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Strain field evolution during dynamic recrystallization nucleation; A case study on ice

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A B S T R A C T

Nucleation mechanisms occurring during discontinuous dynamic recrystallization (DDRX) is investigated by Digital Image Correlation (DIC) during creep experiment on polycrystalline columnar ice. Thanks to the columnar microstructure, discrimination of the nucleus can be done without ambiguity comparing *pre-* and *post-* deformation texture. *In-situ* DIC analyses are performed around a triple junction were nucleation occurred to follow strain field evolution. Strain field evolution appears strongly linked to nucleation mechanisms, local grain boundary migration and sub-grain boundary formation such as tilt sub-grain boundaries and kink bands. Nucleation processes are correlated with strong strain heterogeneities well characterized by the principal strains evaluated by DIC. It was possible to follow nucleus growth through the evolution of strain localization along the new grain boundaries. Kink bands act as a buffer zone close to the triple junction and accommodate shear parallel to the **c**-axis. The local strain field appears to be efficiently redistributed by recrystallization processes which create a new microstructure more compatible with the local stresses.

1. Introduction

Ice is an hexagonal material in which deformation mainly occurs by dislocation glide along the basal plane conferring a strong viscoplastic anisotropy to the crystal [1]. This anisotropy induces strong inter- and intra- granular strain field heterogeneities during deformation of polycrystalline ice [2]. Recrystallization processes in ice are similar to those in rocks or metals [3,4]. Depending on the deformation conditions, various forms of recrystallization can occur as normal grain growth, dynamic continuous recrystallization or dynamic discontinuous recrystallization (DDRX) [5].

In most materials, DDRX occurs during deformation at high temperature when a critical deformation condition is reached [6]. It is characterized by a competition between dislocation accumulation with deformation, and reduction of dislocation density by new grain nucleation and grain boundary migration. DDRX is well known to have a strong impact on deformation behavior and texture development in metal as magnesium [7] [8] and copper [9] for instance. In ice, DDRX induces drastic microstructure modification toward highly interlocked grains and texture evolves toward a multi-maxima girdle oriented around 30° to 35° from the compression axis [10–12].

A typical strain curve for compression creep of a granular polycrystalline ice sample deformed in common laboratory conditions ($0.8; \mathrm{MPa} > \sigma > 0.1\ \mathrm{MPa}$, $10^{-6}\ \mathrm{s}^{-1} > \dot{\varepsilon}_{min} > 10^{-8}\ \mathrm{s}^{-1}$) is characterized by a fast decrease of strain rate during primary creep to reach a minimum between 1% and 2% macroscopic strain, called secondary creep, and finally an accelerated tertiary creep, to reach a steady state after 10% [11,13]. This acceleration is attributed to DDRX which becomes the dominant process after 1% strain. The nearly constant creep rate reached after about 10% strain is attributed to the balance between recrystallization processes (nucleation and GB migration) and strain hardening. Although DDRX mechanisms dominate the macroscopic behavior after 1% strain, nucleation was observed locally in ice during primary creep [14] similarly to what was reported in other materials such as copper [15]. It shows that local strain and stress at intergranular scale play an important role for nucleation.

Nucleation processes in ice can take various form during DDRX. Tilt sub-grain boundaries and kink bands are observed in ice deformed in the laboratory [16–19,14]. A tilt sub-grain boundary (TSGB) is a rearrangement of basal edge dislocations into low-energy configurations inducing a **c**-axis misorientation through the sub-grain boundary. TSGB, well characterized using Electron

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BackScatter Diffraction (EBSD) [19], are straight sub-grain boundaries parallel to the **c**-axis direction. The combination of two tilt sub-grain boundaries in opposite rotation direction forms a kink band [20,16]. Very similarly to twins in hexagonal metals, kink bands enable to accommodate local shear, here parallel to the **c**-axis [21] but kink boundaries do not share the same crystallographic axis such as twins, and can accommodate a large range of misorientations.

Other nucleation processes are observed or suggested from both laboratory and ice-core texture analyses. Bulging, related to strain induced grain boundary migration, leads to nucleus with orientation close to the neighbor grains [22–24]. Some "spontaneous" nucleation was also suggested by Duval et al. [25] from calculation of the impact of long-range internal stresses (modeled by dislocation pile-ups) in drastically decreasing both nucleation critical radius and saddle point energy. Observation of nucleus with an orientation very different from the neighbor grains, for instance in deep ice cores at depth were texture is strong [26,27] could be explained by this "spontaneous" nucleation. We will see in the present work that similar observations are made during experimental creep.

As nucleation is a key point to a better understanding of DDRX impact on microstructure and texture development [6], many efforts are made to investigate nucleation processes in regard to nucleus orientation [7,28] and nucleation localization [29,15,30]. Due to optical or electronic microscopy methods used for orientation mapping, most nucleation studies are 2D and *post*-deformation. These characterizations therefore give no information about the time dynamics, and the evolution of internal stress and strain field in relation with DDRX processes.

Strain field measurements are currently obtained by Digital Image Correlation (DIC) which is an efficient tool to investigate strain field heterogeneities at *intra*-granular scale. For example, it was used recently to explore strain field evolution during twinning formation in magnesium [31] or to understand the link between strain field and texture heterogeneity in zirconium [32]. In ice, DIC was recently used to characterize the evolution of strain field heterogeneities during primary creep, i.e. until 1% macroscopic strain [2]. Evidence was given of strong strain localizations (up to ten times the macroscopic strain) close to GBs and triple junctions, but also of the lack of correlation between the local strain and the orientation relative to the imposed stress. Strong strain localization at the end of the transient creep was analyzed as the precursor of DDRX processes, and could explain the occurrence of tertiary creep after only 1% macroscopic strain.

In order to better understand the nucleation mechanisms and their kinetics in ice during DDRX, we present a way to investigate nucleation processes and their impact on local strain field by coupling Digital Image Correlation with *pre*- and *post*- deformation texture analyses on polycrystalline columnar ice, that offers a 2D-1/2 microstructure [2]. Besides the precise observation of some nucleation processes, this coupling evidences the strain redistribution induced and driven by recrystallization mechanisms, in order to accommodate the strong stress heterogeneities close to grain boundaries and triple junctions.

2. Experimental procedure

Polycrystalline columnar ice samples were elaborated following Grennerat et al. [2]. They are formed of parallel columnar grains with **c**-axes lying close to the sample surface plane ($\pm 15°$) [33,2], and typical in-plane grain dimension of about 10 mm. Rectangular samples ($8.5 \times 8.5 \times 1.5\ mm^3$) were deformed perpendicularly to the column axis by compression creep until 3% macroscopic strain (Fig. 1) along the vertical axis (**y**-axis, Fig. 2) in a cold room at $-7\ °C$

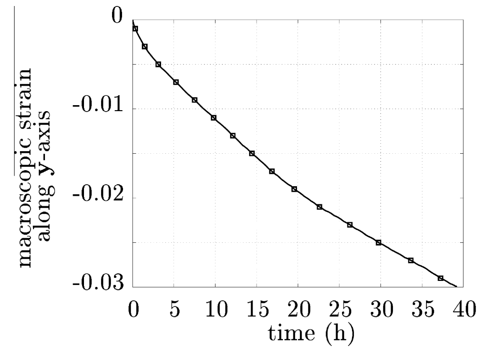

Fig. 1. Macroscopic strain response for the creep test performed on the sample shown in Fig. 2. The symbols show the position of the DIC analyses presented in Fig. 4.

and under uniaxial stress of 0.5 MPa. The contact between the press and the ice sample is made through Teflon sheets in order to avoid friction at the interfaces. The lateral surfaces are stress free.

The macroscopic response recorded by the strain curve is slightly different from the one described for granular polycrystalline ice in Section 1. Contrary to isotropic granular ice samples, the columnar ice microstructure used here do not constitute a representative volume element (RVE). The creep response (Fig. 1) therefore reflects a strong influence of the microstructure on the macroscopic behavior.

Sample microstructure and texture were measured using an Automatic Ice Texture Analyser (AITA) [34,35], an optical technique measuring the **c**-axis (or optical axis) coordinates $(sin(\phi)cos(\theta), sin(\phi)sin(\theta), cos(\phi)$, azimuth θ and colatitude ϕ). Thanks to the 2D-1/2 columnar microstructure, *pre*- and *post*-deformation texture analyses enable to discriminated new grains in the microstructure without ambiguity (Fig. 2).

During the experiment, DIC analysis was performed over a reduced area around a triple junction (Fig. 2) where nucleation is more likely to occur [15]. A detailed description of the DIC technique applied on ice is given in Grennerat et al. [2]. Pictures of the sample surface (marked with an appropriate speckle [2]) were taken every 10 min with a resolution of 18; μm pix^{-1}. The displacement and strain fields were extracted using 7D software described in [36] with a grid step and a pattern size of 12 pixels. This choice provided a spatial strain field resolution of 0.22 mm pix^{-1} that appeared sufficient with respect to the grain size (cm scale), in order to access the *intra*-granular strain field.

From the displacement field componant $\vec{\delta} = u.\vec{x} + v.\vec{y}$, where $\vec{x}$ and $\vec{y}$ are unit vectors along the x and y directions, the strain components are extracted by calculating $\varepsilon_{xx}, \varepsilon_{yy}, \varepsilon_{xy}$ using Green–Lagrange expression, and solid rotation $\omega_{xy} = \frac{1}{2}\left(\frac{\partial u}{\partial y} - \frac{\partial v}{\partial x}\right)$ using the approximation of small rotation. Grennerat et al. [2] showed that the main uncertainties could be evaluated by correlating two images acquired without any displacement. In our experimental conditions, such uncertainties are $\sigma_{\varepsilon_{xx}} = 2.2 \times 10^{-3}, \sigma_{\varepsilon_{yy}} = 1.6 \times 10^{-3}$, $\sigma_{\varepsilon_{xy}} = 2.6 \times 10^{-3}$ and $\sigma_{\omega_{xy}} = 2.8 \times 10^{-3}$.

From the strain field components, we define an equivalent strain field as:

$$\varepsilon_{eq} = \sqrt{\frac{2}{3}\left(\varepsilon_{xx}^2 + \varepsilon_{yy}^2 + 2\varepsilon_{xy}^2\right)} \tag{1}$$

with an uncertainty $\sigma_{\varepsilon_{eq}} = 2.5 \times 10^{-3}$.

Please note that we will refer to the macroscopic strain (or macro strain) as being the strain measured at the whole sample