



# Rheology, microstructure and crystallographic preferred orientation of matrix containing a dispersed second phase: Insight from experimentally deformed ice



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## ABSTRACT

We utilize in situ neutron diffraction to continuously track the average grain size and crystal preferred orientation (CPO) development in ice, during uniaxial compression of two-phase and pure ice samples. Two-phase samples are composed of ice matrix and 20 vol.% of second phases of two types: (1) rheologically soft, platy graphite, and (2) rigid, rhomb-shaped calcite. The samples were tested at 10 °C below the ice melting point, ambient pressures, and two strain rates ( $1 \times 10^{-5}$  and  $2.5 \times 10^{-6} \text{ s}^{-1}$ ), to 10 and 20% strain. The final CPO in the ice matrix, where second phases are present, is significantly weaker, and ice grain size is smaller than in an ice-only sample. The microstructural and rheological data point to dislocation creep as the dominant deformation regime. The evolution and final strength of the CPO in ice depend on the efficiency of the recrystallization processes, namely grain boundary migration and nucleation. These processes are markedly influenced by the strength, shape, and grain size of the second phase. In addition, CPO development in ice is further accentuated by strain partitioning into the soft second phase, and the transfer of stress onto the rigid second phase.

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## 1. Introduction

Correct interpretation of microstructure in terms of deformation mechanisms and flow properties is crucial in deriving reliable information on the rheology of the continental lithosphere, which is mainly composed of polymineralic rocks. Commonly, the patterns and strength of crystal preferred orientation (CPO), together with grain size, are used to determine kinematics of flow (e.g., Lister and Hobbs, 1980), deformation conditions (e.g., Passchier and Trouw, 2005) and active deformation mechanisms (e.g., Fliervoet et al., 1997; Linckens et al., 2011). While experiments and numerical models provide a relatively good understanding of microfabric evolution in monomineralic aggregates (Jessell, 1988), the dynamics of

grain size evolution and CPO development in polymineralic rocks are still not fully known.

Many microstructural studies focus on the effect of second phases on the grain size of the deforming polymineralic matrix (e.g., Herwegh et al., 2011 and references therein). However, only a limited number of studies were designed to investigate their effect on the CPO of the matrix phase (e.g., Tullis and Wenk, 1994; Ebert et al., 2007; Austin et al., 2014; Little et al., 2015; Hunter et al., 2016). Within natural shear zones, CPO of the dominant matrix phase in polymineralic rocks is usually weaker, and grain size is smaller, compared with neighbouring monomineralic rocks, deformed at the same conditions (Herwegh et al., 2011). Similar relationships have been observed in experiments (e.g., Wilson, 1984; Tullis and Wenk, 1994; Austin et al., 2014).

A weaker CPO of matrix with second phase, compared to monomineralic matrix, can be explained in different ways. (i) Grain boundary sliding (GBS) accompanied by diffusion creep or dislocation glide was locally activated, and a CPO that developed at low strains, was weakened at higher strains (e.g., Fliervoet et al., 1997; Svahnberg and Piazzolo, 2010; Kilian et al., 2011). (ii) The bal-

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ance between the processes responsible for CPO development in the matrix with a second phase, and monomineralic aggregates is different (Ebert et al., 2007). (iii) The amount of strain accommodated by the matrix varies in samples with different composition, due to load transfer or strain partitioning (e.g., Renner et al., 2007; Hunter et al., 2016).

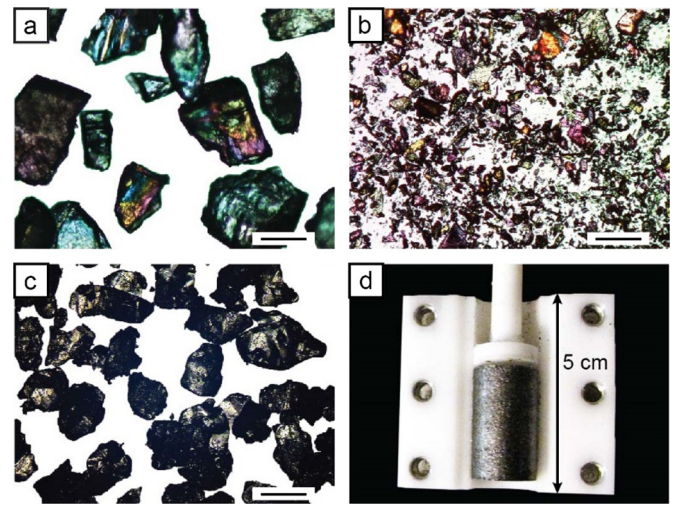
To better understand the mechanics behind “CPO weakening” in the matrix with a second phase, we conducted a series of uniaxial compression experiments on ice with two types of particles: rheologically soft, platy graphite, and rigid, rhomb-shaped calcite. Using a neutron diffraction facility, we have been able to record CPO and grain size evolution of the ice matrix, along with the mechanical data, at incremental strain steps of  $\sim 0.6\%$  strain up to 20% shortening. This continuous recording of data has the advantage over previous experiments, which recorded only initial and final stages, and allows assessment of the relationships between the changing microstructure, CPO and rheology of the matrix containing a second phase.

## 2. Experiments

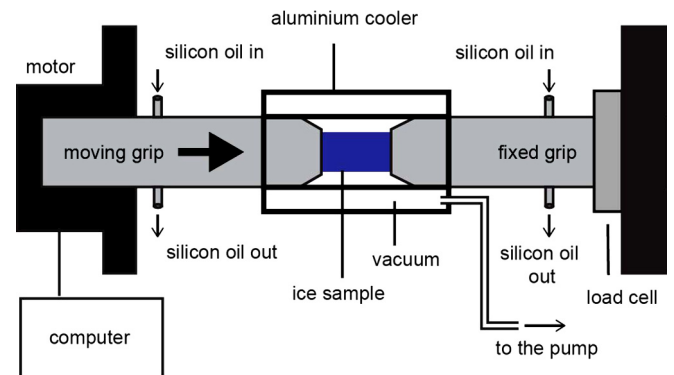
### 2.1. Materials

Ice, used here as the matrix phase, has a high visco-plastic anisotropy and deforms through slip on basal (0001) planes in  $\langle 11\text{--}20 \rangle$  direction (e.g., Glen and Perutz, 1954; Steinemann, 1958). Other slip systems in ice are rarely observed as they require two orders of magnitude higher shear stresses to activate (Wilson et al., 2014). Ice has been recognised as a valuable analogue for minerals with high viscoplastic anisotropy, such as quartz and olivine (Wilson et al., 2014). Ice is also a geological material in its own right, and in the majority of ice masses, zones and layers with a large spectrum of impurities can be found. Strain rates observed in natural ice (i.e.,  $10^{-8}\text{--}10^{-10} \text{ s}^{-1}$ ; Goldsby, 2006), are closer to those accessible in the laboratory ( $10^{-5}\text{--}10^{-7} \text{ s}^{-1}$ ), compared to rocks common in the lithosphere. Even at high homologous temperatures ( $T_h$ ), there is no chemical reaction between ice and a second phase, allowing evaluation of the physical processes alone. We use deuterium ( $\text{D}_2\text{O}$ , “heavy water”) ice, instead of  $\text{H}_2\text{O}$  ice, as  $\text{D}_2\text{O}$  ice has significantly better signal to noise ratio for neutron scattering, with little absorption (Piazolo et al., 2013). Importantly,  $\text{D}_2\text{O}$  ice has similar structural characteristics (Peterson and Levy, 1957) and rheology (Wilson et al., 2014) to  $\text{H}_2\text{O}$  ice.

For the second phase material we use calcite and graphite, representing analogue materials for a rheologically hard phase of isotropic shape, and a rheologically soft phase of anisotropic shape, respectively (Fig. 1a–c). At applied experimental conditions, calcite is brittle and much stronger than ice (de Bresser et al., 2002). Naturally deformed rocks often consist of minerals of contrasting physical properties, e.g. calcite and quartz in marbles, olivine and pyroxene in peridotites. Therefore, ice–calcite mixtures can be viewed as an analogue for such rocks. The platy graphite has a structure similar to mica, requiring very low shear stresses for the basal slip activation, even at low temperatures ( $<1 \text{ MPa}$ ; Soule and Nezbeda, 1968). Consequently, the ice–graphite mixture is a good analogue for quartz–mica mylonites and mica-bearing meta-psammities, as well as graphite-bearing quartz- and marble-mylonites. We used calcite crystals of 150–355  $\mu\text{m}$  and  $<150 \mu\text{m}$  in diameter, and graphite flakes of 150–355  $\mu\text{m}$  in diameter (Fig. 1a–c). Ice–calcite and ice–graphite samples were prepared by filling and pressing the pre-prepared mixtures of ice with 20 vol.% of second phase into custom-made cylindrical moulds of 2.5 cm diameter and 5 cm height (Fig. 1d). Samples were prepared in a cold room ( $T$ :  $-10^\circ$  to  $-20^\circ \text{ C}$ ) at the Bragg Institute in the Australian Nuclear Science and Technology Organisation (ANSTO). For a detailed sample preparation description see supplementary



**Fig. 1.** The powders (a–c) and custom-made mould used for sample preparation (d); the scale is 250  $\mu\text{m}$ . a: Coarse-grained calcite ( $150 < d < 355 \mu\text{m}$ ); b: fine-grained calcite ( $d < 150 \mu\text{m}$ ); c: platy graphite ( $150 < d < 355 \mu\text{m}$ ); d: cylindrical sample of ice with 20% graphite in a mould topped with the perforated plug.



**Fig. 2.** A scheme showing the deformation set-up, including the load frame assembly, the cooling system and the position of the load cell.

material S1. Deformed samples were stored at  $-80^\circ \text{ C}$  and any handling was performed at  $-20^\circ \text{ C}$  or colder, to ensure that no annealing or recovery took place.

### 2.2. Experimental techniques and data analysis

We deformed samples in uniaxial compression with the compression axis ( $\sigma_1$ ) parallel to the long axis of the sample cylinder (Fig. 2). Experiments were performed at constant displacement rate to axial strains of 10 and 20%. The displacement rate was calculated to obtain a starting strain rate of  $2.5 \times 10^{-6}$  and  $1.0 \times 10^{-5} \text{ s}^{-1}$  (Table 1). In this contribution we mainly focus on results obtained at strain rate  $10^{-6} \text{ s}^{-1}$ . To calculate the stress exponent for the flow law of ice mixtures, we combine the rheological data from both strain rates. The experimental temperature was set to  $-7^\circ \text{ C}$ , corresponding to  $0.96 T_h$  for  $\text{D}_2\text{O}$ . Sublimation of ice on both grips causes friction ( $f$ ) of  $\sim 0.1 \text{ kN}$  between the grips and the aluminium cooler (Fig. 2). The load ( $F$ ) was measured at small time steps ( $<2 \text{ s}$ ) on the fixed grip (Fig. 2). Stress ( $\sigma$ ) was calculated using the relationship

$$\sigma = (F - f)/A$$

where  $f$  is friction, and  $A$  is the increasing area of the cylinder, assuming uniform strain and constant sample volume. The calculated sample diameter was identical to the sample diameter, measured

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