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Deformation Microstructures in an Antarctic Ice Core (EDML) and in Experimentally Deformed Artificial Ice

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Abstract: Deformation microstructures in an Antarctic ice core (EDML) and in experimentally deformed artificial ice, which together comprise a great variety of conditions and parameters, are reconsidered and compared. Data presented here cover grain substructure and shape. Despite the different flow conditions surprising similarities in these observations indicate intracrystalline slip as the deformation carrier in natural as well as in experimentally deformed ice. Similar subgrain-boundary shapes and arrangements in both cases indicate characteristic types, which suggest that non-basal dislocations may play a significant role in the deformation of ice. Subgrain-boundary density and grain-boundary shapes show that a difference between processes in creep tests and in the Antarctic ice sheet is the efficiency of recovery and dynamic recrystallization.

Key words: low angle grain boundary, creep test, dynamic recrystallization, recovery, sublimation grooves

1 Introduction

Studies on ice flow conditions and physical properties have been performed for several decades in natural ice from deep ice cores [e.g. 1, 2, 3] as well as in artificially produced and experimentally deformed ice [e.g. 4, 5, 6, 7]. Microstructure investigations in these works are usually concerned with the texture (grain size, grain morphology) and crystal orientation fabrics on the grain scale. However, as the preponderant deformation mechanism in ice is intracrystalline slip [e.g. 6, 8, 9], most deformation is indeed carried out on a smaller scale range, the subgrain scale within individual grains. Investigations on this high resolution usually lack the overview which enables statistics over a significant ice volume [10, 11]. The microstructure mapping method represents therefore a good compromise, by offering insight into small scale structures at high resolutions over a considerable amount of material [12].

Microstructures provide evidences of recrystallization and deformation and therefore provide information on the operation of these processes. A sequence with depth of three recrystallization regimes is often recited for polar ice sheets [e.g. 9, 14, 13], and disputed by recent observations [15, 16]. Deformation independent normal grain growth in the upper hundreds of meters is supposed to be followed by rotation recrystallization (continuous dynamic recrystallization) in the middle part of the ice sheet's depth and by discontinuous dynamic recrystallization (migration recrystallization) in the lowermost region. Rotation recrystallization results from progressive misorientation of grain parts, which are formed by the so-called polygonization mechanism [e.g. 17]. This process divides grains under the action of localized stresses into two or more misoriented subgrains. Thus, the rotation recrystallization process which should dominate the middle depth region of an ice sheet is highly correlated to subgrain-boundary formation processes. Therefore these features deserve special interest and investigation.

The aim of this article is to reconsider subgrain-boundary data and grain substructure observations obtained from Antarctic ice as well as from deformed artificial samples [18, 16] and to try a first comparison to show evidences which microprocesses represent the different flow behaviour under high and low stresses. Arguments for the activity of intracrystalline slip in low and high stress regimes involving basal and maybe even non-basal glide can be given by subgrain-boundary observations.

2 Samples

Creep experiments using ice samples free of bubbles, impurities and deformation features have been conducted under uniaxial compression in the stress range 0.18 - 0.52MPa and the strain range 0.5 - 8.6% at approximately

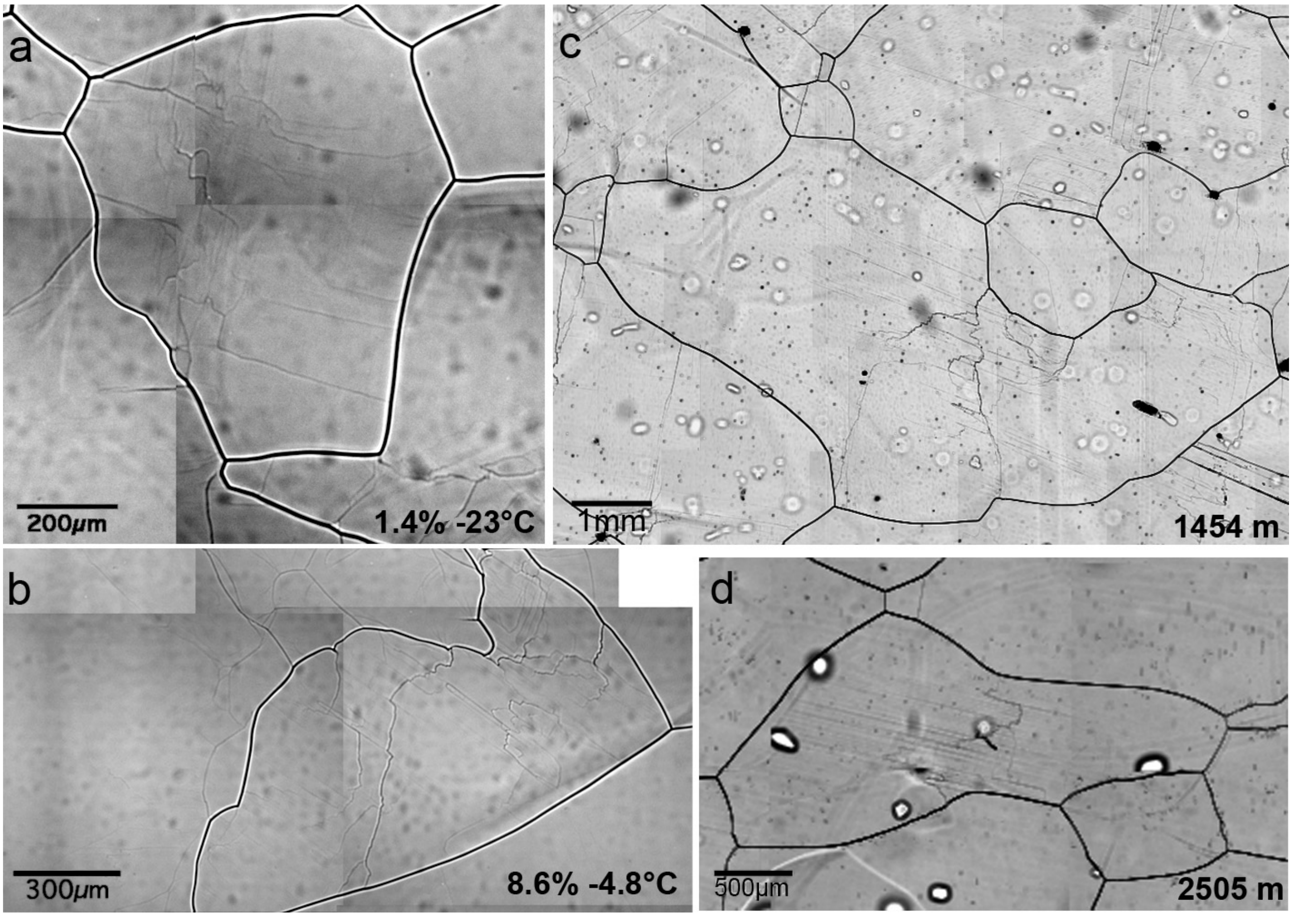

Figure 1: *Microstructure mapping examples from vertically cut thick sections.* a & b: *creep experiment samples.* a: *deformed at* $-23\,^{\circ}C$ *with* $0.35MPa$ *until* 1.4% *strain.* b: *deformed at* $-4.8\,^{\circ}C$ *with* $0.52MPa$ *until* 8.6% *strain.* c & d: *EDML ice core samples. Depths are given. (Some photographs are reprinted from the Journal of Glaciology with permission of the International Glaciological Society.)*

-5°C and -20°C [18]. Initial samples were isotropic and had small grain sizes ($\approx 0.6\text{mm}^2$). Microstructures of deformed samples were examined by microstructure mapping [12] which reveals grain boundaries and subgrain boundaries as shallow sublimation grooves (examples see Fig. 1) by thermal etching [19]. This technique is highly sensitive in detection of very low angle boundaries ($< 0.5°$, verified by X-ray Laue diffraction). However, there are some limitations to be mentioned. A symmetric dihedral angle of equilibrium thermal grooving can only be achieved if the (sub)grain-boundary plane is perpendicular to the sublimated surface [20]. Any other angle between these two planes, leads to an asymmetric angle of the groove, which has a noticeable effect on the quality of the groove line observed/photographed under the microscope. The groove line appears lighter-grey and sometimes a little diffuse. Although all subgrain boundaries are revealed by this method, some might be overseen by the observer under unfavourable conditions, due to these quality differences. As the subgrain boundaries seem to occur mainly in planes of some, non-random orientations inside the crystal lattice (see Paragraph 3) such low-quality grooves can alter the statistics on subgrain-boundary occurrence. Another important limitation is that this technique does not give information on misorientations. Although the groove depth depends on the misorientation, the above mentioned effect caused by the angle between the sublimation surface and the (sub)grain-boundary plane does not allow any quantitative conclusions concerning misorientations. Therefore, further high-resolution crystallographic methods are needed in combination with microstructure mapping.

This observation method was also used for natural ice samples from the $\approx$ 2775m long EDML (EPICA Dronning Maud Land) core acquired at Kohnen station (75°00.104′S, 0°04.07′E) between 2001 and 2006. At this site the annual accumulation rate is 64kgm^{-2} per year [21] and mean temperatures at the surface are ≈ -45°C and ≈ -3°C at the bore hole bottom [22]. Surface velocities are in the order of 0.7ma^{-1} [23]. Variable grain sizes [16] and impurity contents [24] are observed along the ice core.

The microstructure discussions here are focused on

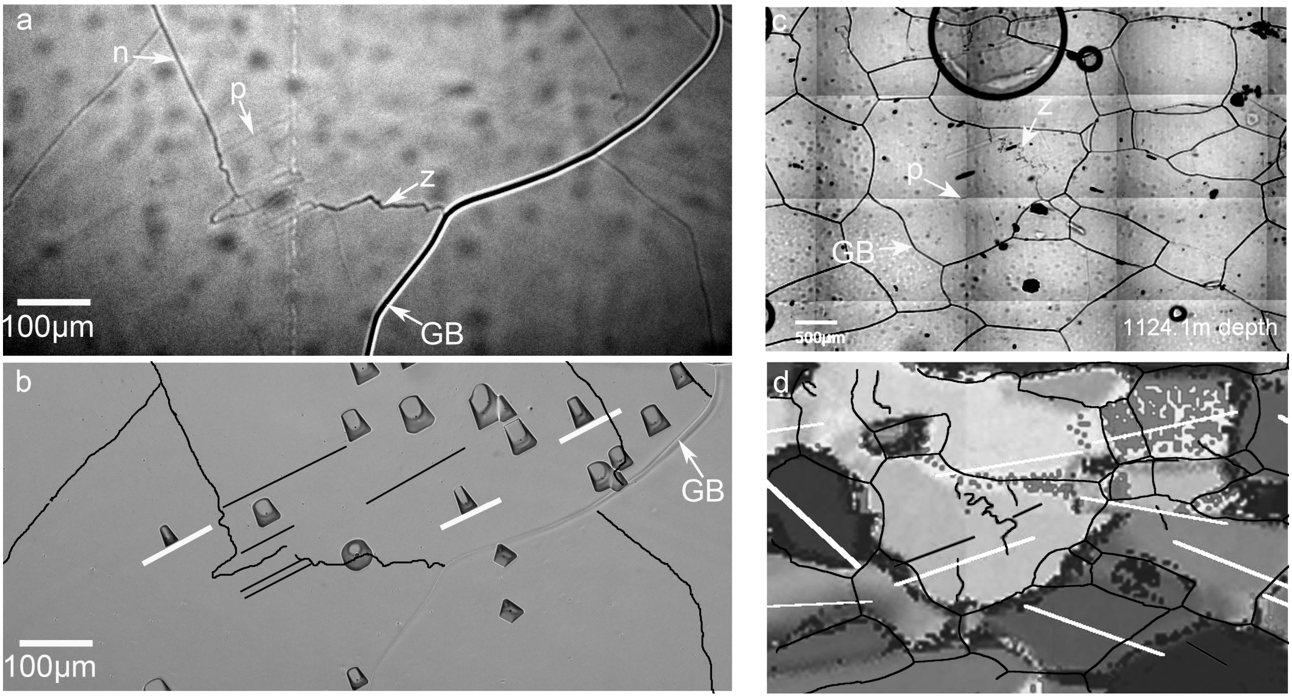

Figure 2: *Subgrain-boundary types and crystal orientation.* a: *Creep-test sample.* b: *Same section as* a*, after application of etch pit method. Subgrain boundaries (black lines) are drawn after* a*. Basal plane traces (white bars) are drawn after etch pits.* c: *EDML sample.* d: *C-axis orientations encoded as AVA (AchsenVerteilungsAnalyse) image obtained from the same section as* c *with automatic fabric analyzer [27] (color version available from authors). Basal plane traces (white bars) are drawn after c-axes measurements. Subgrain boundaries and grain-boundary network (black lines) are drawn after* c*. Imperfect fit of grain-boundary network with AVA-image is due to oblique grain boundaries. (GB=grain boundary, p=parallel type, z=zigzag type, n=normal polygonization type subgrain boundary as described by Nakaya [26]). (Some photographs are reprinted from the Journal of Glaciology with permission of the International Glaciological Society.)*

grain shape and subgrain-boundary observations, because these have been so far rarely considered.

3 Subgrain-boundary types

In consideration of the significantly different formation and deformation conditions in terms of e.g. temperature, impurities, grain size, strain and strain rate of the observed ice samples it is surprising at first sight that the same types of subgrain boundaries were found in experimentally deformed ice as well as in natural ice [18, 16]. These characteristic types can be distinguished after their shapes and arrangements (Fig. 1) and with respect to the crystal lattice orientation (Fig. 2). It is known from other materials that the orientation of subgrain boundaries depends on the orientation of slip systems of dislocations accumulating in the grain [25]. As detailed observation and analysis of these characteristic arrangements can be useful to study dislocation action in polycrystalline ice, the observed types shall be described here in detail.

The subgrain-boundary formation in ice was first described by Nakaya [26]. Bending of the basal plane, rearrangement of basal edge dislocations into walls and subsequent splitting of the grain lead to formation of subgrain boundaries [28, 29]. This process produces straight subgrain boundaries normal to the basal plane. Such arrangement of subgrain boundaries (here called n-type subgrain boundaries) can indeed be observed in the sublimation grooves revealed by microstructure mapping (see example in Fig. 2a, b). First X-ray Laue measurements confirm that the majority of subgrain boundaries arranged in this configuration indicate a misorientation obtained by rotation around one a-axis [30]. They are interpreted as basal tilt boundaries [18]. Actually this type of subgrain boundary is most rare in artificial creep test samples as well as in polar ice (Fig. 3a,b). Characteristically n-type subgrain boundaries are attached to another type characterized by its irregular, usually zigzag shape.

Subgrain boundaries of this related zigzag or step shape (z-type) are also arranged at an angle to the basal plane, with short segments running parallel to this plane (see example in Fig. 2). Typically z-subgrain boundaries do not cross a grain completely but fade out at some distance to the high angle grain boundary. They can be interpreted as a sequence of boundaries with a diversity of dislocation types involved (e.g. basal edge and non-basal edge dislocations) [18]. Clearly further investigations are necessary, which become available with high resolution full crystal orientation methods [31, 32, 33, 34].

The third distinguishable type is called p-type subgrain

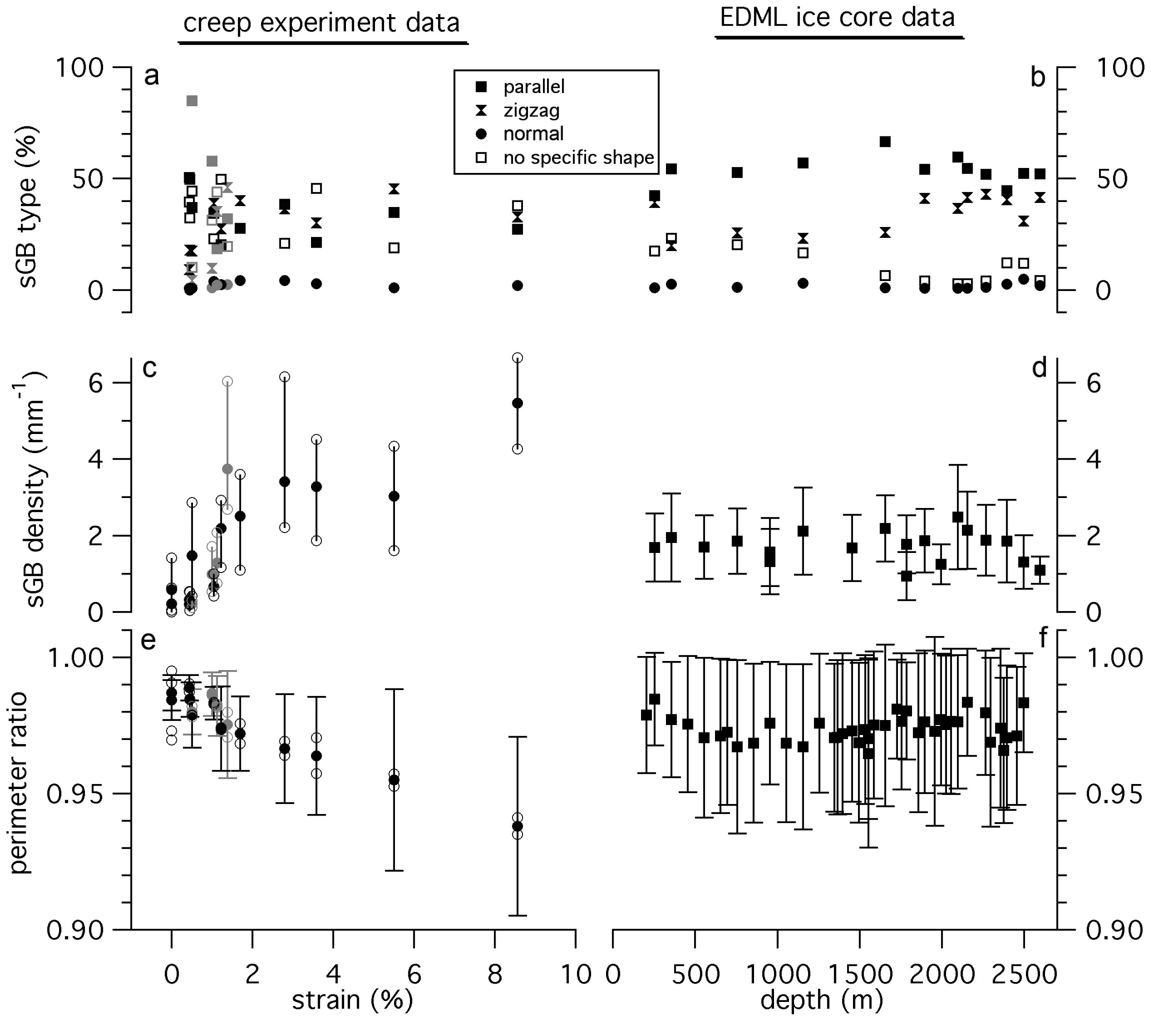

Figure 3: *Evolution of microstructure parameters.* a&b: *Subgrain-boundary type frequency.* c&d: *Mean subgrain-boundary density.* e&f: *Mean grain perimeter ratio indicating irregularity of grains (see text). Abscissa is identical in right (*b, d, f*) and in left (*a, c, e*) column. Error bars give the standard deviation.* Left: *during creep experiments. Open circles indicate the scatter of data betweeen four to six measured sections. Black markers represent experiments at $\approx -4.5°C$, grey markers at $\approx -23°C$.* Right: *ice sheet samples (EDML). (Partly reproduced from the Journal of Glaciology with permission of the International Glaciological Society.)*

boundary, because it appears in swarms exactly parallel to each other. Besides the alignment in swarms, its parallel arrangement with respect to the basal plane of the crystal lattice is remarkable (Fig. 2). In consideration of their arrangement and shape three possible explanations can be given: basal twist boundaries, non-basal tilt boundaries or micro-shear zones [18, 16, 35]. Basal twist boundaries are composed of screw dislocations in the basal plane. The commonness of this dislocation type in ice has been shown [e.g. in 10, 36, 37]. These subgrain boundary types produce misorientations which can be described by rotation around the c-axis. Indeed preliminary X-ray Laue measurements confirm their existence [30].

Non-basal tilt boundaries can be imagined as made up of non-basal edge dislocations. They have been confirmed by preliminary X-ray Laue measurements as well [30], indicating a rotation around one a-axis. The process producing such a high amount of non-basal dislocations to build up subgrain boundaries is still not clear and will be topic of further investigations.

The alternative explanation as micro-shear zones describes shearing-off of a prominent part of a grain, which is penetrating a neighbour (Fig. 7c in [16]). This shear occurs along the basal plane that is aligned parallel to long grain-boundary chains. This process was observed during the deformation of octachloropropane [38] and considered as a possible cause for sudden change in ice softness in the depth range 2385m to $\approx$ 2575m of EDML [35].

As mentioned in Section 2, there is a dependence of sublimation on cutting orientation due to the fact that best thermal grooving can be obtained if the boundary is perpendicular to the surface, whereas an oblique intersection of boundary and surface produces oblique and shallow grooves. Thus, the arrangement and shapes of sublimation grooves along subgrain boundaries is most easily observable if the sublimation surface is almost parallel (within $\approx 30°$) to the c-axis. Therefore a considerable amount of unidentifiable types is observed (Fig. 3a,b), which, in the case of ice core samples decreases with depth (Fig. 3b), as fabric enhancement enables vertical sections with many grains "nicely" oriented for sublimation.

3.1 Implications on micromechanisms

It is interesting that the types of observed ice substructures are the same in deformed artificial ice and ice from a deep Antarctic ice core, especially as the experiments reached at most secondary creep with small strains whereas in polar ice large strains prevail. First of all, the similarity in subgrain-boundary observations prove directly the relevance of dislocation creep in polar ice [supporting 6, 8, 9] and under the chosen experimental conditions.

Moreover, due to the observation of the similarity of grain substructures and the high mechanical anisotropy of ice it can be assumed that these structures indeed are characteristic traces of deformation processes displaying a material peculiarity in its response to creep. If there is even a very small component of shear stress in the basal plane of a crystal, which is likely in the highly complex stress configurations for grains in polycrystalline material, the ice crystal responds firstly by the activation of the dominant slip system in the basal plane. Other slip systems should contribute much less to deformation, because of the peculiar properties of dislocations in ice [37]. However, non-basal slip as a basal-slip-accommodating process is most important to provide strain compatibility among neighbouring grains, to avoid the occurrence of microcracks and voids [6, 39, 40]. The significant occurrence of subgrain boundaries which cannot only be explained by arrangement of basal dislocations (p and maybe z in Fig. 3a,b) gives first experimental evidence of the importance of non-basal glide. Further investigations including subgrain-boundary lengths of all types and full lattice misorientation measurements can bring the question forward, how much non-basal glide is possible or necessary under different deformation conditions in polycrystalline ice.

4 Subgrain-boundary density, Grain-boundary morphology and Strain/Stress localization

The first characteristic similarities observed above encourage us to try a comparison of creep experiments and ice sheet samples with more parameters.

As a parameter to describe the occurrence of subgrain boundaries, a subgrain-boundary density is defined as the total subgrain-boundary length per area, which can be obtained by image analysis of microstructure images [12] (examples see Fig. 1). In experimentally deformed ice samples subgrain-boundary formation in creep tests correlates to strain during primary creep stage which lasted until ≈ 1 to 2% strain (Fig. 3c) due to the production and interaction of dislocation walls and subgrain boundaries which act as obstacles for dislocation movement [18]. This demonstrates the connection of crystal-substructure evolution with isotropic hardening [18], which is due to short-range interactions between dislocations and represents the irreversible component of strain during transient creep [6]. Microstructure mapping of EDML ice core thick sections reveals an invariance in subgrain-boundary occurrence with depth (Fig. 3d,f). Hence, subgrain formation is permanently active at all depths [16].

A measure for the irregularity of grains is the perimeter ratio: the ratio of convex perimeter against real perimeter (Fig. 1 in [18]). A most regular object (e.g. circle, ellipse or rectangle) has perimeter ratio =1. Lower values describe the degree of irregularity. The correlation of irregularity of grains with strain in creep tests (Fig. 3e) is clear. Compared to the values of grains from ice core samples they can exhibit extremal values (Fig. 3e,f). Similar to subgrain-boundary density the mean perimeter ratio does not change significantly with depth compared to the variation of data suggesting that the resultant driving pressure for grain-boundary migration is nearly the same at all depths [16].

Furthermore, distributions of subgrain boundaries inside grains are very similar. In both cases crystal substructures are stronger and preferably accumulated close to grain boundaries forming a highly heterogeneous substructure distribution [18, 16]. The accumulation at prominent parts of complex grain-boundary geometries observed in ice sheets and creep test samples strikingly suggests that strain accumulation is the rule rather than the exception in deforming ice in general. The frequent occurrence of grain-boundary curvatures and the surplus of subgrain boundaries on their convex side and hence the excess of driving pressure exerted by internal strain energy over driving pressure exerted by grain-boundary tension indicates that locally high dislocation densities can occur [18].

4.1 Implications on micromechanisms

Experiments have been conducted at stresses between 0.18 and 0.52MPa. Compared with the polar ice sheets, where driving stresses are typically lower than 0.1MPa, measured deformation rates are rather high. Besides differences in strain rates, total strains in experiments reached a maximum of $\approx 8.6\%$ only, whereas much higher deformation is expected in the ice core samples. Furthermore other possible variables influencing creep or deformation related recrystallization (e.g. impurities, temperature, grain size, fabrics) cover a striking range of values along the ice core and in the samples. Therefore it is most surprising that mean subgrain-boundary density is of the same order in experiments (≈ 0 to 4mm^{-1}, see Fig. 3c) and in EDML ice core (≈ 1 to 3mm^{-1}, see

Fig. 3d). The appearance of subgrain boundaries proves the important role of dislocation creep under the range of deformation conditions, because subgrain boundaries are composed of arrays of dislocations [e.g. 28, 29]. The irregularity of the shapes of grain boundaries suggest difficulties to carry out grain boundary sliding.

A slight difference can be observed in the scattering of measured values. While the subgrain-boundary density data from creep experiments cover a range of $\approx 6\text{mm}^{-1}$, the data from the EDML ice core vary only within $\approx 3\text{mm}^{-1}$ (Fig. 3c,d). However, here a statistical effect cannot be excluded definitely as the creep test samples are smaller and therefore the examined area is smaller than in ice core samples.

Together with the slightly lower average values in ice deformed at low stresses in the ice sheet the data-scattering difference can be explained by processes which reduce the dislocation density. They can act more extensively with the prolonged duration of creep in the slowly deforming ice sheet. Montagnat and Duval [8] incorporated these processes in a deformation model. The two main processes are recovery and dynamic recrystallization which compete, because both are driven by the stored internal strain energy. Dynamic recrystallization can be defined as a *"deformation-induced reworking of the grain sizes, shapes or orientations"* [see 17, p. 179]. Conversely, recovery takes place at a smaller scale within individual ice grains.

Two primary processes are involved in recovery: annihilation of dislocations and rearrangement of dislocations [41]. Annihilation can remove dislocations by connecting two dislocations of opposite signs. The rate of dislocation annihilation is dependent on the ease of operation of climb and cross slip, because two dislocations with the same Burgers and line vectors but opposite signs have to share identical glide planes to meet and annihilate [29]. However, the low stacking fault energy of ice [37], which leads to wide dissociation of dislocations in the basal plane, makes climb and cross slip difficult [41] and thus the recovery by dislocation arrangement is probably more effective than the recovery by annihilation.

Rearrangement of dislocations creates lower energy configurations and leads to formation of dislocation walls and subgrain boundaries (polygonization). This process is very active in ice as can be observed by the frequent occurrence of subgrain boundaries. Further gathering of dislocations leading to subsequent subgrain rotation, and thus giving way to continuous dynamic recrystallization, converts subgrain boundaries into grain boundaries. Therefore the higher subgrain-boundary density values in experimentally deformed samples compared to EDML samples (Fig. 3c,d) does not necessarily suggest higher amount of rearrangement of dislocations during creep tests. However, a surplus of small angle misorientations of neighbouring grains with respect to random grain-pair misorientations was not found (Fig. 3 in [16]) and thus intensive further gathering of dislocations and subsequent subgrain rotation cannot be proven. Ice sheet ice does not completely work harden, viz. reach maximum subgrain-boundary density (Fig. 3c,d).

As another important process, which heals the material from dislocations, grain-boundary migration plays an important role. Grain boundaries can sweep away dislocations and subgrain boundaries which are located in front of a moving grain boundary [42]. This process is less subtle and restores the materials properties more efficiently than recovery, because it can remove dislocations effectively which are typically accumulated locally due to strain localization [43, 44, 45, 46, 18, 16]. That this strain induced grain-boundary migration (SIBM as one mode of dynamic recrystallization) indeed plays an important role can easily be observed by the irregularity of grains (examples see Fig. 1). These complex grain geometries evolve due to bulging of grain boundaries in experimentally deformed ice as well as in polar ice [18, 16]. This is part of an intense feed-back process where complex geometries lead to complicated stress interaction at grain boundaries in polycrystalline ice, which lead to strain localization and localized dislocation formation; that restart again bulging of grain boundaries. The difference between experimentally-deformed and ice core samples in terms of the degree of irregularity of grains (Fig. 3e,f) gives insight in the delicate balances of the competing recovery and SIBM under slow and fast deformation. Irregularity during creep tests clearly increases with strain (Fig. 3e), which reflects the dislocation production directly. In these high stress regimes recovery seems to be relatively slow compared to the generation of defects by deformation. During application of low stresses grains are still significantly irregular (Fig. 3f), but as total accumulated strains should be higher in the ice sheet than in experiments, recovery, which reduces dislocation density without SIBM, seems to inhibit the evolution of highest irregularities of grains (Fig. 3e,f).

Microstructure data indicate that the relation of dislocation-production rate to dislocation-reduction rate is significantly higher in experimentally deformed samples, which leads to more extreme values for subgrain-boundary density and for grain-boundary irregularity (Fig. 3c-f). The comparison of the two data sets indicates that the reason for different high-stress and low-stress behaviour might be that recovery is mainly time dependent, whereas dislocation production is deformation dependent. Annihilation, which is difficult to carry out in ice, also occurs with aging [36] and may thus play some role in the ice sheet. However, further investigations to cover the plurality of probable variables (e.g. impurities) influencing these processes are necessary.

5 Conclusions

Studies on grain substructures in deformed ice samples reveal the effects and impact of intracrystalline deformation processes. The similarities in grain-substructure observations in experimentally deformed and Antarctic ice identify dislocation creep as a deformation carrier, whereas the more moderate values and scatters of subgrain-boundary densities and grain-boundary irregularities in ice sheets indicate that dislocation density-decreasing processes (recovery and dynamic recrystallization) play a far more important role under low-stress conditions. Recovery by rearrangement of dislocations can be identified in subgrain-boundary formation. Among dynamic recrystallization mechanisms, strain-induced grain boundary migration can be detected by bulging grain boundaries and the degree of irregularity of the grains. The delicate interplay of dislocation production, recovery and dynamic recrystallization controls the degree of work hardening and therefore the softness of the material, apart from fabric evolution.

The amount of non-basal dislocations should be much smaller than that of basal dislocations, which are responsible for dislocation creep of ice, but significant amounts of non-basal dislocations, enough even to form subgrain boundaries have to be taken into account to explain all the observed subgrain-boundary types.

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