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In situ X-ray tomography densification of firn: the role of mechanics and diffusion processes.

Alexis Burr^{a,b,*}, Pierre Lhuissier^a, Christophe L. Martin^a, Armelle Philip^{b,*}

^a*Univ. Grenoble Alpes, CNRS, Grenoble INP¹, SIMaP, 38000 Grenoble, France*

^b*Univ. Grenoble Alpes, Grenoble INP¹, CNRS, IRD, IGE, 38000 Grenoble, France*

Abstract

One of the most efficient proxy methods for paleoclimatology consists of obtaining data previously preserved within polar ice cores. Models for past climate reconstruction are based in particular on the characterization of entrapped gases in ice closed pores. Improving the temporal accuracy of these models requires a better understanding of firn densification mechanisms. In particular, the interplay between viscoplastic deformation and diffusion processes for pore closure is not well understood. In this work, we describe the first in situ laboratory densification experiments on polar firn retrieved from Antarctica with live characterization by X-ray tomography. *Our in situ tests allow for the first time to approximately access the process of pore closure in ice, which takes thousands of years to occur in Antarctica, from visualizations and quantitative analyses of short time laboratory experiments. The parameters of pore separation and closure and the microstructural changes that accompany them are monitored.* We show that densification of polar

*Corresponding authors

Email addresses: alexis.burr@simap.grenoble-inp.fr (Alexis Burr),
armelle.philip@univ-grenoble-alpes.fr (Armelle Philip)

¹Institute of Engineering Univ. Grenoble Alpes

firn and pore closure could be replicated at higher strain rate and warmer temperature. Experiments allow the viscoplastic part of the firn deformation to be decoupled from the diffusion mechanisms that occur at high temperature. Our results show that density alone is not sufficient to predict the close-off density at which gases get entrapped. More generally, the method laid out here may find useful application in the domain of high temperature powder compaction, for which pore closure and grain growth are significant process parameters.

Keywords: firn, X-ray tomography, pore closure, in situ compaction

1 Introduction

2 Polar firn contains a large part of the history of atmospheric air in its
3 pores [1]. Thanks to the progressive densification of snow to ice, gases are
4 entrapped in firn, and progressively buried down in the ice core. This pro-
5 cess occurs by gradual pore closure with the age of gases being distributed
6 for a given depth. Thus, dating a particular atmospheric composition, and
7 relating it to changes in past climate temperature is challenging. In partic-
8 ular, improving the evaluation of the gas–ice age difference requires a better
9 understanding of firn densification and gas-trapping mechanisms.

10 From a climate point of view, the firn column can be divided into three
11 zones [2]. Top layers constitute a convective zone for air as snow is highly
12 permeable and subjected to wind. Below, gas transport occurs by molecular
13 diffusion and gravitational fractionation within the firn. When reaching the
14 lock-in depth (LID), gravitational fractionation stops [1], and gases enter the
15 lock-in zone (LIZ). Along these three zones, porosity is decreasing, which

16 leads to progressive closure and separation of pores to form individual closed
17 pores. At the close-off depth (COD), these pores are fully isolated from the
18 surface, i.e. air can no longer be pumped out from the firn.

19 These changes in gas transport mechanisms [originate](#) from different den-
20 sification mechanisms. First, snow undergoes grain rearrangement and sin-
21 tering until a density $\rho = 550 \text{ kg.m}^{-3}$ is reached. Further densification takes
22 place by viscoplastic deformation of grains. The order of magnitude of the
23 densification rate being typically between 10^{-12} and 10^{-9} s^{-1} depending of
24 the polar sites, Maeno and Ebinuma [3] proposed that deformation proceeds
25 by a combination of dislocation creep, which is the dominant mechanism,
26 and diffusion creep (boundary or lattice diffusion depending on the temper-
27 ature). The last stage of densification is porosity-related. When the density
28 exceeds about 840 kg.m^{-3} , pores are closed and further shrinking is driven by
29 the difference between their internal pressure and the larger external stress.

30 As pore closure is a very slow process, there is no direct observation of
31 this phenomenon in the field as it would take many decades to capture it.
32 However, ex situ characterizations of post mortem firn samples have been
33 performed, in particular with the use of X-ray tomography [4, 5, 6, 7]. This
34 enabled the amount of closed pores to be quantified and to relate it to gas
35 transport in the firn. The significant geometrical evolution of pores with
36 depth can also be characterized. Still, the closure process and its accom-
37 panying microstructural evolution can only be assumed in ex situ observa-
38 tions, while in situ observations in real time would give much more valuable
39 information on the phenomena at play. Therefore, in situ observations un-
40 der laboratory conditions provide very promising tests to study densification

41 mechanisms and pore closure within a reasonable time frame. To our best
42 knowledge, no in situ compaction of firn under X-ray microtomography has
43 been attempted yet. In other words, pore closure has not been followed in
44 situ during mechanical tests.

45 Mechanical tests on firn are relatively scarce. Some laboratory experi-
46 ments on dense snow and natural firn have been reported in the literature.
47 For instance, Landauer [8] performed uniaxial tests up to 630 kg.m^{-3} density
48 and recorded stresses of the order of 0.3 MPa for strain rates of $4 \cdot 10^{-4} \text{ s}^{-1}$.
49 Ebinuma and Maeno [9] carried out creep tests on denser laboratory snow
50 for various initial densities (above 550 kg.m^{-3} , similar to natural firn), and
51 went further than 850 kg.m^{-3} . They concluded that above a critical density,
52 which depends on the applied stress, dislocation creep governs deformation
53 [10]. Meussen et al. [11] offer a more recent and complete work on creep
54 experiments on natural firn with a large range of tested densities. Results
55 for densities of approximately 850 kg.m^{-3} exhibited less than 1% of strain
56 for an eighty-hour experiment under 0.5 MPa and at -20°C . For those large
57 densities (close to the close-off density), firn exhibited a similar behavior to
58 polycrystalline ice. In short, in these tests, applied stresses are about the
59 same order of magnitude than in the firn core (ranging from 0.1 to 0.7 MPa)
60 but those experiments were too lengthy to allow for X-ray tomography. These
61 results exemplify the range of mechanical responses of our material from the
62 extremely porous to the extremely dense firn and provide the necessary con-
63 ditions for displacement-controlled tests. Indeed, decreasing by a factor 10
64 the time of the experiment will lead to significantly larger stresses. Disloca-
65 tion creep is also expected to be the dominant microstructural mechanism

66 under such conditions.

67 Rolland Du Roscoat et al. [12] were the first to perform in situ X-ray
68 diffraction contrast tomography on snow samples under slow creep, allow-
69 ing grains to be differentiated from one another (with stresses lower than
70 0.14 MPa and initial densities lower than 500 kg.m^{-3}). These authors fol-
71 lowed the intragranular deformation of snow grains as small intragranular
72 disorientations were observed. These results emphasized that the anisotropic
73 viscoplasticity of ice crystals should be considered even for snow. Other com-
74 paction tests on snow also revealed that deformation is mainly controlled by
75 pressure sintering, but is also dependent on the density and on grain spe-
76 cific surface area [13, 14, 15]. Still, in situ studies of snow mostly consist of
77 following time lapse metamorphism for different types of snow [16] and with
78 controlled thermal gradients or other particular conditions [17, 18]. The ul-
79 timate goal is usually to compute snow effective properties [19] or model
80 snow behavior [20, 21, 22]. Moreover, curvature change is a key feature of
81 snow metamorphism and its investigation reveals underlying processes at
82 work [23, 24]. In particular, rounding and smoothing of snow grains along
83 with grain growth and specific surface area decrease, demonstrate time and
84 temperature influence [25, 26].

85 For firm typical densities (ranging from 550 kg.m^{-3} to 912 kg.m^{-3}), the
86 evolution of pore curvature, and time and temperature effects should be
87 noticeable in laboratory conditions (day-long experiment at -10°C). In this
88 work, we investigate the effects of time, temperature and densification-rates
89 on pore closure, microstructure evolution, and pore curvatures for large initial
90 densities ($\rho > 780 \text{ kg.m}^{-3}$). In particular, we [make an attempt to discern the](#)

91 effects of dislocation creep and diffusion creep by using two relevant firn
92 parameters: the closed porosity ratio and the connectivity index. To that
93 end, in situ compaction tests are performed on natural firn characterized by
94 X-ray tomography.

95 **Methods**

96 *Strategy*

97 This work aims at monitoring, in a time frame of a few hours, the densi-
98 fication of polar firn by X-ray micro-tomography. This means experimental
99 strain rates ($\approx 10^{-6}$ - 10^{-5}s^{-1}) six or seven orders of magnitude larger than
100 during natural densification in polar ice cap, as well as warmer temperature
101 (-10°C in laboratory instead of -55°C in the East Antarctica plateau for in-
102 stance). Power law creep is the dominant mechanism for such strain rates at
103 high temperature. Diffusion mechanisms and processes such as sintering or
104 grain growth are not expected to occur in an eight-hour experiment. To as-
105 sess the impact of the different mechanisms on firn, experimental tests and in
106 situ inspection were carried out. Ex situ characterization was first performed
107 and published in Burr et al. [7], thus providing a reference state for the firn
108 from Dome C (East Antarctica plateau)². The ex situ data set is composed
109 of 29 samples extracted from the same ice core at different depths, above and
110 below the close-off depth. Ex situ samples were not subjected to any treat-
111 ment. In contrast, we refer to in situ samples when samples extracted from
112 the ice core are thereafter mechanically tested or thermally treated. In situ

²Volsol ice core, grant ANR NT09-431976-VOLSOL)

Figure 1: The experimental strategy developed in this work. Ice age against density for Dome C [27] together with the proposed paths in the experimental procedure: mechanical densification under oedometric compaction at -10°C followed by ageing (heat treatment) at -2°C . Creep tests on firn are also performed at -2°C .

113 compacted samples are artificially densified at -10°C by means of an oedo-
 114 metric compaction device, while being characterized by X-ray tomography.
 115 Additionally, a few samples are kept at high temperature at -2°C for five
 116 months and analyzed afterward. A third set of in situ samples are submitted
 117 to creep also at -2°C for five months. This procedure is illustrated by the
 118 evolution of the ice core age with density in Fig. 1. The compaction test den-
 119 sifies the material, while the heat treatment at -2°C ages it. The creep tests
 120 attempt to couple stress and temperature for densification. The accompany-
 121 ing microstructures of these samples are compared with ex situ samples from
 122 the same density, corresponding to different firn depths: 79.87 m, 89.5 m and
 123 94.5 m.

124 *Compaction device*

125 A special cold cell was developed and adapted to a compaction device to
126 perform mechanical tests on the firn. A motor controls the vertical motion
127 of a piston via gears. A load cell is mounted under the piston and a software
128 enables the displacement of the piston to be controlled in a rather large
129 range of velocities (between 0.1 and 10 $\mu\text{m.s}^{-1}$). The cold cell is fixed on the
130 compaction device. It consists of a cold air input and a cover that bears the
131 load (Fig. 2). The sample is set between the piston and the cover. The cover
132 was designed to follow several specifications. It should sustain a sufficient
133 load, be thermally insulated and should moderately absorb X-rays.

134 Figure 2 shows the apparatus inside the laboratory tomograph. Cold air
135 flows through the cell at -10 °C thanks to the combination of an air dryer and
136 a cryostat. The diameter of firn samples are adjusted to the PMMA socket
137 with a lathe, in order to impose oedometric conditions before subsequent
138 treatment. These conditions are required to avoid issues that could occur for
139 such a granular material in uniaxial compression (grains may detach easily
140 during the compaction and lead to catastrophic failure). An oedometric test
141 is a compaction test, which reproduces satisfactorily the strain state imposed
142 in ice caps and soils, and which models in this work an artificial densification
143 of firn.

144 *Tested samples*

145 Ex situ and in situ samples are 12 mm in diameter and their height is in
146 the 20–30 mm range. All samples are characterized at -10 °C in an Easytom
147 XL nano tomograph from RX Solutions, at an acceleration voltage of 60 kV
148 and a target current of 166 μA . Scans have a helical geometry enabling the

Figure 2: The apparatus inside the laboratory tomograph with the compaction device highlighted in a red ellipse. The firn sample is shown inside its PMMA socket (oedometric conditions). Cold air flows in the cell at -10°C .

149 sample to be imaged on its full length. Multiple scans on the same ex situ
 150 sample have barely shown any effect of the X-rays and of the air flow on the
 151 pore shape nor density [28]. Voxel size is $12\mu\text{m}$ for ex situ conditions and
 152 $15\mu\text{m}$ for in situ conditions. After 3D image reconstructions with the filtered-
 153 back projection algorithm, all image analyses and parameter determination
 154 were carried out using the free software Fiji [29] combined with the plugin
 155 Analysis 3D [30]. To comply with the method proposed in Burr et al. [7], a
 156 cylindrical region of interest of 9.6 mm in diameter is extracted from volume

157 image after thresholding of the ice phase.

158 In situ compacted samples were tested at different displacement rates.
159 Tests were interrupted to scan samples at different densities and ensure a
160 stable microstructure. After 10 minutes of viscoplastic relaxation, a 25-
161 minute scan was performed, to avoid blurred 3D images. Interruptions of
162 tests roughly lead to a 30 % stress drop (see SI for a stress-time curve). Four
163 samples were successfully scanned during in situ compaction. They originate
164 from the same slice of the Dome C ice core at 79.92 m depth and are detailed
165 in Table 1. The nomenclature of samples relates to their depth in the ice
166 core. When samples 80₃-comp, 80₄-comp, 80₅-comp and 80₆-comp were in
167 oedometric conditions, the displacement rate of the piston was kept constant
168 leading to strain rates in the range $[2 \cdot 10^{-6} \text{s}^{-1}, 1.5 \cdot 10^{-5} \text{s}^{-1}]$. Displacement
169 rates were set so that there were twice the one of 80₃-comp for 80₄-comp and
170 80₆-comp, and three times larger for 80₅-comp. Note that strain rates above
171 $\approx 4 \cdot 10^{-5} \text{s}^{-1}$ lead to micro-cracks within the firm. Care was taken to ensure
172 a correct positionning of samples before the desired speed could be applied.
173 This delicate pre-positioning of samples could last up to 5 hours, leading to
174 a total compaction time of 8 to 10 hours. Digital volume correlation on grey
175 level images indicated that radial strain reached between 1 % and 2.5 % in
176 the first few hours of the test, corresponding approximately to an axial strain
177 $\varepsilon \approx 4 \%$ [28].

178 After compaction, samples are denoted with the suffix -comp. The par-
179 ticular sample 80₆ is submitted to two consecutive treatments: first a com-
180 paction (80₆-comp) and then a heat treatment (80₆-comp-temp). Table 1
181 lists the initial designations and densities of all tested samples as well as

Figure 3: Experimental strategy throughout this work. The red path denotes heat treatment with no applied stress. The green path denotes densification under mechanical compaction. The purple path denotes densification under creep for 5 months. Samples are denominated by their initial depth in the ice core. For example, sample 80_6 was drilled at 80 m depth, compacted (80_6 -comp) and finally submitted to heat treatment (80_6 -comp-temp).

182 their designations and densities after a 5 months heat treatment at -2°C (de-
 183 noted with a suffix -temp). These temperatures and duration were chosen to
 184 keep a reasonable time frame. Note that samples 80_2 and 90_2 crept during
 185 heat treatment as a load was applied on them (denoted with a suffix -creep,
 186 axial stress ≈ 0.3 , and ≈ 0.7 MPa, respectively), in contrast to samples 80_1
 187 and 90_1 which only aged at elevated temperature without external stress.
 188 The heat treatment was performed in two hermetic boxes with large hu-
 189 midity content to avoid sublimation (RH74% and RH99.9%, respectively).
 190 Samples inside their PMMA socket were also wrapped in plastic foils to limit

191 sublimation. Figure 3 illustrates the various treatments applied on samples
192 80 and 90.

193 *Microstructure*

194 After experiments, all in situ tomographic samples are cut in half (-comp,
195 -temp and -creep), and the thin section of each half is analyzed (typically
196 $\approx 25\mu m$). Microstructures and grain orientations are revealed thanks to po-
197 larized light using an Automatic Ice Texture Analyzer (AITA) [31]. Mean
198 grain size is then determined for each thin section by averaging the surface
199 area of all grains (using Fiji [29]). For ex situ samples, slices of ice core
200 retrieved at the same depth than in situ samples are analyzed with thin sec-
201 tions of 20x20 mm². Therefore the mean grain size of ex situ samples is more
202 statistically representative than for in situ tomographic samples (algorithm
203 for image analyses and evolution with depth of the ex situ microstructure
204 and c axis orientations are given in Burr [28]).

Ex situ samples			In situ samples					
Initial name	Depth (m)	ρ_k (kg.m ⁻³)	State 1	Final strain rate (s ⁻¹)	Final stress or dead load (MPa)	ρ_1 (kg.m ⁻³)	State 2	ρ_2 (kg.m ⁻³)
95 ₀	94.5	829.7						
80 ₁	79.87	786.3	80 ₁ -temp		0	784.8		
80 ₂	79.92	794.1	80 ₂ -creep		≈ 0.3	810.3		
80 ₃	79.92	797.8	80 ₃ -comp	$3.4 \cdot 10^{-6}$	≈ 3	825.1		
80 ₄	79.92	785.0	80 ₄ -comp	$7.6 \cdot 10^{-6}$	≈ 4.8	867.8		
80 ₅	79.92	784.8	80 ₅ -comp	$9.3 \cdot 10^{-6}$	≈ 5	852.2		
80 ₆	79.92	-	80 ₆ -comp	$\approx 4 \cdot 10^{-6}$	≈ 5.7	828.3	80 ₆ -comp-temp	825.6
90 ₁	89.5	822.3	90 ₁ -temp		0	823.6		
90 ₂	89.5	821.0	90 ₂ -creep		≈ 0.7	829.5		

Table 1: Nomenclature of samples. Ex situ samples are reference states, used as initial states of in situ samples (before any treatment). In situ samples are samples submitted to a treatment. Denotations -comp, -temp and -creep refer respectively to in situ compacted samples at -10 °C, in situ heat treated samples at -2 °C and samples that crept at -2 °C. Sample 80₆ is first compacted at -10 °C and then heat treated at -2 °C, hence is denoted -comp-temp. Samples 80_{3,4,5} are scanned regularly throughout compaction and state 1 refers to their final strain step. Though strain rates and stresses evolve during compaction tests, only the final ones are shown (see supplementary file for curves). Samples come from a slice of Dome C ice core from depths in the range 80 to 95 meters.

205 *Curvature*

206 Curvature is supposed to be the driving force of snow metamorphism
 207 (surface energy minimization). On 3D images, it is usually described with
 208 the mean and Gaussian curvatures [32, 23]. Here we take advantage of two
 209 other measures of local shape introduced by Koenderink and Doorn [33]. The
 210 shape index is a measure of the local shape that also differentiates convexities
 211 from concavities. It ranges from -1 to $+1$. The curvedness is a positive value
 212 that quantifies the amount of curvature of the shape. It differentiates well
 213 flat surfaces from acute shapes for instance. These measures of shape were
 214 successfully used in the metallurgy community, to quantify dendritic growth
 215 [34] or to compare dendrites depending on the solidification method used
 216 [35].

217 In the following, curvatures are investigated in the porosity reference
 218 framework, such that the surface of an entrapped spherical bubble is con-
 219 sidered convex. This is important as we adopt the convention $\kappa_1 > \kappa_2$ for
 220 principal curvatures. The shape index S writes :

$$S = \frac{2}{\pi} \arctan \left(\frac{\kappa_1 + \kappa_2}{\kappa_1 - \kappa_2} \right) \quad (1)$$

221 Regarding the curvedness C , it is equal to 0 for a perfectly planar surface
 222 (infinite curvature radius), and increases as the curvature radius decreases.
 223 Its formulation is:

$$C = \sqrt{\frac{\kappa_1^2 + \kappa_2^2}{2}} \quad (2)$$

224 Local principal curvatures are computed with the software Avizo [36].
 225 The generation of the surface mesh implies the choice of a mesh size. It is
 226 achieved by selecting a smoothing parameter in Avizo (set to 3). For the

227 actual calculation of curvatures, the user also has to determine how many
 228 neighbors are taken into account for the local curvature. Using the ten closest
 229 neighbors for local curvatures leads to about 10 to 20 minutes of computation
 230 time (Burr [28] for more details). The computation of the curvedness and
 231 the shape index gives a 2D histogram which can be interpreted with a scaled
 232 probability density $P(S, C)$. In the following, we chose a normalization such
 233 that $P(S_{max}, C_{max})=1$ which means that the local surface characterized by
 234 the corresponding values S_{max} and C_{max} is the most abundant in the sample.

235 *Initial state - ex situ samples*

236 Figure 4 shows microstructural information for samples from 80 and 90 m
 237 depths at their initial state. Figure 4 is used as a reference state for com-
 238 parison with in situ samples. The classification of shapes depending on the
 239 value of S in the range $[-1; 1]$ is also exemplified as layouts in Fig. 4. For
 240 instance, a perfectly spherical pore (cap-like shape) would have a shape index
 241 equal to 1, and a perfectly cylindrical pore (ridge-like shape) would have a
 242 shape index equal to 0.5. Figure 4b1 shows more closed pores (illustrated in
 243 red) than Fig. 4a1 as the sample was taken 10 meters deeper. In both cases,
 244 pores are usually rounded with smooth surfaces (Fig. 4a1-b1), as depicted by
 245 S-C maps in Fig. 4a2-b2. Indeed, their shape span through the saddle-ridge
 246 and the dome-like shape (between a ridge and a cap). Looking at the most
 247 abundant local surface in the sample, for which $P(S_{max}, C_{max})=1$, one can
 248 also distinguish the small shift of S_{max} towards increasing S between samples
 249 coming from 80 meters ($S_{max} < 0.5$) and 90 meters depth ($S_{max} > 0.5$). Vari-
 250 ation of the curvedness is relatively low, with the same horizontal shift of the
 251 maximum curvedness than for S_{max} . Grains are quite equiaxed and depth

clearly impacts grain size: 90 m deep samples exhibit larger grains than 80 m deep samples (left hand side of Fig. 4a3-b3). In fact, firn grain size increases monotonously with depth in Dome C firn [28, 7]. Finally, orientations of **c** axes towards the ice core direction (loading direction) are already discernible for all samples from 80 m and 90 m (see right hand side of Fig. 4a3-b3).

Results

Closed porosity ratio and connectivity index

Pore closure evolution has been classically investigated by using the closed porosity ratio [38, 39, 40, 41]. Recently, we have introduced a parameter defined as the connectivity index, which was found more reliable than the closed porosity ratio to predict the close-off depth and density [7]. The connectivity index is defined by the ratio of the volume of the largest pore to the total pore volume of a sample, and its quantification is less dependent on the analyzed volume, heterogeneities and voxel size than the closed porosity ratio. The closed porosity ratio is defined by the ratio of the closed pore volume to the total pore volume. They both work reversely and vary between 0 and 100 %, but the closed porosity ratio is fully dependent of the status of pores (close or open), which is sensitive for the small volumes typically obtained with X-ray tomography in this work. Its quantification is carried out at the scale of the sample and depends on the cut-pore ratio. It does not give values that can be used to characterize a firn layer. That being said, both parameters are calculated and discussed here.

Figure 5a shows the evolution of the closed porosity ratio with density and Figure 5b shows the connectivity index for both types of samples. Ex

Figure 4: Typical initial microstructures and references for samples (a) 80_k and (b) 90_k (with k ranging from 1 to 6 for samples 80_k and being 1 or 2 for samples 90_k). Layouts are shown as reading guides. Visualization of open pores (white) and closed pores (red) in a subvolume (a1),(b1) (side view $6.72 \times 6.72 \text{ mm}^2$). (a2),(b2) Curvature maps. (a3),(b3) Left hand side : thin sections observed by polarized light illustrating grain size. Right hand side : c axis reconstruction of the same thin section using the AITA python toolbox [37] (pores and reconstructed pixels with low quality are in white).

Figure 5: Evolution of the closed porosity ratio (a) and of the connectivity index (b) with density for ex situ and in situ samples. Blue dots depict ex situ samples [7], and green crosses, diamonds and squares depict all strain steps of in situ compacted samples 80_{3,4,5}-comp. Blue and green dashed lines serves only to guide the eyes. All other in situ samples are displayed by arrows that link their initial states to their final states. Different steps of compaction of sample 80₄-comp are also visualized with open pores in white and closed pores in red (with $\rho_A = 785.0 \text{ kg.m}^{-3}$, $\rho_B = 839.8 \text{ kg.m}^{-3}$ and $\rho_C = 867.8 \text{ kg.m}^{-3}$). See Fig. 3 and Table 1 for sample color and nomenclature, and supplementary for full in situ visualization of sample 80₄-comp.

276 situ points from Burr et al. [7] and their trend have been added in blue, while
 277 in situ compacted samples are displayed in green. The red and purple colors
 278 correspond respectively to in situ -temp and -creep samples along with their
 279 initial states. All points linked by an arrow are the same extracted subvol-
 280 umes at different states. However, to avoid superimposing arrows, samples
 281 80_{3,4,5}-comp and their intermediary steps are displayed by green crosses, di-
 282 amonds and squares, respectively. When required for analyses, error bars
 283 have been added. These were calculated by investigating the effects of image
 284 processing, voxel size, sample size and pixel connectivities on the studied
 285 parameters [7]. Relative errors are 0.15 % for ρ , 3.1 % for the closed porosity
 286 ratio and 0.11 % for the connectivity index. In situ points have been obtained
 287 with very different strain rates, but interestingly they all fall on a master
 288 curve (displayed by a green dashed line) for the closed porosity ratio and the
 289 connectivity index. For any given density, the fraction of closed porosity is
 290 larger for ex situ samples. For instance, at 830 kg.m⁻³, approximately 24.4 %
 291 of pores are closed for ex situ samples, whereas for in situ compacted samples
 292 there are only 4.5 % of closed pores. Accordingly, the connectivity index is
 293 the largest for in situ compacted samples. In other words, pores are closing
 294 at larger densities for in situ compacted samples than for ex situ samples.
 295 Tomographic images Fig. 5 illustrate this pore closure process. Note that
 296 the minimum value obtained for the connectivity index is 9.3 % with a den-
 297 sity value of 867.8 kg.m⁻³ (sample 80₄-comp was the most compacted one
 298 at the end of the experiment). We have shown that the close-off depth and
 299 its corresponding density could be predicted from the abrupt drop and the
 300 final plateau of the connectivity index curve (see Burr et al. [7] for details

301 on the determination procedure). Here, the final plateau is not reached at
 302 840 kg.m^{-3} for in situ samples, meaning that percolation is still possible after
 303 compaction up to at least 870 kg.m^{-3} . This means that using density as the
 304 only indicator of the close-off is not correct. Microstructure and densification
 305 history also play an important role in determining close-off. It is likely this
 306 microstructure effect is partly taken into account when using the temperature
 307 of a polar site as a determination parameter for the air volume entrapped in
 308 closed pores (e.g. Martinerie et al. [42]).

309 Figure 5 also displays the closure of pores after heat treatment (red ar-
 310 rows). In fact, a green arrow representing compaction, links 80_6 to 80_6-comp ,
 311 illustrating a density increase without closing much pores. But, from 80_6-
 312 comp to 80_6-comp-temp , the closed porosity ratio increases from 3.3 to 6.7 %
 313 while the density only slightly decreases. In contrast to the closed poros-
 314 ity ratio, the connectivity index drops from 74 to 63 %. Depending on the
 315 conditions they were submitted to, samples exhibit different trends. Indeed,
 316 the density stayed constant within error bars between 80_1 and 80_1-temp , or
 317 between 90_1 and 90_1-temp , but an increase of the closed porosity ratio is only
 318 noticed between 90_1 and 90_1-temp . Creep tests on samples 80_2 and 90_2 both
 319 exhibit an increase in density. However, only the creep test on the deepest
 320 sample do close some pores (90_2-creep), while they do not for sample 80_2-
 321 creep . Consistently, the connectivity index decreases for sample 90_2-creep
 322 and stagnates for sample 80_2-creep . Pore size being rather large for sample
 323 80_1 , the 5 months heat treatment was not sufficient to have pore channels dis-
 324 connecting from one another, compared to sample 90_1 . In brief, compaction
 325 tests lead to an increase in density without disconnecting much pores, while

326 increasing temperature tends to separate pores for a given density.

327 Visual inspection of 3D views (see Fig. 4-5-6) gives valuable qualitative in-
328 formation on the porous structure. However, in the following section, a more
329 precise evaluation of the morphological differences is realized by quantifying
330 these changes in terms of curvatures of pores.

331 *Curvature*

332 Figure 6 shows a close-up view within the porosity networks for different
333 samples of approximately the same density ($\rho = 828 \pm 2 \text{ kg.m}^{-3}$), but re-
334 sulting from different process histories. The separation of globular portion
335 of pores is highlighted in blue for samples 80₆-comp and 80₆-comp-temp.
336 Blue ellipses point to sharp dead ends in Fig. 6b1 that appear smaller and
337 rounder in Fig. 6c1. Green ellipses highlight thin channels (Fig. 6b1) that
338 have disappeared after heat treatment (Fig. 6c1). The accentuated curvature
339 after compaction test is illustrated by zoom-ins on the pore network. Sam-
340 ples 95₀ (Fig. 6a2) and 90₂-creep (Fig. 6d2) do not exhibit marked curved
341 pores, whereas sample 80₆-comp (Fig. 6b2) exhibits sharp pore shapes, char-
342 acterized by a larger curvedness on pore edges. After heat treatment, sam-
343 ple 80₆-comp-temp exhibit smoother edges as emphasized by red ellipses in
344 Fig. 6c2. The curvedness clearly shows that the sharp shapes and pointy
345 dead-ends are starting to round. Concavities also seem to be reduced and
346 some pore channels are disappearing, thus indicating pore closure during heat
347 treatment.

348 This morphological evolution of pores is confirmed quantitatively by cur-
349 vature maps in Figs. 6a3 to d3. As observed on 3D images, the ex situ sample
350 95₀ depth is very similar to 90₂-creep, which was crept at -2 °C during five

Figure 6: Comparison of pore morphologies (rows 1-2) and local curvatures (rows 2-3) for samples of approximately equal density ($\rho = 828 \pm 2 \text{ kg.m}^{-3}$) but resulting from different process histories. (a) 95₀, (b) 80₆-comp, (c) 80₆-comp-temp and (d) 90₂-creep. Blue and green ellipses highlight pore closure processes. Red ellipses indicate the smoothing of pores after heat treatment. **White ellipses emphasize isolines around $P(S,C) \approx 0.5$.** See Table 1 for sample nomenclature.

351 months. Most of the local surfaces have shapes between the saddle-ridge and
 352 the dome, with low curvedness. In comparison to Fig. 4a2 and Fig. 4b2,
 353 the distributions of local shapes of those samples (95₀, 80₆-comp, 80₆-comp-
 354 temp and 90₂-creep) are slightly shifted towards the cap-like shape ($S = 1$)
 355 as samples come from a deeper slice (94.5 m instead of 79.92 m) or are denser
 356 (829 kg.m^{-3} instead of 785 kg.m^{-3}).

357 In contrast to Fig. 4a2-b2, Fig. 6b3 displays a larger distribution of local
 358 pore shapes after compaction. In addition, this [curvature map \(80₆-comp,](#)
 359 [Fig. 6b3\)](#) looks very similar to all other in situ compacted samples (80₃-comp,
 360 [80₄-comp and 80₅-comp](#)). Negative values are much more prevalent than for
 361 Figs. 6a3 (95₀) and 6d3 (90₂-creep), indicating a non-negligible proportion
 362 of concavities. This is due to the flattening of pores during the compaction.
 363 Similarly to Fig. 6c3 (80₆-comp-temp), the dominant local shape of Fig. 6b3
 364 (80₆-comp) is the cylinder as S_{max} is centered around $S = 0.5$. The distri-
 365 bution is more widespread for 80₆-comp than for 80₆-comp-temp, but the
 366 most striking difference comes from the curvedness. [The maximum curved-](#)
 367 [ness value of the isoline \$P\(S,C\)=0.5\$ is roughly 14% larger after compaction](#)
 368 [than after heat treatment, as pointed out by white ellipses on Fig. 6.](#) This
 369 is a quantitative assessment of the evolution of the acute shapes observed
 370 in Fig. 6b2 (80₆-comp) towards the smoother shapes of pores observed in
 371 Fig. 6c2 (80₆-comp-temp).

372 *Grain size*

373 Compaction tests also impacts ice grains according to [microstructural](#)
 374 [analyses.](#) In fact, Fig. 7 focuses on four microstructures representing different
 375 [firn states \(ex situ reference state, in situ -comp, -comp-temp and -creep\).](#)

376 Note that we have access to the grain size only at the end of the in situ
 377 experimental tests. The ex situ sample 95₀ exhibits a larger mean grain
 378 size than the in situ compacted sample 80₅-comp (see Fig. 7a1 and 7b1).
 379 Overall, the mean grain size of an ex situ sample is always larger compared
 380 to the mean grain size of an in situ compacted sample with the same or larger
 381 density. This is simply because of the duration of the compaction experiment,
 382 which is too short to notice any grain growth. The mean grain size of sample
 383 80₃-comp is similar to its value before compaction (if compared to Fig. 4a3,
 384 $\langle \bar{A} \rangle = 0.82 \text{ mm}^2$), but the final strain was small ($\varepsilon = 5.90 \%$). It has a lower
 385 value around 0.40 mm^2 for samples 80₄-comp and 80₅-comp (with standard
 386 deviations equal to the mean grain size for all samples). The decrease in
 387 mean grain size for samples 80₄-comp and 80₅-comp could be consistent with
 388 dynamic recrystallization which can be expected for the temperature and
 389 strain-rate conditions imposed here [43]. Dynamic recrystallization would
 390 also explain the many small grains observed in Fig. 7b1. Moreover, features
 391 of dynamic recrystallization have been shown even for ex situ samples [28],
 392 and more generally in some firn cores [44]. However, the size of the thin
 393 sections ($L = 18 \text{ mm}$, $l = 8 \text{ mm}$) for these in situ samples was small with
 394 less than a hundred grains, possibly affecting representativeness. In any case,
 395 grain growth is excluded in these eight-hour experiments. The study of the
 396 **c** axis orientations in the Dome C firn (ex situ samples) shows the initial
 397 development of fabric with the **c** axes becoming aligned with the vertical -
 398 core axis (see full fabric analysis for ex situ samples in Burr [28]). In the
 399 case of the compaction test 80₅-comp (Fig. 7b2), the fabric is a little more
 400 pronounced than the ex-situ sample 95₀ (Fig. 7a2). This is coherent with a

Figure 7: Comparison of the microstructures and **c** axis orientations of (a1)-(a2) 95₀, (b1)-(b2) 80₅-comp, (c1)-(c2) 80₆-comp-temp and (d1)-(d2) 90₂-creep.

401 preferential mechanism of dislocation creep during in situ compaction test.
 402 However, asserting sample texture is not appropriate when only a few grains
 403 are observable as for the 90₂-creep sample for instance (Fig. 7d1). In any
 404 case, quantifying the rotation of **c** axes and the strength of the fabric is
 405 beyond the scope of this work.

406 Despite the rather high storage temperature at -2 °C, no noticeable dif-
 407 ferences were observed when comparing samples 80₁-temp and 90₁-temp to

408 their reference states Fig. 4a3 and Fig. 4b3 respectively. Thus, there are
409 not shown. However samples 80₂-creep and 90₂-creep (Fig. 7d1) that were
410 crept at -2°C for five months, have a larger mean grain size than initially
411 (compare Fig. 7d with Fig. 4b3), but also larger than those only heat treated
412 (80₁-temp and 90₁-temp) or than ex situ sample of the same density 95₀
413 (compare Fig. 7d1 and Fig. 7a1). As for sample 80₆-comp-temp (Fig. 7c1),
414 features of recrystallization are hardly noticeable, and its grain size is similar
415 to the one shown in Fig. 4a3. It is difficult to evaluate in which proportion
416 dynamic recrystallization occurred.

417 Discussion

418 In situ characterization revealed large differences with ex situ results both
419 for pore morphology and for pore closure. In particular, during in situ com-
420 paction tests, rate dependency was observed, with stresses about one order
421 of magnitude larger than in firn cores (from which the samples are taken).
422 The strain rates used in these tests are also large compared to polar firn
423 densification rates (about 6 orders of magnitude larger). The durations in-
424 volved during in situ tests are much too short for slow thermally activated
425 processes to occur. The eight-hour in situ experiment represents about a
426 thousand years of densification in Dome C for the same reached density. It is
427 not possible to reproduce grain growth and sintering during this short time.
428 The differences in closed porosity ratio and connectivity index observed be-
429 tween ex situ and in situ compacted samples come from this rate dependency.
430 Thus, our tests decoupled the effect of viscoplastic deformations but thereby
431 excludes also potential dynamic feedbacks between diffusion processes and

432 [dislocation glide](#). According to the sharpening of pore morphology, texture
433 observations and strain rates, dislocation creep was the dominant mechanism
434 during compaction. It has been previously shown that such mechanism oc-
435 curs essentially by basal glide for ice and is strongly anisotropic (see Burr
436 et al. [45], Chauve et al. [46] for recent updates on that subject).

437 [To counteract these differences, some in situ samples were kept at -2 °C.](#)
438 Figures 5 and 6 prove that high temperature and time facilitate pore closure
439 and pore rounding. But, closure also depends on the initial microstruc-
440 ture. Indeed, between samples 90₁ and 90₁-temp, curvature variations are
441 small. These are much less important than for samples 80₆-comp and 80₆-
442 comp-temp for instance. Therefore, surface minimization (defined by the
443 difference between the mean curvature and the local curvature), which is
444 one of the driving forces for pore closure is different for these samples. De-
445 spite this difference, closure occurs for both 90₁-temp and 80₆-comp-temp
446 samples (Fig. 5). We have shown that closure was heterogeneous between
447 approximately 87 m and 98 m for ex situ firn due to the presence of small
448 pore channels [7]. This means that closure is particularly accelerated in a
449 certain range of depths, namely for a particular type of microstructure. More
450 samples should be tested to reach a firmer conclusion on this topic.

451 Sample 80₆-comp-temp (Fig. 7c1), [which was compacted and then heat](#)
452 [treated](#), exhibits a grain morphology surprisingly close to 80₁-temp (only
453 heat treated), [thus close to its reference state Fig. 4a3](#). However, in agree-
454 ment with the anisotropic plasticity, the **c** axes of 80₆-comp-temp are still
455 more aligned with the loading direction (Fig. 7c2). For all compacted sam-
456 ples (80₃-comp, 80₄-comp, 80₅-comp and 80₆-comp-temp), no clear sign of

457 recrystallization is noticed.

458 Indeed, in situ compaction experiments are rather fast ($>10^{-6} \text{ s}^{-1}$) with
459 large stresses (from 1 MPa to 5 MPa, [see SI](#)) while the creep experiment
460 is closer to what Chauve et al. [47] carried out, with stresses smaller than
461 1 MPa. However, crystal nucleation along grain boundaries such as necklaces
462 of grains is not observed for any of our experiments contrary to what these
463 authors observed in columnar ice [47]. Capolo [48] also observed drastic
464 recrystallization after a few hours of creep under $\approx 0.3 \text{ MPa}$ stress on ice
465 tricrystal. Serrated grain boundaries (evidence of grain boundary migration)
466 are also not noticed in contrast to observations from Montagnat et al. [49] or
467 Capolo [48]. However resolution of our images may hinder close inspection.
468 Examination of thin sections reveals that there are less small grains for sample
469 80₆-comp-temp than for ex situ and in situ compacted samples. This could
470 mean an effective annealing of the sample excluding nucleation or rotation
471 recrystallization. Nevertheless, we would have expected grains to grow in
472 that case and we do not see a particularly marked increase in grain size.
473 Again, the small number of observable grains calls for caution.

474 Samples 80₂-creep and 90₂-creep reached vertical strains of $\approx 6.6\%$ and
475 11.9% , respectively. Therefore secondary creep should be reached, and dy-
476 namic recrystallization should take place [49, 50]. In addition, as the grain
477 size is larger than initially, it is likely that high temperature also activated
478 diffusion mechanism during creep. Meanwhile, according to the very few
479 grains observed, the **c** axes of these samples do show a quite strong girdle
480 textures (stronger for 90₂-creep [shown in Fig. 7d2](#) than for 80₂-creep). This,
481 combined with the microstructure of Fig. 7d1 reminds of grain boundary mi-

gration mechanism, which happens in the deepest part of ice cores when large grains interlock as dynamic recrystallization proceeds [51, 52, chap. 6]. High-resolution images and precise measurements of the orientation with the help of EBSD should enhance our comprehension of the underlying mechanisms [49, 47].

Conclusion

In this paper, we presented three different experiments in order to discern the mechanisms of densification of polar firn. In situ oedometric compaction tests of Dome C firn were carried out under X-ray at -10°C during a few hours. It was possible to characterize the evolution of pore curvature during densification and to follow the closure of pores. The connectivity index appears to be a good indicator of pore closure phenomena as it is not dependent of cut pores at the sample boundary as is the closed porosity ratio [7]. Since, by construction, lab experiments will never be able to reproduce the evolution on climatological time scales, it is important to exploit the rate-temperature equivalence as much as possible for aiming at it. Therefore, the second experiment consisted in studying the effect of temperature on firn through heat treatment (ageing at -2°C) during a few months. The third one involved creep tests, i.e. compaction of firn performed at high temperature (-2°C) during 5 months to combine the two previous experiments.

It is reported here that in situ compaction at high strain rates (between $2 \cdot 10^{-6} \text{ s}^{-1} < \dot{\epsilon}_z < 1.5 \cdot 10^{-5} \text{ s}^{-1}$, larger than natural strain rates in Antarctica) results in densification with a reduced volume of closed pores compared to the natural conditions of the ice cap for the same firn density. In addition,

506 the connectivity index decreases, but at higher densities than for the ex
507 situ profile. On the contrary, heat treatments lead to pore closure without
508 significantly changing the density. Creep tests allow to combine at the same
509 time the densification and the closure of pores.

510 These experiments highlight that densification of firn in ice cap activates
511 both physical mechanisms: it is an interplay between dislocation glides and
512 diffusion processes. The dislocation glides lead to intragranular deformation
513 with $\mathbf{c}$ axis orientation evolution (the ice viscoplastic behavior is strongly
514 anisotropic) and to an increase in density.

515 The second mechanism leads to atomic diffusion, especially on the sur-
516 face, which makes it possible, for example, to smooth pores by minimizing
517 curvature and increasing the size of the grains. This last mechanism closes
518 also some pores but it does not seem to change density in the range investi-
519 gated in our experiments. Therefore, microstructure evolution of polar firn
520 should depend on the history of each of the parameters influencing the local
521 flow of ice cap. In return, the microstructure and not only the density should
522 ultimately influence the capture of gases. In conclusion, we believe that the
523 anisotropy of ice viscoplasticity should be considered in polar firn densifi-
524 cation models as it would better represent ice crystal deformation than the
525 common use of the isotropic power law of ice [e.g., 53], and may ultimately
526 give different past climate reconstructions. It is also noteworthy that pore
527 closure could be modeled by curvature minimization given appropriate diffu-
528 sion coefficient. The physical model proposed by Flin et al. [24, 54] for snow
529 metamorphism could be adapted for firn. This would help understand coa-
530 lescence or separation of pores. It would also offer an alternative approach to

531 the classical use of empirical parameterizations of the closed-porosity ratio
532 when modeling gas transport in firn [55].

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543 References

- 544 [1] M. Battle, M. L. Bender, T. Sowers, P. P. Tans, J. Butler, J. Elkins,
545 J. Ellis, T. J. Conway, N. Zhang, P. Lang, A. Clarke, Atmospheric gas
546 concentrations over the past century measured in air from firn at the
547 South Pole, *Nature* 383 (1996).
- 548 [2] T. Sowers, M. Bender, D. Raynaud, Y. Korotkevich, $\delta^{15}\text{N}$ of N_2 in
549 air trapped in polar ice: A tracer of gas transport in the firn and a
550 possible constraint on ice age-gas age differences, *Journal of Geophysical*
551 *Research: Atmospheres* 97 (1992) 15683–15697.

- 552 [3] N. Maeno, T. Ebinuma, Pressure Sintering of Ice and Its Implication
553 to the Densification of Snow at Polar Glaciers and Ice Sheets, *Journal*
554 *Physical Chemistry* 169 (1983) 4103–4110. doi:10.1021/j100244a023.
- 555 [4] S. A. Gregory, M. R. Albert, I. Baker, Impact of physical properties and
556 accumulation rate on pore close-off in layered firn, *Cryosphere* 8 (2014)
557 91–105. doi:10.5194/tc-8-91-2014.
- 558 [5] R. Lomonaco, M. Albert, I. Baker, Microstructural evolution of fine-
559 grained layers through the firn column at Summit, Greenland, *Journal*
560 *of Glaciology* 57 (2011) 755–762. doi:10.3189/002214311797409730.
- 561 [6] C. F. Schaller, J. Freitag, O. Eisen, Gas enclosure in polar firn follows
562 universal law, *Climate of the Past Discussions* 1 (2017) 1–12.
- 563 [7] A. Burr, C. Ballot, P. Lhuissier, P. Martinerie, C. L. Martin, A. Philip,
564 Pore morphology of polar firn around closure revealed by x-ray
565 tomography, *The Cryosphere* 12 (2018) 2481–2500. doi:10.5194/
566 tc-12-2481-2018.
- 567 [8] J. K. Landauer, The Creep of Snow under Combined Stress, *Journal of*
568 *Rheology* 2 (1958) 175. doi:10.1122/1.548829.
- 569 [9] T. Ebinuma, N. Maeno, Experimental studies on densification and
570 pressure-sintering of ice, *Annals Of Glaciology* 6 (1985) 83–86. doi:10.
571 3189/1985AoG6-1-83-86.
- 572 [10] T. Ebinuma, N. Maeno, Particle Rearrangement and dislocation creep
573 in a snow-densification process, *Le Journal de Physique Colloques* 48
574 (1987) C1–263–C1–269. doi:10.1051/jphyscol:1987137.

- 575 [11] B. Meussen, O. Mahrenholtz, H. Oerter, Creep of polar firn, Cold
576 Regions Science and Technology 29 (1999) 177–200.
- 577 [12] S. Rolland Du Roscoat, A. King, A. Philip, P. Reischig, W. Ludwig,
578 F. Flin, J. Meyssonier, Analysis of Snow Microstructure by Means of X-
579 Ray Diffraction Contrast Tomography, Advanced Engineering Materials
580 13 (2011) 128–135. doi:10.1002/adem.201000221.
- 581 [13] X. Wang, I. Baker, Observation of the microstructural evolution of
582 snow under uniaxial compression using X-ray computed microtomogra-
583 phy, Journal of Geophysical Research: Atmospheres 118 (2013) 12,371–
584 12,382. doi:10.1002/2013JD020352.
- 585 [14] S. Schleef, H. Löwe, X-ray microtomography analysis of isothermal den-
586 sification of new snow under external mechanical stress, Journal of
587 Glaciology 59 (2013) 233–243. doi:10.3189/2013JoG12J076.
- 588 [15] S. Schleef, H. Löwe, M. Schneebeli, Hot-pressure sintering of low-density
589 snow analyzed by X-ray microtomography and in situ microcompression,
590 Acta Materialia 71 (2014) 185–194. doi:10.1016/j.actamat.2014.03.
591 004.
- 592 [16] M. Schneebeli, S. A. Sokratov, Tomography of temperature gradient
593 metamorphism of snow and associated changes in heat conductivity,
594 Hydrological Processes 18 (2004) 3655–3665. doi:10.1002/hyp.5800.
- 595 [17] P. P. Ebner, M. Schneebeli, A. Steinfeld, Tomography-based monitor-
596 ing of isothermal snow metamorphism under advective conditions, The
597 Cryosphere 9 (2015) 1363–1371. doi:10.5194/tc-9-1363-2015.

- 598 [18] K. Hammonds, R. Lieb-lappen, Z. Courville, A. Song, X. Wang, I. Baker,
599 Laboratory investigations on the thermophysical properties of the ice-
600 snow interface while under a controlled temperature gradient, in: Pro-
601 ceedings, International Snow Science Workshop, Banff, 2014, pp. 35–42.
- 602 [19] N. Calonne, F. Flin, C. Geindreau, B. Lesaffre, S. Rolland du Roscoat,
603 Study of a temperature gradient metamorphism of snow from 3-D
604 images : time evolution of microstructures , physical properties and
605 their associated anisotropy, The Cryosphere 8 (2014) 2255–2274.
606 doi:10.5194/tc-8-2255-2014.
- 607 [20] C. Chandel, P. K. Srivastava, P. Mahajan, Micromechanical analysis
608 of deformation of snow using X-ray tomography, Cold Regions Science
609 and Technology 101 (2014) 14–23. doi:10.1016/j.coldregions.2014.
610 01.005.
- 611 [21] A. Wautier, C. Geindreau, F. Flin, Linking snow microstructure
612 to its macroscopic elastic stiffness tensor: A numerical homogeniza-
613 tion method and its application to 3-D images from X-ray tomogra-
614 phy, Geophysical Research Letters 42 (2015) 8031–8041. doi:10.1002/
615 2015GL065227.
- 616 [22] A. Wautier, C. Geindreau, F. Flin, Numerical homogenization of the
617 viscoplastic behavior of snow based on X-ray tomography images, The
618 Cryosphere 11 (2017) 1465–1485.
- 619 [23] F. Flin, J. B. Brzoska, B. Lesaffre, C. Coléou, R. A. Pieritz, Three-
620 dimensional geometric measurements of snow microstructural evolution

- 621 under isothermal conditions, *Annals of Glaciology* 38 (2004) 39–44.
622 doi:10.3189/172756404781814942.
- 623 [24] F. Flin, J. B. Brzoska, The temperature-gradient metamorphism
624 of snow: Vapour diffusion model and application to tomographic
625 images, *Annals of Glaciology* 49 (2008) 17–21. doi:10.3189/
626 172756408787814834.
- 627 [25] T. U. Kaempfer, M. Schneebeli, Observation of isothermal metamor-
628 phism of new snow and interpretation as a sintering process, *Journal of*
629 *Geophysical Research* 112 (2007) D24101. doi:10.1029/2007JD009047.
- 630 [26] M. Kerbrat, B. Pinzer, T. Huthwelker, H. W. Gäggeler, M. Ammann,
631 M. Schneebeli, Measuring the specific surface area of snow with X-
632 ray tomography and gas adsorption: comparison and implications for
633 surface smoothness, *Atmospheric Chemistry and Physics Discussions* 7
634 (2007) 10287–10322. doi:10.5194/acpd-7-10287-2007.
- 635 [27] D. Veres, L. Bazin, A. Landais, H. Toyé Mahamadou Kele, B. Lemieux-
636 Dudon, F. Parrenin, P. Martinerie, E. Blayo, T. Blunier, E. Capron,
637 et al., The Antarctic ice core chronology (AICC2012): An optimized
638 multi-parameter and multi-site dating approach for the last 120 thou-
639 sand years, *Climate of the Past* 9 (2013) 1733–1748. doi:10.5194/
640 cp-9-1733-2013.
- 641 [28] A. Burr, Investigation of pore closure during polar firn densification,
642 Ph.D. thesis, Université Grenoble Alpes, 2017. URL: [https://tel.](https://tel.archives-ouvertes.fr/tel-01725405)
643 [archives-ouvertes.fr/tel-01725405](https://tel.archives-ouvertes.fr/tel-01725405).

- 644 [29] J. Schindelin, I. Arganda-carreras, E. Frise, V. Kaynig, T. Pietzsch,
645 S. Preibisch, C. Rueden, S. Saalfeld, B. Schmid, J.-y. Tinevez, D. J.
646 White, V. Hartenstein, P. Tomancak, A. Cardona, Fiji - an Open Source
647 platform for biological image analysis, *Nature Methods* 9 (2013). doi:10.
648 1038/nmeth.2019.Fiji.
- 649 [30] V. Boulos, V. Fristot, D. Houzet, L. Salvo, P. Lhuissier, Investigat-
650 ing performance variations of an optimized GPU-porting granulome-
651 try algorithm To cite this version : Investigating performance varia-
652 tions of an optimized GPU-porting granulometry algorithm, in: De-
653 sign and Architectures for Signal and Image Processing (DASIP), 2012
654 Conference on, Oct 2012, Karlsruhe, Germany, 2013, pp. 1–6. URL:
655 <https://hal.archives-ouvertes.fr/hal-00787861>.
- 656 [31] M. Peternell, D. S. Russell-Head, C. J. L. Wilson, A technique for
657 recording polycrystalline structure and orientation during in situ de-
658 formation cycles of rock analogues using an automated fabric analyser,
659 *Journal of Microscopy* 242 (2011) 181–188. doi:10.1111/j.1365-2818.
660 2010.03456.x.
- 661 [32] F. Flin, J.-B. Brzoska, B. Lesaffre, C. Coléou, R. A. Pieritz, Full
662 three-dimensional modelling of curvature-dependent snow metamor-
663 phism: first results and comparison with experimental tomographic
664 data, *Journal of Physics D: Applied Physics* 36 (2003) A49–A54.
665 doi:10.1088/0022-3727/36/10A/310.
- 666 [33] J. J. Koenderink, A. J. V. Doorn, Surface shape and curvature scales,
667 *Image Vision Comput.* 10 (1992) 557–564.

- [34] J. Gibbs, K. A. Mohan, E. Gulsoy, A. Shahani, X. Xiao, C. Bouman,
M. De Graef, P. Voorhees, The Three-Dimensional Morphology of Growing Dendrites, *Scientific reports* (2015) 1–9. doi:10.1038/srep11824.
- [35] R. Daudin, S. Terzi, P. Lhuissier, J. Tamayo, M. Scheel, N. H. Babu,
D. G. Eskin, L. Salvo, Particle-induced morphological modification of Al alloy equiaxed dendrites revealed by sub-second in situ microtomography, *Acta Materialia* 125 (2017) 303–310. doi:10.1016/j.actamat.2016.12.005.
- [36] Avizo, Avizo lite 9.2.0, <https://www.fei.com/software/avizo-for-materials-science/>, 2017.
- [37] T. Chauve, Aita toolbox, <https://github.com/ThomasChauve/aita>, 2017.
- [38] J. Schwander, J.-M. Barnola, C. Andri, M. Leuenberger, A. Ludin,
D. Raynaud, B. Stauffer, The age of the air in the firn and the ice at Summit, Greenland, *Journal of Geophysical Research* 98 (1993) 2831–2838. doi:10.1029/92JD02383.
- [39] C. M. Trudinger, I. G. Enting, D. M. Etheridge, R. Francey, V. A. Levchenko, L. P. Steele, Modeling air movement and bubble trapping in firn, *Journal of Geophysical Research* 102 (1997) 6747–6763.
- [40] J. P. Severinghaus, M. O. Battle, Fractionation of gases in polar ice during bubble close-off: New constraints from firn air Ne, Kr and Xe observations, *Earth and Planetary Science Letters* 244 (2006) 474–500. doi:10.1016/j.epsl.2006.01.032.

- 691 [41] L. E. Mitchell, C. Buizert, E. J. Brook, D. J. Breton, J. Fegyveresi,
692 D. Baggenstos, A. Orsi, J. Severinghaus, R. B. Alley, M. Albert, R. H.
693 Rhodes, J. R. McConnell, M. Sigl, O. Maselli, S. Gregory, J. Ahn, Ob-
694 serving and modeling the influence of layering on bubble trapping in
695 polar firn, *Journal of Geophysical Research Atmospheres* 120 (2015)
696 2558–2574. doi:10.1002/2014JD022766.
- 697 [42] P. Martinerie, D. Raynaud, D. M. Etheridge, J. M. Barnola, D. Maza-
698 udier, Physical and climatic parameters which influence the air content
699 in polar ice, *Earth and Planetary Science Letters* 112 (1992) 1–13.
700 doi:10.1016/0012-821X(92)90002-D.
- 701 [43] P. Duval, M. F. Ashby, I. Andermant, Rate-Controlling Processes in the
702 Creep of Polycrystalline Ice, *Journal of Physical Chemistry* 87 (1983)
703 4066–4074.
- 704 [44] S. Kipfstuhl, S. H. Faria, N. Azuma, J. Freitag, I. Hamann, P. Kauf-
705 mann, H. Miller, K. Weiler, F. Wilhelms, Evidence of dynamic recryst-
706 tallization in polar firn, *Journal of Geophysical Research: Solid Earth*
707 114 (2009) 1–10. doi:10.1029/2008JB005583.
- 708 [45] A. Burr, W. Noël, P. Trecourt, M. Bourcier, F. Gillet-Chaulet, A. Philip,
709 C. L. Martin, The anisotropic contact response of viscoplastic monocrys-
710 talline ice particles, *Acta Materialia* 132 (2017) 576–585. doi:10.1016/
711 j.actamat.2017.04.069.
- 712 [46] T. Chauve, M. Montagnat, S. Piazzolo, B. Journaux, J. Wheeler,
713 F. Barou, D. Mainprice, A. Tommasi, Non-basal dislocations should

- 714 be accounted for in simulating ice mass flow, *Earth and Planetary Sci-*
715 *ence Letters* 473 (2017) 247–255. doi:10.1016/j.epsl.2017.06.020.
- 716 [47] T. Chauve, M. Montagnat, F. Barou, K. Hidas, A. Tommasi, D. Main-
717 price, Investigation of nucleation processes during dynamic recrystalliza-
718 tion of ice using cryo-EBSD, *Philosophical Transactions of the Royal*
719 *Society A: Mathematical, Physical and Engineering Sciences* 375 (2017)
720 20150345. doi:10.1098/rsta.2015.0345.
- 721 [48] L. Capolo, Contribution à l’étude des hétérogénéités de déformation
722 viscoplastique de la glace Ih mono et multi cristalline : essais de com-
723 pression in-situ sous rayonnement X, Ph.D. thesis, Université Joseph
724 Fourier - Grenoble 1, 2007.
- 725 [49] M. Montagnat, T. Chauve, F. Barou, A. Tommasi, B. Beausir, C. Fres-
726 sengeas, Analysis of Dynamic Recrystallization of Ice from EBSD Ori-
727 entation Mapping, *Frontiers in Earth Science* 3 (2015) 1–13. doi:10.
728 3389/feart.2015.00081.
- 729 [50] T. Chauve, M. Montagnat, P. Vacher, Strain field evolution during dy-
730 namic recrystallization nucleation; A case study on ice, *Acta Materialia*
731 (2015). doi:10.1016/j.actamat.2015.08.033.
- 732 [51] P. Duval, O. Castelnau, Dynamic Recrystallization of Ice in Polar Ice
733 Sheets, *Journal de Physique IV* 5 (1995) 197–203.
- 734 [52] E. M. Schulson, P. Duval, et al., Creep and fracture of ice, volume 1,
735 Cambridge University Press Cambridge, 2009.

- 736 [53] C. Bréant, P. Martinerie, A. Orsi, L. Arnaud, A. Landais, Modelling
737 the firn thickness evolution during the last deglaciation: constraints on
738 sensitivity to temperature and impurities, *Climate of the Past* 13 (2017)
739 833–853. doi:<https://doi.org/10.5194/cp-13-833-2017>.
- 740 [54] F. Flin, N. Calonne, R. Denis, R. Caneill, B. Lesaffre, A. Dufour,
741 A. Philip, J. Roulle, S. R. Roscoat, C. Geindreau, 3D Growth Rates
742 from Tomographic Images : Local Measurements for a Better Under-
743 standing of Snow Metamorphism - Application to Isothermal Meta-
744 morphism and Comparison with a Phase-Field Model, in: American
745 Geophysical Union (AGU) Fall Meeting, American Geophysical Union
746 (AGU) Fall Meeting, San Fransisco, USA, 14-18 December, 2015, 2015.
747 doi:10.1002/2015GL063541.Flin.
- 748 [55] C. Buizert, P. Martinerie, V. V. Petrenko, J. P. Severinghaus, C. M.
749 Trudinger, E. Witrant, J. L. Rosen, A. J. Orsi, M. Rubino, D. M.
750 Etheridge, L. P. Steele, C. Hogan, J. C. Laube, W. T. Sturges, V. A.
751 Levchenko, A. M. Smith, I. Levin, T. J. Conway, E. J. Dlugokencky,
752 P. M. Lang, K. Kawamura, T. M. Jenk, J. W. C. White, T. Sow-
753 ers, J. Schwander, T. Blunier, Gas transport in firn: Multiple-
754 tracer characterisation and model intercomparison for NEEM, Northern
755 Greenland, *Atmospheric Chemistry and Physics* 12 (2012) 4259–4277.
756 doi:10.5194/acp-12-4259-2012.