1 Behavior of Saline Ice under Cyclic Flexural Loading

2 Andrii Murdza¹, Erland M. Schulson¹, Carl E. Renshaw^{1,2}

¹Thayer School of Engineering, Dartmouth College, Hanover, NH, USA, 03755

4 ²Department of Earth Sciences, Dartmouth College, Hanover, NH, USA, 03755

5 Correspondence to: Andrii Murdza (Andrii.Murdza@dartmouth.edu)

6 Abstract. New systematic experiments reveal that the flexural strength of saline S2 columnar-grained ice loaded 7 normal to the columns can be increased upon cyclic loading by about a factor of 1.5. The experiments were 8 conducted using reversed cyclic loading over ranges of frequencies from 0.1 to 0.6 Hz and at a temperature of -10 °C 9 on saline ice of two salinities: 3.0±0.9 and 5.9±0.6 ‰. Acoustic emission hit rate during cycling increases with an 10 increase of stress amplitude of cycling. Flexural strength of saline ice of 3.0±0.9 ‰ salinity appears to increase 11 linearly with increasing stress amplitude, similar to the behavior of laboratory-grown freshwater ice (Murdza et al., 12 2020b) and to the behavior of lake ice (Murdza et al., 2020a). The flexural strength of saline ice of 5.9±0.6 ‰ 13 depends on the vertical location of the sample within the thickness of an ice puck; i.e., the strength of the upper 14 layers, which have a lower brine content, was found to be as high as three times that of lower layers. The fatigue life 15 of saline ice is erratic. Cyclic strengthening is attributed to the development of an internal back stress that opposes 16 the applied stress and originates possibly from dislocation pileups.

17 **1. Introduction**

Fatigue of materials is a subject of practical importance in engineering and has been widely studied (Bathias and Pineau, 2013; Broek, 1986; Schijve, 2009; Suresh, 1998). Fatigue refers to changes in material properties resulting from cyclic loading. Fatigue strength of crystalline materials is typically controlled by microcrack initiation and subsequent growth that leads to failure.

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23 It is not surprising that fatigue appears to play an important role in sea ice mechanics. For example, the 24 Arctic and Antarctic floating ice covers and ice shelves are subjected to cyclic loading from ocean swells that can 25 penetrate deeply into an ice pack and potentially result in the breakup of the ice cover (Squire, 2007). Such events, 26 where under the action of surface waves a floating ice cover exhibited sudden breakup into smaller pieces, have 27 been repeatedly witnessed and described (Shackleton, 1982; Liu and others, 1988; Prinsenberg and Peterson, 2011; 28 Asplin and others, 2012; Collins and others, 2015; Kohout and others, 2016; Hwang and others, 2017). Ice cover 29 breakup leads to a decline in albedo (Pistone and others, 2014; Zhang and others, 2019) and to the acceleration of 30 melting. Also, smaller ice floes attenuate ocean waves less effectively than does the parent solid ice cover, thereby 31 endangering coastal zones to erosion. Given the retreat of the sea ice cover and the attendant increase in oceanic 32 fetch, larger waves are expected to develop; correspondingly, the remaining ice cover is expected to be subjected to 33 episodes of greater cyclic loading. The potential for fatigue failure is thus increasing.

Cyclic loading may also play an important role in other scenarios. For instance, during ice-structure interactions (Jordaan, 2001; Hendrikse and Metrikine, 2016; O'Rourke and others, 2016; Jordaan and others, 2008) the structure itself, such as a light-house, may be weakened **or damaged** to a degree that depends on the strength of the ice. Other examples are runways and roads that are built by freezing water on cold oceans, rivers and lakes and subsequently subject to cyclic loading. Therefore, it is important to understand the behavior of ice under cyclic loading.

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42 Currently, the effects of cyclic loading on the physical and mechanical properties of sea ice and on the 43 susceptibility of the material to fatigue are poorly constrained. Tabata and Nohguchi (1980) conducted experiments 44 on sea ice sampled from Lake Saroma, Hokkaido, Japan and from Barrow, Alaska. They loaded the ice cyclically 45 under uniaxial compression between two specified stress levels under a variety of combinations of strain rate (from 46 10^{-5} s⁻¹ to 10^{-2} s⁻¹), temperature (from -2 °C to -24 °C) and orientation (horizontal and vertical). They found that 47 with a decrease of average stress and with a decrease of amplitude, the time to failure increases; and by lowering the 48 temperature, the time to failure and the number of cycles also increases.

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50 Other evidence of the weakening of sea ice under wave-driven in situ cyclic loading is discussed by 51 Haskell and others (1996), Bond and Langhorne (1997), Langhorne and others (1998), (1999), (2001). In these 52 works the authors obtained an S-N fatigue curve (S, upper peak stress of cycling; N, number of cycles imposed to 53 failure), typical of curves obtained from engineering materials, i.e. for lower stress amplitude more cycles are 54 needed for failure. The authors stated that the endurance limit, that is the stress amplitude below which the sea ice 55 can withstand an unlimited number of cycles, is approximately one-half the failure stress of non-cycled ice.

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The constitutive behavior of saline ice under cyclic loading was also investigated previously (Cole, 1995, 1998; Cole et al., 1998, 2002; Cole and Dempsey, 2004; Cole and Durell, 1995; Dempsey et al., 2003; Wei et al., 2020); specifically, inelastic deformation of sea ice was explored and interpreted in terms of a dislocation-based mechanism. In these works the authors investigated the effect of temperature (from -5 to -50 °C), microstructure (total porosity varied from 14 to 104 ppt), cyclic stress amplitude (from 0.04 to 0.8 MPa), loading frequency (from 10^{-3} to 1 Hz), and dry isothermal vs floating specimens on the response of the ice. However, the strength of ice after it had been cycled was not measured.

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Nothing more (to our knowledge) has been reported on the fatigue of sea ice. The topic is absent from a critical review by Squire (2007) and from two recent books on ice (Schulson and Duval, 2009; Weeks, 2010).

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The behavior summarised above indicating the weakening of ice under cyclic loading, obtained from experiments conducted on saline and sea ice, might possibly account for the sudden breakup of natural ice covers. However, this behavior appears in conflict with the behavior of freshwater ice under cyclic loading (Cole, 1990;

Gupta et al., 1998; Iliescu et al., 2017; Iliescu and Schulson, 2002; Murdza et al., 2019, 2020b, 2020a). In those 71 72 experiments, it was discovered that the ice flexural strength increases upon repetitive loading, followed by the 73 recovery of the cyclic-induced increment in strength to the original non-cycled strength upon post-cycling annealing. 74 This difference in the behavior of the two kinds of ice could perhaps be attributed to the presence of defects in 75 sea/saline ice, such as brine pockets, brine channels and non-penetrating microcracks. Such defects serve as stress 76 concentrators, thereby lessening the need to nucleate cracks to the degree that fatigue life may be governed primarily 77 by crack propagation. The strengthening of ice is of more than scientific interest, reflected, perhaps in an 78 interesting comment of an arctic engineer who reported that builders of ice roads never trust the ice until it 79 had been "worked in" (Masterson, 2018).

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Therefore, given that limited information about the behavior of sea/saline ice under cyclic loading and given the discrepancy in behavior of fresh and sea/saline ice, we conducted a study under controlled conditions in the laboratory on the flexural behavior of saline ice. In this paper, we describe the experiments in which beams of S2 columnar-grained saline ice of two salinities $(3.0\pm0.9 \text{ and } 5.9\pm0.6 \%)$ were subjected at -10 °C to four-point, reverse cycling at ~0.1-0.6 Hz and then, after several hundred or more cycles, were bent to failure, provided the beams did not break during cycling. We chose the rate of cycling to simulate the vibration frequency of a natural sea ice cover (Collins et al., 2015).

88 2. Experimental procedure

89 2.1 Ice growth and characterization

90 We studied saline ice of two melt-water salinities: 3.0 ± 0.9 and 5.9 ± 0.6 ppt, where \pm sign indicates standard 91 deviation. We produced the ice in the laboratory in an 800 L circular polycarbonate tank in the manner described 92 previously (Golding et al., 2014). Briefly, solutions containing 17.5 ± 0.2 ppt and 35 ± 0.2 ppt (parts per thousand, 93 or ‰) of the commercial product "Instant Ocean" salt mixture were prepared and then frozen unidirectionally 94 downward over a period of about 7 days by using a top-placed cold plate maintained at a temperature T = -95 20±0.1 °C. Before bringing the cold plate into contact with the salt-water solution, the top surface of the 96 solution was seeded with freshwater ice fragments of $\sim 0.3 - 1$ mm in diameter. This procedure produced pucks 97 ~ 1 m in diameter and ~ 0.3 m thick. For practical considerations, the bottom, skeletal layer of ice of about 7-10 cm 98 was discarded as it was slushy and weak; we also believe that the skeletal layer does not play a significant role in 99 supporting load. The top layer of ice of a few centimeters was also discarded because it was seeded and its 100 grain size was considerably smaller and its microstructure thus different from the rest of the ice puck. Melt-101 water salinity was measured using a calibrated YSI Pro30 conductivity and salinity probe.

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Figure 1 shows the microstructure of the ice. Table 1 lists its density and the **average** grain size **of the test specimens described below.** Figure 2 shows stereographic projections of the orientation of the crystallographic caxes. The ice is characterized by columnar-shaped grains whose growth texture is marked by c-axes confined within 106 about 15° of the horizontal plane of the parent ice puck and randomly oriented within that plane. In other words, the

107 ice is termed S2, after Michel and Ramseier (1971), and is similar to natural first-year sea ice (for comparison, see

108 Figure 3.7 of Schulson and Duval (2009)). The grain size noted above is the averge diameter of the columnar-shaped

109 grains, ranging from about 2 to 7 mm in Figure 1.

110 **2.2. Growth features**

111 The ice contained both sub-mm sized brine pockets and supra-mm sized drainage channels, reminiscent of natural sea ice. Figures 3 and 4 show examples. The ice of lower salinity $(3.0\pm0.9 \text{ ppt})$ had fewer defects of both 112 113 kinds. Some of the ice of higher salinity $(5.9\pm0.6 \text{ ppt})$ possessed channels whose size was almost as large as the 114 grain diameter. The defects scattered light to the degree that in bulk form the ice had an overall opaque appearance. 115 When observed in thin section (~1mm) the ice exhibited to the naked eye distinct linear whitish features which we 116 took to be sets of interconnected brine pockets that could possibly be filled with very fine-grained ice. The ice of 117 higher salinity possessed more of these features, especially near the bottom of the parent puck (which was the last 118 part to solidify). Our sense is that these features served as stress concentrators, particularly ones that traversed the 119 width of the test specimen (described below), thereby weakening the ice. Indeed, as will become apparent, samples 120 obtained from near the bottom of a puck of higher salinity $(5.9\pm0.6 \text{ ppt})$ had relatively low flexural strength.

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Because the ice of both salinities exhibited a different appearance from the top and bottom of the parent puck, in preparing test specimens for flexing we distinguished them by their position (depth from top surface) within the ice puck from which they were prepared,

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127 Table 2.

128 **2.3. Sample preparation and test setup**

129 Once the ice had been grown, it was cut into blocks of dimensions ~ $10 \times 30 \times 20 \text{ cm}^3$, where the longest 130 and the shortest dimensions are in the horizontal plane of the original ice puck, perpendicular to the direction 131 of growth. The blocks were stored in a cooler (at -10±0.5 °C) on their side (such that columnar-shaped grains were 132 oriented horizontally) to reduce brine drainage for periods of time of about 1-10 weeks.

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Specimens for flexing were manufactured from the ice blocks in the form of thin beams of dimensions $h \sim 16$ mm in thickness (parallel to the long axis of the grains), $b \sim 85$ mm in width, and $l \sim 300$ mm in length. The test specimens were allowed to equilibrate to the test temperature of -10 ± 0.5 °C for at least 24 hours before testing.

A detailed description of the specimens' preparation and loading can be found elsewhere (Iliescu et al., 139 2017; Murdza et al., 2018, 2019, 2020b). To summarize: The ice beams were flexed up and down under 4-point 140 loading under constant displacement rate using a servo-hydraulic loading system (MTS model 810.14) to which we attached a custom-built 4-point loading frame, Figure 5. A load cell, calibrated for both tension and compression,
and a linear variable differential transformer (LVDT) gauge were used for measurements of load and displacement
of the upper surface of the ice beam during cycling.

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Acoustic emissions were recorded during cycling using a PCI-2 18-bit A/D system; its frequency response was 3 kHz–3 MHz and its minimum acoustic emission (AE) amplitude detection threshold was set to 45 dB. We used a micro 30STC sensor (9.5 mm diameter, 11 mm thickness) which was attached to the top surface of an ice beam with a rubber band. Vacuum grease was used as the coupling agent between the sensor and the ice surface.

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The experiments were performed in a cold room at a temperature of -10° C and at an outer-fiber centerpoint displacement rate of 0.1 mm s⁻¹ (or outer-fiber strain rate of about 1.4×10^{-4} s⁻¹). This displacement rate resulted in an outer-fiber stress rate in the range from ~ 0.3 to 0.5 MPa s⁻¹, outer-fiber stress amplitude in the range from 0.35 to 1.2 MPa, outer-fiber strain amplitude in the range from ~ 1 to 5 x 10⁻⁴ and frequencies in the range from 0.1 to 0.6 Hz (i.e. periods from ~10 to 1.5 sec). The period, as already noted, is similar to the period of ocean swells (Collins et al., 2015). The major outer-fiber stress σ_f was calculated from the relationship:

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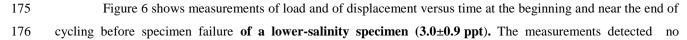
$$\sigma_f = \frac{3PL}{4bh^2} \tag{1}$$

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where *P* is the applied load and *L* is the distance between the outer-pair of loading cylinders (shown in Figure 5b) and is set by the geometry of the apparatus to be L = 254 mm.

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162 We used two different loading procedures, as we did earlier in our study of S2 freshwater ice. Type I 163 loading was a completely reversed stress cycle with constant stress amplitude and mean stress of zero. Type II was 164 similar to Type I but incorporated an increasing multi-level (or step-level) stress amplitude. This second type of 165 loading essentially consisted of several Type I steps of increasing stress amplitudes. In the present study for stress amplitudes below 0.7 MPa we used Type I loading. To cycle ice samples at stress amplitudes above 0.7 MPa, we 166 first pre-conditioned specimens through step-loading Type II procedure at progressively higher stress amplitude 167 168 levels, i.e. we cycled specimens for ~300 times at each of the following stress amplitudes: 0.7, 0.75, 0.8, 169 0.85 MPa and so on either until failure occurred or until a specific value of stress amplitude set by the 170 operator (see Iliescu et al. (2017) and Murdza et al. (2018) for details). To change stress amplitude the loading was 171 stopped for ~15 sec to change settings. After pre-conditioning, the specimens were cyclically loaded according 172 Type I loading at least 300 times and generally for ~2000 times, since no change in strength was observed beyond a 173 few hundred cycles (see below).



- 177 softening. According to Bažant et al. (1984) softening is a decline of stress at increasing strain or, in our case,
- 178 an increase of strain during cycling at constant stress amplitude during the tests). The absence of detectable
- softening during cycling of the saline ice is reminiscent of the absence of softening during the cycling of freshwater
- 180 ice (Iliescu et al., 2017; Murdza et al., 2020b).

181 **3. Results and Observations**

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3.1. Flexural strength of non-cycled ice

183 The flexural strength of non-cycled saline ice of both salinities was measured at -10 °C and at a nominal 184 outer-fiber center-point displacement of 0.1 mm s⁻¹. The results are listed in

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187 Table 2. Failure more often occurred at random locations between the two inner loading cylinders 188 and less often either below or slightly outside the loading cylinders. The reason for the latter location was the 189 presence prior to testing of a significant concentration of whitish features at loading cylinders which served as 190 stress concentrators and along which the failure ultimately occurred (similar to Figure 4). The average and 191 standard deviation of the measured flexural strength of saline ice of lower salinity (3.0±0.9 ppt) are 0.96±0.13 MPa. 192 The strength of the lower salinity ice did not correlate systematically with the depth of the parent puck from which 193 ice beams were prepared. The measured strength compares favorably with the value of 0.85 ± 0.20 MPa reported by 194 Timco and O'Brien (1994) for sea ice of similar salinity, as can be seen in Figure 7. Brine volume fraction ν_b was 195 calculated according to Frankenstein and Garner (1967):

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$$\nu_b = 0.001 * S\left(\frac{49.185}{|T|} + 0.532\right),\tag{2}$$

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where *T* is temperature in degrees Celsius between -0.5 °C and -22.9 °C and *S* is melt-water salinity (in ppt) of the ice.

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201 The average and standard deviation of the measured flexural strength of saline ice of higher salinity 202 (5.9±0.6 ppt) are 0.98±0.36 MPa. The measured values (Figure 7) deviate slightly towards higher values compared 203 to the data of Timco and O'Brien (1994), although scatter is significantly greater than is the scatter in the strength of the ice of lower salinity $(3.0\pm0.9 \text{ ppt})$. This may be explained by the greater degree of interconnectivity of brine 204 205 pockets at the bottom of an ice puck (discussed above and shown in Figures 3 and 4). Indeed, the flexural strength 206 of the higher-salinity specimens appears to depend on the depth of ice from which beams were prepared, 207 Table 2. This result shows how much the strength of ice is sensitive to flaws and defects. Given that larger bodies 208 usually contain larger defects, the flexural strength of sea ice on the medium and large scale, in the field (Karulina et 209 al., 2019) for instance, is expected to be lower than on the smaller scale of the present experiments.

211 We also compare our measurements of flexural strength with the tensile strength of sea ice. For this 212 purpose, and as we did in our previous work on freshwater ice (Iliescu et al., 2017; Murdza et al., 2020b), flexural 213 strength is divided by 1.7 (Ashby and Jones, 2012). This factor reflects the fact that the volume of the material 214 which is subjected to the highest stress in bending is smaller than in uniaxial tension; thus, the largest defect which 215 governs the failure may not be near the surface of a bent specimen. Upon dividing the flexural strength of the non-216 cycled saline ice of lower salinity by 1.7, we found the average across-column tensile strength from our experiments 217 to be 0.96 ± 0.13 MPa/ $1.7 = 0.56\pm0.08$ MPa. This value compares favorably with the values 0.56 ± 0.06 MPa and 218 0.63±0.12 MPa reported by Richter-Menge and Jones (1993) for the tensile strength of columnar-grained first-year sea ice of 4.1±0.3 ppt salinity loaded uniaxially across the columns at a temperature of -10 °C and strain rates of 10^{-5} 219 and 10^{-3} s⁻¹. Recall that in the present experiments the outer-fiber strain rate was about 1.4 x 10^{-4} s⁻¹ which is within 220 the range reported by Richter-Menge and Jones (1993). This agreement between direct and indirect measurements of 221 222 tensile strength lends confidence that our lab-grown saline ice is a reasonably faithful analogue of natural sea ice.

223 **3.2.** Flexural strength versus number of reversed cycles under constant low stress amplitude

224 To determine whether there is a relationship between flexural strength and number of cycles imposed under 225 a constant low stress amplitude, we performed via Type-I loading a series of experiments on saline ice of lower salinity (3.0±0.9 ppt) at -10 °C at an outer-fiber center-point displacement rate of 0.1 mm s⁻¹ at a low stress 226 227 amplitude of 0.35 MPa; i.e., at an amplitude less than one-half the flexural strength of non-cycled ice. Figure 8 228 shows the results. The number of cycles varied from about 100 to 14000. The average strength and standard 229 deviation of all data from Figure 8 are 0.96±0.23 MPa. As noted above the strength and standard deviation of non-230 cycled ice are 0.96±0.13 MPa. In other words, no strengthening was detected upon cycling up to 14000 times at a 231 stress amplitude of 0.35 MPa. For freshwater ice (Murdza et al., 2020b), we found that once the number of cycles at 232 a given low stress amplitude exceeded 300, the number of cycles had no significant effect on the flexural strength, 233 implying that a kind of saturation of strength developed. Given that result and the new results for saline ice, we 234 followed the practice in the present study of cycling more than 300 times, often as many as 2000 times, before 235 bending the ice to failure.

3.3. Flexural strength versus stress amplitude

The flexural strength increases with stress amplitude. Figure 9 shows measurements obtained from saline ice of both salinities cycled at -10 °C at an outer-fiber displacement rate of 0.1 mm s⁻¹. For comparison, data from laboratory grown freshwater ice (Murdza et al., 2020b) of S2 character and from lake ice of the same character (Murdza et al., 2020a) are also shown. The relationship between the flexural strength, σ_{fc} and cycled stress amplitude, σ_a , for saline ice appears to be a linear one and, within experimental scatter, to have essentially the same sensitivity to stress amplitude as freshwater ice; namely:

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$$\sigma_{fc} = \sigma_{f0} + k\sigma_a \quad , \tag{2}$$

where k = 0.68 is a constant. For freshwater ice the non-cycled flexural strength is $\sigma_{f0} = 1.75$ MPa compared with $\sigma_{f0} = 0.96$ MPa for the saline ice. There is, perhaps, in Figure 9 a hint that for saline ice there is a threshold of about 0.4 MPa that must be exceeded to detect strengthening. Interestingly, this apparent threshold is similar in magnitude to the stress that marks the onset of significant AE activity under cyclic loading of sea ice cores (Cole and Dempsey, 2006). Although saline ice is weaker than freshwater ice, it appears that upon cycling its strength increases at the same rate as freshwater ice.

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252 Although the rate of strengthening with stress amplitude appears to be the same for sline ice and freshwater 253 ice, the maximum increase in strength in the case of saline ice of lower salinity (3.0±0.9 ppt) is significantly lower. 254 We were able to strengthen saline ice by about 50% of the non-cycled strength compared with about 100% for 255 freshwater ice (Murdza et al., 2020b). Another point is that we almost were not able to cycle specimens at stress 256 amplitudes greater than the flexural strength of non-cycled material, whereas in the case of freshwater ice we were 257 able to cycle at stress amplitudes significantly greater than flexural strength of non-cycled ice. Indeed, the maximum 258 cycled stress amplitude we were able to reach in the case of saline ice of lower salinity $(3.0\pm0.9 \text{ ppt})$ during all tests 259 was 1.1 MPa, which is not statistically different from the non-cycled flexural strength of 0.96±0.13 MPa.

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For saline ice of lower salinity $(3.0\pm0.9 \text{ ppt})$, there is no evidence that the flexural strength of both noncycled and cycled ice is significantly affected by the depth of ice from which ice beams were harvested. For saline ice of higher salinity $(5.9\pm0.6 \text{ ppt})$, however, the flexural strength of both non-cycled and cycled ice appears to depend on the depth of ice from which beams were prepared, Figure 10. Indeed, the flexural strength of specimens from the bottom and from the top of an ice puck of higher salinity $(5.9\pm0.6 \text{ ppt})$ differs by ~3 times (~0.4 MPa vs ~1.4 MPa).

267 **3.4. Fatigue behavior**

268 Although the specimens from which the data in Figure 9 were obtained did not fail during cycling, other specimens cycled under similar consitions did fail while being cycled. Results from such tests (on of saline ice of 269 lower salinity (3.0±0.9 ppt) at -10°C and 0.1 mm s⁻¹ outer-fiber displacement rate) allowed us to construct S-N 270 271 fatigue curve, shown in Figure 11. The number of cycles here is the number of cycles to failure during cycling at 272 the last stress amplitude level and not the total number of cycles. At most the S-N curve showd only a weak 273 systematic dependence of the number of cycles to failure on stress amplitude. Indeed, for the same stress amplitude 274 of ~ 0.9 MPa, fatigue failure occurred after as few as <10 cycles and after as many as a few thousand cycles. 275 Statistical analyses to test the hypothesis that the slope in Figure 11 is zero resulted in a p-value equal ~ 0.06 . 276 Therefore, there is only a marginally significant effect of number of cycles on the stress at which failure occurred. 277 We attribute this variability in fatigue life to the variability in microstructure from specimen to specimen.

279 That said, a note of caution is appropriate. The data in Figure 11 should not be viewed as fatigue data in the 280 usual sense; i.e., in the way such data are viewed when obtained from other materials (e.g., metals and alloys) that 281 exhibit classical fatigue behavior. In those cases, before cycling, all specimens are assumed to have the same 282 thermal-mechanical history. That was not the case here for the saline ice, as most of the samples were pre-283 conditioned according to Type II procedure before they were cycled at the last stress level where they failed while 284 cycling. In other words, in order to get fatigue failure, we were increasing stress amplitude by small increments of 285 ~0.05 MPa and allowed a sufficient number of cycles at each stress level (~500-1000) before we reached a fatigue 286 failure.

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The question to address here is why we did not obtain a classical S-N curve? We suggest that the classical mechanism of fatigue, i.e. accumulation of damage, is not in play in our tests and some other process is controlling fatigue life.

291 **3.5 Microstructural observations of samples after fatigue failure**

292 In an attempt to reveal deformation damage in the form of microscracks, we examined using thin-section 293 optical microscopy (up to 50x magnification) the microstructure of specimens of the lower salinity ice $(3.0\pm0.9 \text{ ppt})$ 294 after they had failed during cycling; i.e., failed in fatigue. Three thin sections were prepared from four specimens in 295 order to ensure a greater probability of observing microcracks growing from brine pockets or brine channels, should 296 they be present. The plane of the thin section was parallel to the long axis of the columnar grains and parallel to the 297 direction of the greater normal stress. This plane was taken as the best plane to observe possible cracks. Thin 298 sections were observed using non-polarized light. We found no evidence of microcracks starting from brine pockets 299 or from other defects. In fact, we found no microcracks at all. It appears, therefore, that slow crack growth is not 300 a significant contribution to the fatigue life of the beams of the laboratory-grown saline ice that we studied.

301 **3.6. Acoustic emissions**

Acoustic emissions (AE) during repetitive loading of ice have been previously recorded and analyzed in laboratory and in situ (Langhorne and Haskell, 1996), (Cole and Dempsey, 2006, 2004; Lishman et al., 2020; Murdza et al., 2020b). Langhorne and Haskell (1996) suggested that the emissions originate either from dislocation breakaway or from microcracking associated with dislocation motion.

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In contrast to freshwater ice, where no sound was detected until failure (Murdza et al., 2020b), continuous emission was detected while cycling at constant stress amplitude. Figure 12 shows the cummulative acoustic emissions, or "hits", as a function of time for ice that was cycled reversely at a constant stress amplitude of 0.5 MPa. As can be seen, the hit rate (or hits per unit time), which is the slope of the curve in Figure 12, is about the same for the duration of the experiment.

Interestingly, the hit rate depends on stress amplitude during cycling. Figure 13 shows this behavior . The greater is the stress amplitude, the greater is the hit rate. However, during cycling below about 0.2 MPa no hits were detected.

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Figure 13 also indicates that the hit rate is independent of the sequence of different stress amplitudes. The numbers in Figure 13 show the order of cycling at different stress amplitudes; i.e., firstly we cycled ice at higher stress amplitudes (0.5-0.8 MPa), then at lower stress amplitudes (0.2-0.4 MPa). The results showed an increase in the hit rate as stress amplitude increases, regardless of the sequence of cycling.

321 **4. Discussion**

322 The results obtained from the experiments described in this paper show that the flexural strength of saline 323 ice can be increased upon reversed cyclic loading. Therefore, the same set of questions as for the freshwater ice 324 should be addressed here, i.e.: What governs the flexural strength of saline ice? Does crack propagation or crack 325 nucleation control the tensile strength? First of all, to understand the behavior of saline ice, it is important to 326 recognize that flexural strength in the present experiments is governed by the tensile strength, although greater by a 327 factor of about 1.7 (Ashby and Jones, 2012). Secondly, the apparent absence of remnant microcracks within the two 328 parts of broken samples (Section 3.5) indicates that crack nucleation controls the flexural strength, just as it appears 329 to do for freshwater ice. Indeed, this seems reasonable given the fact that freshwater ice comprises of ~95% by 330 volume of the saline ice we studied. Within the freshwater component, there is almost no solubility of salts (Weeks 331 and Ackley, 1986). The remainder of the saline ice is a mixture of air and brine. As was shown earlier, the 332 microstructure of saline ice that we grew is closely similar to the microstructure of sea ice. Pores lower the saline ice 333 strength (Sammis and Ashby, 1986). However, the behavior of S2 saline ice under cyclic loading is essentially the 334 same as the behavior of S2 freshwater ice (Murdza et al., 2020b), i.e. its strength increases at the same rate as 335 freshwater ice upon cycling under a given amplitude of the outer fiber stress. Hence, it is reasonable to assume that 336 the strengthening mechanism for the saline ice is similar to that for the freshwater ice. In our earlier work (Murdza 337 et al., 2020b) we proposed that strengthening might be due to the development of an internal back stress that 338 originates from either dislocation pileups or grain boundary sliding. However, one reviewer suggested the 339 possibility of a different strengthening mechanism. Due to the inherent weakness of the saline ice 340 microstructure, the microstructural stress relief may occur through localized damage via microcracking 341 mentioned above. More research, however, is needed to examine this hypothesis.

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The maximum degree of strengthening in the case of saline ice is significantly lower than that for the freshwater ice, although the slopes of the two data sets (rate of strength increase with increasing cyclic amplitude) in Figure 9 are nearly equivalent. That difference may be explained by the structure of saline ice which limits maximum possible strengthening. **Given the significantly greater number of stress concentrators in saline ice**, **such as brine pockets and channels, the propensity for failure during cycling is greater in saline ice** (Sammis and Ashby, 1986), thereby limiting the development of the back stress. Indeed, in the present study failure of

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Flexural experiments conducted on saline ice of higher salinity (5.9±0.6 ppt) showed the importance of brine features. Samples that were manufactured from the bottom of the ice puck were characterized by more frequent whitish interconnected features (taken to be interconnected brine pockets) that often were the path for easy crack propagation. Often samples were so weak that they failed before testing simply by handling. Interestingly, there were no interconnected features in samples prepared from the top of an ice puck, which resulted in a difference of more than a factor of three in strength between samples from top and bottom. Samples produced from saline ice of lower salinity (3.0±0.9 ppt) also had whitish features; however, these features were spread more uniformly (on a

specimens during cycling occurred more frequently than in the study on freshwater ice (Murdza et al., 2020b).

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360 It is worth noting again that a significantly greater fraction of saline ice samples failed in fatigue while pre-361 conditioning compared with freshwater ice. This may be explained by the fact that freshwater ice was essentially 362 free from pores, brine pockets and other defects. Based on this observation, it appears that crack growth is not a 363 significant contribution to the fatigue life of saline ice under the conditions of our experiments.

macroscopic scale) across the sample, resulting in little difference in strength between the bottom and top samples.

364

On the origin of the acoustic emissions, there are at least four possible sources of the noise detected. 365 366 One is from microcracking. We imagine that microcracks form in regions of mechanical weakness which 367 results in accumulation of damage that we detected via the AE method. Specifically, the brine drainage 368 whitish features discussed above in the test specimens constitute regions of high porosity and thus provide 369 favorable sites for the concentration of such damage. Failure may occur when one of these sites can no longer 370 support the applied stress and a microcrack emerges from the damage zone and propagates. It is possible 371 that newly formed microcracks are stable until a critical length is reached (Cannon et al., 1990; Schulson et 372 al., 1991), at which point the crack growth ensues. The reason that microcracks were not observed under the 373 optical microscope may be because they filled up with liquid brine upon formation which results in a loss of 374 contrast. A second possible explanation for the acoustic emissions is the motion and friction of very fine 375 particles of ice which may have been entrapped inside brine drainage features, as mentioned above. A third 376 possibility is microcracking along grain boundaries due to grain boundary sliding (Elvin and Shyam Sunder, 377 1996; Goldsby and Kohlstedt, 1997; Mulmule and Dempsey, 1997; Schulson et al., 1997; Weiss and Schulson, 378 2000). A fourth possible explanation consistent with the non-history dependence of the hit rate (Figure 13) is a 379 kind of water-hammer effect in which brine entrapped within pockets impacts the wall, first in one direction 380 and then another. None of these possibilities can be evaluated based upon the limits of the present 381 observations. We refrain, therefore, from further speculation on this point.

382

Returning to the observations noted in the Introduction, and to the results obtained from imposed, in situ cyclic loading experiments on sea ice beams by (Bond and Langhorne, 1997; Haskell et al., 1996; Langhorne et al., 385 1998, 1999), the question is: Why does ice fail in the field under wave action and under imposed cyclic loading, but

- 386 strengthen upon cycling in our experiments in the laboratory? Although we do not know the process through which
- the ice sheet failed in the field, we expect that there are many micro and macro cracks in natural sea ice. Indeed,

thermally-induced tensile stresses can induce thermal cracking in floating ice sheets (Evans and Untersteiner, 1971).

389 Therefore, our sense is that the difference in ice behavior under cyclic loading in situ in the field (Bond and

Langhorne, 1997; Langhorne et al., 1998) and in the laboratory in the present study is due to other types of defects

391 other than brine channels and pockets that are generated in the field as a result of thermo-mechanical history of ice.

392 **5.** Conclusions

From new, systematic experiments on the flexural strength of sub-meter sized beams of S2 columnargrained saline ice stressed principally across the columns through reversed cyclic loading at a temperature of -10 °C and frequencies in the range from 0.1 to 0.6 Hz, it is concluded that:

- (i) The flexural strength of saline ice can be increased upon reversed cyclic loading by as much as 1.5 times.
- 397 (ii) The flexural strength of ice subsequent to cycling scales linearly with the amplitude of the outer-fiber398 stress.
- 399 (iii) The fatigue life of saline ice is erratic and does not obey classical S-N behavior.
- 400 (iv) Crack growth is not a significant contribution to the fatigue life of saline ice.
- (v) There is high variability in structure and strength through the thickness of a saline ice puck of higher
 salinity (5.9±0.6 ppt).

403 (vi) Given the lack of definitive proof of the underlying failure mechanism in saline ice, the increase in 404 flexural strength of freshwater ice and saline ice attributable to pre-failure load cycling is roughly 405 equivalent.

- 406 (vii) Acoustic emission hit rate during cycling at a constant stress amplitude is about constant.
- 407 (viii) Acoustic emission hit rate during cycling increases with an increase of stress amplitude of cycling.

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- 413
- 414 **Author contributions:** AM, ES and CR designed the experiments and AM carried them out. AM prepared the 415 manuscript with contributions from all co-authors.
- 416
- 417 **Competing interests:** The authors declare that they have no conflict of interest.

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569 Table 1. Physical properties of as-grown saline ice.

1.08

0.97

1.09

Average

0.96±0.13

	Material	Density	[kg m ⁻³] Average	salinity [ppt]	Grain size [mm]
	Saline ice (lower salinity)	878	±11 3.	0±0.9	3.8±0.9
	Saline ice (higher salinity)	897	±10 5.	9±0.6	3.6±1.1
0					
1					
2					
3					
4 7	Table 2. Flexural strength of non-cycled saline ice at -10°C and a displacement rate of 0.1 mm/s.				
4]	Table 2. Flexural strength of no	n-cycled saline ice	at -10°C and a displacement	nt rate of 0.1 mm/s.	
4	Flex strength of ice of lower salinity	n-cycled saline ice Depth [cm]	at -10°C and a displacement Flex strength of ice of l salinity (5.9±0.6 ppt) [.	nigher Depth [6	cm]
4	Flex strength of ice of		Flex strength of ice of l	nigher Depth [6	
4	Flex strength of ice of lower salinity (3.0±0.9 ppt) [MPa]		Flex strength of ice of I salinity (5.9±0.6 ppt) [.	nigher MPa] Depth [o	2.5
4]	Flex strength of ice of lower salinity (3.0±0.9 ppt) [MPa] 1.08		Flex strength of ice of I salinity (5.9±0.6 ppt) [0.45	nigher MPa] Depth [o 20 - 22	2.5 20
4	Flex strength of ice of lower salinity (3.0±0.9 ppt) [MPa] 1.08 0.86		Flex strength of ice of I salinity (5.9±0.6 ppt) [0.45 0.53	nigher MPa] Depth [o 20 - 22 17.5 - 2	2.5 20 15
+ 」	Flex strength of ice of lower salinity (3.0±0.9 ppt) [MPa] 1.08 0.86 1.06		Flex strength of ice of I salinity (5.9±0.6 ppt) [0.45 0.53 0.62	Digher MPa] Depth [or 20 - 22 17.5 - 2 12.5 - 2	2.5 20 15 0

1.26

1.44

1.17

Average

 0.98 ± 0.36

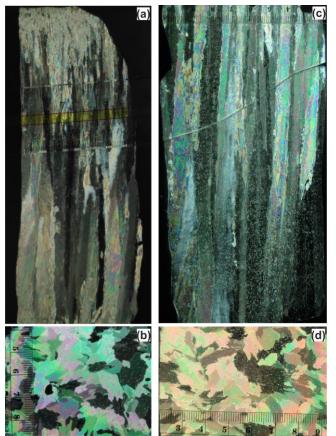
2.5 - 5

1 - 2.5

10-13.5

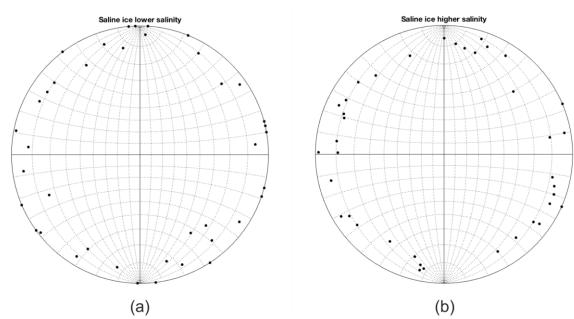
6.5 - 10

3-6.5



577578Figure 1. Photographs of a vertically-oriented (a) and a horizontally-oriented (b) thin-sections (~1mm) of columnar-
grained, saline ice of lower salinity (3.0±0.9 ppt) as viewed between crossed-polarized filters; photographs of a vertically-

580 oriented (c) and a horizontally-oriented (d) thin-sections of saline ice of higher salinity (5.9±0.6 ppt).



581(a)(b)582Figure 2. Stereographic projection plots of crystal c-axis {0001} orientations in saline ice of lower (3.0±0.9 ppt) salinity (a)583and saline ice of higher (5.9±0.6 ppt) salinity (b).

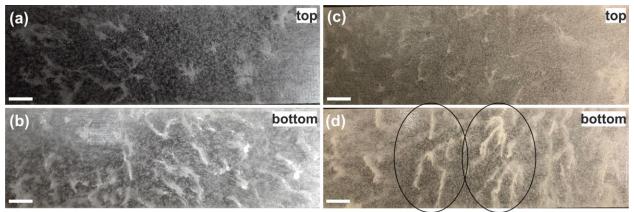
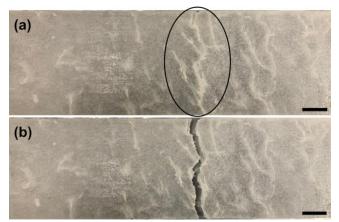


Figure 3. Photographs of saline ice samples of lower salinity (3.0±0.9 ppt) from the top (a) and bottom (b) of an ice block 586 and saline ice samples of higher salinity (5.9±0.6 ppt) from the top (c) and bottom (d) of an ice block. The concentration of 587 whitish features along the width of a sample in (d) is shown inside circles which is a predominant place for a crack to

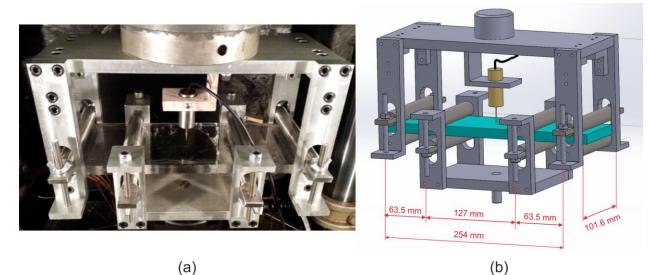
588 initiate. The columnar grains run in and out of the images. Scale bars: 20 mm.



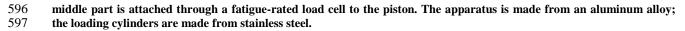
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Figure 4. Photograph of a sample from the bottom of an ice block of higher salinity (5.9±0.6 ppt) before cycling (a) and

591 after (b) failure. Note a crack that propagated along whitish features in the area in (a) depicted by the circle. Scale bars: 592 20 mm.



593 594 Figure 5. Photograph (a) and sketch (b) of the four-point bending apparatus connected to an MTS hydraulic testing 595 system (Iliescu et al., 2017; Murdza et al., 2020b). The upper part is attached to the frame of the machine while the mobile



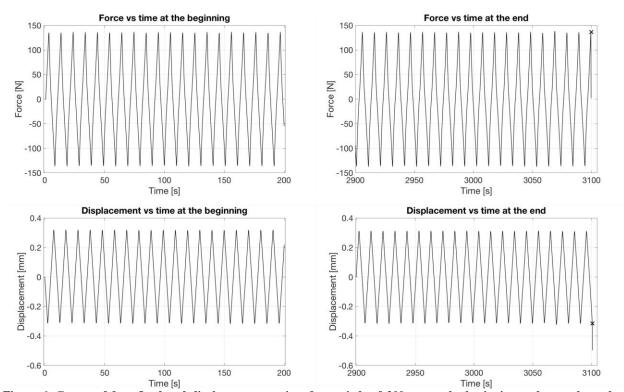
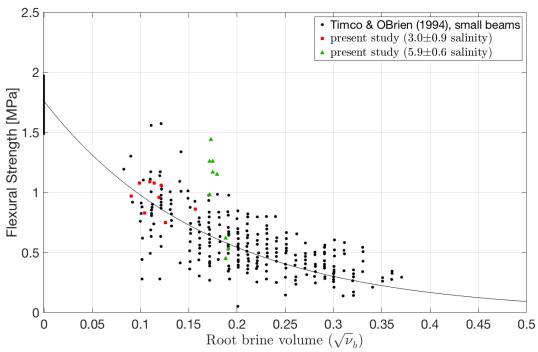


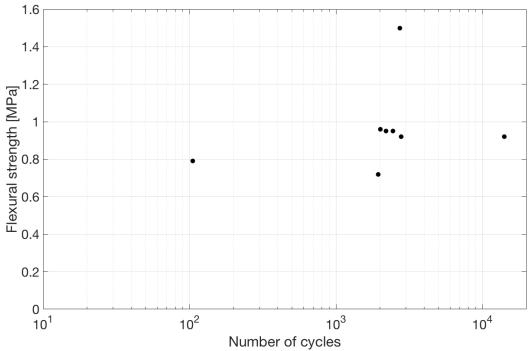
Figure 6. Curves of force/load and displacement vs. time for periods of 200 s near the beginning and near the end of cycling before fatigue failure occurred. Marker symbol "x" denotes a moment of specimen failure. Force of ~135 N 601 corresponds to ~1.2 MPa.

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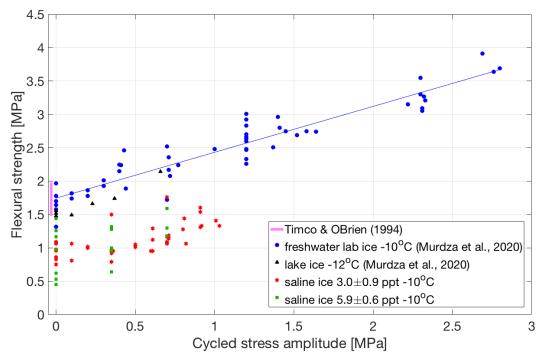




602 603 Figure 7. Flexural strength of saline ice as a function of root brine volume for the ice grown in the present study and for 604 data from Timco and O'Brien (1994) for comparison.

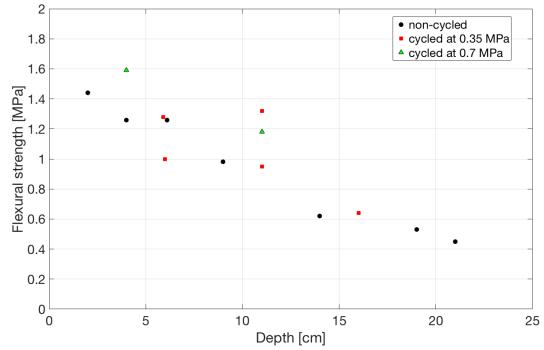


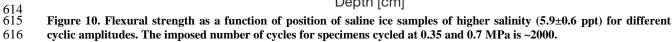
605 606 607 Figure 8. Flexural strength and the corresponding number of cycles imposed for saline ice of lower salinity (3.0±0.9) ppt cycled at 0.35 MPa outer-fiber stress amplitude at -10 °C and 0.1 mm s⁻¹ outer-fiber center-point displacement rate.



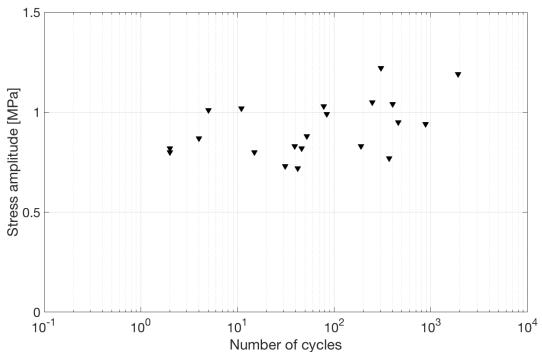


608 609 Figure 9. Flexural strength of freshwater ice and saline ice of lower (3.0±0.9 ppt) and of higher (5.9±0.6 ppt) salinity as a 610 function of reverse-cycled stress amplitude. Freshwater ice laboratory and lake data are taken from (Murdza et al., 2020b, 611 2020a). Red five-pointed stars and green squares represent tests performed on saline ice of lower and higher salinities, respectively, at 0.1 mm s⁻¹ and -10°C. During all depicted tests the ice did not fail during cycling and was broken by 612 613 applying one unidirectional displacement until failure occurred.





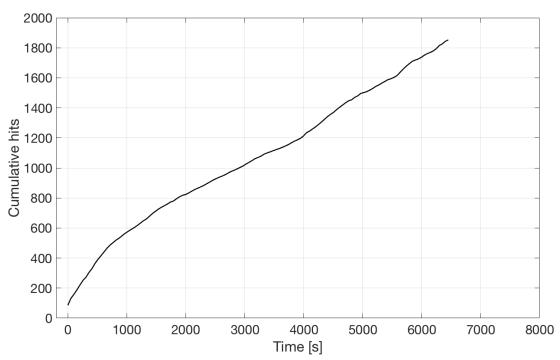
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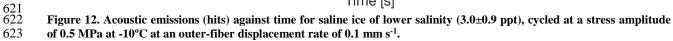


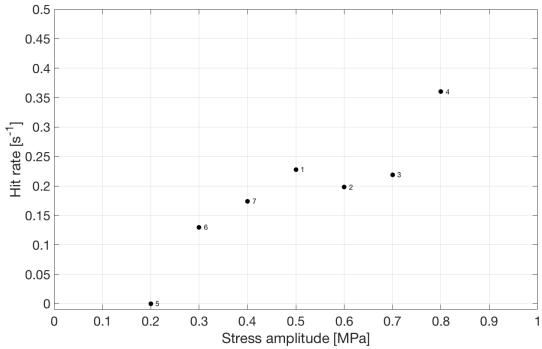


617 618 Figure 11. Stress amplitude as a function of the number of cycles to fatigue fracture for saline ice of lower salinity 619 (3.0±0.9 ppt) tested at -10°C and 0.1 mm s⁻¹ outer-fiber center-point displacement rate.









624Stress amplitude [MPa]625Figure 13. Hit rate as a function of cycled stress amplitude for saline ice sample of lower salinity (3.0±0.9 ppt). Numbers626show the order of cycling at different stress amplitudes.