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# Freeze Bond Failure Under Tensile Stress due to Flexural Loading

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## ABSTRACT

The flexural strength of ice plates bonded together by freezing water (freeze-bond) is investigated in this work. Freshwater S2 columnar grained ice was used as a parent material to be bonded; water of salinities ranging from 0 to 35 ppt was used to generate bonds. Freezing occurred in air at temperatures ranging from -3 to -25 °C and under compression of about 4 kPa for periods of time varying from 0.5 to ~100 hrs. The bond strength was measured under 4-point bending. The study revealed that the flexural strength of bonded ice decreases with an increase in both salinity and temperature. The flexural strength of freshwater bonds is similar or higher than the flexural strength of intact parent material after less than 0.5 hrs freezing. The strength of the saline ice bonds levels off within ~6-12 hours of freezing.

KEY WORDS: Ice mechanics; Flexural strength; Failure; Freeze bond; Rheology

#### **1. INTRODUCTION**

Freeze bonds are common features in Arctic and Antarctic regions. They form between separate ice bodies (ice floes, ice rubble blocks, etc.) coming into contact owing to compression or shear of ice sheets or to ocean waves. Freeze bond formation is attributed to sintering and thermal freezing. The strength of freeze-bonds affects the mechanical properties of refrozen ice floes and ice rubble piles (Boroojerdi et al., 2020b, 2020a; Ettema and Urroz, 1989; Høyland and Møllegaard, 2014; Liferov, 2005; Liferov et al., 2002; Lu et al., 2019; Salganik et al., 2020; Shafrova and Høyland, 2008; Surkov et al., 2001; Surkov and Truskov, 1993). It is, for example, argued that the initial failure of an ice rubble pile is reached when the freeze bonds within it fail. Numerical simulations (Polojärvi and Tuhkuri, 2013) suggest that the individual freeze bonds within deforming rubble do not fail through shear, but rather through tension. Similarly, ocean waves are known to affect mechanical behavior and to activate failure of sea ice covers (Collins et al., 2015; Murdza et al., 2020c; Shen, 2017; Squire, 2020) as, during wave-ice interaction, an ice cover bends up and down and vertical cracks form owing to flexural deformation. After the formation of wave-induced vertical cracks, freeze bonds that form the cracks deform and may fail under a tensile state of stress. It is relevant to ask whether the flexural failure of refrozen ice floes is initiated at freeze bonds.

The strength of freeze bonds has been previously measured only under either compressive or shear loading. To our knowledge, no measurements have been made under tensile loading at time and length scales relevant to problems in ice engineering. For this purpose, we conducted

four-point-bending tests on bonded ice using apparatus described earlier (Murdza et al., 2020c). Freeze bonds were formed between milled surfaces of specimens of freshwater ice (termed the parent material) and bond freezing was performed in air under a compressive stress of ~ 4 kPa. The freezing time ranged from 0.5 hrs to 100 hrs; the ice temperature ranged from -25 to -3 °C; the salinity of the water used to form the bond ranged from 0 to 35 ppt. The results from these experiments are the first set on the failure of freeze bonds under tensile stress.

# 2. EXPERIMENTAL PROCEDURE

Freshwater columnar-grained ice that we used in the present experiments as the parent material was produced at Dartmouth's Ice Research Laboratory in the manner described previously (Golding et al., 2010; Smith and Schulson, 1993). Briefly, tap-water was frozen unidirectionally from top to bottom in an 800 L circular polycarbonate tank, forming pucks of ~1m in diameter and ~0.3 m in thickness. Freezing occurred under a top-loaded cold plate maintained at  $T = -20\pm0.1$  °C over a period of about 7 days. Before bringing the cold plate into contact with the water, the top surface of the water was seeded with freshwater ice crystals of  $\sim 0.3 - 1$  mm in diameter. The ice generally was bubble-free, columnar-grained with the S2 growth texture; thin-section analysis showed that the c-axes were randomly oriented within the horizontal plane of the ice and confined more or less to that plane. The average column diameter, as measured in a plane normal to the direction of growth using the linear intercept method, was  $5.5\pm1.3$  mm. The ice density was  $914.1\pm1.6$  kg·m<sup>-3</sup> (Golding et al., 2010). Once the ice had been grown, it was stored in plastic cooler boxes in a cold room at -10°C. A detailed description of the growing procedure of ice, its characterization, and further specimen preparation and loading can be found elsewhere (Iliescu et al., 2017; Murdza et al., 2018, 2019, 2020c).

Samples were prepared by milling from the ice pucks thin plates of dimensions of  $h \sim 15$  mm in thickness (parallel to the long axis of the grains),  $b \sim 85$  mm in width, and  $l \sim 300$  mm in length. Specimens were allowed to equilibrate to the test temperature for at least 24 hours prior to testing.

With an interest in mimicking the freeze bonds forming into vertical cracks within a broken and refrozen ice cover, we cut the plates perpendicular to their long axis into two parts using a band saw. The sawn surfaces were milled after cutting. The two parts of the specimen were then placed in a cold room with a temperature of  $+2^{\circ}$ C for a few minutes. The next step was to initiate freeze-bond formation and growth. For this purpose, the milled surfaces of two parts of a sample were sprayed with a fine mist of water at a temperature of  $+2^{\circ}$ C and brought into contact in a freeze-bonding rig (shown in Figure 1). In addition, we injected ~ 0.1 ml of water to the bond with a syringe to ensure uniform wetting of the surfaces. Finally, the freeze-bonding rig was moved to another cold room with a desired test temperature.

Figure 1 shows a photograph of the freeze-bonding rig. The rig had a system consisting of two plastic bars, two springs and metal fixation for applying a desired compressive load to the bond during freezing. In the present experiments, a confining pressure of  $\sim 4$  kPa was chosen, which is in accordance with the maximum hydrostatic pressure within submerged 10-meter-thick ice rubble mass (Ettema and Schaefer, 1986). The rig was kept in a cold room of the desired temperature, varying from -25°C to -3°C, during freezing. The base and the T-shaped walls of the rig were made from an acrylic material having low heat conductivity, ensuring the heat flux in the bond area was mainly along the long axis of the sample. All materials of the rig were

such that the frictional resistance between them and ice was low. This enabled good control of the confining pressure and sample alignment.



Figure 1. Photograph of the freeze-bonding rig with an ice sample having the shape of a thin plate.

To investigate the effect of salinity on the bond strength, we used both freshwater and saline water for spraying and wetting the milled surfaces of ice specimens to be in contact. Saline water salinities ranged from 2 to 35 ppt (parts per thousand by weight). Saline water was prepared in the manner described previously (Golding et al., 2010, 2014), i.e. by adding the commercial product "Instant Ocean" salt mixture to the tap water. Salinity was measure using a calibrated YSI Pro30 conductivity salinity meter.

After a desired time of freezing, varying from 0.5 to  $\sim 100$  h, we removed the freeze-bonded sample from the rig and measured its flexural strength under four-point bending. For this purpose, a servo-hydraulic loading system with a custom-built four-point loading frame was utilized (see Iliescu et al. (2017); Murdza et al. (2018), (2019), (2020c), (2020a), (2020d) for details). The hydraulic actuator was driven under displacement control and a calibrated load cell was used to measure the load.

The experiments were performed at an outer-fiber center-point displacement rate of 0.1 mm s<sup>-1</sup> (or outer-fiber strain rate of about 1.4 x 10-4 s<sup>-1</sup> according to linear-elastic first-order beam theory). This displacement rate resulted in an outer-fiber stress rate of about 1 MPa s<sup>-1</sup>. The major outer-fiber stress  $\sigma_f$  was calculated as:

$$\sigma_f = \frac{3PL}{4bh^2} , \qquad (1)$$

where *P* is the applied load and *L* is the distance between the outer pair of loading cylinders and is set by the geometry of the apparatus to be L = 254 mm. It is important to note that in all experiments described in this paper the bond formation and breaking of bonded ice occurred at the same temperature.

### **RESULTS AND OBSERVATIONS**

### Flexural strength of parent material

The flexural strength of pristine freshwater ice samples of the same kind as in the present work was measured previously (Murdza et al., 2020c) using the same apparatus as here. The authors reported that the average and standard deviation of the measured across-column flexural strength at -3 and -10 °C are  $1.42\pm0.16$  and  $1.67\pm0.22$  MPa, respectively. The average and standard deviation of the flexural strength of freshwater ice at temperatures below -4.5 °C reported by Timco and O'Brien (1994) are  $1.73\pm0.25$  MPa, which compares favorably with the values obtained by Murdza et al. (2020b).

#### Flexural strength of bonded ice

The experiments on freshwater bonds were conducted at -3 and -10 °C. The results are listed in Table 1. In all experiments, the failure occurred outside the bond and within the parent material, even after only 0.5 hrs of freezing, indicating that the newly formed bond is stronger than the pristine material. As the results in Table 1 show, the obtained values of flexural strength are not statistically significantly different from the flexural strength of pristine freshwater ice samples mentioned above.

Table 1. Results from testing freshwater bond experiments. The time here is the bond formation time, the strength is the flexural strength.

Temperature [°C]	Time [hrs]	Strength [MPa]
-10	24	1.43
-10	25	1.39
-10	24	1.28
-10	3	1.58
-3	1.5	1.02
-3	1.5	1.28
-3	0.5	1.4

Figures 2 and 3 show the results from the experiments performed to investigate the effect of the water salinity used to create the freeze bond on bond strength. Figure 2 shows that the strength of the saline bonds increases over time and levels off, or saturates, after about 6-12 h. These results show that the strength of the saline bonds is significantly lower than the strength of the freshwater S2 ice used as the parent material.

Figure 3 shows the effect of salinity of the water used to generate the freeze-bond at -3 °C on its saturated strength at -3 °C. The bond strength decreases rapidly with an increase in salinity and no freezing occurs once the salinity of the salt water used to generate the bond reaches ~25 ppt. Figure 3 additionally shows an exponential curve taken from Timco and O'Brien (1994) for the flexural strength of saline ice. It is important to notice that the fit by Timco and O'Brien (1994) yields lower values than the measured bond strength values in the present study for the whole range of salinities used. Likewise, the actual strength values for the freshwater bonds are greater than the ones suggested by Figure 3, since the failure in these cases occurred outside the bond, indicating that the bond is stronger than the parent material. Both saline and freshwater bonds that develop through freezing appear to reach strengths higher than that of S2

type parent material of the same salinity (strength of saline parent material is assumed to be the same as in Timco and O'Brien (1994)).

Figure 4 illustrates the effect of temperature on saturated bond strength. The experiments were conducted on specimens having bonds made from water of salinity 20 ppt at temperatures ranging from -3 °C to -25 °C. The strength vs temperature trend appears, to a first approximation, to be roughly linear. Three out of the four specimens at -25 °C failed outside of the bond with a measured strength of  $1.61\pm0.12$  MPa, which is close to 1.89 MPa measured earlier at -25 °C on the same type of freshwater ice (Murdza and others, 2020). Figure 4 also suggests that no freezing occurs at temperatures above about -3 °C.

Figure 5a-c shows an example of an ice samples after failure. Figure 5a shows a case where the crack had initiated at the bond and started to propagate along it, but then deviated from it and continued to grow through the parent material. Figure 5b shows a close up of a bond face after the most common type of failure, which occurred along the bond. In this case, both surfaces of the failed freeze-bond had a fairly uniform "blurry" appearance, which indicates that the failure occurred through the ice of the bond. It was also fairly usual for the samples having low salinities, low temperatures and long freezing times, that the crack initiated and started to propagate along the bond and then slightly deviated and moved parallel to the bond but inside the parent material, as shown by Figure 5c.



Figure 2. Flexural strength as a function of freezing time for bonded ice prepared from salt water of 35 ppt salinity at -10°C (in red) and from salt water of 12 ppt salinity at -3°C (in black).



Figure 3. Flexural strength at -3 °C of bonded ice as a function of the salinity of the salt water from which the bond was formed. The solid pink line indicates the flexural strength  $1.42\pm0.16$  MPa of parent freshwater ice at -3 °C (Murdza et al., 2020c). A red dashed line is taken from Timco and O'Brien (1994) for the ice at -3 °C. A blue dashed line is a fit to the present data.



Figure 4. Flexural strength of bonded ice as a function of temperature for bonds formed from water of salinity of 20 ppt. Triangular-shaped points at -25 °C indicate that actual bond strength is greater than that of the parent material as the failure occurred outside the bond.



Figure 5. Photographs of an ice sample right after forced failure (a); the saline bond surface of 10 ppt after a crack propagated fully through the bond (b) and partially through the parent material (c).

#### DISCUSSION

The above results are the first measurements to be reported for the strength of freeze bonds under tensile (flexural) loading. Although the experiments were performed under flexural loading, they provide unique data on the tensile strength of the freeze bonds. As was shown earlier (Murdza et al., 2020c), the flexural strength of ice under the loading conditions described in the present work is governed by tensile strength. The only difference is that the flexural strength of brittle materials is greater by a factor of about 1.7 than tensile strength (Ashby and Jones, 2012). This factor of 1.7 was obtained based on statistical analysis of stress distribution within the material and volume of the material that is subjected to maximum stress. The ratio between the flexural and the tensile strength was also confirmed to be 1.7 for freshwater columnar-grained ice (Carter, 1971; Murdza et al., 2020c, 2020b, 2021). By using this factor of 1.7 to scale the values for saturated bond strengths shown in Figure 2 leads to tensile strength values of about 0.3 MPa and 0.18 MPa for bonds at -10 °C and -3 °C, respectively.

As mentioned above, in samples with freshwater bonds failure initiated and propagated outside of the bond. This result suggests that the strength of the freshwater bond is greater than the strength of pristine freshwater S2 ice. This result may be due to the difference in ice microstructure in the bond and parent S2 ice, specifically, ice in freeze bond may have a significantly smaller grain size. This argument is consistent with the results obtained by Schulson and others (1984) who showed that tensile strength depends on the grain size, decreasing as grain size increases. A difference in grain size can also explain why the strength versus salinity curve from Timco and O'Brien (1994) is below the curve obtained in the present study (Figure 3).

Both temperature and salinity affect the flexural strength of bonded ice samples. The data suggest an exponential function similar to the one suggested in Timco and O'Brien (1994) to represent the salinity effect on the bond strength (Figure 3). The trend of strength versus temperature for saline bonds (Figure 4) appears, to a first approximation, to be roughly linear.

# SUMMARY

New systematic experiments on the flexural strength of freeze bonds were. The bonds were grown in the air under 4 kPa confining pressure. The parent material was S2 columnar-grained freshwater ice. The salinity of the water used to generate the bond varied from 0 to 35 ppt and freezing temperatures from -3 to -25 °C. It is concluded that:

- (i) Freshwater bond strength exceeds the strength of parent ice in less than 0.5 h upon freezing.
- (ii) The saline bonds reach their saturated strength within about 6-12 h of freezing.
- (iii) An increase in bond salinity and in freezing temperature leads to a decrease in bond strength.
- (iv) The relationship between bond strength and its salinity is exponential and similar to the one suggested by Timco and O'Brien (1994).
- (v) No freezing occurs once the salinity of the water used to generate the bond reaches values of about ~25 ppt at -3 °C.

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