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

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## 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

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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

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## 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  $10^{-9} \text{ s}^{-1}$  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  $840 \text{ kg.m}^{-3}$ , 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  $630 \text{ kg.m}^{-3}$  density  
48 and recorded stresses of the order of  $0.3 \text{ MPa}$  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  $550 \text{ kg.m}^{-3}$ , similar to natural firn), and  
51 went further than  $850 \text{ kg.m}^{-3}$ . 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  $850 \text{ kg.m}^{-3}$  exhibited less than 1% of strain  
56 for an eighty-hour experiment under  $0.5 \text{ MPa}$  and at  $-20^\circ\text{C}$ . 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  $0.7 \text{ MPa}$ )  
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  $500 \text{ kg.m}^{-3}$ ). 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 firn typical densities (ranging from  $550 \text{ kg.m}^{-3}$  to  $912 \text{ kg.m}^{-3}$ ), the  
86 evolution of pore curvature, and time and temperature effects should be  
87 noticeable in laboratory conditions (day-long experiment at  $-10^\circ\text{C}$ ). 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 ( $\approx 10^{-6} - 10^{-5}\text{s}^{-1}$ ) 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)<sup>2</sup>. 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

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<sup>2</sup>Volsol ice core, grant ANR NT09-431976-VOLSOL)

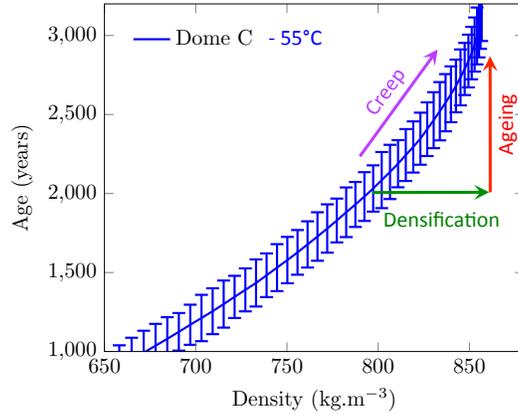


Figure 1: The experimental strategy developed in this work. Ice age against density for Dome C [27] together with the proposed paths in the experimental procedure: mechanical densification under oedometric compaction at  $-10^{\circ}\text{C}$  followed by ageing (heat treatment) at  $-2^{\circ}\text{C}$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

113 compacted samples are artificially densified at  $-10^{\circ}\text{C}$  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  $-2^{\circ}\text{C}$  for five  
 116 months and analyzed afterward. A third set of in situ samples are submitted  
 117 to creep also at  $-2^{\circ}\text{C}$  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  $-2^{\circ}\text{C}$  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  $\mu\text{m}\cdot\text{s}^{-1}$ ). 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  $-10^\circ\text{C}$  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^\circ\text{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  $\mu\text{A}$ . Scans have a helical geometry enabling the

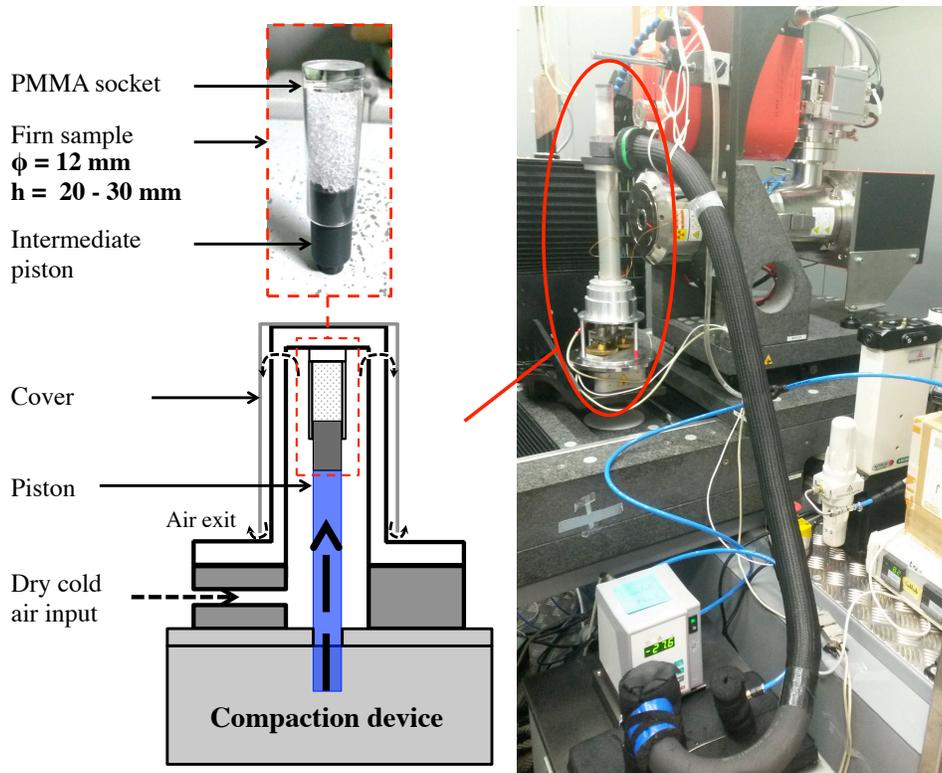


Figure 2: The apparatus inside the laboratory tomograph with the compaction device highlighted in a red ellipse. The firn sample is shown inside its PMMA socket (oedometric conditions). Cold air flows in the cell at  $-10^{\circ}\text{C}$ .

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  $9.6\text{ mm}$  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 80<sub>3</sub>-comp, 80<sub>4</sub>-comp, 80<sub>5</sub>-comp and 80<sub>6</sub>-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 80<sub>3</sub>-comp for 80<sub>4</sub>-comp and  
170 80<sub>6</sub>-comp, and three times larger for 80<sub>5</sub>-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 positioning 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 80<sub>6</sub> is submitted to two consecutive treatments: first a com-  
180 paction (80<sub>6</sub>-comp) and then a heat treatment (80<sub>6</sub>-comp-temp). Table 1  
181 lists the initial designations and densities of all tested samples as well as

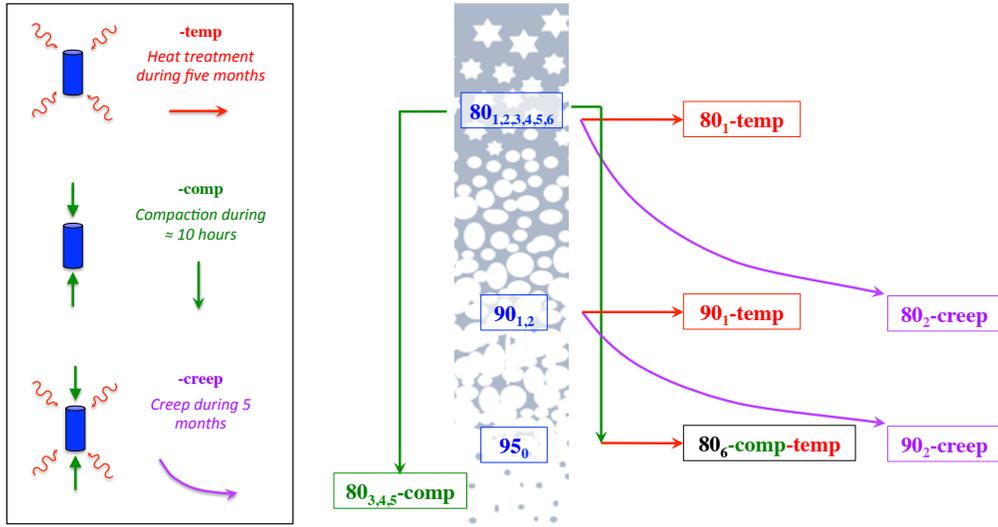


Figure 3: Experimental strategy throughout this work. The red path denotes heat treatment with no applied stress. The green path denotes densification under mechanical compaction. The purple path denotes densification under creep for 5 months. Samples are denominated by their initial depth in the ice 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  $-2^\circ\text{C}$  (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  $\approx 0.3$ , and  $\approx 0.7$  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 mm<sup>2</sup>. 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)	$\rho_k$ (kg.m <sup>-3</sup> )	State 1	Final strain rate (s <sup>-1</sup> )	Final stress or dead load (MPa)	$\rho_1$ (kg.m <sup>-3</sup> )	State 2	$\rho_2$ (kg.m <sup>-3</sup> )
95 <sub>0</sub>	94.5	829.7						
80 <sub>1</sub>	79.87	786.3	80 <sub>1</sub> -temp		0	784.8		
80 <sub>2</sub>	79.92	794.1	80 <sub>2</sub> -creep		≈ 0.3	810.3		
80 <sub>3</sub>	79.92	797.8	80 <sub>3</sub> -comp	$3.4 \cdot 10^{-6}$	≈ 3	825.1		
80 <sub>4</sub>	79.92	785.0	80 <sub>4</sub> -comp	$7.6 \cdot 10^{-6}$	≈ 4.8	867.8		
80 <sub>5</sub>	79.92	784.8	80 <sub>5</sub> -comp	$9.3 \cdot 10^{-6}$	≈ 5	852.2		
80 <sub>6</sub>	79.92	-	80 <sub>6</sub> -comp	$\approx 4 \cdot 10^{-6}$	≈ 5.7	828.3	80 <sub>6</sub> -comp-temp	825.6
90 <sub>1</sub>	89.5	822.3	90 <sub>1</sub> -temp		0	823.6		
90 <sub>2</sub>	89.5	821.0	90 <sub>2</sub> -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 80<sub>6</sub> is first compacted at -10 °C and then heat treated at -2 °C, hence is denoted -comp-temp. Samples 80<sub>3,4,5</sub> 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

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

## 257 Results

### 258 *Closed porosity ratio and connectivity index*

259 Pore closure evolution has been classically investigated by using the closed  
260 porosity ratio [38, 39, 40, 41]. Recently, we have introduced a parameter  
261 defined as the connectivity index, which was found more reliable than the  
262 closed porosity ratio to predict the close-off depth and density [7]. The  
263 connectivity index is defined by the ratio of the volume of the largest pore to  
264 the total pore volume of a sample, and its quantification is less dependent on  
265 the analyzed volume, heterogeneities and voxel size than the closed porosity  
266 ratio. The closed porosity ratio is defined by the ratio of the closed pore  
267 volume to the total pore volume. They both work reversely and vary between  
268 0 and 100 %, but the closed porosity ratio is fully dependent of the status  
269 of pores (close or open), which is sensitive for the small volumes typically  
270 obtained with X-ray tomography in this work. Its quantification is carried  
271 out at the scale of the sample and depends on the cut-pore ratio. It does not  
272 give values that can be used to characterize a firn layer. That being said,  
273 both parameters are calculated and discussed here.

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

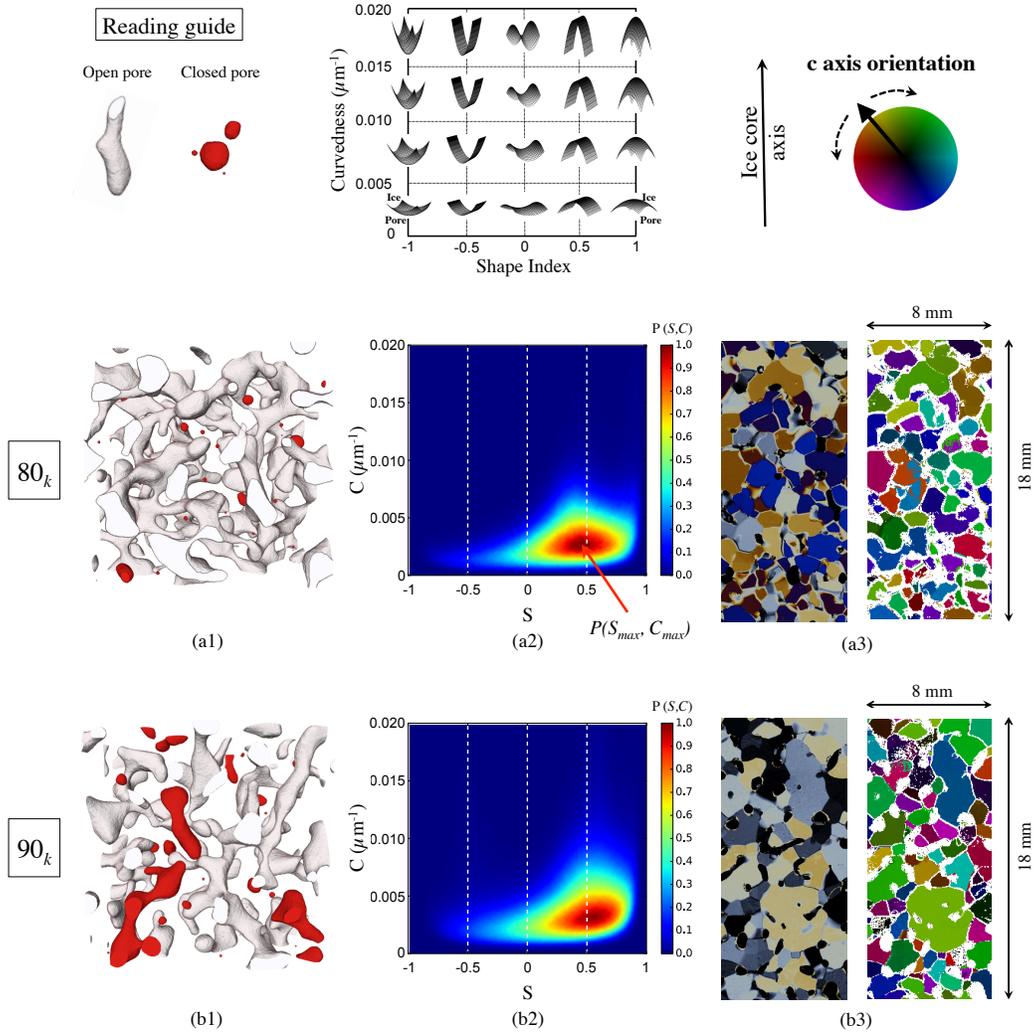


Figure 4: Typical initial microstructures and references for samples (a)  $80_k$  and (b)  $90_k$  (with  $k$  ranging from 1 to 6 for samples  $80_k$  and being 1 or 2 for samples  $90_k$ ). Layouts are shown as reading guides. Visualization of open pores (white) and closed pores (red) in a subvolume (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).

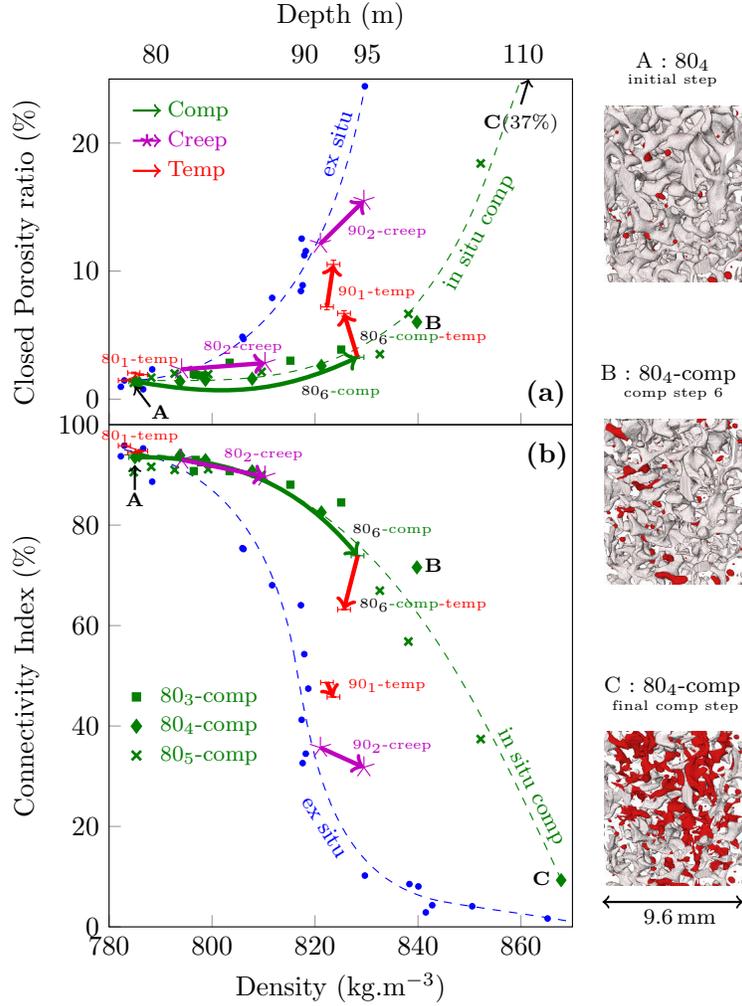


Figure 5: Evolution of the closed porosity ratio (a) and of the connectivity index (b) with density for ex situ and in situ samples. Blue dots depict ex situ samples [7], and green crosses, diamonds and squares depict all strain steps of in situ compacted samples  $80_{3,4,5}$ -comp. Blue and green dashed 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  $80_4$ -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  $80_4$ -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<sub>3,4,5</sub>-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  $\rho$ , 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<sup>-3</sup>, 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<sup>-3</sup> (sample 80<sub>4</sub>-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  $840 \text{ kg.m}^{-3}$  for in situ samples, meaning that percolation is still possible after  
303 compaction up to at least  $870 \text{ kg.m}^{-3}$ . 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  $80_6\text{-comp}$ ,  
311 illustrating a density increase without closing much pores. But, from  $80_6\text{-}$   
312  $\text{comp}$  to  $80_6\text{-comp-temp}$ , 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  $80_1\text{-temp}$ , or  
317 between  $90_1$  and  $90_1\text{-temp}$ , but an increase of the closed porosity ratio is only  
318 noticed between  $90_1$  and  $90_1\text{-temp}$ . 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 ( $90_2\text{-creep}$ ), while they do not for sample  $80_2\text{-}$   
321  $\text{creep}$ . Consistently, the connectivity index decreases for sample  $90_2\text{-creep}$   
322 and stagnates for sample  $80_2\text{-creep}$ . 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 80<sub>6</sub>-comp and 80<sub>6</sub>-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 95<sub>0</sub> (Fig. 6a2) and 90<sub>2</sub>-creep (Fig. 6d2) do not exhibit marked curved  
341 pores, whereas sample 80<sub>6</sub>-comp (Fig. 6b2) exhibits sharp pore shapes, char-  
342 acterized by a larger curvedness on pore edges. After heat treatment, sam-  
343 ple 80<sub>6</sub>-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 95<sub>0</sub> depth is very similar to 90<sub>2</sub>-creep, which was crept at -2 °C during five

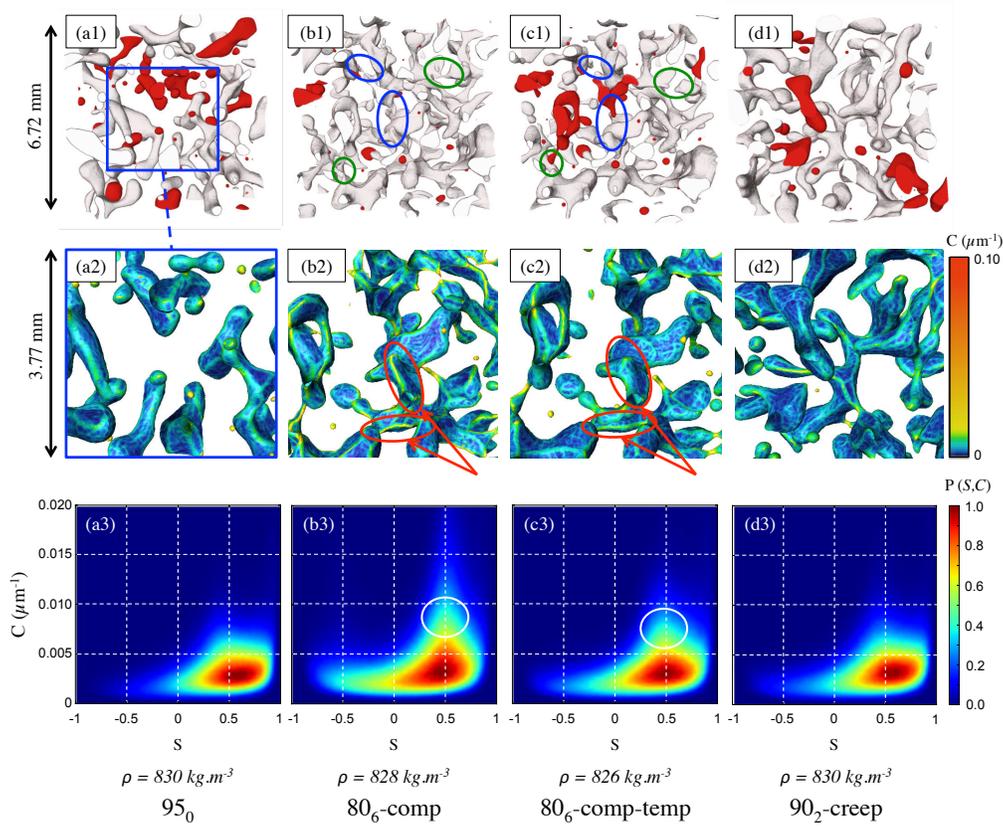


Figure 6: Comparison of pore morphologies (rows 1-2) and local curvatures (rows 2-3) for samples of approximately equal density ( $\rho = 828 \pm 2 \text{ kg.m}^{-3}$ ) but resulting from different process histories. (a) 95<sub>0</sub>, (b) 80<sub>6</sub>-comp, (c) 80<sub>6</sub>-comp-temp and (d) 90<sub>2</sub>-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 (95<sub>0</sub>, 80<sub>6</sub>-comp, 80<sub>6</sub>-comp-  
354 temp and 90<sub>2</sub>-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 ( $829 \text{ kg.m}^{-3}$  instead of  $785 \text{ kg.m}^{-3}$ ).

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 \(80<sub>6</sub>-comp,](#)  
359 [Fig. 6b3\)](#) looks very similar to all other in situ compacted samples (80<sub>3</sub>-comp,  
360 [80<sub>4</sub>-comp and 80<sub>5</sub>-comp](#)). Negative values are much more prevalent than for  
361 Figs. 6a3 (95<sub>0</sub>) and 6d3 (90<sub>2</sub>-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 (80<sub>6</sub>-comp-temp), the dominant local shape of Fig. 6b3  
364 (80<sub>6</sub>-comp) is the cylinder as  $S_{max}$  is centered around  $S = 0.5$ . The distri-  
365 bution is more widespread for 80<sub>6</sub>-comp than for 80<sub>6</sub>-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 (80<sub>6</sub>-comp) towards the smoother shapes of pores observed in  
371 Fig. 6c2 (80<sub>6</sub>-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 95<sub>0</sub> exhibits a larger mean grain  
378 size than the in situ compacted sample 80<sub>5</sub>-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 80<sub>3</sub>-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  $0.40 \text{ mm}^2$  for samples 80<sub>4</sub>-comp and 80<sub>5</sub>-comp (with standard  
386 deviations equal to the mean grain size for all samples). The decrease in  
387 mean grain size for samples 80<sub>4</sub>-comp and 80<sub>5</sub>-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 80<sub>5</sub>-comp (Fig. 7b2), the fabric is a little more  
400 pronounced than the ex-situ sample 95<sub>0</sub> (Fig. 7a2). This is coherent with a

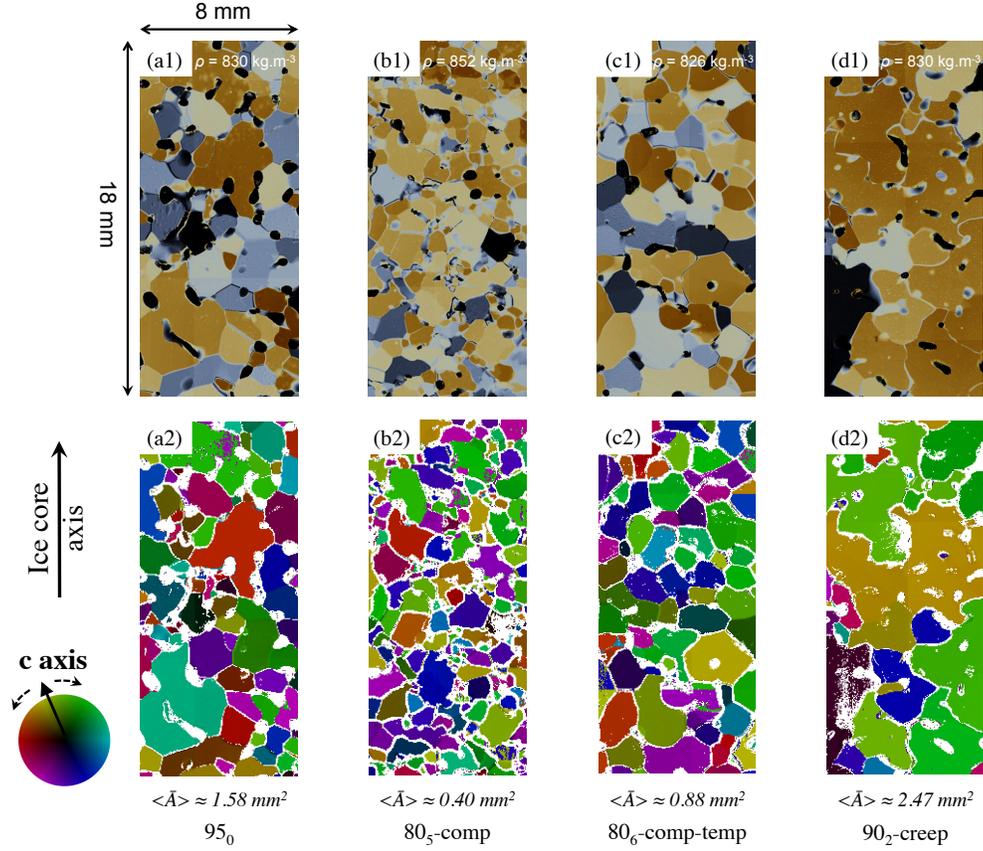


Figure 7: Comparison of the microstructures and  $c$  axis orientations of (a1)-(a2) 95<sub>0</sub>, (b1)-(b2) 80<sub>5</sub>-comp, (c1)-(c2) 80<sub>6</sub>-comp-temp and (d1)-(d2) 90<sub>2</sub>-creep.

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 90<sub>2</sub>-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 80<sub>1</sub>-temp and 90<sub>1</sub>-temp to

408 their reference states Fig. 4a3 and Fig. 4b3 respectively. Thus, there are  
409 not shown. However samples 80<sub>2</sub>-creep and 90<sub>2</sub>-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 (80<sub>1</sub>-temp and 90<sub>1</sub>-temp) or than ex situ sample of the same density 95<sub>0</sub>  
413 (compare Fig. 7d1 and Fig. 7a1). As for sample 80<sub>6</sub>-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 90<sub>1</sub> and 90<sub>1</sub>-temp, curvature variations are  
441 small. These are much less important than for samples 80<sub>6</sub>-comp and 80<sub>6</sub>-  
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 90<sub>1</sub>-temp and 80<sub>6</sub>-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 80<sub>6</sub>-comp-temp (Fig. 7c1), [which was compacted and then heat](#)  
452 [treated](#), exhibits a grain morphology surprisingly close to 80<sub>1</sub>-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 80<sub>6</sub>-comp-temp are still  
455 more aligned with the loading direction (Fig. 7c2). For all compacted sam-  
456 ples (80<sub>3</sub>-comp, 80<sub>4</sub>-comp, 80<sub>5</sub>-comp and 80<sub>6</sub>-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 80<sub>6</sub>-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 80<sub>2</sub>-creep and 90<sub>2</sub>-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 90<sub>2</sub>-creep [shown in Fig. 7d2](#) than for 80<sub>2</sub>-creep). This,  
481 combined with the microstructure of Fig. 7d1 reminds of grain boundary mi-

482 gration mechanism, which happens in the deepest part of ice cores when large  
483 grains interlock as dynamic recrystallization proceeds [51, 52, chap. 6]. High-  
484 resolution images and precise measurements of the orientation with the help  
485 of EBSD should enhance our comprehension of the underlying mechanisms  
486 [49, 47].

## 487 **Conclusion**

488 In this paper, we presented three different experiments in order to **discern**  
489 the mechanisms of densification of polar firn. In situ oedometric **compaction**  
490 tests of Dome C firn were carried out under X-ray at  $-10^{\circ}\text{C}$  during a few  
491 hours. It was possible to characterize the evolution of pore curvature dur-  
492 ing densification and to follow the closure of pores. The connectivity index  
493 appears to be a good indicator of pore closure phenomena as it is not depen-  
494 dent of cut pores at the sample boundary as is the closed porosity ratio [7].  
495 **Since, by construction, lab experiments will never be able to reproduce the**  
496 **evolution on climatological time scales, it is important to exploit the rate-**  
497 **temperature equivalence as much as possible for aiming at it. Therefore,**  
498 **the second experiment consisted in studying the effect of temperature on firn**  
499 **through** heat treatment (**ageing at  $-2^{\circ}\text{C}$** ) during a few months. The third one  
500 involved creep tests, i.e. compaction of firn performed at high temperature  
501 ( $-2^{\circ}\text{C}$ ) during 5 months to combine the two previous experiments.

502 It is reported here that in situ compaction at high strain rates (between  
503  $2 \cdot 10^{-6} \text{ s}^{-1} < \dot{\epsilon}_z < 1.5 \cdot 10^{-5} \text{ s}^{-1}$ , larger than natural strain rates in Antarctica)  
504 results in densification with a reduced volume of closed pores compared to  
505 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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