grains are short, fairly straight and, therefore, more favourable for GBS.

6. Summary and conclusions

The SEM/EBSD technique has been used to analyse the full crystallographic orientation of original quartz parent grains and recrystallized daughter grains in a mylonitic quartzite which was deformed at greenschist facies conditions. The parent grains have two orientation populations. Parent grains of orientation 1 have given rise to neighbour–daughter grains of orientation 1 and parent grains of orientation 2 having produced neighbour–daughter grains of orientation 2. Neighbour–daughter grains of both orientation 1 and 2 surround most of the parent grains. Most misorientation angles between parents and neighbour–daughter grains are between 10° and 30°. The parent grains show internal deformation in the form of undulose extinction and subgrain boundaries, which have misorientations axes parallel to [c]. Such subgrain walls can be formed by dislocations on (c)〈a〉 slip systems, but if this is the case, they must have some twist character. The subgrain boundary misorientations are greater than 2°. There is no systematic increase in misorientation from the centre of the parent grains to the edges. The average parent subgrain size is 16 μm, whereas the average daughter grain size is 12 μm. The subgrains are significantly larger than the neighbour–daughter grains. The neighbour–daughters show no internal deformation and are polygonal in shape. These data are summarised in a cartoon (Fig. 10). The conceptual model listed below shows one way in which the microstructure could have formed:

(1) Parent grains deform by dislocation creep accompanied by recovery and subgrain rotation.
(2) Nucleation of the neighbour–daughter grains takes place via bulging during strain-induced grain boundary migration. The sizes of daughter grains that are being formed via bulging are not controlled by the internal subgrain size of the parent and tend to be smaller than the subgrain size.
(3) Full isolation of the daughter grains is achieved by the development of a bridging subgrain boundary and its conversion by progressive misorientation into a grain boundary.
(4) The neighbour–daughter grains are then rotated further during grain boundary sliding to give the high observed misorientations between parents and daughters and the weakening of crystallographic control on misorientation axes. Recrystallized grains are able to slide past each other causing neighbour switching and, hence, mixing of orientation populations.

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