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# Influence of porosity on ice dynamic behavior as assessed by spalling tests

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## Abstract

The impact of hailstones remains a major issue due to the damage caused by hailstorms against aeronautical or space structures. During a such impact, an intense fragmentation develops in the hailstone due to dynamic tensile loading at high strain-rates which influences the mechanical loading transmitted to the impacted structure. Up to now, the work of Saletti et al. (2019) is the only survey providing a robust and reproducible set of experiments about the dynamic tensile strength of ice, however the microstructure influence was out the scope of this experimental study. Though, the hailstone microstructures are generally charaterised by a high level of porosity. It is the reason why the influence of this microstructural parameter on the dynamic behavior of polycrystalline ice needs to be investigated. The aim of this paper is to study the role of porosity on the tensile behavior of polycrystalline ice by applying the same experimental set-up and measurements processing than in Saletti et al. (2019) to low-porosity (LP) and high-porosity (HP) microstructures. To do so, spalling tests with a Hopkinson bar apparatus were conducted and fragmentation processes were carefully analyzed by means of an Ultra High Speed Camera, an Automatic Ice Texture Analyser and a micro-Computed Tomograph. The experimental results show that the tensile strength is sensitive to the applied strain rate for both types of micro-structures over the range  $24 s^{-1}$  to  $120 s^{-1}$ . A weakening effect of porosity is also observed, the lowly porous specimens being significantly stronger than the highly porous ones. The large

27 spherical pores present in the LP specimens are seen to contribute to this strength decrease.  
28 Although fragmentation analysis did not allow to clearly assess the role of these porosities on  
29 the crack initiation and propagation processes, the numerous clues such as the high number of  
30 cracks detected, the tortuosity of crack paths and the presence of porosities on the crack surfaces  
31 seem to validate the hypothesis of porosities playing a key role during ice fragmentation.

## 32 **1 Introduction**

33 Dynamic interactions between ice and man made structures are of concern in many situations,  
34 such as icebreaker ships crushing ice floe in polar regions, serac falls, atmospheric ice impacting  
35 buildings, aircrafts or even space shuttles. For the latter, NASA classified atmospheric ice as a  
36 potential source of damage after the catastrophic Columbia space shuttle break down in 2003.  
37 An accurate understanding of the ice dynamic strength failure is of interest to determine and to  
38 model the force generated during an impact and thus to provide relevant scaling of the structures  
39 of concern.

40

41 Dynamic confined compressive and tensile loadings are expected to develop in brittle mate-  
42 rials under impact (Forquin et al., 2015; Forquin, 2017). Ice is known to behave as a brittle  
43 material at high strain-rates (Schulson and Duval, 2009), its mechanical response to dynamic  
44 confined compression and tension is thus essential and needs to be considered to get an accurate  
45 modelling. Up to now, with the improvement of computing capacities, numerous models have  
46 been developed to describe the behavior of polycrystalline ice during an impact event. Most of  
47 them adopted an elasto-plastic or elasto-visco-plastic approach, coupled with failure criterions  
48 (Carney et al., 2006; Pernas-Sánchez et al., 2012; Tippmann et al., 2013; Dousset, 2019). The  
49 ice behavior is assumed to be pressure dependent and the volumetric and deviatoric response of  
50 ice is different according to the direction of loading, compression or tension. The first models  
51 were manually adjusted by changing the material input parameters, the hardening modulus, the  
52 plastic failure strain and the compressive and tensile failure pressures, until a good correlation is  
53 reached between numerical simulations and experimental data (Kim and Kedward, 2000; Keune,  
54 2004; Park and Kim, 2010). Thanks to the Split Hopkinson Pressure Bar technique (SHPB),  
55 the compressive yield stress sensitivity to strain-rate of ice was investigated (Kim and Keune,

2007; Shazly et al., 2009; Wu and Prakash, 2015). These experimental results, although scattered, reveal a strain-rate hardening behavior that has been implemented in recent modelling works (Pernas-Sánchez et al., 2012; Tippmann et al., 2013). These models are based on the strong hypothesis of a transition from a solid-to-liquid behavior of ice when reaching a tensile failure criterion. Above this threshold, ice is supposed to behave as a fluid, and not carry any shear or tensile stress. In most of the concerned models, the tensile failure stress is considered to be strain-rate insensitive and is either taken from quasi-static experiment results or determined by a numerical parametric study. Indeed, for strain-rates lower than  $10^{-1} \text{ s}^{-1}$ , the ice tensile strength remains constant and ranges between 0.5 MPa to 3 MPa, depending on the microstructural properties (Petrovic, 2003). However, other brittle materials, such as concrete and ceramics, are characterized by a dynamic tensile strength strongly dependent on the loading rate (Schuler et al., 2006; Erzar and Forquin, 2011, 2014; Zinszner et al., 2015). In order to take that into consideration, Ortiz et al. (2015) adapted the initial Mazars model (Mazars, 1984) to dynamic loadings and considered an ice tensile strength evolving similarly to the compressive strength with strain rate, but with values of strength one-tenth lower. While being the first attempt to catch the dynamic tensile behavior of ice, this approach was not based on any measurements performed under dynamic tensile conditions. This lack of knowledge is problematic as the tensile strength of ice material is the main driver of the fragmentation processes during impact events. This material property should be at the forefront among all the input parameters that should be identified in order to get satisfying modelling.

76

To our knowledge, only two studies experimentally investigated the response of ice dynamically loaded under tension. Lange and Ahrens (1983) tested ice specimens via plate impact experiments and measured a tensile strength of 17 MPa at a strain rate of  $10^4 \text{ s}^{-1}$ . Saletti et al. (2019) studied the strain rate sensitivity of ice dynamic tensile strength by performing spalling tests with a Hopkinson bar apparatus on isotropic polycrystalline ice. They observed a clear strain-rate hardening, the ultimate spalling strength was found to change from 2.3 MPa to 16.3 MPa for strain rates between  $30 \text{ s}^{-1}$  and  $141 \text{ s}^{-1}$ . Both studies have given little weight to the impact of defaults and specificities of the microstructures of the studied specimens. Under quasi-static tensile loading, grain size, crystallographic orientations and porosity, are expected

86 to impact the mechanical response of polycrystalline ice (Schulson and Duval, 2009). In par-  
87 ticular, Kermani et al. (2008) and Timco et al. (1994) showed that the presence of porosity in  
88 atmospheric and sea ice respectively, significantly decreases their quasi-static tensile strength.  
89 Hailstone microstructures appear to be complex, and to strongly depend on the formation his-  
90 tory, but clearly always present some porosity with a dispersed pore size distribution (see Macklin  
91 et al. (1977) for instance). More broadly, in many quasi-brittle materials, cracks are supposed  
92 to be triggered on the microstructural defects under tensile loading, such as porosities, grain  
93 boundaries, inclusions, sintering defects, etc.. At high strain-rates in tension an intense multiple  
94 fragmentation occurs as a result of the activation of numerous microstructural defects (Hild et al.,  
95 2003). In polycrystalline ice, and more specifically in hailstones, these microstructural defects  
96 can either be porosities, grain boundaries, impurities, micro-cracks, for instance. As mentioned  
97 before, owing to the presence of a high and complex porosity in hailstones, porosities might  
98 be the dominating defect population influencing the behavior of ice under dynamic loading.  
99 Quantifying how the defect of the microstructure can act upon ice fragmentation processes is  
100 therefore a key step to reach a better understanding of the behavior of ice under dynamic loading.

101

102 The present study will aim to evaluate how the porosity affects the dynamic tensile response  
103 of polycrystalline ice. To do so, two type of polycrystalline ice specimens, presenting different  
104 levels of porosity but similar grain size and shape, have been manufactured. The dynamic be-  
105 havior of both types of specimen has been evaluated using the spalling technique on Hopkinson  
106 bar, according to the experimental procedure described in Saletti et al. (2019). The specimen  
107 growth method, the procedure for microstructure characterization and the spalling set-up are  
108 presented in section 2. In section 3.3, the dynamic tensile strength sensitivity to the strain-rate  
109 and porosity is analyzed. The influence of the porosity on the induced failure mechanisms is  
110 addressed in the discussion part via the analysis of crack initiation and propagation processes.

## 111 **2 Materials and methods**

### 112 **2.1 Specimen growth and machining**

113 The two studied materials are made of artificial polycrystalline granular ice grown in laboratory  
114 and are characterized by a microstructure with a low porosity (LP)( $\approx 1 - 2\%$ ) and a microstruc-

115 ture with an high porosity (HP)( $\approx 7 - 10\%$ ) respectively. Both microstructures are obtained  
116 using two growth techniques inspired by the one described in Barnes et al. (1971). Specimens are  
117 grown out of isotropic seeds made of crushed ice (with a maximum particle diameter of  $2\text{ mm}$ )  
118 surrounded by water at  $0^\circ\text{C}$ . The slurry is placed on a Peltier element ( $-15^\circ\text{C}$ ), in a  $0^\circ\text{C}$  room, to  
119 grow gently from bottom to top, and avoid internal stresses during freezing. To obtain the LP  
120 microstructure, the isotropic seed is pumped before adding the water. The air trapped between  
121 the snow grains is maintained in order to produce the HP microstructure. The LP specimens  
122 have no visible porosities and are slightly opaque, whereas large porosities are visible in HP  
123 specimens. After unmolding, the specimens are stored during 24 hours in a  $0^\circ\text{C}$  environment.  
124 This annealing stage aims to relaxe freezing stress and to homogeneize the grain size. Finally,  
125 after lathing and milling,  $120\text{ mm}$  long and  $45\text{ mm}$  diameter cylindrical specimens are obtained.  
126 The geometric and density features of the specimens used in the spalling tests are listed in Table  
127 1.

## 128 **2.2 2D analysis of the microstructure : the Automatic Ice Texture Analyser** 129 **(AITA)**

130 Several LP and HP specimens, not tested here but assumed to be representative of the whole sets,  
131 were machined in thin square sections of approximately  $50 \times 50\text{ cm}^2$  and  $0.3\text{ }\mu\text{m}$  in thickness.  
132 Owing to the birefringent property of ice, the optical method of AITA (Wilson et al., 2003)  
133 gives access to the orientation of the **c**-axis (the optical axis) of the hexagonal crystal cell. The  
134 current assumption of a relative isotropic behavior in the plane normal to the **c**-axis (the basal  
135 plane) renders sufficient the knowledge of **c**-axis orientations. The AITA provides the **c**-axis  
136 orientations at a pixel scale set here to  $20\text{ }\mu\text{m}$ , and therefore offers orientation images that  
137 enable to extract the 2D microstructure fabric and the crystallographic texture (plotted with  
138 orientation color-scaled images, see Figure 5).

## 139 **2.3 3D analysis of the microstructure : micro Computed X-ray Tomography** 140 **(microCT)**

141 MicroCT is a convenient non destructive method to collect the 3D microstructure of a biphasic  
142 material when phases are X-Ray contrasted, and hence the porosities size and shape. Quantify-

<b>Spec Id</b>	Length ( <i>mm</i> )	Diameter ( <i>mm</i> )	Density ( <i>kg/m</i> <sup>3</sup> )
HP01	119.7	44.9	857
HP02	120.2	44.9	840
HP03	120.3	44.9	847
HP04	119.6	44.9	818
HP05	119.5	45.1	834
HP06	119.7	45.1	844
HP07	118.0	45.0	844
HP08	119.8	45.2	834
HP09	119.7	45.0	848
HP10	120.0	44.9	843
HP11	119.9	45.0	835
HP12	119.0	45.0	833
HP13	120.9	45.0	840
HP14	120.6	45.6	805
LP01	119.7	45.1	906
LP02	119.9	45.1	917
LP03	120.1	45.1	921
LP04	119.9	45.0	908
LP05	119.6	45.1	912
LP06	119.2	45.0	906
LP07	120.0	45.3	910
LP08	120.6	45.2	918
LP09	120.0	45.0	910
LP10	119.0	44.9	908
LP11	119.5	45.0	914
LP12	110.7	44.8	908

Table 1: Dimensions and densities of the specimens presented in this study. The calculation of the density using the mass and volume of the specimen delivers a typical uncertainty of individual values about  $\pm 5 \text{ kg.m}^{-3}$ .

143 ing damage in post-mortem specimens can also be achieved with microCT. Numerous scans with  
144 different resolutions were performed on HP and LP specimens in order to assess the homogeneity  
145 and the reproducibility of the pore structure. Here we present the scan configuration used with  
146 three HP specimens and three LP specimens scanned prior and after spalling tests. Detailed  
147 observations are given in the section 3.2 and 3.6.

148

### 149 **2.3.1 Description of the microCT configuration (TomoCold)**

150 Scans were performed in a cold room set at  $-20^{\circ}\text{C}$  with the tomograph TomoCold DeskTom130  
151 RX Solutions, specifically adapted to cold temperatures at the CNRM-CEN (Centres d'Etudes de  
152 la Neige), Grenoble. A voxel size of  $27\ \mu\text{m}$  was chosen, resulting from a compromise between the  
153 volume to be analyzed and the minimal resolution required to detect porosities in LP specimens.  
154 The X-ray tube was powered by a current of  $238\ \mu\text{A}$  and a voltage of  $60\ \text{kV}$ . The detector was  
155 composed of  $1920\ \text{pixels} \times 1536\ \text{pixels}$  with a physical pixel size of  $127\ \mu\text{m}$ . Each scan consisted  
156 of four stacks composed of 1440 radiographs covering a  $360^{\circ}$  rotation for every stack. One  
157 radiograph was computed as the average of two frames captured at three frames per second.  
158 The whole process for every scan lasted 1 h 15 min and the scanned specimens were subjected  
159 to spalling tests ( $\varnothing \times h = 45 \times 120\ \text{mm}$ ).

### 160 **2.3.2 Image post-processing**

161 A 3D image is reconstructed from the acquired radiographs. A ring filter with a 20 voxel kernel  
162 was applied in order to remove ring features that are artefacts from the acquisition. After re-  
163 construction the scans are composed of approximately  $4500 \times 1660 \times 1660$  voxels, whose value  
164 spreads out between 0 (black) and 65536 (white). An enhancement of the contrast prior to  
165 conversion into 8 bit images was made in order to have reproducible grey level histograms. This  
166 operation allowed to apply identical thresholding method for each scan of the same microstruc-  
167 ture (more below). Typical images (8 bits) of both microstructures are shown on Figures 1a and  
168 2a. Examples of corresponding grey level probability density function are highlighted for each  
169 microstructure.

170

171 The threshold step allows to separate the voxels into two phases, i.e. air and ice, accord-



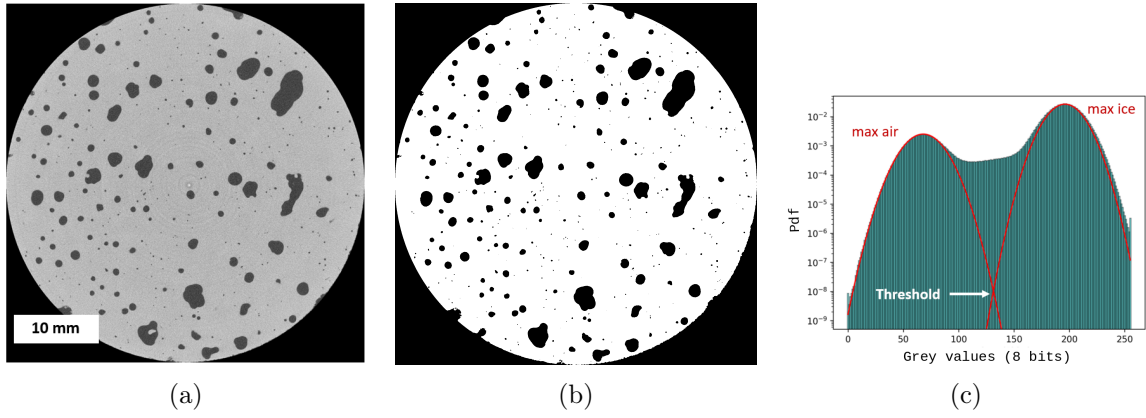


Figure 1: Slice of HP microstructure perpendicular to the specimen axis: (a) 8 bits image (b) Binary image (c) Probability Density Function of voxel grey levels

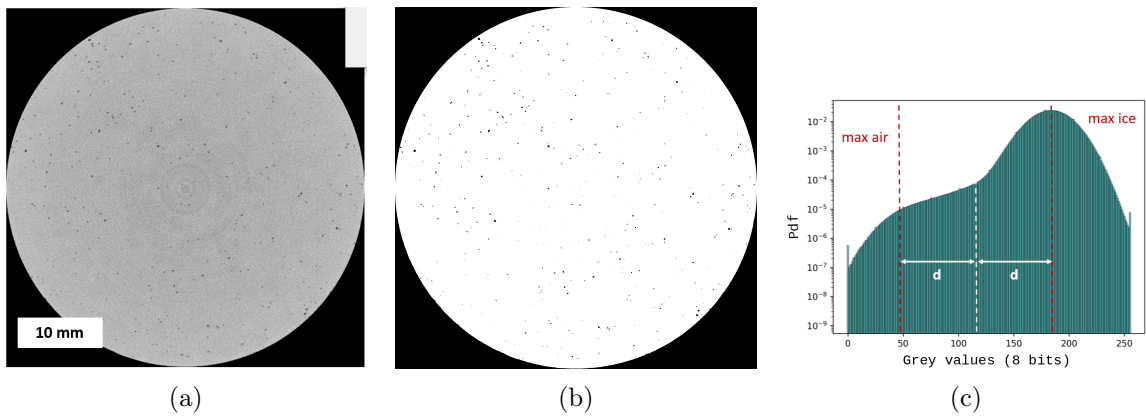


Figure 2: Slice of LP microstructure perpendicular to the specimen axis: (a) 16 bits image (b) Binary image *Need to add scale !!!*

172 ing to their grey level value. This operation has to be conducted with caution to minimize the  
173 error related to the image noise. HP microstructure histograms present two distinct maxima  
174 associated with each phase. Assuming that images are undergoing a Gaussian noise (related to  
175 the microCT device), the threshold value minimizing the voxel error is the intersection between  
176 the two Gaussian functions fitting each phase (red curves on Figure 1c). Finding a relevant  
177 threshold is more tricky for histograms associated to LP specimens, as the porosity density and  
178 size are much smaller. To do so, a pseudo-maximum for the air phase is manually picked, the  
179 threshold value is chosen to be the middle value between this maximum and the ice phase max-  
180 imum. See Figures 1b and 2b to have an overview of the resulting binary images.

181

182 The segmentation stage has been performed on the binary images using SPAM (The Software for  
183 the Practical Analysis of Materials), a Python package currently in development in the 3SR labo-  
184 ratory (Ref SPAM Eddy). During this stage each group of isolated or aggregated black voxels is  
185 labelled with a corresponding integer. Due to the presence of impulse noise (sparsely occurring  
186 white and black voxels) only labels whose volume exceeds 10 voxels, namely  $0.19 \text{ mm}^3$  here,  
187 were taken into account and considered as porosities. The results of the analysis are presented  
188 in the Section 3.2.

## 189 **2.4 The spalling technique**

### 190 **2.4.1 Experimental set-up**

191 The spalling test on Hopkinson bar is a convenient technique to measure the tensile strength  
192 of quasi-brittle materials at strain-rate up to  $200 \text{ s}^{-1}$  (Klepaczko and Brara, 2001; Erzar and  
193 Forquin, 2010; Saletti et al., 2019) and to characterize the stress-strain relationship and tensile  
194 fracture energy of geomaterials (Pierron and Forquin, 2012; Lukić et al., 2018; Forquin et al.,  
195 2019b). A short compressive pulse generated by the impact of a cylindrical projectile against an  
196 incident bar propagates along the bar. When it reaches the interface bar/specimen a part of it  
197 is reflected because of an impedance discontinuity whereas the other part is transmitted to the  
198 specimen. The transmitted pulse is then reflected on the free surface as a tensile pulse creating a  
199 dynamic tensile loading inside the specimen when the amplitude of the latter is higher than the  
200 compressive pulse one. The core of the specimen undergoes an elastic deformation up to brittle

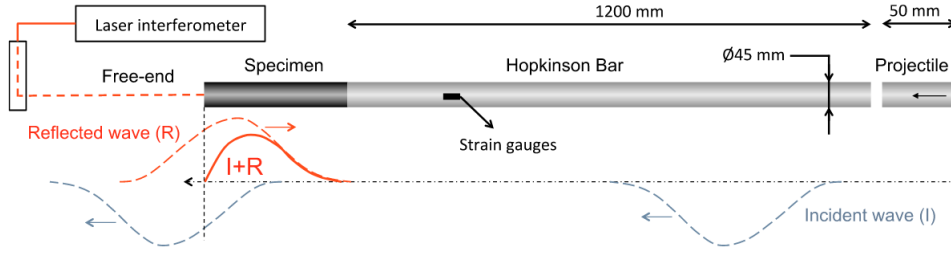


Figure 3: Scheme of the spalling test set-up used in the experiments, from Saletti et al. (2019)

201 fragmentation due to an intense dynamic tensile stress. Obviously this method is suitable for  
 202 materials which compressive strength is higher than the tensile strength as it is the case for ice  
 203 in the quasi-static regime (Schulson and Duval, 2009).

204

205 The configuration used here (Figure 3) is the same than the one described in Saletti et al.  
 206 (2019) where further informations can be found. The projectile is 50 *mm* long and 45 *mm* in  
 207 diameter and has a spherical-cap-ended nose acting as a pulse shaper, in order to smooth the  
 208 loading pulse (Erzar and Forquin, 2010). The incident bar is 1200 *mm* long with the same  
 209 diameter as the projectile. Both the projectile and the bar are made of high-strength aluminium  
 210 alloy (yield strength  $> 450 \text{ MPa}$ ) with a longitudinal wave speed  $C$  of  $5078 \text{ m.s}^{-1}$ , a density  
 211 of  $2800 \text{ kg.m}^{-3}$  and an elastic modulus equal to  $72.2 \text{ GPa}$ . The Hopkinson bar is instrumented  
 212 with a strain gauge to measure the compressive pulse applied to the specimen. A reflective paper  
 213 is glued at the free-end of the specimen allowing the measurement of the particle velocity of this  
 214 rear face by a laser interferometer. The tests are filmed with an ultra high speed (UHS) camera  
 215 (Kirana model from Specialised Imaging). For each test, 180 images with an inter-frame of  $1 \mu\text{s}$   
 216 are obtained, the sensor resolution used is  $924 \text{ pixels} \times 768 \text{ pixels}$ .

217

218 As the major difficulty being the ambient temperature of the spalling room that far exceeds  
 219 the ice melting temperature, several arrangements were made. The specimens are stored in a  
 220 freezer set at  $-30^\circ\text{C}$  located nearby their final position in the experimental set-up. A protocol  
 221 was established in order to ensure that less than 45 seconds elapse between the time when the  
 222 specimen is taken out from the freezer and the time when it is loaded in the spalling test appa-  
 223 ratus. The full time of handling was systematically recorded for every test and remained below  
 224 45 seconds. A cylinder made of the same aluminium alloy as the one of the input bar is glued

225 on the specimen during its preparation in the cold room. This cylinder is 45 mm in diameter  
226 and two lengths were used, 30 mm and 40 mm. Its main roles are (i) to avoid a thermal shock  
227 between the ice specimen and the Hopkinson bar which is at room temperature; (ii) to delay the  
228 melting of the specimen on its face in contact with the bar. The post-mortem specimens were  
229 immediately taken back in the freezer after experiment when possible, namely if the fragmenta-  
230 tion was not too intense.

231

## 232 2.4.2 Signal processing

### 233 *Spalling strength to failure*

234

235 As described in details in Erzar and Forquin (2010) and Saletti et al. (2019), from the sig-  
236 nal measured one can use the linear acoustic approximation (Novikov et al., 1966) to deduce the  
237 ultimate spall strength  $\sigma_T$  of the specimen prior to fracture :

$$\sigma_T = \frac{1}{2} \rho_{ice} C_{ice} \Delta V_{pb}, \quad (1)$$

238 where  $\rho_{ice}$  is the specimen density and  $C_{ice}$  the longitudinal wave velocity in the specimen.  
239 The latter is computed by performing an inter-correlation between the rising stage of the strain  
240 gauge and laser signals to determine the time of propagation between the gauge location and the  
241 specimen free surface, the distance being perfectly known.  $\Delta V_{pb}$  represents the pullback velocity  
242 corresponding to the difference between the maximum velocity and the velocity at rebound that  
243 are measured on the rear face of the specimen.

244

245 The Novikov approximation is relevant under certain assumptions. The deformation prior to  
246 fracture is assumed to be purely elastic in the core of the specimen likewise in the fragment  
247 bounded between the first fracture plane and the free surface. The specimen is assumed to un-  
248 dergo 1D longitudinal stress only, dispersion effects are neglected. In addition, a linear response  
249 is supposed prior the tensile stress peak, and the compressive and tensile Young's moduli are  
250 supposed to be equal (Forquin and Lukic 2018).

251

253

254 Gluing strain-gauges on an ice surface is challenging, especially in these experimental condi-  
 255 tions where each second matters. The strain rate is estimated by means of elastic numerical  
 256 simulations that are performed with the software ABAQUS-explicit. The material velocity pro-  
 257 file of the free surface of each test (red curve in Figure 4a) is converted into an elastic profile  
 258 (black curve in Figure 4a). This is achieved by keeping the slope of the tensile phase (that  
 259 follow the maximum value of particle velocity profile) before the expected occurrence of spalling  
 260 fracture characterized by the velocity rebound. By using the equation 1, a corresponding stress  
 261 can be computed from the material velocity at a given time  $t$ . This stress profile is used as a  
 262 loading pulse in the numerical simulations. The exact density and dimensions of each specimen  
 263 was used for each computation. Figure 4b shows the type of curve obtained from the simula-  
 264 tion to evaluate the strain rate in the tensile phase prior failure. For each test, the values are  
 265 extracted from the results of the numerical simulation at the approximate location of the first  
 266 appearance of cracks, thanks to the image sequence filmed during the test. The curve is valid  
 267 up to the spall strength identified with equation 1. To get the minimum, maximum and mean  
 268 value of the strain rate, the authors considered the stress interval which begins with the tensile  
 269 phase ( $\sigma > 0$ ) and ends with the ultimate spall strength measured (see gray zone in Figure  
 270 4b).

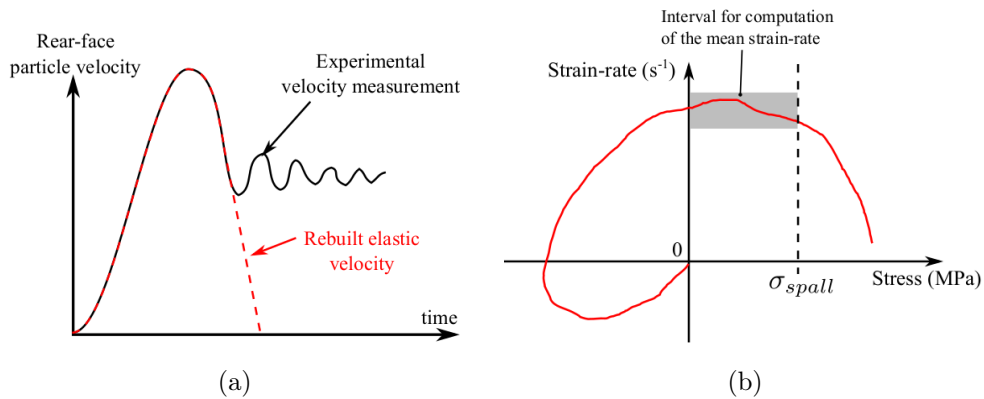


Figure 4: (a) Rebuilt of the rear-face velocity to reproduce an virtual elastic loading in numerical simulations. (b) Interval of data taken into account to compute the mean strain-rate of a test (from Saletti et al. (2019))

### 271 **2.4.3 Experimental indicators of test validity**

272 Different indicators were considered to assess the quality of each test. As the major risk is the  
273 initiation of cracks due to compressive loading, the images of the UHS camera were carefully  
274 analyzed in order to detect any compressive damage. The post-mortem specimen part near the  
275 ice-bar interface is also checked with naked eyes, when specimens were fragmented in few pieces.  
276 Another source of error is the quality of the interfaces between (i) the aluminium insert and the  
277 specimen and (ii) the incident bar and the aluminium insert. For (i), the presence of water due  
278 to premature melting of the ice is possible. For (ii), the risk is a slight orientation mismatch  
279 between the bar and the insert, an alignment defect could lead to the generation of a moment at  
280 the interface. In both cases, although the specimen is still undergoing compression and tension,  
281 the input stress is not completely transmitted. The procedure used here to quantify the quality  
282 of these interfaces is the same as the one described in Saletti et al. (2019). The experimental  
283 ratio of the transmitted energy with the incident energy  $\alpha_{exp}$  is compared with a theoretical  
284 value  $\alpha_0$  function of the bar and specimen impedances. The quality of the interfaces is assumed  
285 to be satisfying when the ratio  $\alpha_{exp}/\alpha_0$  is close to 1. The ratios are presented in Table 2 for  
286 every succesful spalling test.

## 287 **3 Results**

### 288 **3.1 2D characterization of the microstructure**

289 The porosity being the parameter we focus on, we took care to provide specimens characterized  
290 by similar microstructures and crystallographic textures. Examples of thin sections analyzed  
291 with AITA are presented in Figure 5. We verified that the textures are isotropic and that grains  
292 are rather equiaxed. Grain areas have been measured by contour extraction and morphological  
293 tools from the orientation color-coded images obtained with AITA. The mean grain sizes in both  
294 microstructures range between 1 and 2 *mm*. This analysis shows that no significant difference  
295 is observed between the LP and HP microstructures and texture properties.

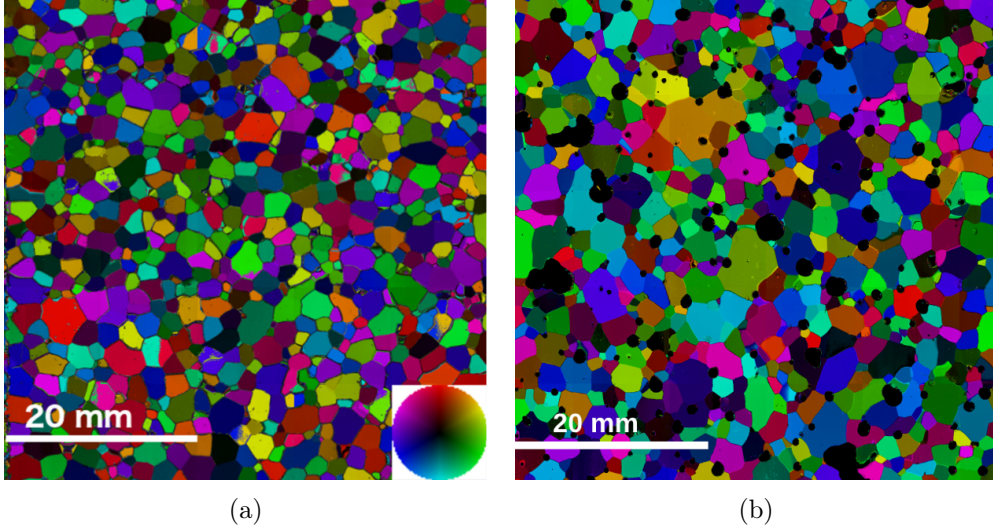


Figure 5: (a) and (b) Microstructures represented colour-coded with the [0001] crystallographic axis ( $\mathbf{c}$ -axis) orientation of LP and HP specimens respectively, as measured with the Automatic Ice Texture Analyzer from thin sections.

### 3.2 3D characterization of the microstructure with microCT

The low porosity specimens scanned for this study are LP10, LP11, and LP12, and the high porosity ones are HP12, HP13 and HP14. Figure 6 shows the distributions of porosity size and geometry for both microstructures as a function of a porosity equivalent radius. This equivalent radius is defined as the radius of a sphere whose volume is the one of the porosity. The coefficient of sphericity  $\Psi$  is computed as

$$\Psi = \frac{\pi^{1/3}(6V_p)^{2/3}}{A_p} \quad (2)$$

with  $V_p$  and  $A_p$  the volume and the surface of the ideal ellipsoid fitting the porosity. The technique described in Ikeda et al. (2000) was applied to recover the length of the principal axes. The main difference between the two microstructures is the presence of some large spherical porosities in the HP specimens, which equivalent radii exceed  $0.3 \text{ mm}$ . These large porosities are likely to exist prior to ice growth in the ice slurry mixture of HP specimens. Smaller porosities take random ellipsoid shapes and their formation is probably related to air trapped in the water. In the following, porosities will be split into two populations : the population **A** (large spherical porosities) that only exist in HP specimens and the population **B** (small porosities with random ellipsoidal shapes), observed in both microstructures. Note that for a

311 scan at a given resolution, the uncertainty on porosity size and shape increases as the porosity  
 312 size decreases. However scans performed at higher resolutions (not presented here) confirmed  
 313 the random shape feature associated with population **B**.

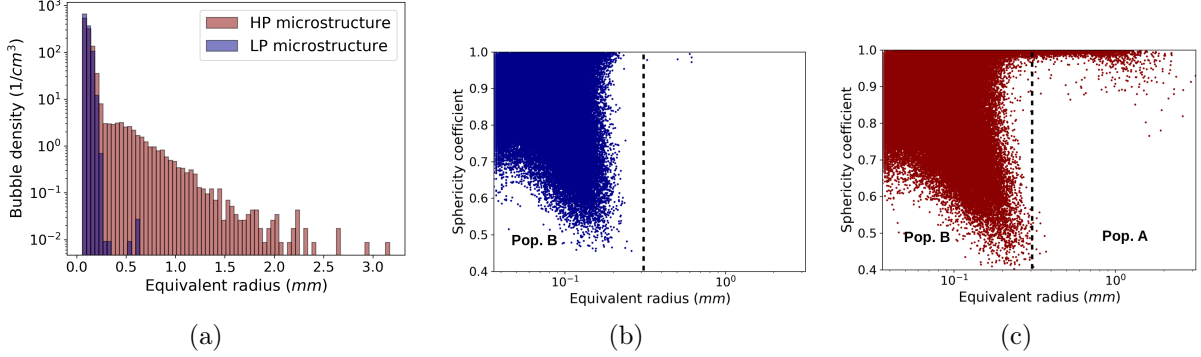


Figure 6: (a) porosity size distribution of LP11 (blue) and HP13 (red) specimens according to the porosity equivalent radius. Sphericity coefficient of each porosity for (b) LP and (c) HP specimens. The black dotted lines delimit the population **A** and **B**

### 314 **3.3 Tensile strength measurements**

315 The specimens LP12 and HP14 failed during the compression phase and were thus dismissed.  
 316 The low quality of laser signals from LP10 and HP12 tests prevented the computation of their  
 317 spalling strength. Consequently, we will treat only 12 spalling tests performed on HP speci-  
 318 mens and 11 tests on LP specimens in the following. The relevant parameters directly measured  
 319 or deduced from experimental signals, as detailed in section 2.4.2, are given in Table 2. The  
 320 data obtained from LP specimens are extracted from Saletti et al. (2019) except LP09 and LP11  
 321 which are new tests. The strain rates range between 24 and 112  $s^{-1}$  and between 41 and 120  $s^{-1}$   
 322 for HP and LP specimens respectively. The measured spalling strengths, i.e. tensile strengths,  
 323 vary between 0.8 to 3.0  $MPa$  and from 1.9 to 5.3  $MPa$  in the considered range of strain rate  
 324 for HP and LP specimens, respectively.

325

326 The results presented in Figure 7 highlight an increase of the tensile strength with strain  
 327 rate. A strength weakening with increasing porosity is also observable, LP ice appearing almost  
 328 twice stronger than HP ice for the highest strain rates. Few tests on HP specimens exceed a  
 329 strain rate of 80  $s^{-1}$  as these specimens are more likely to fail in compression at high loading  
 330 rates. Overall it is difficult for both microstructures to reach strain rates higher than 120  $s^{-1}$



<b>Spec Id</b>	$V_p$ ( $m.s^{-1}$ )	$C_{ice}$ ( $m.s^{-1}$ )	$\sigma_{spall}$ (MPa)	$\dot{\epsilon}$ ( $s^{-1}$ )	$\alpha_{exp}/\alpha_0$
LP01	6.3	3040	4.34	107	1.04
LP02	3.3	2667	1.90	41	0.94
LP03	5.1	2917	4.02	121	1.05
LP04	5.3	2992	4.40	110	1.04
LP05	5.8	2963	4.08	108	1.07
LP06	5.7	2881	4.33	120	1.07
LP07	3.9	3010	3.79	83	1.08
LP08	3.8	2794	3.16	69	1.07
LP09	7.7	3216	5.30	87	1.03
LP11	3.2	2972	3.66	43	0.97
HP01	2.9	2743	1.33	36	0.99
HP02	4.1	2669	2.14	90	1.09
HP03	2.9	2371	1.48	31	1.03
HP04	2.9	2673	1.68	54	1.10
HP05	2.9	2645	1.43	53	1.02
HP06	3.9	2589	2.96	99	1.06
HP07	3.2	2636	2.02	53	1.14
HP08	2.4	2482	1.40	24	1.03
HP09	4.0	2674	1.80	112	1.07
HP10	2.5	2530	1.52	29	1.05
HP11	4.0	2753	2.13	60	0.99
HP13	2.6	2174	0.83	33	0.80

Table 2: Projectile velocity  $V_p$ , acoustic wave velocity into the specimen  $C_{ice}$ , spalling strength to failure  $\sigma_{spall}$  and calculated strain rate in the tensile phase  $\dot{\epsilon}$  for LP and HP specimens, as obtained from the treatment of laser and strain-gauges signals.  $\alpha_{exp}/\alpha_0$  is the ratio between the experimental and theoretical transmitted energy

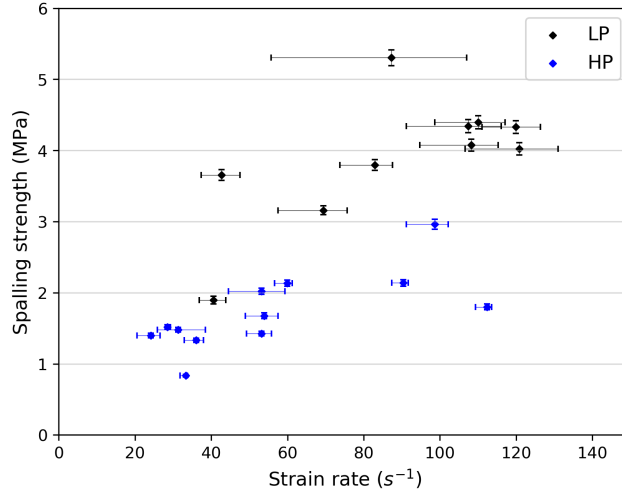


Figure 7: Tensile strength as a function of the strain rate. The displayed intervals on the curve correspond to the minimum and maximum values of strain rate measured during the tensile phase according to the elastic simulations. The marker (in between the intervals) corresponds to the mean values of the determined strain rates

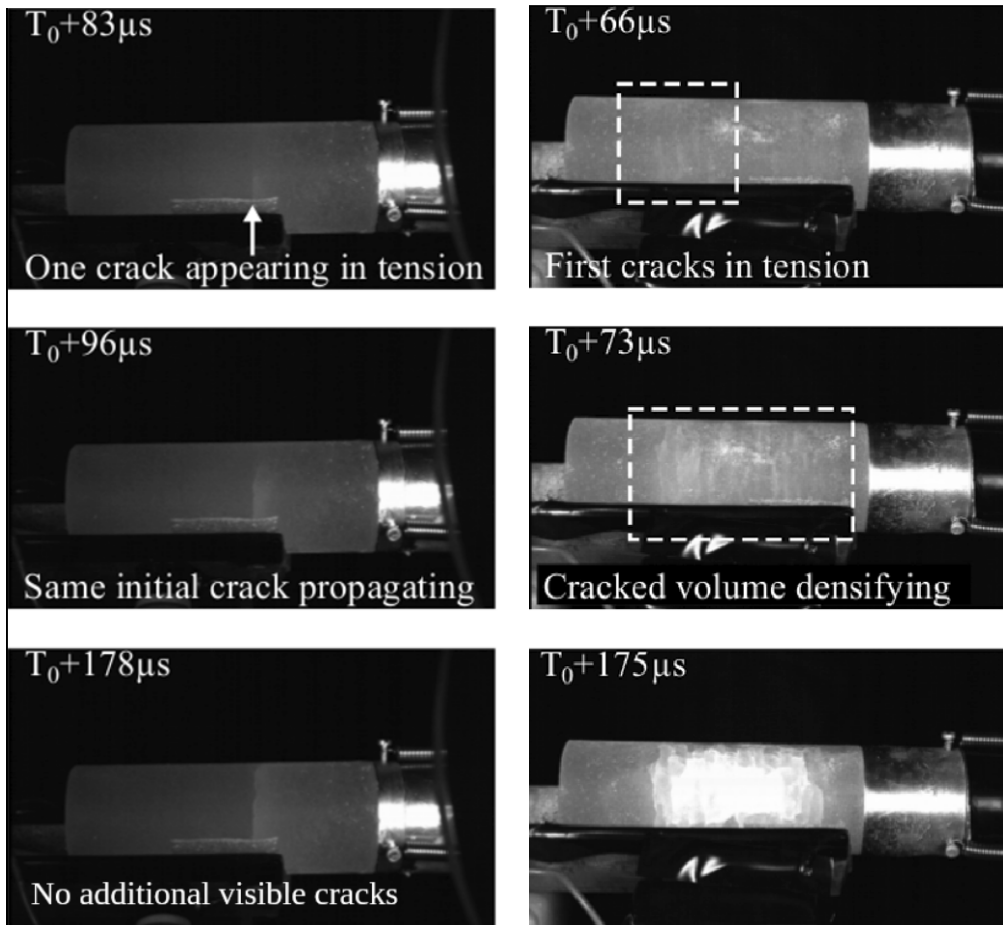
331 with the experimental configuration used here.

### 332 3.4 Fragmentation analysis from UHS camera

333 The UHS camera allowed to observe the crack propagation in the volume of the specimen  
 334 during each test. Although a qualitative piece of information, it enables to distinguish two  
 335 main scenarios as a function of the strain rate. At low strain rates the ice experiences a single  
 336 fragmentation occurring randomly in the volume loaded in tension as it can be observed in  
 337 Figures 8a and 9a. At higher strain-rates the specimens undergo intense multiple fragmentations.  
 338 In Figures 8b and 9b one can see several cracks oriented perpendicularly to the specimen axis  
 339 expanding in an initially restricted zone (white dashed-line rectangle). In the next steps of the  
 340 test, this zone of damage spreads out toward the bar side of the specimens as observed on the  
 341 last images. Finally, the higher the strain rate, the higher the crack density. However the quality  
 342 of the images did not allow to find significant variations of the fragmentation pattern between  
 343 LP and HP specimens.

### 344 3.5 Fragmentation analysis from AITA measurements

345 After spalling tests, post-mortem specimens were first scanned with the microCT (see section  
 346 3.6) and then brought back in cold rooms (-15°C) to perform thin sections and analyze the



(a) # LP02 ( $41 \text{ s}^{-1}$ )

(b) # LP04 ( $110 \text{ s}^{-1}$ )

Figure 8: Fracture in LP specimens for two different strain rates.

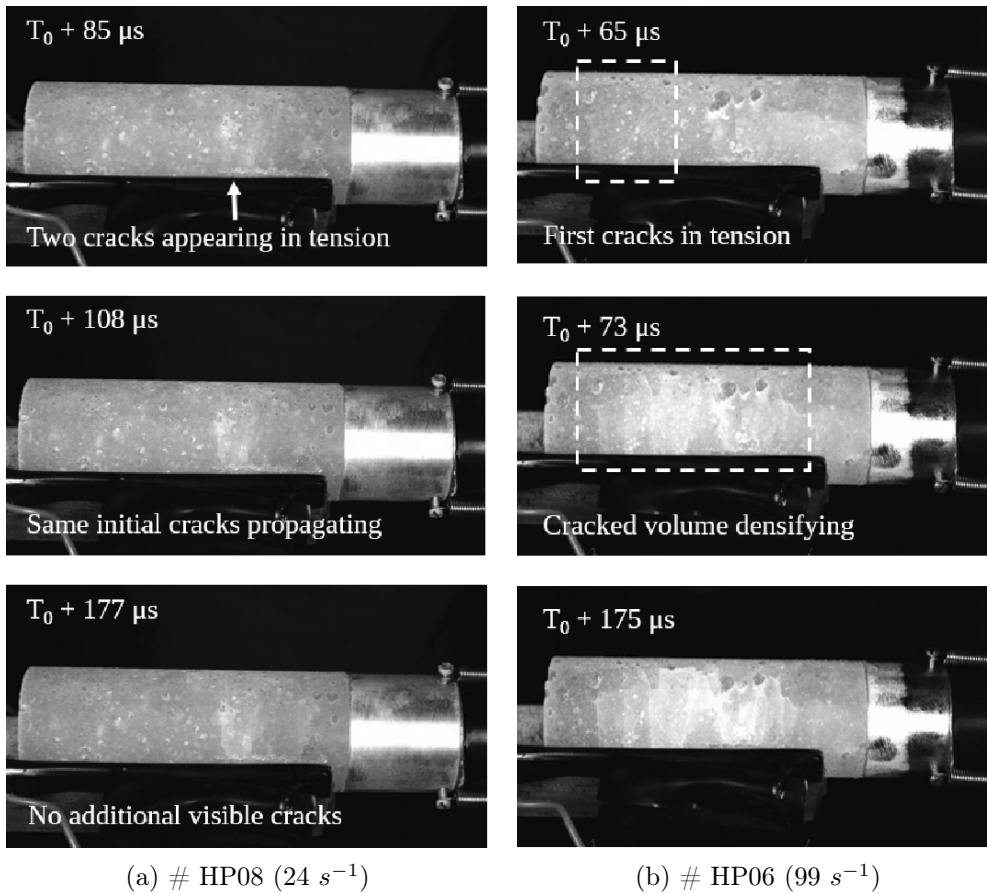


Figure 9: Fracture in HP specimens for two different strain rates.

347 cracked zones by using the Automatic Ice Texture Analyser. Areas of interest were selected  
348 around the fracture planes which could be located by eye. As described in section 2.2, 0.3 mm  
349 thick thin sections were made but a great care was taken not to create additional cracks during  
350 the operation. Pictures of the post-mortem specimens are shown in Figures 10 and 11, together  
351 with the corresponding thin sections analyzed with AITA. The cracks being very thin and there-  
352 fore hardly visible on the pictures, there were manually highlighted by white lines added on the  
353 images. For specimens that fractured into two distinct fragments, a small gap was left between  
354 the two parts during the manufacturing of the thin sections (see Figures 10b and 11b).

355

356 Although one main plane of fracture is always observable, numerous additional cracks seem  
357 to have been triggered during the tests, which orientations are roughly perpendicular to the  
358 specimen principal axis. In each thin section a branching phenomenon or cracks coalescence can  
359 be seen, especially in the LP10 specimen. No clear evidence appears in our observations about  
360 a likely influence of the microstructure in the crack junction/separation as no cracks follow the  
361 grain boundaries and no distinct direction shift could be observed at a triple junction. However,  
362 in the analyzed HP microstructures, the cracks seem to follow a route through the largest porosi-  
363 ties or to be triggered from these porosities. The crack tip is generally located in a porosity,  
364 at least on the 2D images and for cracks not crossing the entire specimen. These observations  
365 have to be considered with caution considering the high density of large porosities in the vol-  
366 ume and the fact that these images provide only 2D information. Regarding LP specimens, no  
367 evidence about some relationship between porosities and crack initiation and propagation could  
368 be established, due to a too low spatial resolution of the AITA analysis and the high density of  
369 small pores.

370

371 Unfortunately, the measurement chains were not successful for LP10 and HP12 : it was not  
372 possible to process these tests to get the spalling strength. Nevertheless, they both experienced  
373 tension during the test and UHS camera analysis reveals that no compression fractures occurred.  
374 Consequently their fragmentation pattern can still be presented in this paper as we consider they  
375 are representative of the micro-scale processes at play.

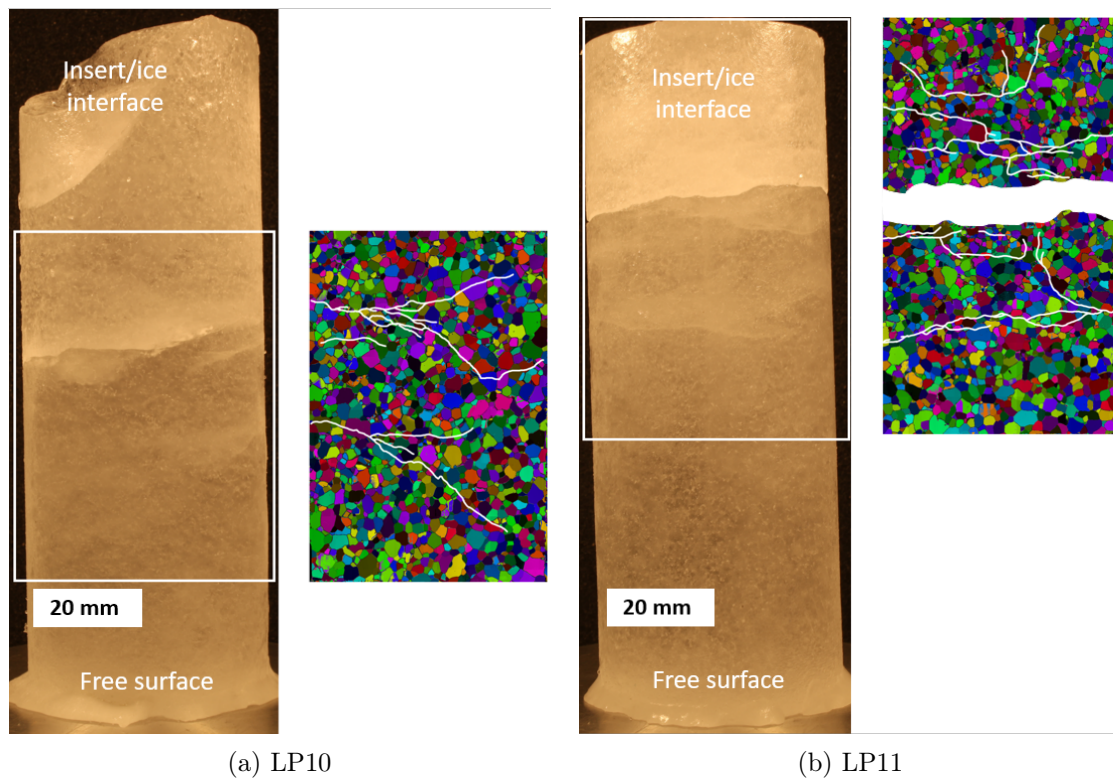


Figure 10: Post-spalling pictures and orientation color-coded microstructures of the cracked zones for two LP specimens. White lines on thin sections are manually added to make visible the crack network.

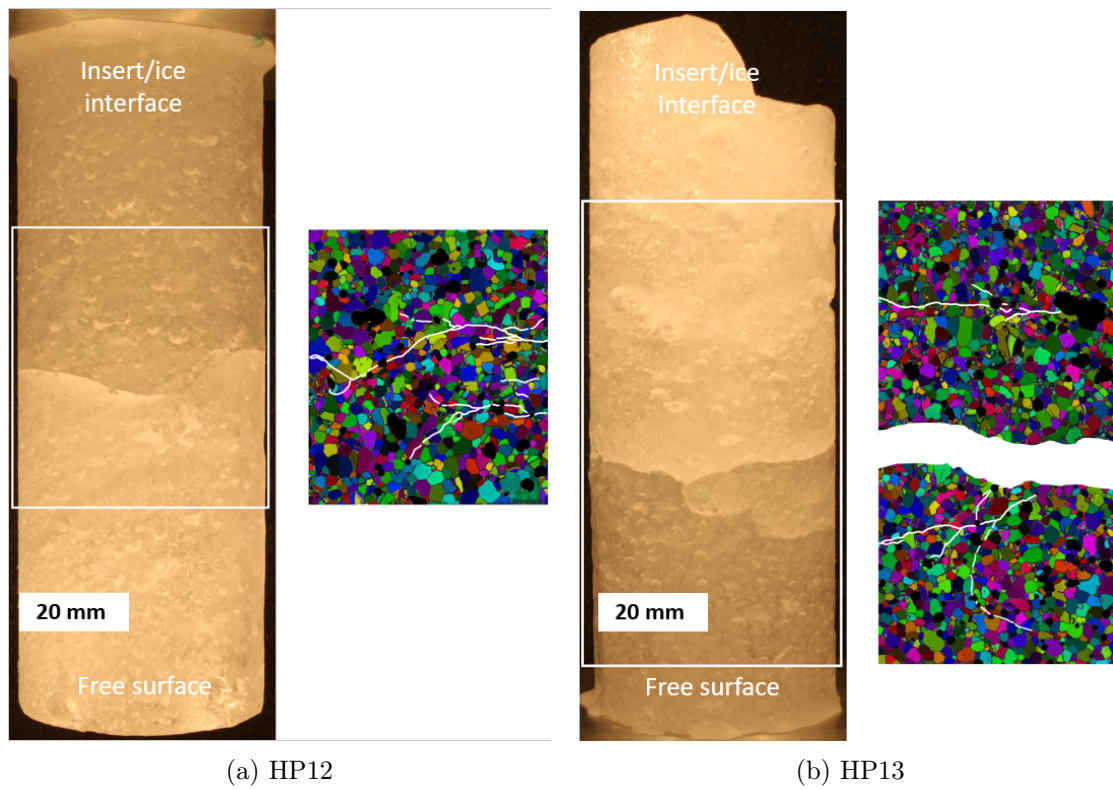


Figure 11: Post-spalling pictures and orientation color-coded microstructures of the cracked zones for two HP specimens. White lines on thin sections are manually added to make visible the crack network.

### 376 **3.6 Fragmentation analysis from micro-CT measurements**

377 LP10, LP11, HP12 and HP13 specimens were scanned within a few hours following the spalling  
378 tests, using the set-up and conditions described in section 2.3. The intense fragmentation during  
379 the spalling tests of LP12 and HP14 specimens prevented from carrying them to the micro-CT  
380 device. LP11 and HP13 specimens fractured into two pieces, fragments were put together back  
381 by taking care of a good match between them prior to scanning. We can expect some cracks  
382 healed between the spalling test and the micro-CT scans, as commonly observed when ice is  
383 subjected to a temperature gradient (Colbeck, 1986).

384

385 Examples of microCT images of cracked zones are presented in Figures 12 and 13. Except  
386 for fracture planes separating two fragments, the crack thickness appears not to exceed several  
387 microns. In addition, the grey values associated to crack volume lie in the ice value range, as  
388 shown on images (1) and (2) of Figure 12a, where impulse noise makes difficult or impossible  
389 to isolate the cracks. Consequently only the main fracture planes were efficiently identified. To  
390 do so, digital image correlation has been performed with SPAM on HP specimens by using the  
391 initial volumes scanned prior to spalling test and the fragmented volumes analyzed post-test.  
392 The transformation matrix from DIC treatment allowed to adjust the position of the two vol-  
393 umes. The only difference between the two scanned volumes being the fragmentation pattern,  
394 the largest cracks could be identified by subtracting the two 3D images. The software SPAM  
395 gives access to the exact position of each voxel in the volume, porosities in contact with the  
396 different fracture planes could thus be collected. Results are shown on images (2) and (3) of  
397 Figures 13a and 13b. A simple thresholding method was applied for LP11 specimen to isolate  
398 the main fracture plane.

399

400 The crack observed in the LP11 specimen (Figure 12b) is almost perpendicular to the tensile  
401 loading direction, with no noticeable direction shift, and covers the entire specimen thickness.  
402 The same pattern is observable on 2D images obtained with micro-CT for the LP20 specimen.  
403 The situation is different for the fracture plane of specimen HP12 where the crack, although  
404 approximately perpendicular, is more tortuous and eventually stops before crossing the total  
405 thickness. Two cracks could be isolated in the HP13 specimen, one perpendicular to the speci-



406 men axis and another oriented at around  $30 - 40^\circ$  from this axis. The junction between these  
 407 cracks occurred nearby the specimen surface. From qualitative observations, the crack surfaces  
 408 seem to be slightly rougher in the HP microstructures than in the LP microstructures.

409

410 As for the 2D analyses performed in section 3.5, we can hardly conclude on a clear influence of  
 411 porosity on crack initiation and propagation. However porosities were detected on the surface  
 412 of each crack in every specimen analyzed from microCT 3D reconstructions.

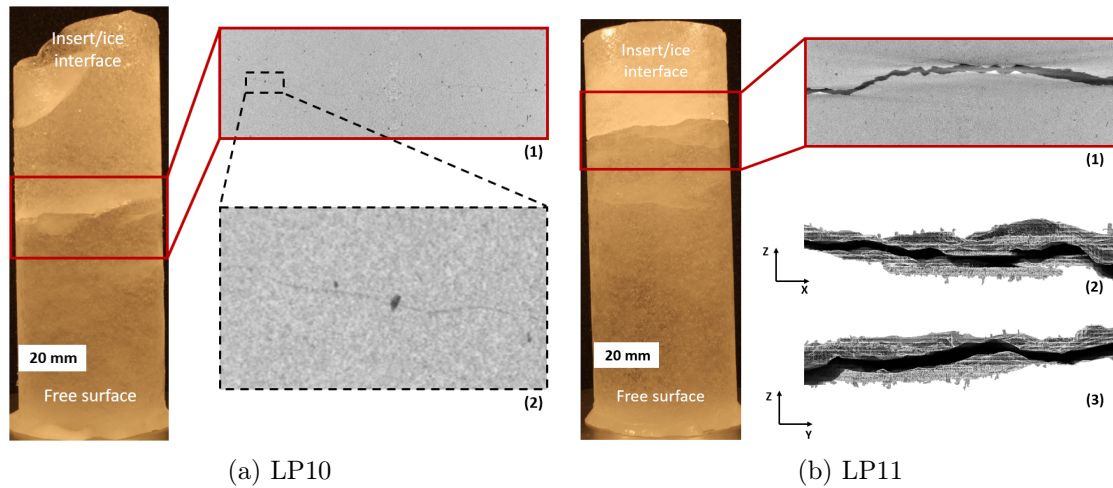


Figure 12: Observation of crack areas from microCT scans. a) specimen **LP10** : (1) 16 bits 2D image of the crack area, (2) Focus on the crack path. b) specimen **LP11** : (1) 16 bits 2D image of the crack area, (2) and (3) 3D crack visualisation according to XZ and YZ planes respectively.

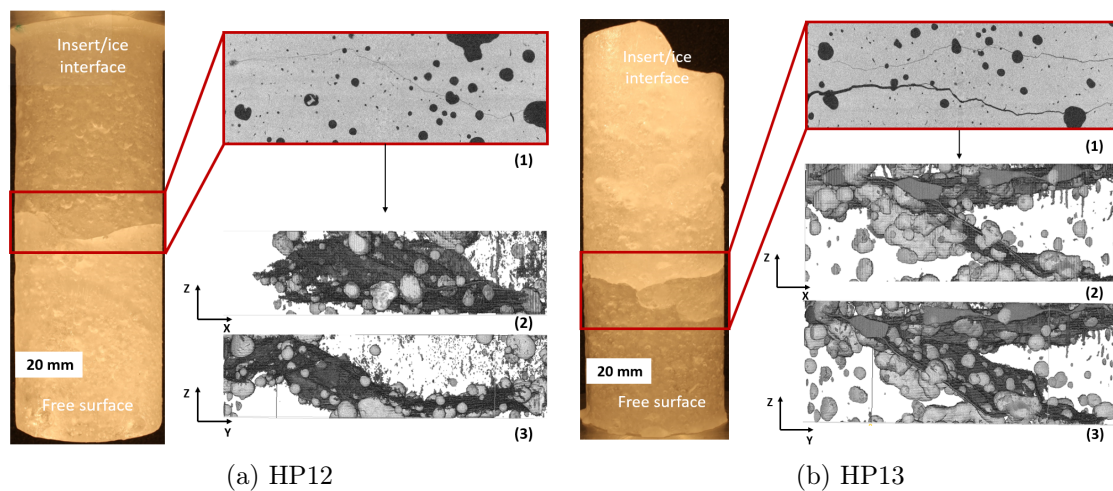


Figure 13: a) and (b): (1) 16 bits image of cracked zone, (2) and (3) 3D crack visualisation according to XZ and YZ planes respectively. The porosities in contact with crack networks are shown in white. *Figures en cours de modification (suppression du bruit et amélioration du contraste pores/fissures)*

## 413 4 Discussion

### 414 4.1 Strain-rate dependency of ice dynamic tensile strength

415 Results shown on Figure 7 evidence a strain-rate sensitivity of the dynamic tensile strength of  
416 ice, as already shown by Saletti et al. (2019) for the microstructure with a low porosity. This  
417 strain-rate hardening is a common feature found in many other brittle materials, such as con-  
418 crete, ceramics, or rocks (Cho et al., 2003; Schuler et al., 2006; Zinszner et al., 2015; Saadati  
419 et al., 2016). Under quasi-static sollicitations, ice strength has been shown to be strain-rate in-  
420 sensitive and is rather subjected to microstructural property dependency (Schulson and Duval,  
421 2009; Schulson, 2001). Based on results obtained from direct tensile tests and bending tests in  
422 the literature (Petrovic, 2003; Schulson and Duval, 2009; Timco and Weeks, 2010) , together  
423 with the results presented here, the transition between a strain-rate insensitive and a strain-rate  
424 sensitive response seems to occur approximately between  $10^{-1} s^{-1}$  and  $10^1 s^{-1}$ . This transi-  
425 tion phenomenon exists in many other brittle materials that show a probabilist behavior at low  
426 strain-rates and a deterministic behavior at higher rates of loading (Hild et al., 2003; Forquin  
427 and Hild, 2010). Despite the narrow strain-rate range covered here ( $24 s^{-1}$  to  $120 s^{-1}$ ), poly-  
428 crystalline ice appears to follow the same trend, as the dispersion of tensile strength associated  
429 with spalling tests appears much lower than the one observed in the quasi-static regime (Timco  
430 et al., 1994).

431

432 To explain this macroscopic behavior, a point of view at a microscopic scale can be used. Indeed,  
433 microstructural defects (such as porosities, grain boundaries, micro-cracks, etc) are frequently  
434 considered as responsible for crack triggering in brittle materials under tension. The micro-  
435 scopic stress required to activate a critical defect is a complex function of the defect nature,  
436 size, orientation, etc... (Jayatilaka and Trustrum, 1977). Under quasi-static conditions, a single  
437 crack is likely to be responsible for the complete specimen failure. In the dynamic range, brittle  
438 materials undergo a multiple fragmentation, as observed on ice during spalling tests (Section  
439 3.4) with a crack density increasing with strain rate. Denoual and Hild (2000) and Forquin and  
440 Hild (2010) introduced the concept of "critical defect obscuration" and "local weakest link hy-  
441 pothesis" to describe the fragmentation process in brittle solids and to predict the crack density  
442 and the tensile strength dependency with the applied strain rate. The theory lies on the growth

443 of obscuration zones which correspond to areas in which stress-release waves develops due to  
444 unstable cracks activated from the non-obscured critical defects. At low strain-rates, damage  
445 propagates to the full specimen prior to local stress increase, preventing any additional crack  
446 triggering. At high strain-rate, the limited crack speed makes possible a local stress increase,  
447 leading to the activation of a large number of cracks that propagate in small zones up to crack  
448 coalescence. Only when damaged volumes link up will the complete fragmentation of the ma-  
449 terial occur, which results into a higher macroscopic strength. As a consequence, crack density  
450 and strength is expected to increase with strain-rate.

451

452 The observations performed here on polycrystalline ice are in agreement with Forquin and Hild  
453 (2010) approach. Indeed, the high speed camera observations reveal an increase in the number  
454 of failure planes with strain rate (see Figures 8 and 9). Cracks are approximately perpendicular  
455 to the loading direction, meaning that they are mainly opening in mode I as a result of a brittle  
456 response to the tensile loading.

457

458 3D images of post-mortem specimens (Figures 12b, 13a and 13b) reveal rough and irregular  
459 profiles of crack at the microstructure scale, while the linear elastic fracture mechanics theory  
460 would predict straight cracks perpendicular to the maximum tensile stress, as long as the cracks  
461 do not interact with each other. On top of the impact of porosity on the crack propagation routes  
462 discussed in Section 4.2, the crack roughness could result from the coalescence of several small  
463 cracks. Although the limitations of our observation methods (AITA and microCT) and possible  
464 microcrack proofing, prevent from a quantitative evaluation of crack density, the observation  
465 of a relatively high number of small cracks in the vicinity of the main fracture plane would be  
466 consistent with the former hypothesis. Furthermore, would a single crack be responsible for the  
467 specimen failure, the failure process would take the minimum time for the crack to propagate  
468 through the specimen, that can be estimated at around  $20 \mu\text{s}$  (equal to  $r_{spec}/0.38C_{ice}$ ,  $0.38C_{ice}$   
469 being the supposed crack speed [Ref](#)) for specimen of radius 22.5 mm. In the case of specimen  
470 LP11, for instance, the time interval in the tensile phase to reach failure (roughly equal to  
471  $\sigma_{spall}/E\dot{\epsilon}$ ) was about  $9 \mu\text{s}$ , that is about twice shorter than the time for a crack to propagate  
472 from the specimen axis to its outer surface.

473

474 Based on these observations, we can hypothesise that the pullback velocity measured by the  
475 laser interferometer on the free surface of the specimen is indeed the result of the growth and  
476 interaction of several cracks, very likely triggered on critical defects that are supposed to be the  
477 porosities, and that the coalescence results into a macroscopic fracture plane.

## 478 **4.2 Porosity effects on ice dynamic tensile strength**

479 The strain-rate effect discussed above, although noticeable for both microstructures, is less pro-  
480 nounced for the HP microstructure. Both LP and HP microstructures have been designed to  
481 be similar in terms of grain size and shape, and crystallographic orientation, but to present  
482 two different porosities. The main difference between the two microstructures come from the  
483 presence, in the HP specimens, of large spherical porosities (population **A**). We therefore make  
484 the hypothesis that the difference observed in dynamic tensile strength between the two types  
485 of specimens arises, in a direct or indirect way, from the role of porosities. Porosities are mi-  
486 crostructure defects and therefore potential areas of stress concentration during tensile loading.  
487 Two processes could be involved, namely initiation of new cracks or/and the influence of porosi-  
488 ties on crack propagation.

489

### 490 *Crack initiation*

491

492 Several studies point out a link between the size of a microstructural defect and a corresponding  
493 critical stress for crack nucleation. Liu (1997) showed that increasing the pore size for a given  
494 porosity induced a decrease in the compressive strength of porous ceramics. Chao et al. 1992  
495 evidenced that the fracture strength distribution in sintered silicon nitride under tensile loading  
496 could be predicted from the pore size distributions. Recently, Forquin et al. (2019a) hypothesized  
497 that the porosities population found in an ultra-high performance concrete, a ceramic and porous  
498 ice could be considered to model single and multiple fragmentation properties of these materials.

499

500 Under dynamic loadings, as described in the DFH obscuration model (Denoual and Hild, 2000;  
501 Forquin and Hild, 2010), the differences observed in the dynamic responses of two materials

502 having distinct microstructural properties would arise from the activation of different defect  
503 populations. In this study, if we assume that the porosities are the only critical defects of the  
504 microstructures considered and that the threshold stress for crack triggering is depending on  
505 the pore size, the porosity weakening effect could be due to a premature fragmentation in HP  
506 specimens. The porosities belonging to population **A** (present only in HP specimens) would  
507 initiate cracking phenomena at lower stresses than porosities from population **B** (present in  
508 both types of microstructures). At very high strain-rates (not reached here) and if the afore-  
509 mentioned hypothesis are still valid, one could expect similar dynamic behavior from LP and  
510 HP specimens as the pore size distributions for small porosities tend to be the same in both  
511 microstructures.

512

513 Although the 2D or 3D analyses of post-mortem specimens presented here are not accurate  
514 enough to clearly identify the role of porosities in crack triggering, we observed a large number  
515 of porosities from population **A** located along cracks in HP specimens. In addition, porosities  
516 from population **B** adjacent to cracks in LP specimens were also detected on the microCT im-  
517 ages (see for instance the image 2 of Figure 12a) . On top of that, cracks propagate through  
518 the grains (see Figures 5a and 5b) implying that grain boundaries or at a triple junction are not  
519 major initiation areas.

520

### 521 *Crack propagation*

522

523 As shown previously (Section 3.4), and as usually observed for quasi-brittle materials, the crack  
524 density is increasing with strain-rates. The main explanation comes from the limited crack ve-  
525 locity that restricts the growth of obscured volumes making possible the activation of more  
526 cracks on non-obscured critical defects. However, under quasi-static loadings or at moderate  
527 strain-rates when the material is not highly fragmented (as the ones undergone by LP10, LP11,  
528 HP12 and HP13 for instance), crack propagation could be dependent on the presence of mi-  
529 crostructural defects. Renard et al. (2009) showed that mechanical heterogeneities (such as pores  
530 and grains) in rocks may affect a fracture propagation by acting on the stress field near a crack  
531 tip under quasi-static loading. They observed an attraction phenomenon from porosities on

532 the final crack path hence a subsequent amplitude of its out-of-plane fluctuations. Chen et al.  
533 (2016) investigated the effect of pores on the stress-intensity factors of the crack tips in porous  
534 ceramics, by using the J-integral numerical method. Pores in front of the propagating cracks  
535 was found to promote crack propagation by enhancing the stress intensity factor of the crack  
536 tip. For ice material, Smith et al. (1990) experimentally established a porosity weakening of  
537 ice fracture toughness, implying porosity would facilitate crack propagation. Here, rough and  
538 wavy crack paths described in section 3.6 tell us about a likely role of porosity on crack prop-  
539 agation prior to failure. The presence of large spherical porosities on the crack path (Figures  
540 11 and 13) could illustrate how porosities would distort the crack propagation resulting in the  
541 roughness and out-of-plane fluctuations observed on crack surfaces of HP specimens. Also, the  
542 redistribution of the stress-field ahead of a crack tip could potentially activate new micro-cracks  
543 on porosities into the vicinity of the crack (Ref). Based on the work of Smith et al. (1990) and  
544 regarding the high density of porosities from population **A**, we can expect significantly lower  
545 fracture toughness in HP specimens compared to LP ones. Thus, micro-cracks coalescence is  
546 probably more efficient in the highly porous specimens, hence a faster damage propagation in  
547 the latters inducing lower tensile strength to failure.

548

## 549 **5 Conclusion**

550 Following on the work of Saletti et al. (2019), we performed spalling tests on polycrystalline ice.  
551 Specimens with two different levels of porosity were considered in order to study the porosity  
552 effect on the dynamic response of ice under tensile loading. Fragmentation processes were also  
553 investigated on post-mortem specimens by means of an Automatic Ice Texture Analyser (AITA)  
554 and a micro-Tomograph (TOMOCOLD). A strain-rate hardening of the tensile strength and the  
555 crack density have been observed for both microstructures over the range  $24 \text{ s}^{-1}$  to  $120 \text{ s}^{-1}$ ,  
556 as expected for a material exhibiting a brittle behavior. However, porosity seems to weaken  
557 the ice tensile strength, the strain-rate influence being less marked with highly porous ice. The  
558 presence of large spherical porosities in highly porous specimens is thought to be responsible  
559 of the porosity weakening observed, as no additional differences could be detected between the  
560 two microstructures. The role of these porosities has been discussed in term of crack initiation

561 and propagation. We made the assumption that the critical stress susceptible to initiate a  
562 crack from a porosity is function of the porosity size, leading to a premature activation of the  
563 large spherical porosities in highly porous ice. This theory is supported by the high density  
564 of porosities located along crack paths. Rough crack surfaces observed in 2D and 3D analysis  
565 also show that porosities potentially accelerated the crack propagation and crack coalescence.  
566 All these observations tend to confirm the role of porosity as a key parameter to describe the  
567 dynamic behavior of ice subjected to tensile loading.

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