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## The influence of water on deformation microstructures and textures in synthetic NaCl measured using EBSD

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## Abstract

Wet NaCl with > 10–15 ppm water shows weakening behaviour compared with dry NaCl containing <5 ppm of water. At strains greater than about 0.1 this weakening is associated with recrystallization but at lower strains there is also considerable weakening that is thought to be associated with pressure solution creep. The development of textures and microstructures in wet, synthetic NaCl polycrystals deformed at elevated temperatures has been investigated using electron backscattered diffraction (EBSD). At very low natural strains (0.07), textures, grain shapes and average misorientations of subgrain boundaries in wet NaCl differ to those found in dry NaCl deformed under similar conditions. In wet NaCl, cube shaped grains, oriented in a hard orientation for slip on the  $\langle 110 \rangle \{110\}$  system, grow and produce a well defined sub texture, with  $\langle 100 \rangle$  poles at 45° to the compression axis. An estimation of strain in wet NaCl was made using average misorientation values of subgrain boundaries. We estimate about 55% of the total strain was accommodated by dislocation creep in wet NaCl at 0.07 strain, the remaining strain being accommodated by pressure solution. At higher strains dynamic recrystallization occurs forming a  $\langle 100 \rangle$  fibre texture. This texture can be explained by preferential nucleation of initially strain free grains, which occurs after a critical strain in the most highly deformed, soft-orientation grains, that is grains which have  $\langle 100 \rangle$  parallel to the compression axis.

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## 1. Introduction

During plastic deformation at low temperatures (20–200 °C) and at laboratory strain rates, significant weakening is reported to occur in both natural and synthetic polycrystalline NaCl when intercrystalline brine is present (see Fig. 1) (Spiers et al., 1986; Urai et al., 1986; Peach et al., 2001). Above strains of about 0.1 weakening is associated primarily with dynamic recrystallization by enhanced fluid assisted grain boundary migration (Urai, 1983; Spiers et al., 1986; Ter Heege et al., 2005a). Only very little water, in excess of about 10–15 ppm, is needed to cause weakening (Watanabe and Peach, 2002; Ter Heege et al., 2005a): in such cases, the term wet NaCl is used here. The microscopic distribution of brine in polycrystalline NaCl is complex and has many important implications (see, for instance, Shenk and Urai (2004)). Under conditions of hydrostatic equilibrium, water is localized along grain boundaries and at triple junctions as isolated inclusions and tubes in accordance with dihedral angle considerations (Holness and Lewis, 1997). The pockets of water are thought to spread out into a thin film along grain boundaries during deformation due to dynamic wetting (Urai, 1983; Peach et al., 2001), increasing boundary diffusivity and enhancing grain boundary migration, which otherwise would not occur until temperatures above about 500 °C (Guillopé and Poirier, 1979; Franssen, 1993). Recent work shows that wetting of boundaries also occurs under static conditions when there is sufficient driving force for grain boundary migration (Shenk and Urai, 2004).

We use the term dynamic recrystallization to describe the growth of initially strain free grains that occurs in wet NaCl after a critical strain. Even at very low strains before the onset of widespread dynamic recrystallization, synthetic wet NaCl is 30–50% weaker than dry NaCl at strain rates of about  $10^{-7} \text{ s}^{-1}$ . At these low strains, the microstructures of both materials contain subgrain boundaries (Watanabe and Peach, 2002) indicating that some strain was accommodated by dislocation creep. The weakening could be related to an effect of water on either dislocation creep or on the operation of enhanced intergranular deformation in the wet material.

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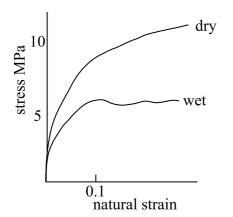


Fig. 1. Schematic diagram of stress versus strain for wet and dry polycrystalline NaCl deformed in compression, showing that wet NaCl is weaker than dry NaCl.

Calculations by Ter Heege (2002) suggest that at 0.07 strain up to 70% of the deformation could have been accommodated by fluid-enhanced grain boundary diffusion creep, or pressure solution (Spiers et al., 1990; De Meer et al., 2005). Though possibly coupled to pressure solution, grain boundary sliding is another possible mechanism that may also contribute to strain (Stokes, 1966).

Identifying active deformation mechanisms from microstructural signatures is a crucial step in obtaining mechanismbased rheological laws for geological materials (such as NaCl), which are suitable for extrapolation to natural conditions. In the absence of internal markers, identifying different deformation mechanisms from microstructures can be difficult (Passchier and Trouw, 1996), especially when more than one deformation mechanism operates. Diffusion creep can sometimes be recognised from segregation and overgrowth microstructures. In experimentally deformed material, grain boundary sliding can be inferred from split cylinder studies (Schmid et al., 1980), from internal marker particles in grains (Ree, 1994) and from the occurrence of diamond grain structures (Urai, 1987). In naturally deformed rocks grain boundary sliding is difficult to detect and possible indicators, such as a diamond grain structure, have been reviewed by White (1977), Drury and Humphreys (1988) and Ree (1994). Electron back scattered diffraction (EBSD) mapping is a particularly useful technique for determining changes in texture, microtexture and microstructure caused by dislocation creep (Randle, 1992; Humphreys et al., 2001) that occur in both naturally and experimentally deformed materials. Here the term microtexture means a population of orientations related to microstructure (Randle, 1992).

Trimby et al. (2000b) showed that misorientation angle distributions were significantly different for wet and dry NaCl at high strains because dynamic recrystallization removed subgrain boundaries in wet NaCl. In recent work on NaCl by Pennock et al. (2002) and Pennock and Drury (2005), EBSD mapping was used to determine the misorientation angle distributions of subgrain boundaries in dry NaCl as a function of strain. The average misorientation angle was found to be a useful parameter for describing the changes in the subgrain boundary misorientation distribution with strain (Hughes et al.,

1997; Pennock et al., 2005); at low natural strains up to 0.5, a power law relationship was found to exist between strain and average misorientations of subgrain boundaries. These results indicate that, with suitable calibration, average subgrain boundary misorientations could offer a method for estimating the strain accommodated by dislocation creep in dry NaCl. Furthermore, if only limited grain boundary migration occurs, average misorientations could be used to estimate the strain accommodated by dislocation creep in wet NaCl during multimechanism deformation, providing there is no intragranular effect of water on dislocation mobility.

Textures (lattice preferred orientations) also differ in deformed dry and wet NaCl. Inverse pole figures (IPFs) of the compression axis show a maximum intensity in the  $\langle 110 \rangle$ direction for dry NaCl 125–550° (Franssen and Spiers, 1990; Trimby et al., 2000b; Pennock et al., 2004) whereas in wet NaCl, at similar temperatures a (100) fibre texture occurs (Trimby et al., 2000b). In wet and dry NaCl at strains above about 0.1, this texture difference is caused by dynamic recrystallization, which occurs in wet NaCl, whereas a deformation texture is retained in dry NaCl (Trimby et al., 2000b). In a model, based on a self-consistent viscoplastic theory of polycrystalline plasticity, Wenk et al. (1997) and Lebensohn et al. (2003) investigated the effect of recrystallization and different deformation conditions in plastically anisotropic materials. The texture predictions for extrusive deformation using their model were in general agreement with textures observed in extruded NaCl, although the finite element methodology for averaging the polycrystalline response to deformation gave a better correlation to experimental data. The self consistent recrystallization model supports the idea that preferential nucleation occurs in grains that are more highly deformed (because they are in a soft orientation). In consequence, soft orientation grains commonly dominate recrystallization textures. Textures can be determined on a microstructural level using EBSD and used to test the current models of texture development.

Within the dislocation creep regime, nuclei for recrystallization in NaCl are most likely formed within highly deformed grains (Lebensohn et al., 2003). Subgrains are ubiquitous in NaCl deformed in the range of 125-500 °C and could be important nucleation sites for recrystallization (Humphreys and Hatherly, 1996; Humphreys, 2004). Subgrain boundaries in NaCl have widely varying misorientations and spatial distributions that vary with grain orientation and surrounding grain orientation (Trimby et al., 2000b; Pennock et al., 2005). Using EBSD, Pennock et al. (2002) and Pennock and Drury (2005) showed that individual subgrains in dry NaCl deformed at 165 °C were surrounded by segments of subgrain boundaries with a wide range of misorientations. Above strains of about 0.15, three types of subgrain boundaries were identified, depending on the boundary misorientation and distribution. Subgrain boundaries that surrounded equiaxed subgrains were ubiquitous at strains above about 0.15 and generally had lower misorientations ( $<5^{\circ}$ ), whereas extended subgrain boundaries rapidly developed higher misorientations even at low strains and were associated with triple points or dissected grains.

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