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Post-deformational annealing at the subgrain scale: Temperature dependent behaviour revealed by in-situ heating experiments on deformed single crystal halite

V.E. Borthwick*, S. Piazolo

Department of Geology and Geochemistry, Stockholm University, Svante Arrhenius väg 8C, Stockholm 10691, Sweden

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ABSTRACT

The dynamics of substructures, which encompass all structures present at the subgrain-scale, were investigated by static, in-situ annealing experiments. Deformed, single crystal halite was annealed inside a scanning electron microscope at temperatures between 280 and 470 °C. Electron backscatter diffraction maps provided detailed information about crystallographic orientation changes. Three temperature dependent regimes were distinguished based on boundary misorientation changes. In regime I (280–300 °C) some low angle boundaries (LABs), i.e. with 1°–15° misorientation, increase in misorientation angle, while others decrease. In regime II (~300 °C) all LABs undergo a decrease in misorientation angle. Regime III (>300 °C) is defined by enhancement of the subgrain structure as remaining LABs increase and some undergo a rotation axis change. Throughout regimes I and II, new LABs develop, subdividing subgrains. LABs could be divided into four categories based on annealing behaviour, orientation and morphology. We suggest that these observations can be directly related to the mobility and activation temperature of climb of two dislocation groups introduced during deformation. Therefore, with in-depth investigation of a substructure with known deformation geometry, we can infer ratios of dislocation types and their post-deformation and post-annealing location. These can potentially be used to estimate the post-deformational annealing temperature in crystalline materials.

1. Introduction

Interpretation of microscale behaviour is key to developing a greater understanding of tectonic processes. Examination of the microstructure of a rock can give insight into the processes that occurred during its deformation history and the conditions under which these processes took place. The inherent problem in microstructural interpretation is that we are viewing the "frozen-in" final microstructure, a result of the accumulation of a sequence of processes. In particular, post-deformational annealing can drastically change the microstructure by growth of new strain-free grains and reorganisation of grain boundaries (e.g. Heilbronner and Tullis, 2002). However, at the same time, these changes have the potential to provide evidence of the time-temperature path of the rock, as well as its rheological evolution.

An important part of post-deformational annealing is substructural rearrangement. Deformation is accommodated by the storage of defects in the crystal lattice. Strain energy is stored as point defects, dislocations and dislocation arrays (subgrain boundaries or grain boundaries) (Passchier and Trouw, 2005). The driving force for post-deformational annealing is the reduction of the stored energy of the system (Urai et al., 1986; Drury and Urai, 1990; Baker, 2000). This recovery is driven by the interaction of dislocations via their long-range stress fields and occurs through two main processes, annihilation of dislocations of opposite signs and polygonisation, where dislocations align to form low energy arrays (LABs) (Gottstein, 2004; Humphreys and Hatherly, 2004). As dislocations align, their areas of distortion overlap so that, with increasing misorientation the energy per dislocation decreases (Hull and Bacon, 2001). These processes are facilitated by dislocation climb, which is thermally activated and can limit mobility at lower temperatures. In particular, climb is important for dislocations in a boundary to rearrange to decrease spacing (Hull and Bacon, 2001). Once a polygonised substructure is attained, the stored energy can be further lowered by a coarsening of the substructure to reduce total boundary area (Humphreys and Hatherly, 2004). In particular, symmetrical tilt boundaries can move by glide of the edge dislocations that comprise the boundary (Humphreys and Hatherly, 2004). Mobility in this case is high, and

* Corresponding author. Fax: +46 (0) 8 6747897.
E-mail address: verity.borthwick@geo.su.se (V.E. Borthwick).

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boundary migration can occur even at low temperatures (Parker and Washburn, 1952). It is important to note that the energies of LABs are strongly dependent on both misorientation and the boundary plane; therefore changes in the misorientation and/or boundary plane may also result in energy reduction even if the total grain boundary length increases (Piazolo et al., 2004).

LABs have in general not received as much attention as high angle grain boundaries in geological materials. Previous experiments carried out on polycrystalline halite (Bestmann et al., 2005; Piazolo et al., 2006) indicated that substructural elements did not behave exactly as predicted by the above outlined classical theory, where LABs are generally expected to undergo a stable increase in misorientation once formed, progressing on to subgrain growth to reduce the total boundary length (Humphreys and Hatherly, 2004). Bestmann et al. (2005) and Piazolo et al. (2006) observed LABs both increasing and decreasing in misorientation, rearranging within grains as well as in some cases dissipating completely.

Recently developed techniques in the field of microstructural analysis enable us to investigate substructural behaviour in more detail. Electron backscatter diffraction (EBSD) (Prior et al., 1999 and references therein) allows us to fully characterise misorientation axes and angles between grains and subgrains. There have been a number of studies of microstructures taken from various stages of annealing in both geological materials (e.g. calcite) (Barnhoorn et al., 2005) and metals (Ferry and Humphreys, 1996, 2006; Huang and Humphreys, 2000, 2001; Huang et al., 2000). In-situ heating within the scanning electron microscope (SEM) (Le Gall et al., 1999) and analysis with EBSD are thus an essential addition, providing a powerful tool for "real-time" microanalysis of structural changes during annealing (Humphreys, 2001; Seward et al., 2002; Piazolo et al., 2005). At present a handful of studies have been carried out using this method, on materials including titanium (Seward et al., 2004), aluminium (Huang and Humphreys, 1999; Piazolo et al., 2005; Kirch et al., 2008) rocksalt (Bestmann et al., 2005; Piazolo et al., 2006), copper (Mirpuri et al., 2006; Field et al., 2007) and Al–Mn alloys (Lens et al., 2005).

NaCl was chosen as the experimental material for this study. Halite plays a significant role in fold-and-thrust belts, delta tectonics, basin evolution and hydrocarbon accumulation, as well as being a possible medium for storage of nuclear waste (Franssen, 1993 and references therein; Rempe, 2007; Schléder and Urai, 2007). The development of subgrain-scale microstructures in halite occurs at experimentally attainable conditions ($\sim$20 MPa and $>$200 °C) (Senseny et al., 1992) and is similar to that occurring at higher temperatures and pressures in silicates, making it a good analogue material (Guillope and Poirier, 1979; Drury and Urai, 1990). Due to its ionic-bonded, cubic crystal structure, NaCl provides a simple starting point for studying these complex processes.

In this contribution, we aim to provide a comprehensive characterisation of the substructural dynamics of a crystalline geological material during post-deformational annealing. On the basis of this information we attempt to recognise key features occurring at different annealing temperatures, which may be used for interpreting post-deformational annealing conditions in natural samples. In particular, by recognising different types of LABs and their respective behaviour during annealing we aim to improve our understanding of substructural development.

2. Methods

2.1. Sample preparation

The sample (TL1) was prepared from a single crystalline rod of melt grown high purity sodium chloride provided by *Harshaw Chemical Co.* (van der Linden, 2002). Using a single crystal removed the possibility of rapid high angle grain boundary migration removing the substructure (Piazolo et al., 2006) and the high purity meant that boundary pinning by impurities would be less likely to occur (Smith, 1948). Rectangular samples with a size of $\sim 7 \times 10 \times 15$ mm were cleaved along {100} faces. These particular edge ratios were chosen to limit dislocation glide to two sets of perpendicular planes (Fig. 1a) (Davidge and Pratt, 1964). Due to the asymmetry of the sample one set of glide planes will be activated more easily. With this chosen geometry the sample is more relevant to geological materials with low symmetry crystallography, which tend to have a well-developed dominant slip system with a number of subsidiary slip systems.

The single crystals were deformed as follows: during a period of four days the sample was heated in steps of 20–25 °C per hour to a maximum temperature of 660 °C. After heating, the sample was cooled to the deformation temperature of 453 °C. TL1 was then deformed under uniaxial compression to a final strain of 0.165 at a strain rate of $6.9 \times 10^{-6} s^{-1}$. After deformation the sample was unloaded and allowed to cool to room temperature, taking $\sim$1 h. As fluid content at the boundaries can have a large effect on boundary mobility, the samples were kept dry in a desiccator to reduce the possibility of atmospheric absorption of water. Sections of deformed halite ($6 \times 5.5 \times 1.5$ mm) were cleaved using a razor-blade (Fig. 1b). The cleaved surfaces gave good quality electron backscatter diffraction patterns (EBSPs).

2.2. Experimental setup

The analysis areas for in-situ annealing experiments were selected close to the centre of each slice, where deformation was most intense (Fig. 1b). Two main areas of interest were chosen, areas in the central deformation zone (CDZ), where subgrains were near equiaxed and numerous (Fig. 1c) and areas in more peripheral zones (PZ) where subgrains were organised into elongate bands (Fig. 1d). The annealing experiments were performed in an XL30 environmental field emission gun SEM. Samples were held in place on the heating stage by a metal plate which served to increase the connectivity of the sample to the heating stage (Fig. 2a). Sample TL1_A2 had some dry silver paint on the bottom surface, which may have slightly increased conduction to the sample. In correspondence with annealing behaviour we estimate that this resulted in an increase of $\sim$10 °C at the sample surface and furnace temperatures have been adjusted for this. The sample was tilted at a 70° angle to the electron beam to optimise the EBSD signal. The sample temperatures are estimated to be within $\pm$15 °C of the furnace temperature. Furnace temperatures are quoted in this paper; samples were heated under high vacuum ($\sim 5.4 \times 10^{-6}$ mBar) to temperatures of 280–470 °C in a number of steps. A number of different heating approaches were taken (Fig. 2b). We chose to investigate annealing at temperatures below that of the experimental deformation temperature (i.e. 453 °C) in order to examine a deformation-temperature-time path that is experienced commonly by natural rocks. Rocks are generally deformed in a ductile manner at depth at high temperatures. When deformation ceases the rock is subject to lower than deformation temperatures for long periods. In order to separate distinctive behaviour at differing temperatures it was necessary to steadily increase the temperature of annealing towards that of deformation. Had we started at higher temperatures, evidence of lower temperature regimes, which may be significant in natural settings would have been erased.

2.3. Data acquisition

EBSD mapping was conducted during annealing to gather information about changes in crystallographic orientation. Analysis areas with a high density of subgrains were selected from examination