

# Observation of microstructures of atmospheric ice using a new replica technique

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## Abstract

A replication technique has been developed to study the microstructure of atmospheric ice based on the use of nail varnish rather than more harmful materials. The potential of the technique was demonstrated by obtaining and reporting microstructures for impact ice grown on metal surfaces in an icing tunnel under a range of cloud conditions. The technique reveals grain structure, growth striations, porosity and etch features which may indicate an aspect of crystallographic orientations.

### Keywords:

atmospheric ice, impact ice, microstructure, grain size

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1 In order to study the mechanical response of atmo- 27  
2 spheric ice including adhesion to a substrate, knowledge 28  
3 of the ice's microstructure is of prime importance. At- 29  
4 mospheric ice by its nature is very different from other 30  
5 types of ice and varies significantly in its microstruc- 31  
6 ture. Impact ice is formed by the freezing of super- 32  
7 cooled droplets when they strike a surface. Lake ice, 33  
8 sea ice or other types of ice grown by cooling a body 34  
9 of water are dominated by the fact that the cooling is 35  
10 limited by thermal conduction effects. This generally 36  
11 results in producing much coarser grain size, modifying 37  
12 grain aspect ratio and influencing pore size and distribu- 38  
13 tion compared to impact ice. 39

## 1. Previous work

14  
15 While knowledge of the microstructure of ice is im- 43  
16 portant for understanding the fracture process and ice 44  
17 adhesion, as it is in many structural materials, only a 45  
18 small number of authors have reported such data. This 46  
19 is perhaps due to the difficulty of handling a material 47  
20 which is easily broken and modified by a slight change 48  
21 of temperature. 49  
22 Different methods to observe ice microstructure have 50  
23 been reported by Blackford (2007). Depending on the 51  
24 methods used, different level of details can be studied 52  
25 and they all present their advantages and drawbacks. 53  
26 The most direct method, and most commonly used, is 54

55 to make and observe a thin slice of ice under a micro- 56  
57 scope using reflecting or transmitted light. With a reso-  
lution of  $1\ \mu\text{m}$ , this method allow the observation of the  
size, shape and distribution of the grains as well as the  
location and distribution of the impurities. The use of  
polarized light gives more information on the crystallo-  
graphic orientation of each grains (Durand, 2006). The  
main drawbacks of the methods resides in the prepara-  
tion of thin sections of ice and in avoiding any transfor-  
mation of the ice microstructure with time.

To overcome the difficulties of handling and keeping  
the sample intact, several authors (Nasello et al., 1987;  
List et al., 1970; Higuchi, 1958; Fortin and Perron,  
2009) have used a replication technique based on a so-  
lution of formvar dissolved in a solvent such a chlo-  
roform. This is known to be toxic and carcinogenic  
(Takahashi and Fukuta, 1987). Other authors (Taka-  
hashi and Fukuta, 1987; Kriston et al., 2016) have used  
an alternative to formvar. Takahashi and Fukuta have  
successfully replicate ice crystal using common plastics  
dissolved in solvent and discussed the ability of each  
combination. Best results were obtained using plexi-  
glas with ethylene dichloride or trichloroethylene and  
polystyrene with 1,1,1-trichloroethane or carbon tetra-  
chloride. While these solvents have the advantage of  
remaining clear, compared to formvar, due to the ab-  
sence of water soluble ingredients, they are still harm-  
full. Kriston et al. reported work on the effect of the  
friction of rubber tyre on ice. They replicated the ice  
surface by using a dental casting material called vinyl  
polysiloxane. They obtained a negative replica of the

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58 ice surface which was then used as a mould to obtain  
59 a positive replica using polyurethane. Depending on  
60 the level of detail required, optical microscope, SEM  
61 or white light interferometer was used. Grain and sub-  
62 grain boundaries, etch pits, frost and scratches due to  
63 the effect of multiple sweeping of rubber on ice were  
64 observed.

65 In the present paper, a similar technique has been tested  
66 but by replacing the solution of formvar with less harm-  
67 full nail varnish. While nail varnish composition gener-  
68 ally includes some kind of alcohol and acetone, the  
69 proportion of these in some nail varnish is low enough  
70 to not cause any damage to the ice surface. A descrip-  
71 tion of the method and the level of detail that can be  
72 obtained is presented in the following.

## 73 2. Methodology

74 Samples of ice were produced in Cranfield Icing Tun-  
75 nel (Hammond, 2003) as a by product of mechanical  
76 tests. Ice was accreted on the front face of cylinders  
77 made of either Titanium alloy Ti-6Al-4V or Aluminium  
alloy Al2024-T3 (figure 1). The substrate surfaces were

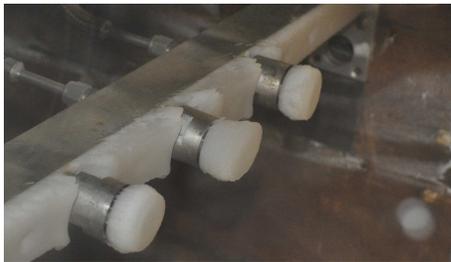


Figure 1: Samples of ice accreted on the front face of metallic cylinders ( $T=-15^{\circ}\text{C}$ ,  $V=80\text{ m.s}^{-1}$ ,  $\text{LWC}=0.3\text{ g.m}^{-3}$ ,  $\text{MVD}=20\text{ }\mu\text{m}$ )

78 polished to mirror finish with a resulting roughness of  
79  $0.3\text{ }\mu\text{m}$  (Ra). The pieces of ice were detached from the  
80 metallic surface using a hammer and a chisel and po-  
81 sitioned at the bottom of a chest freezer straight after  
82 removal. The removal was completely adhesive mean-  
83 ing that the substrate surface was visually free of ice.  
84 The ice pieces were kept in loose fitting plastic bags in  
85 the freezer at a temperature of  $-18^{\circ}\text{C}$  for a couple of  
86 weeks before the surface were replicated using the nail  
87 varnish technique. During this resting time, sublima-  
88 tion will have occurred revealing the grain boundaries  
89 as this process is faster along the grain boundaries than  
90 within the body of the grain. A layer of nail varnish  
91 was applied to the surface of interest using the small  
92 brush provided with the nail varnish bottle. After a dry-  
93 ing time of 14 to 20 hours, the nail varnish layer was  
94

95 peeled off the ice giving the replica including imprint of  
96 the peaks and groves present on the ice surface. Three  
97 different types of replica were collected representing  
98 different orientations and regions of each ice specimen.  
These are shown in figure 2.

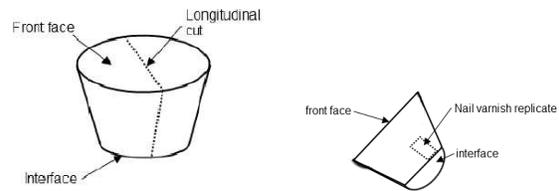


Figure 2: surface replica of the ice sample

99 The three types of replicas collected are referred as  
100 follows:  
101

- 102 - the surface which was in contact with the substrate  
103 (called the interface)
- 104 - the surface at the other extremity (called the front  
105 surface)
- 106 - a longitudinal surface which was obtain by cutting  
107 the ice piece normal to the substrate surface

108 In most of the samples, the interface and the front sur-  
109 face were both replicated directly without the need to  
110 prepare the sample further. When the surface was not  
111 flat enough (it is mostly the case with the front surface  
112 of glaze ice), the ice surface was manually polished us-  
113 ing carbide paper. As this process leaves some groves  
114 onto the surface, the ice piece needed to be left in the  
115 freezer for a further week to allow the sublimation pro-  
116 cess to remove them. The longitudinal surface was cut  
117 in a cooled environment ( $-15$  to  $-20^{\circ}\text{C}$ ) using a saw  
118 with relatively large teeth to help the sawing action. The  
119 saw was also cooled prior to any sample cutting. One  
120 of the two halves was chosen and further ground with  
121 fine grain metalurgical grade silicon carbide paper (180  
122 grit). The sample was then left to sublimate for several  
123 days in the freezer. The nail varnish was applied using  
124 from the interface side to the front surface (figure 2).  
125 Replicas were prepared for the microscopic observation  
126 by sandwiching them between a microscope slide and  
127 a cover slip. They were observed under transmited un-  
128 polarised light in a microscope and photos taken. The  
129 photos were stitched together using the software iMerge  
130 <sup>1</sup>. The average grain size was obtained, for each case, by

<sup>1</sup>iMerge is an images stacking and mosaic-making software con-  
ceived primarily for astronomical purpose

131 using the mean linear intercept method on ten separate  
132 measurements (standard deviation of 10%).

### 133 3. Description of the microstructure obtained

134 The images collected allow us to see the grain bound-  
135 aries of the ice sample as well as the presence of air  
136 pockets. In some cases, when the grains dimensions are  
137 large, some thermal etch pits becomes visible.

#### 138 3.1. Different surfaces of the same piece of ice

139 Figure 3 to 5 present the three different surfaces of in-  
140 terest of the same piece of ice as described above. The ice  
141 was produced at a relatively high ambient tunnel temper-  
142 ature ( $-5^{\circ}\text{C}$ ), a moderate tunnel wind speed ( $50\text{ m}\cdot\text{s}^{-1}$ )  
143 and relatively low LWC ( $0.3\text{ g}\cdot\text{m}^{-3}$ ). The resulting ice  
144 had a transparent aspect but was not completely glaze.  
145 The deposit temperature, measured with a thermocou-  
146 ple placed inside the metallic cylinder (on which the ice  
147 was accreted on) close to the surface, was slightly be-  
148 low the melting point of ice at  $-1^{\circ}\text{C}$ . The freezing frac-  
149 tion (fraction of the liquid water that becomes ice on  
impingement) was estimated around 0.5.

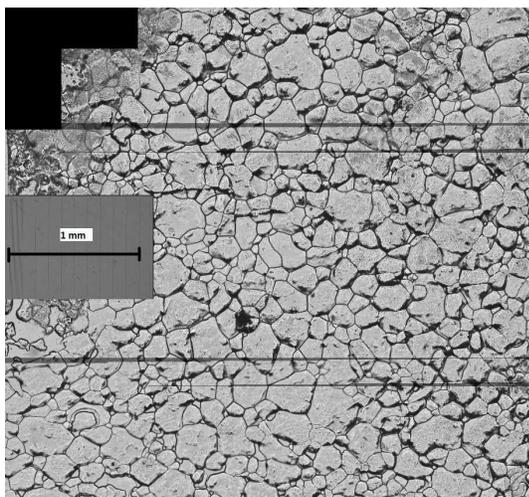


Figure 3: Microstructure of the interface of ice built on a mirror polished surface finish titanium alloy substrate ( $T=-5^{\circ}\text{C}$ ,  $\text{LWC}=0.3\text{ g}\cdot\text{m}^{-3}$ ,  $V=50\text{ m}\cdot\text{s}^{-1}$ ,  $\text{MVD}=20\text{ }\mu\text{m}$ )

150 Figure 3 shows the aspect of the grain at the interface  
151 ice/substrate. The polycrystalline structure of this type  
152 of ice was clearly visible. The average size of the grains  
153 was  $155\text{ }\mu\text{m}$  in the plane of the section. The straight hor-  
154 izontal lines and the fading of light in some part of the  
155 picture is an artefact effect due to the mosaic composi-  
156 tion of the photos. The microstructure of the far side of  
157

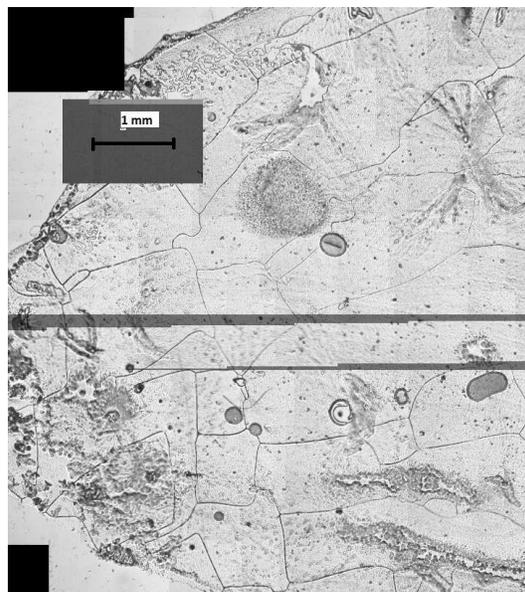


Figure 4: Microstructure of the front face of ice built on a mirror polished surface finish titanium alloy substrate ( $T=-5^{\circ}\text{C}$ ,  $\text{LWC}=0.3\text{ g}\cdot\text{m}^{-3}$ ,  $V=50\text{ m}\cdot\text{s}^{-1}$ ,  $\text{MVD}=20\text{ }\mu\text{m}$ )

158 the ice block, the front side, can be observed in figure  
159 4. Even if the scale is slightly different, it can imme-  
160 diately be seen that the grains are much larger than at  
161 the interface. The average size of the grains was  $1094\text{ }\mu\text{m}$ .  
162 A few round big bubbles can be spotted. These  
163 are water bubbles that have been trapped when the ice  
164 replica was sandwiched between the microscope slide  
165 and the cover slip for the observation under the micro-  
166 scope. More details of the evolution of the grain struc-  
167 ture can be observed in figure 5. This figure represents  
168 the microstructure on a longitudinal cut through the ice  
169 piece. The part closer to the interface is on the bottom  
170 side of the picture while the front face is on the top side.  
171 The grains can be seen to have a columnar structure.  
172 They are much narrower at the interface and gradually  
173 became wider near the front face. The black spots are  
174 the results of crystallisation of the nail varnish replica  
175 and are an artefact obtained when the nail varnish is left  
176 for too long on the ice sample during the replication pro-  
177 cess.

#### 178 3.2. Air bubbles

179 Another feature that can be observed using the nail  
180 varnish replica method is the presence and the size of  
181 air pockets within the ice. These air pockets can be  
182 of different kind and differ mainly by the way they are  
183 formed. One of the kind is due to air contained in

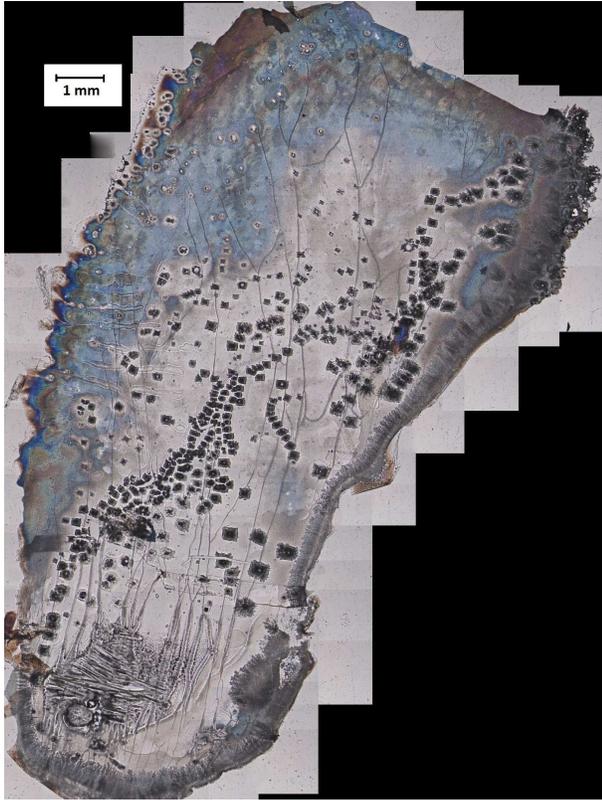


Figure 5: Microstructure of the longitudinal cut of ice built on a mirror polished surface finish titanium alloy substrate ( $T=-5^{\circ}\text{C}$ ,  $\text{LWC}=0.3\text{ g.m}^{-3}$ ,  $V=50\text{ m.s}^{-1}$ ,  $\text{MVD}=20\text{ }\mu\text{m}$ )

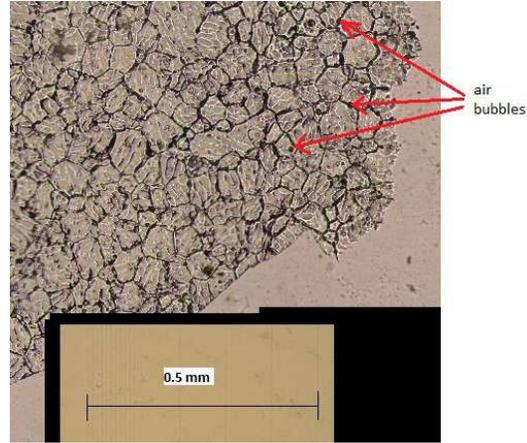


Figure 6: Microstructure of the interface of ice built on a mirror polished surface finish aluminium alloy substrate ( $T=-20^{\circ}\text{C}$ ,  $\text{LWC}=0.3\text{ g.m}^{-3}$ ,  $V=50\text{ m.s}^{-1}$ ,  $\text{MVD}=20\text{ }\mu\text{m}$ )

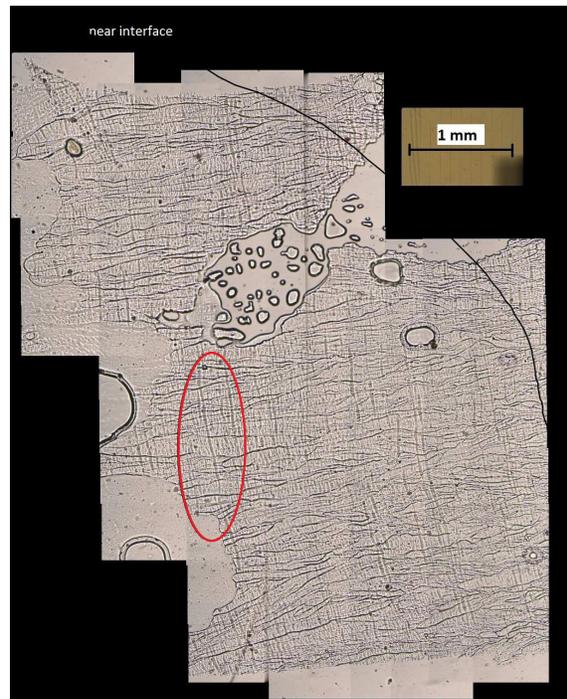


Figure 7: Microstructure of the longitudinal cut of ice built on a mirror polished surface finish titanium alloy substrate ( $T=-15^{\circ}\text{C}$ ,  $\text{LWC}=0.3\text{ g.m}^{-3}$ ,  $V=80\text{ m.s}^{-1}$ ,  $\text{MVD}=20\text{ }\mu\text{m}$ )

184 the water droplets which is dissolved during the ice ac-  
 185 cretion and remained trapped within the ice (Eskandari-  
 186 an and Farzaneh, 2005; Macklin, 1962). Another kind  
 187 which happens mainly with rime ice at low LWC, is  
 188 formed when the water droplets impinge the surface and  
 189 dry patches are created between each droplet.  
 190 Tiny air bubbles can be seen on figure 6 as small black  
 191 dots especially present along the grain boundaries. This  
 192 picture represents the interface part of an ice sample  
 193 grown on mirror polished aluminium alloy at a tunnel  
 194 total temperature of  $-20^{\circ}\text{C}$ , a speed of  $50\text{ m.s}^{-1}$ , a LWC  
 195 of  $0.3\text{ g.m}^{-3}$  and a droplet mean volume diameter of  $20\text{ }\mu\text{m}$ .  
 196 These icing conditions resulted in an ice sample  
 197 which had a white appearance and a freezing fraction of  
 198 1. This means that each droplets coming into contact  
 199 with the metallic surface would freeze completely when  
 200 impinging the surface. This type of ice is commonly  
 201 referred as rime ice. On this particular example, the av-  
 202 erage size of the grains was  $52\text{ }\mu\text{m}$  and the size of the air  
 203 bubbles was around  $10\text{ }\mu\text{m}$ .

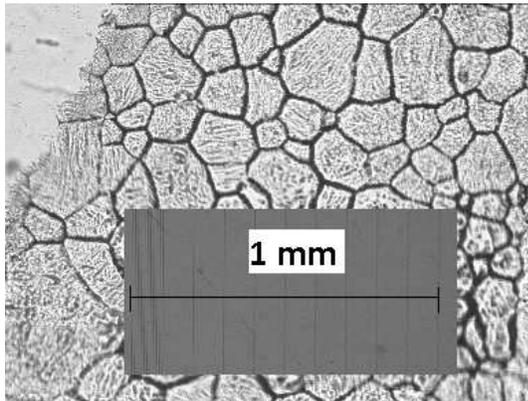
204 Another type of bubbles, more accurately referred

205 as porosity and described previously by Laforte et al.  
 206 (1983), can be spotted on figure 7 (an example of them  
 207 has been circled in black on the figure). This picture  
 208 represents the microstructure of a longitudinal cut of a  
 209 sample of ice grown on mirror polished titanium alloy at  
 210 a temperature of  $-15^{\circ}\text{C}$ . The grains here were columnar

211 and relatively elongated in respect to their width (aspect ratio of around 20). A few of the black round dots described before can be spotted but more interestingly, some faint wavy vertical lines can be observed almost perpendicular to the grain boundaries. These are made of tiny bubbles and represent variations in fine porosity. They also allow us to visualise the advancement of the growth of the ice front.

### 219 3.3. Thermal etch pits

220 The last feature described in this paper have been previously reported by Nasello et al. (1987) and Kriston et al. (2016). It was only observed when the size of the grains were reasonably wide (above  $100 \mu\text{m}$ ).



223 Figure 8: Microstructure of the interface of ice built on a mirror polished surface finish aluminium alloy substrate ( $T=-10^\circ\text{C}$ ,  $LWC=0.8 \text{ g.m}^{-3}$ ,  $V=50 \text{ m.s}^{-1}$ ,  $MVD=20 \mu\text{m}$ )

224 On figure 8, faded greyish non continuous small lines can be spotted inside most of the grains. These are thought to be arrays of thermal etch pits and can give some indication of the orientation of the crystal c-axis.

### 228 4. Conclusion

229 It has been demonstrated that nail varnish may be used to observe the microstructure of atmospheric ice. This method reduced considerably the preparation of the sample compared to more conventional method used previously to observe the microstructure of ice. Furthermore the ice replicas are not destroyed by time as would a thin slice of ice and can be observed as many times as needed.

237 These replicas allow the observation of many features as the size and shape of the grains, the presence of air inclusions, the growth lines of ice and the thermal etch pits that give an indication of the orientation of the c-axis.

242 The technique offers a relatively simple way to observe ice microstructures so that it may be possible for more research on ice feature to be supported with reporting ice microstructural information.

### 246 5. Acknowledgement

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