

Ice Adhesion Shear Tests on Ice-Phobic Surfaces

Robert Gagnon¹, Austin Bugden¹, Matthew Garvin¹, Sigurd Teigen², Brad Elliott³

¹ OCRE/NRC, St. John's, Canada

² Equinor, Stavanger, Norway

³ Equinor, St. John's, Canada

ABSTRACT

Ice adhesion tests were conducted on five substrates with differing ice-phobic coatings. Rectangular-plate ice samples (25–62 cm²), freeze-bonded onto the surfaces, were pushed from one edge (at a nominal rate of 0.5 mm/s) until shear-detachment occurred. Freshwater and saline ice layers were investigated, where the intended test temperatures were -12 °C and -22 °C. For the freshwater ice cases it was found that spraying cold water onto the surfaces led to the formation of ice layers that never fully bonded/contacted the surfaces due to non-uniform freezing and lift-up/delamination. The mild bumpy texture of some of the surfaces and slight curvature of all of the substrates, where liquid could pool in ‘valleys’, contributed to this behavior. Hence, attempts to bond flat pre-shaped ice-plate specimens were unsuccessful. Furthermore, when spraying on smooth surfaces, ice-layer delamination occurred due to freezing of liquid and lift-up at the substrate edges. A thin layer (2–4 mm thickness) of saturated snow applied to the surfaces, however, did freeze and bond because it conformed to the non-flat features of the surfaces. Test results at -12 °C showed a wide variation of ice adhesive strength between the coatings (22–216 kPa). Degradation of the coatings with the number of tests was also noted (i.e. increasing adhesive strength), and was proportional to the adhesive strengths. At -22 °C the preparation method usually led to ice samples that were only partially bonded to the surfaces. In a few cases, video records enabled rough estimates of the contact areas so that approximate adhesive strengths were obtained. For saline ice generated by spraying at -12 °C and -22 °C, lift-off was not evident, however, no freeze-bonding occurred on any of the surfaces because a thin briny liquid layer was present at the interface.

KEY WORDS: Ice adhesion; Shear tests; Ice-phobic surfaces.

INTRODUCTION

Icing is a serious hazard for vessels and offshore structures operating in cold environments (Ryerson, 2011). Freshwater icing accumulations can occur while navigating on large lakes in cold climes, while saline icing occurs in cold-ocean regions. Icing resulting from the freezing of spray generated by wave collisions with the bow of a vessel, or components of a fixed or floating structure, can lead to instability in the case of large accumulations. Even in the case of relatively small accumulations safety and communications equipment is compromised and the level of production may be reduced as outside mobility of crew and equipment is

impaired. Computer models that can forecast the onset and severity of marine icing events are one avenue to help mitigate the problem by aiding avoidance and operational strategies that lessen the effects. Another approach is the use of physical monitoring systems (e.g. Gagnon et al., 2009) to aid crews in real-time assessment of hazard. The removal of icing accumulations from vessels is a difficult and often arduous task, especially if the wave and wind conditions that facilitated the icing still prevail. Since the earliest days of shipping in cold regions to the present time the main method for significant accumulations has been the use of bats and mallets to break the ice from decking and superstructures.

In the past few decades much attention has been garnered by the development of ice-phobic surfaces that have potential for the removal of icing accumulations from surfaces (Golovin et al., 2016). Most of the research so far has focused on aircraft icing and powerline/infrastructure icing. More recently, the shipping and offshore resource development industries, and some of the world's navies, have begun to investigate the potential of these coatings. In keeping with that, Equinor contracted OCRE/NRC to conduct ice adhesion tests on a series of substrates that had differing ice-phobic coatings to determine their efficacy.

OBJECTIVES/CONSIDERATIONS

A series of shear tests was conducted at -12 °C and -22 °C to investigate the effectiveness of five differing ice-phobic coatings, where each coating had been applied to its own specific substrate. The coated boards were labelled as 1, 2, 3, 4, and 5. Note that none of the boards were flat, that is, they all had some degree of curvature (~ 2 m radius of curvature) both lengthwise and laterally. Furthermore, some of the coatings on the boards had an irregular mildly (board 1), or moderately (board 5), bumpy (~ 0.3 – 2.0 cm diameter/length bumps with heights ≤ 0.1 mm) but locally-smooth firm texture, and one had a similarly rough but soft texture (board 4). As will be seen below, these aspects of the boards presented serious challenges in terms of establishing and maintaining good ice/coating bonds for the tests.

Objective/Consideration 1: Determine a suitable method to apply a properly-bonded ice layer on each board so that the shear tests could be conducted.

Objective/Consideration 2: Conduct the shear tests, while ensuring that ice samples were properly bonded to the boards at initiation.

Objective 3: Analyze the shear-test data to determine the adhesive strengths of the various coating/ice bonds so that efficacy levels could be assigned to the coatings.

APPARATUS DESCRIPTION

Figures 1-3 show renderings and photographs of the test equipment. The apparatus was initially designed to accommodate the coated boards supplied by Equinor, with the assumption that the boards were flat. Fortunately the apparatus had enough built-in tolerance to accommodate the non-flat aspects of the actual boards that were supplied, without significantly affecting the test results.

Figures 1-2 show the main components. The actuator was a compact electric screw-driven high-capacity device. This was conveniently controlled using a notebook computer, which enabled the team to perform the tests without having to use a larger hydraulic actuator/pump system. Figures 1 and 3 show a rectangular-shaped ice specimen bonded to one of the boards underneath. At one face of the ice specimen there is an arc-shaped piece of solid aluminum (Pusher) that is ~ 15 mm thick and about 12 cm wide. The flat front face of the Pusher

contacts the ice specimen during the tests and is wide enough to accommodate a variety of ice sample sizes. The Pusher itself is pulled against the ice specimen by an outer rectangular rigid aluminum frame (Yoke). The portion of the Yoke that contacts the Pusher is arc-shaped where it meets the Pusher, but with a greater radius than that of the Pusher's arc. This enables the Pusher to adjust to slight misalignments in the loading direction associated with the orientation and centeredness of the ice sample. The opposite end of the Yoke is attached to a small load cell (via a flexible metal strip) which in turn is connected to the actuator. To prevent potential damage to the board coating and to enable consistent and low-friction gliding of the Yoke and Pusher during the tests, there are strips of paper placed on the board underneath the Yoke at its side edges and under the full expanse of the Pusher. Hence, there is no direct contact between the board and the aluminum pieces during tests, and the paper strips stay in place due to friction between the paper and the coating.

The setup for generating spray-ice layers is shown in Figure 4.

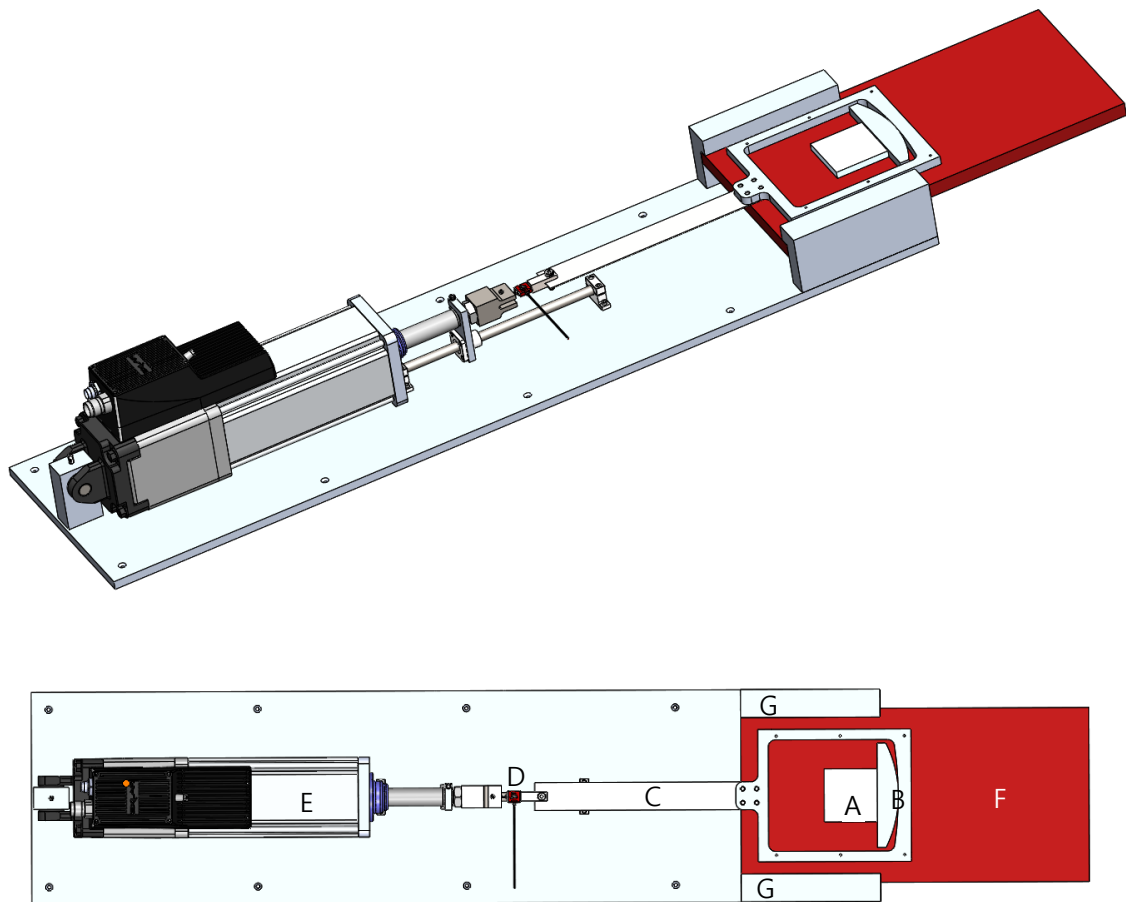


Figure 1. Two views of the test apparatus, bottom image: (A) ice sample; (B) aluminum Yoke and Pusher; (C) flexible metallic strip; (D) load cell; (E) actuator; (F) board with ice-phobic coating; (G) board holder/restrainer.

ICE SAMPLE PREPARATION

For the freshwater ice cases it was found that spraying cold water (near 0 °C) onto the coated-

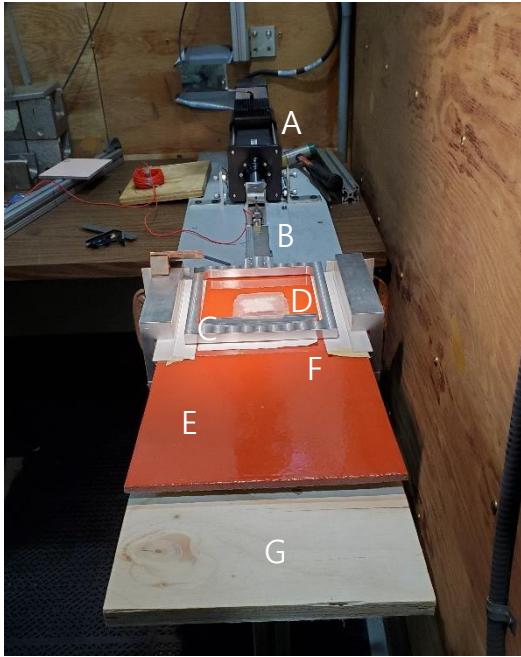


Figure 2. Test setup, length view: (A) actuator; (B) metal pull-strip and load cell; (C) aluminum Yoke and Pusher; (D) ice specimen; (E) board with ice-phobic coating; (F) paper strips; (G) plywood support/spacer board.

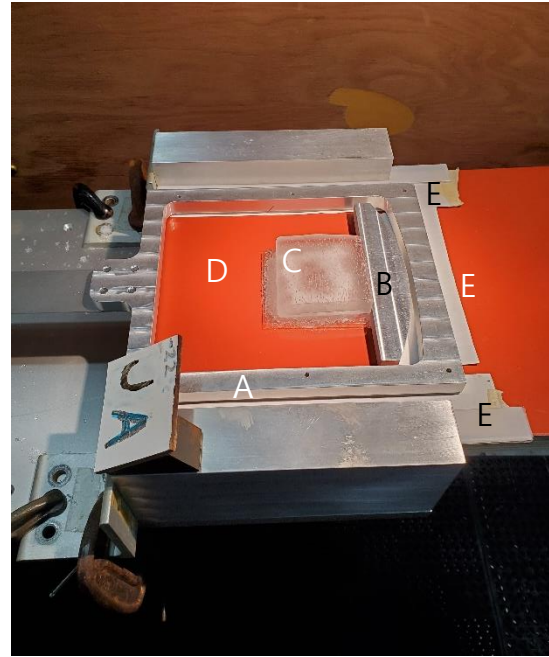


Figure 3. Test setup, side view: (A) Yoke; (B) Pusher; (C) ice specimen; (D) board with ice-phobic coating; (E) low-friction paper strips under Yoke edges and Pusher.

board surfaces led to the formation of ice layers that never fully bonded/contacted the surfaces due to non-uniform freezing and lift-up/delamination (Figure 5). The mild bumpy texture of some of the surfaces and slight curvature of all of the substrates, where liquid could pool in ‘valleys’, contributed to this behavior. Then attempts were made to bond flat pre-shaped ice-plate specimens to the boards, but this was not successful for the same reasons. Furthermore, when spraying water on the boards with smooth surfaces, ice-layer delamination occurred due to freezing of liquid and lift-up at the substrate edges. Having identified these causes of difficulty it was eventually found that a thin layer (2 – 4 mm thickness) of snow could be placed on the surfaces that conformed to their non-flat features. The snow layer was then saturated with water and gently tamped down to insure good conformity with



Figure 4. Spray-icing setup. (left) Boards 1 and 5 receive saline water spray from a manually operated spray nozzle in the Cold Room at an ambient temperature of $-22\text{ }^{\circ}\text{C}$. The nozzle is fed from a handheld pressurized container. The spray targets and nozzle are situated in the airstream of a fan (foreground) that generated a wind speed of $\sim 1.1\text{ m/s}$ at the boards. The nozzle was activated and moved across the boards at roughly 10-20 s intervals. (right) Alternate view of the boards, with icing-layer accumulations, spray nozzle and fan (background).

the underlying surface (Figure 6). A square wooden jig was used to confine the snow at the edges during the process in order to keep the snow in place and get the desired dimensions for the test sample. In the early stages of procedure development, the board, wet-snow layer and jig were then brought into the cold room at -12 °C to freeze up. However, this sometimes led to difficulties removing the wooden jig from the frozen snow-ice layer because the inner walls of the jig were frozen to the ice in some areas. This caused delamination of the ice from the ice-phobic coating when removing the jig. Consequently, the procedure was altered so that after the wet-snow layer was initially created, a thin plastic sheet of transparent ‘Shrink-Wrap’ was introduced between the jig and the wet snow to prevent it from adhering to the inner surface of the jig during freeze-up. Hence, after the snow layer was wetted the jig was momentarily lifted off and the sheet of ‘Shrink-Wrap’ was carefully placed over the layer. Then the jig was put back in place to encompass the periphery of the plastic-covered square patch of wet snow. The layer was tamped down again, and the board with its layer of wet snow inside the confining jig was brought into the testing room where the temperature was -12 °C. After about 10 minutes the wet snow layer had frozen solid and was completely bonded to the coated surface of the board. This was clearly evident because any region that was not bonded, i.e. that had delaminated, would show up as a lighter area due to light refraction and internal reflection effects. Once the layer was frozen, the jig was easily removed without disturbing the ice sample, and then the thin plastic layer was removed. The next stage involved carefully melting away (with a warm aluminum bar) any residual peripheral frozen liquid that had been expelled from the wet-snow layer during freeze-up.



Figure 5. A freshwater ice layer resulting from spraying water onto a board with a red ice-phobic surface coating. The icing layer is of fairly uniform thickness (a few millimeters), however a significant portion of the layer has a whitish color, due to internal reflection, because that portion has delaminated/lifted from the coating. The rest of the ice layer maintained direct physical and optical contact with the coating so that its red color is visible through it. In this case the lifting mechanism was probably due to water freezing and expanding at the lower edge (and possibly far side-edge) of the board.

The next step in preparation was to add an additional solid layer of ice to the top of the frozen snow-ice layer so that the Pusher in the shear-test apparatus would have a sufficiently high vertical wall-face of ice (≥ 13 mm) to push against during the test. Hence, a sufficiently thick piece of freshwater-sheet ice grown in a basin was shaped

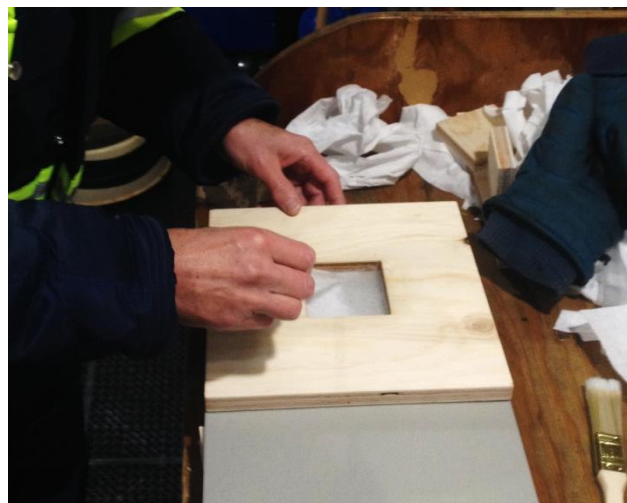


Figure 6. A square-shaped snow layer, confined at its edge by the wooden jig on board 5A, as water from a dropper is used to saturate the layer starting from the right of the image. The saturated snow appears darker than the dry snow. The ambient temperature is near 0 °C.

to the approximate areal size of the snow-ice layer. Some dry snow (3-4 mm thick) was arranged around the top perimeter of the snow-ice layer. The ice piece from the basin ice was then centered and laid on top of the snow and pressed lightly onto it. Then, using a small dropper with cold water in it, several small squirts of water were injected into the peripheral snow between the snow-ice below and the basin-ice above (Figure 7). The wet snow quickly froze onto both the ice layer below and the ice piece above, bonding the two together (Figure 8). This procedure took place in an adjacent room at around -5 °C. The board and ice sample were then brought into the test room at -12 °C to thermalize to test temperature. The final step of preparation was to quickly melt/flatten (within a few seconds) the front vertical face of the test specimen with a warm aluminum bar so that it conformed to the flat vertical face of the Pusher. Within approximately ½ hour the board and ice sample were then carefully placed in the test apparatus while being especially careful not to apply any flexural forces onto the non-flat board (along its length or width) that would lead to delamination of the snow-ice layer from the ice-phobic coating.



Figure 7. A dropper is used to wet the periphery of a layer of snow sandwiched between a snow-ice sample at the bottom and an ice plate on top.



Figure 8. A fully prepared ice sample consisting of the snow-ice sample at the bottom and the ice plate at the top. A peripherally-wetted and frozen layer of snow provides a solid bond between the top and bottom ice components. The residual snow particles on the board from the preparation process will be swept off with a brush before the test.

For saline ice generated by spraying salt water (with seawater salinity (Petrie et al., 1991)) onto the boards at -12 °C and -22 °C, lift-off, such as was observed for freshwater spray ice, was not evident. However, no freeze-bonding occurred on four of the surfaces (boards 1, 2, 3, 5) because a thin briny liquid layer was present at the interface, as observed in video and photographic records (e.g. Figures 9 and 10). Although two saline-ice shear tests were conducted for the relatively-rough surface of board 4, quite low apparent adhesive-strength bonding (8.85 kPa at -12 °C and ~ 14 kPa at -22 °C) was measured. To conduct those tests most of the spray-ice layer on the board was removed by separating portions of the layer that were not wanted, through melting with a warm aluminum bar, and then sliding or prying them off the board with relative ease, leaving only the desired portion that was the 'test sample'. These measured shear test values are suspect, however, and are not considered to be actual adhesive strengths of ice bonding to the surface. They are due, rather, to the force required to shear the unbonded interlocking ice and coating textures at the ice/coating interface in the presence of the brine layer, as described below.

Inspection of the bottoms of the saline icing specimens from these tests on board 4B showed obvious signs of a thin brine layer between the ice and the ice-phobic surface. From the above measurements and observations we conclude that there never was any bonding of the saline spray-ice with the ice-phobic coating on any of the boards. Any perceived ‘adhesion’ was due to imbalance of the brine layer fluid pressure at the coating/ice interface and atmospheric pressure on top of the ice samples when attempting to slide them on the boards in the presence of the non-flat aspects of the coatings and boards. That is, when sliding the ice relative to the board the ice tends to raise slightly from the board, due to the coating bumpiness and board curvatures, thereby reducing the hydrostatic pressure of the liquid layer so that the pressure imbalance with atmospheric pressure on top of the ice generated a normal load to keep the ice pressed towards the board. This led to a ‘jamming’ effect of the ice during sliding that could be misinterpreted as ‘bonding’ of the ice to the ice-phobic surface.

While beyond the scope of the present study, the above considerations do point out that an understanding of what may keep saline ice accumulations ‘attached’ to a surface is more complex than simply whether or not a solid ice/surface bond has been established. The presence of brine, and time dependencies related to its flow, with respect to geometrical aspects of the interfacing surfaces apparently play roles. Note that facilitating the flow of brine out of the spray-ice layers by orienting the boards vertically for an hour after spraying, and in one case several hours, did not lead to solid-solid bonding of the ice and surface coating.



Figure 9. Board 3 with an accumulation of saline spray-ice. The icing layer was applied when the board was lying horizontally. Shortly after that the board was oriented vertically with its side-edge resting on a sheet of plywood. Within a few minutes substantial brine had drained from the icing layer and accumulated in a pool on the plywood at the base of the board. White regions of the icing accumulation indicate areas where some brine had drained out of the ‘spongy’ ice matrix.



Figure 10. A photo of board 3 illustrating how a substantial portion of the spray-ice layer, that was melt-separated from the rest of the layer, had been easily slid off the board by hand when a relatively small shear force was applied at its edge. The portion was then placed back on the board in order to take the photo. Remnants of the thin brine layer that prevented solid-solid bonding of the ice to the board, and facilitated the slippage, can be seen on the board where the ice had previously been.

That is, the thin brine layer at the interface persisted. Furthermore, one of the boards (3) was oriented vertically during the whole spraying procedure, and it also exhibited the brine layer at the ice/coating interface on subsequent inspection.

DATA ANALYSIS AND PRESENTATION

All ice specimens were tested within approximately one hour of their final preparation. From start to finish, the growth and preparation of freshwater ice samples took about 1.5 hours. Saline ice samples required between 2.0 to 3.5 hours for growth (by spraying) and preparation, depending on how much time was allocated for brine drainage when the spray-ice samples were placed in a near-vertical orientation, i.e. 10 minutes, 1 hour or 2 hours.

Figure 11 shows the load record from a typical shear test. The nominal actuator speed and test temperature were 0.5 mm/s and -12 °C respectively. At the initiation of actuator movement, before there is contact of the Pusher with the ice sample, there is a slight load offset established that corresponds to the friction force of the Yoke and the Pusher with the sheets of smooth paper that they slide on during the test. Usually in tests there is also some ‘settling in’ when the Pusher makes contact with the ice face, that is, slight angular adjustments occur between the Yoke, Pusher face and the ice-sample face, both horizontally and vertically. The load rises approximately linearly after the settling-in period to a peak value after which there is an abrupt sharp drop in load signifying breakage of the adhesive bond. Following the load drop there is still a small load associated with the dynamic friction force of the ice sample sliding on the ice-phobic surface. In some tests this force exhibits low-amplitude stick-slip behavior.

Table 1 shows the results of all tests, that is, the results when shear tests were conducted and

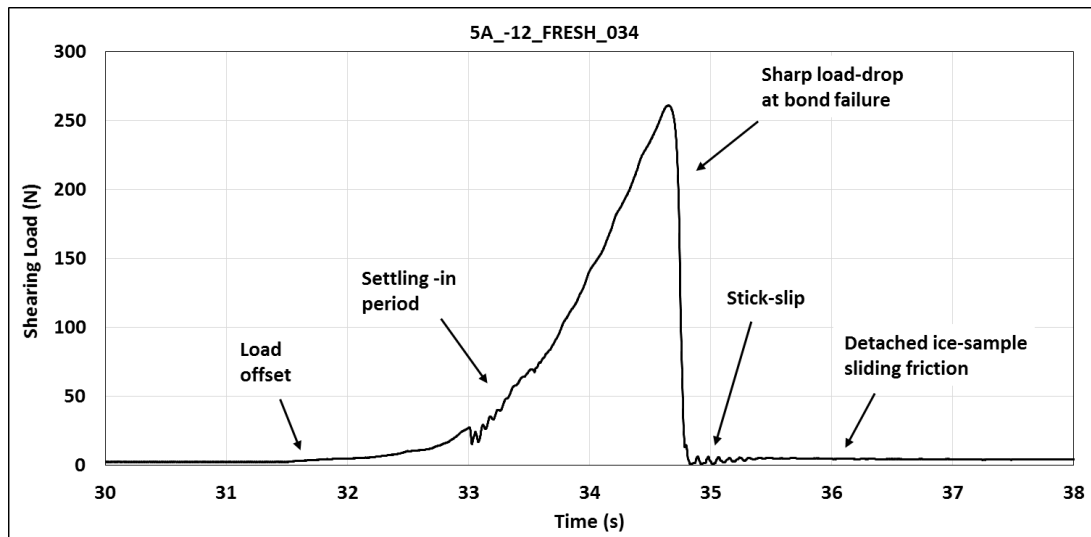


Figure 11. A typical load time-series record from a shear test. A small positive offset (< 5 N) is present at the beginning of the test that corresponds to the sliding friction of the Yoke and Pusher on the underlying paper strips prior to contact with the ice sample. This is followed by a settling-in period during the initial contact when any slight misalignments of the Yoke and Pusher reconcile. Following the detachment of the ice sample, at the sharp drop in load from the peak value, there is some slight friction due to the sliding of the detached sample on the ice-phobic coating, where in some cases stick-slip is also evident.

Table 1. Ice Layer Adhesive Strength Data from the Shear Tests.

Test (board; end; °C; ice type; test #)	Temp. °C	Ice Type (water type / dry base)	Dimensions (length/width) (mm)		Video File #	Date	Peak Load (N)	Shear Strength (kPa)
1A_-12_FRESH_017	-12	Fresh/Snow	74.0	73.8	012	30-Nov-20	121.1	21.6
2A_-12_FRESH_018	-12	Fresh/Snow	73.9	74.0	013	1-Dec-20	305.7	54.4
3A_-12_FRESH_021	-12	Fresh/Snow	42.0	74.0	015	1-Dec-20	584.0	181.1
4A_-12_FRESH_025	-12	Fresh/Snow	74.7	74.7	016	2-Dec-20	787.0	137.3
5A_-12_FRESH_027	-12	Fresh/Snow	76.3	73.7	018	2-Dec-20	138.4	24.0
1A_-12_FRESH_028	-12	Fresh/Snow	77.0	79.5	019	3-Dec-20	155.8	24.8
5A_-12_FRESH_029	-12	Fresh/Snow	76.1	76.8	020	3-Dec-20	202.0	33.7
3A_-12_FRESH_030	-12	Fresh/Snow	51.0	48.7	021	3-Dec-20	557.8	215.8
1A_-12_FRESH_031	-12	Fresh/Snow	77.1	77.0	022	4-Dec-20	162.8	27.4
2A_-12_FRESH_033	-12	Fresh/Snow	76.5	77.0	025	4-Dec-20	425.4	72.2
5A_-12_FRESH_034	-12	Fresh/Snow	77.5	76.6	026	4-Dec-20	258.7	43.6
1A_-22_FRESH_038	-22; sw*	Fresh/Snow	76.8	79.5	029	8-Dec-20	196.2	32.1
5A_-22_FRESH_042	-22; sw	Fresh/Snow	76.0	76.8	--	10-Dec-20	232.0	39.7
1A_-22_FRESH_044	-22; sw	Fresh/Snow	76.8	77.8	035	11-Dec-20	250.0	41.8
5A_-22_FRESH_045	-22; sw	Fresh/Snow	75.5	76.7	--	11-Dec-20	260.0	44.9
4B_-12_SALINE_048	-12	Saline Spray	77.2	81.4	041	15-Dec-20	55.6	8.8***
4B_22_SALINE_050	-22	Saline Spray	110.5	105.0	043	7-Jan-21	169.1	14.6***
2B_-12_SALINE_046	-12	Saline Spray	--	--	039	14-Dec-20	--	No bond
2B_-12_SALINE_047	-12	Saline Spray	--	--	040	14-Dec-20	--	No bond
2B_12_SALINE	-12	Saline Spray	--	--	--	15-Dec-20	--	No bond
1B_-12_SALINE	-12	Saline Spray	--	--	--	16-Dec-20	--	No bond
2B_12_SALINE	-12	Saline Spray	--	--	--	16-Dec-20	--	No bond
3B_-12_SALINE	-12	Saline Spray	--	--	--	18-Dec-20	--	No bond
5B_-12_SALINE	-12	Saline Spray	--	--	--	18-Dec-20	--	No bond
5B_-12_SALINE	-12	Saline Spray	--	--	--	21-Dec-20	--	No bond
1B_-12_SALINE	-12	Saline Spray	--	--	--	21-Dec-20	--	No bond
1B_-22_SALINE	-22	Saline Spray	--	--	--	5-Jan-21	--	No bond
5B_-22_SALINE	-22	Saline Spray	--	--	--	5-Jan-21	--	No bond
3B_-22_SALINE	-22	Saline Spray	--	--	--	6-Jan-21	--	No bond
2B_-22_SALINE	-22	Saline Spray	--	--	--	6-Jan-21	--	No bond
3_-22_SALINE"	-22	Saline Spray	--	--	--	8-Jan-21	--	No bond

* Note: 'sw' refers to sample preparation where thin plastic 'Shrink Wrap' was used to prevent the inner walls of the wooden sample jig from adhering to the ice sample during freeze-up.

** Note: This board was lengthwise vertically oriented during the spraying process.

*** Note: Shear tests were conducted for these two ice samples, however, we do not consider the results to be indicative of true solid-solid bonding between the ice and ice-phobic surface. This was due to the presence of a thin layer of brine at the interface.

also the cases where ice had been prepared but no tests were conducted because it was obvious that no actual solid-to-solid bonding had occurred between the ice and the ice-phobic layers. Figure 12 shows the results of all cases where a shear test had been performed. For the

freshwater ice samples tested at $-12\text{ }^{\circ}\text{C}$ there are obvious differences in the results for all of the tested coatings with respect to the adhesive bond strength between the coating and the ice sample. The ‘best performing’ board was 1A, followed by 5A, 2A, 4A and 3A as progressively ‘poorer performing’ ones. Also noteworthy is that for any particular coating the degradation of its performance is evident with the number of tests on the coating. And furthermore the slope of the degradation trend is more pronounced for the lower performance coatings. This suggests that the coatings that yield higher adhesive strength experience more ‘damage’ during the ice/coating bond breakage due to the higher local interface forces. The exact nature of the ‘damage’ is not known since that was beyond the scope of this study. Similar relative adhesive-strength hierarchy and trends appear at $-22\text{ }^{\circ}\text{C}$ for the two boards tested at that temperature (1A and 5A), even though only rough estimates of the ice/coating contact areas were obtained for three of the four tests. Finally we note that the two lowest-value data points on the graph correspond to tests of saline ice samples on board 4B. While the data points have been included here for completeness since tests were actually performed in these two cases, it is very likely that there was no solid-solid bond between the coating and

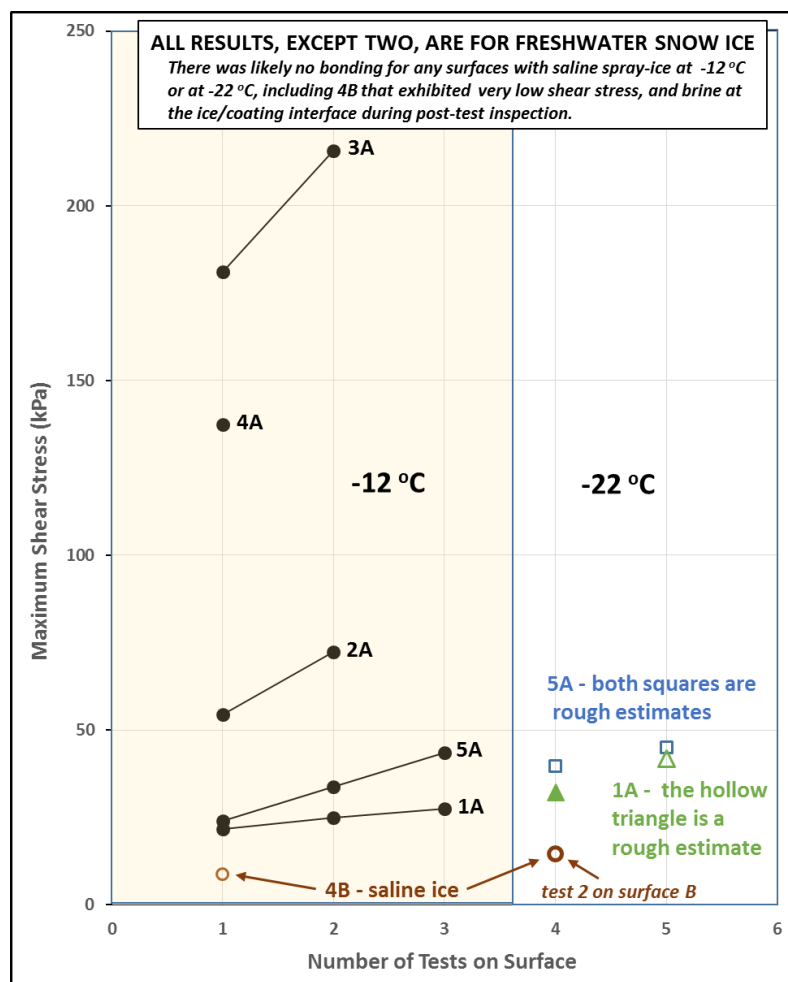


Figure 12. Summary plot of all data obtained from ice samples subjected to shear tests (see Table 1). Note that most of the tests correspond to freshwater ice samples and are considered as valid determinations of bonded adhesive strength. Two tests were for saline ice specimens. However, in these cases the results do not indicate bonded adhesive strength but rather the shear force of a thin layer of brine between the ice and coating surfaces and also the interlocking, but unbonded, shear force of the two rough surfaces.

the ice. The very low shear stress values, compared to freshwater ice samples, and observed presence of brine at the ice/coating interface during post-test inspection support this conclusion.

CONCLUSIONS

Adhesive-strength shear tests for a variety of ice-phobic coatings, using freshwater and saline ice samples, were conducted at -12 °C and -22 °C. Sample preparation, and testing, was challenging due to non-flat aspects of the supplied boards and coatings. But eventually a snow-ice sample preparation technique was devised for the case of freshwater ice samples.

Freshwater ice sample results showed widely varying adhesive strengths between the various coatings, and consistency of results for any particular coating. For the few tests conducted at -22 °C on two coatings there did not seem to be a significant difference from those at -12 °C. Degradation trends of the ice-phobic coatings with the number of tests were evident. And furthermore, the rate of degradation could be intuitively explained by one mechanism, i.e. more damage to the coating occurred per test if the adhesive strength of the coating/ice bond was greater.

Saline ice layers produced by spraying salt water onto the boards did not bond (in a solid-solid manner) to any of the coatings, since a thin layer of brine was always present at the coating/ice interface. What may keep saline ice accumulations ‘attached’ to a surface is more complex than simply whether or not a solid ice/surface bond has been established. The presence of brine, and time dependencies related to its flow, with respect to geometrical aspects of the interfacing surfaces apparently play important roles.

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