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Water Cell Filling/Freezing Test Report

Ice Home Risk Reduction NASA Langley	Ice Home RR Report: Water Cell Filling/Freezing Tests	FINAL, Rev. A Author: T. Caswell

1. Test Objectives

1.1. Relationship to Ice Home Goals

Determine fill and freezing methods at low pressure for best ice clarity and strength. The ice in the Mars Ice Home water holding cells should consist of solid, clear water ice with sufficient strength to be self-supporting and with viscosity low enough to avoid creep of the structure throughout its operational lifetime. The mechanical properties and clarity of the ice are both functions of the size of the crystals in the ice matrix (the "grain size"). Grain size is determined by the rate of cooling of the water cells during freezing (specifically, grain size is initially set by the relative rates of nucleation and growth of ice crystals during freezing) and the temperature at which the cells are maintained following initial freezing (which affects annealing of the ice). These tests are primarily concerned with the former: the initial grain size in the ice matrix after freezing, from which mechanical properties can be estimated based on existing formulae.

The stress at the base of a 15-m tall, vertical ice cell on Mars would be approximately 55 kilopascals. The rheology of pure, polycrystalline water ice is plotted in Fig. 1 according to the composite rheology of Goldsby & Kohlstedt (2001); at 55 kPa, it is apparent that the strain rate depends on the grain size in the ice. The figure demonstrates the importance of ice crystal size in the structural properties of Ice Home water cells.

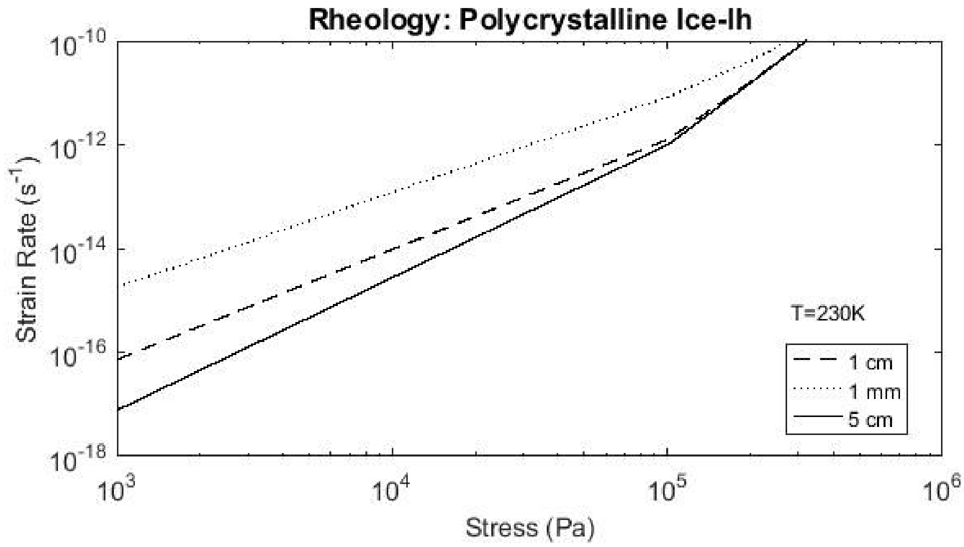


Figure 1: Composite ice rheology, calibrated by the experiments of Goldsby & Kohlstedt (2001), for a temperature of 230 K (approximate Mars ambient).

1.2. Specific Objectives and Rationale

The water holding cells will be outside of the habitation area and thus will be at a pressure less than one Earth atmosphere. In order to reduce risk there is a need to verify that clear, solid ice can be obtained by freezing water at the low pressures expected in the water holding cells, and to assess the mechanical properties of ice formed under low pressure conditions.

In addition to constraints placed on construction of the cells, the gas used to pressurize the water holding cells prior to filling is expected to be filtered air from the Martian atmosphere, which is primarily CO_2. Water cells in the Ice Home will have relief valves to prevent over pressurization, and relief valve settings will be dependent on the minimum pressures needed to freeze water with acceptable properties.

A typical Martian atmospheric pressure is only 0.09 PSI (as opposed to Earth's 14.69 PSI). Although there is much uncertainty in the overall habitat design and the ambient pressure that the cells will be subject to, it is expected that the water holding cells would not require a differential pressure greater than 3 PSIG to maintain the desired shape when initially inflated. This pressure is typical of inflatable structures that do not have to carry significant loads. It is also expected that a differential pressure of less than 0.5 PSIG will be inadequate to maintain a good shape when filling is being performed.

2. Test Apparatus and Methodology

2.1. Apparatus

Filling and freezing tests were conducted in a stainless steel, low-pressure chamber (Fig. 2) inside a commercial chest freezer. The apparatus uses a series of valves to pull vacuum on the NASA-furnished Tedlar bag or the chamber interior, and to introduce water to the bag.

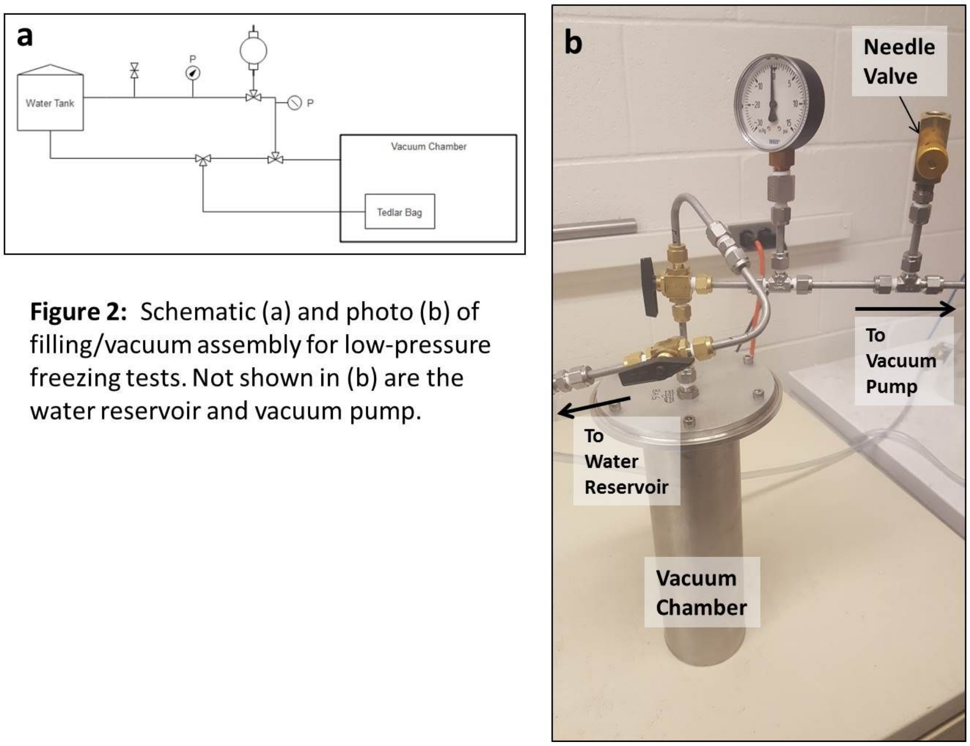


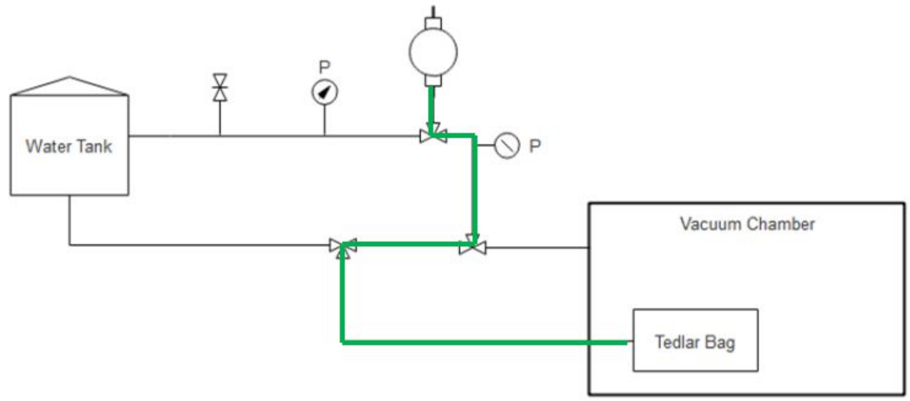
Figure 2: Schematic (a) and photo (b) of filling/vacuum assembly for low-pressure freezing tests. Not shown in (b) are the water reservoir and vacuum pump.

2.2. Test Methodology

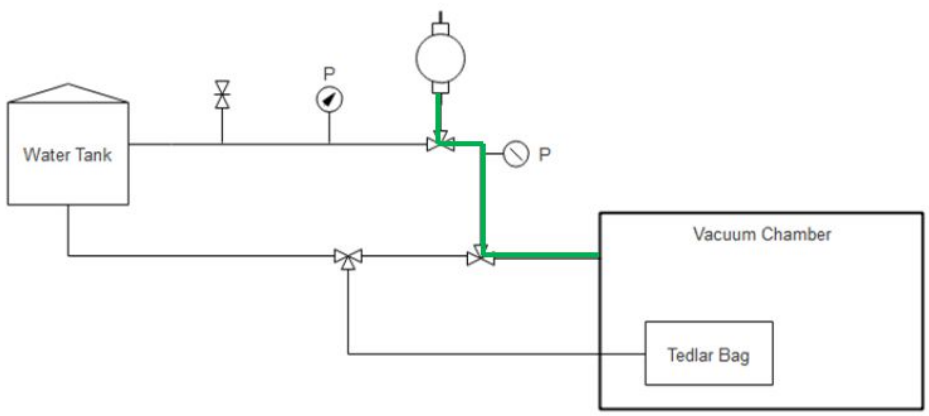
2.2.1. Filling/Freezing Tests

The test procedure is as follows:

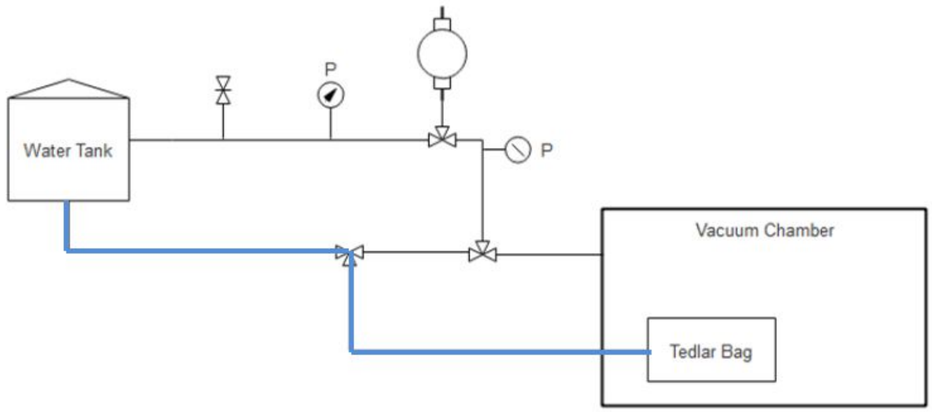
- Deionized water is degassed by boiling in a flask for 5-10 minutes. The flask is sealed, and the water allowed to cool to room temperature overnight.
- All lines in the apparatus are cleared of residual water by flowing air through the lines. The water line between the reservoir and Tedlar bag is evacuated to <1 psi by a vacuum pump to remove air and thereby reduce the introduction of air into the bag during filling.
- The Tedlar bag (Zefon International, Inc., model EG-PP-05) is opened and attached to the apparatus via the built-in sampling port on the bag. The vacuum chamber is then sealed.
- The chamber is placed in the freezer and allowed to cool for at least 1 hour.
- Vacuum is drawn on the bag to evacuate air from the bag.



- The chamber is drawn down to the desired pressure using a vacuum pump. Air is allowed to bleed into the chamber through a needle valve in order to control chamber pressure; the rate is adjusted to stabilize the chamber pressure at the desired level.



- Water is allowed to flow into the Tedlar bag. The backpressure in the water reservoir is prevented from equalizing with ambient pressure, which slows the rate of water introduction to the bag to avoid bursting. To avoid overfilling, <300 mL are introduced to the 500 mL Tedlar bags. The water is initially at room temperature, as colder water can freeze in the apparatus lines and result in a failed test.



- After filling, the water reservoir is isolated by an on/off valve (not shown in the schematic) and disconnected.
- The chamber is left in the freezer overnight. Freezer temperature is recorded via reading a thermometer in the freezer. The vacuum pump is left on, with airflow through the needle valve if necessary, for the first two hours of freezing to ensure stable low-pressure conditions. After that time, the chamber is isolated and the vacuum pump disconnected.
- After >12 hours, the chamber is allowed to equalize with ambient air.
- The ice-filled Tedlar bag is removed from the chamber for microscopy. All bags are stored in a chest freezer (~ -20 C) between freezing and analysis. Because that temperature is still high relative to the freezing point of ice, sections are imaged as soon as possible after freezing to reduce the potential for static annealing to affect the microstructures.

2.2.2. Microscopy

Ice microstructures are evaluated using “thick sections.” 2-4 mm sections are cut from the frozen bags using a band saw in a freezer. Sections are cut from various segments of the bags, including the edges and center, to evaluate spatial variability of ice properties within the bags.

Sections are polished by rubbing with successively finer sandpaper (down to 1200-grit) to remove marks from the saw blade. After polishing, sections are placed on an LED light tablet between cross-polarized films and photographed.

Grain size in the ice is measured via the line-intercept method. A line of known length is drawn across the image of the sample. The number of grain boundaries crossing the line is counted. The grain size is then the length of the line divided by the number of boundaries counted, multiplied by a geometric factor. The geometric factor, in this case 1.3, accounts for the fact that grains are not cut perfectly along their longest axis.

Relative transmissivity of the thick sections was evaluated by measuring the absolute gray values (a measure of total brightness) of the background illumination and the specimen in images using the NIH analysis software ImageJ. These values were measured in images with the cross-polarized films removed.

3. Results

In all tests, a solid mass of ice was produced inside of the Tedlar bag. A small amount of "snow" was also found within the bags, although the amount varied between tests.

A representative ice microstructure is shown in Fig. 3. In general, all bags displayed a region of finer (sub-cm-scale) grains near the topmost surface of the ice (i.e., the liquid/vapor interface inside of the bag). This region gave way to much larger (cm-scale) grains within the bulk of the ice. The transition between grain sizes was sudden, rather than gradual.

Despite the fact that the water was degassed prior to freezing, numerous elongated bubbles were present in each sample.

For both specimens, the absolute gray value of the background illumination and the specimens were not significantly different, indicating that differences in freezing pressure did not produce measurable differences in the transmissivity of ice at the thicknesses studied (1-5 mm).

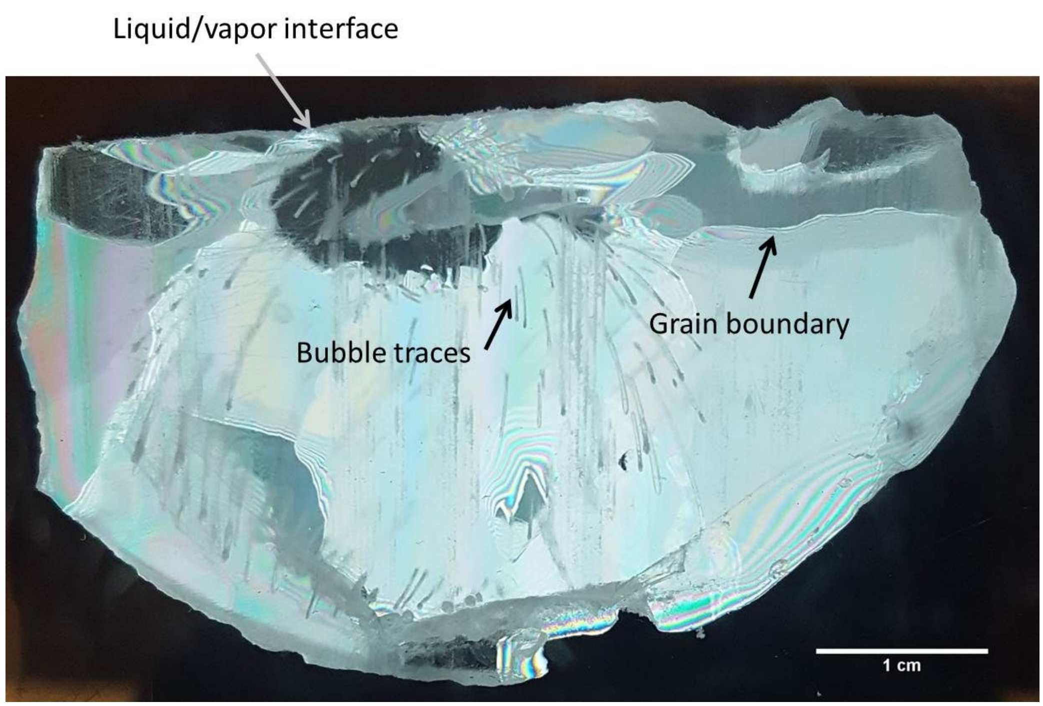


Figure 3: Cross-section along short axis of bag frozen at <1 psi ambient pressure. The top surface in the photo is the liquid/vapor interface.

3.1. Low-pressure (1 psi)

In this test, the bag and chamber were evacuated to the lowest pressure achievable with the apparatus (<1 psi). Water was introduced at room temperature, 25.9 C. The bag and chamber were equilibrated at -12 C.

The chamber and bag were laid horizontally in the chest freezer. Because of a leak in the line through which water was introduced, the bag was only ~⅓ full. Ice formed in both the bag and the chamber (due to the leak) were imaged for comparison. The frozen bag is shown in Fig. 4.

Figure 3 is a thick section cut parallel to the short axis of the bag. The section was cut from the ice adjacent to the sampling port, opposite from the bag label. The grain size in the upper section, closest to the ice-vapor interface, was measured as 0.9 cm. The grain size in the lower portion of the bag was 2.4 cm.

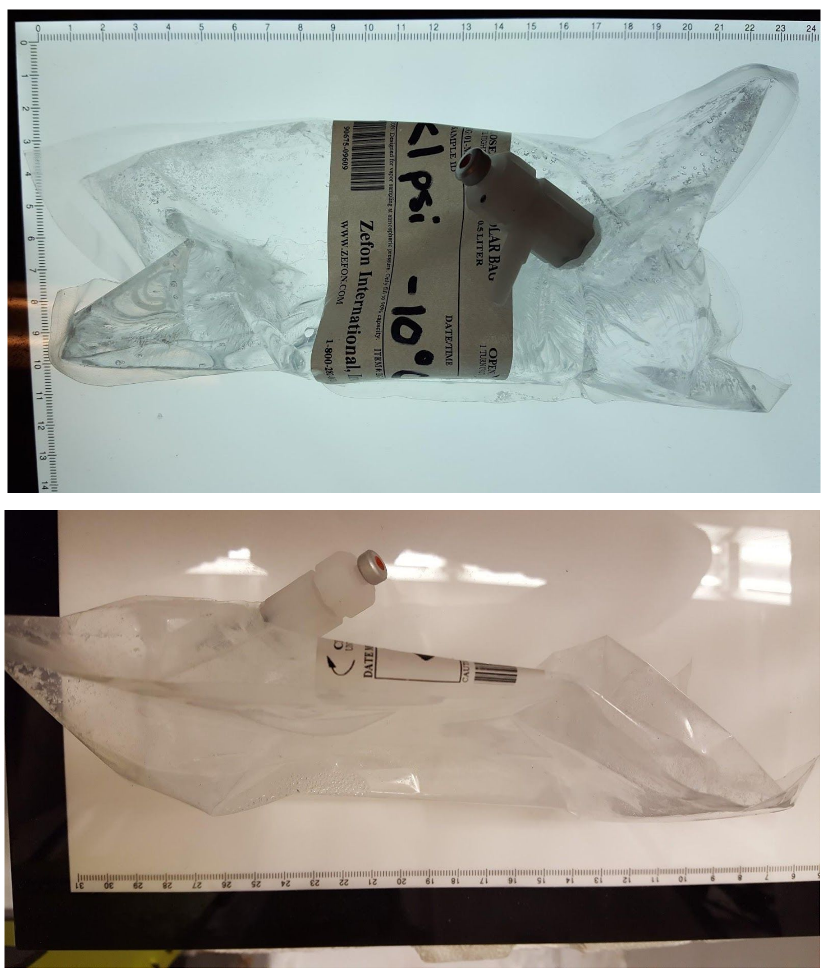


Fig. 4: Low-pressure (<1 psi) bag after freezing.

A section parallel to the long axis of the bag was also cut from the area of the ice adjacent to the sampling port. The section, shown in in Fig. 5, was cut as near as practicable to the ice-vapor interface in order to obtain a cross-section of the smaller grains imaged in Fig. 3. The grain size in this section is ~1.4 cm, indicating that the grains formed in the top layer of the water are plate-like, although many of the grains are anhedral and embayed by other grains. This texture suggests that some grain growth (static annealing) occurred in this section of the ice.

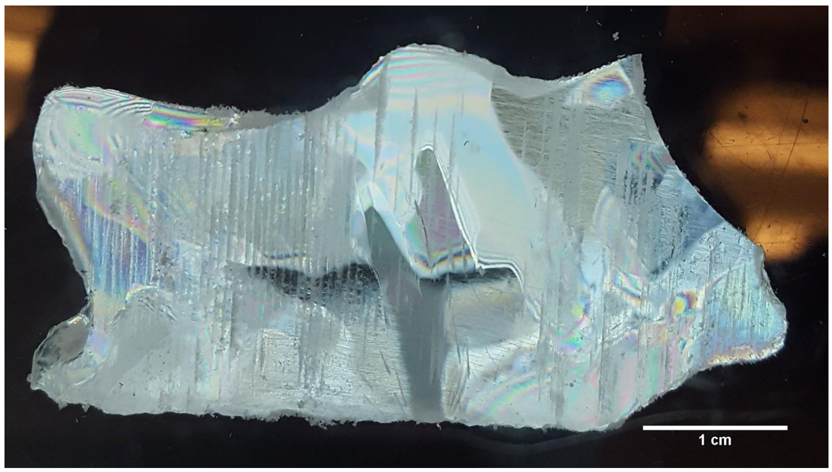


Figure 5: Section parallel to the ice-vapor interface in the bag frozen at <1 psi ambient pressure, cut from near the surface.

3.2. "High" pressure

The bag frozen at higher ambient pressure (~5 psi) exhibits a microstructure with overall similar characteristics to that frozen at low pressure, with a few key differences. First, a small amount of snow was formed within the bag and remained independent of the larger ice mass (Fig. 6). Though a small number of free ice crystals formed in the low-pressure test, the amount formed in this test constituted roughly 1 Tablespoon. The powder may have formed from water droplets condensed or adhered to the inside of the bag during freezing. Some of the grains are needle-shaped, which is common in snowflakes formed in the temperature range of these tests, though the bulk of the grains do not evince a specific crystal habit at the scale of the images collected.

Figure 6: Fine-grained "snow" from the high-pressure freezing test.

Grain sizes measured in cross-sections of the bag interior ranged from 1.4-3.1 cm. Grains in the middle of the ice mass, away from the ice/vapor interface, were elongated parallel to the short axis of the bag (Fig. 6). The texture suggests that the ice crystals nucleated near the edges of the bag and grew inward, consistent with cooling from the outside-in.

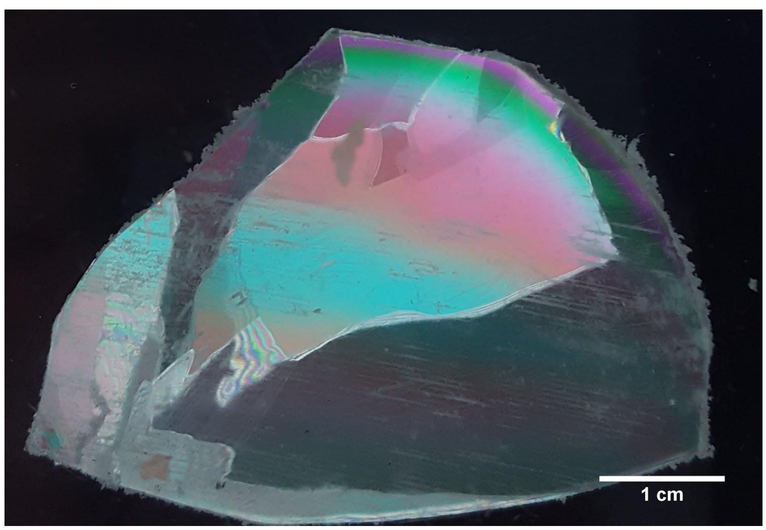


Figure 7: Thick section cut parallel to the short axis of the bag. The curved surface on the right-hand side was in contact with the edge of the bag. Grains are elongated perpendicular to the side of the bag, indicating nucleation near the cold edge of the bag and crystallization inward.

The section of this bag cut near the ice/vapor interface, shown in Fig. 8, exhibits finer grain size than in the <1 psi test. The size of small grains in this section is ~0.5 cm, though there are a few larger (~3.7 cm) grains at the edge of the section.

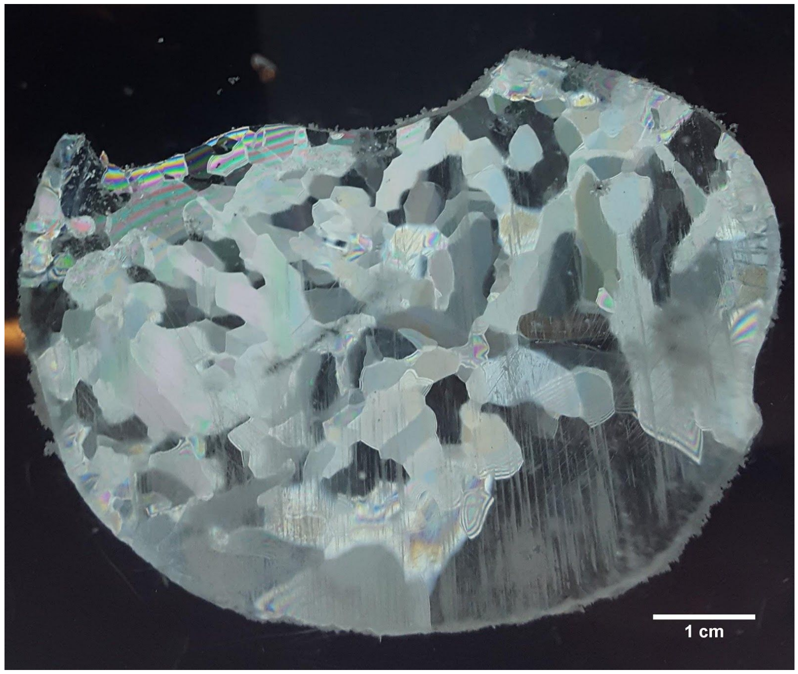


Figure 8: Thick section cut parallel to, and near, the ice/vapor surface in the bag frozen at 5 psi ambient.

4. Discussion

The general texture of the frozen ice packets indicates that an initial "quench" crust forms at the liquid/vapor interface during filling at low pressure, with substantially smaller grains at the interface than the ice in the bulk of the cell. The implication of this finding is that if water is introduced to the cells in small batches, the end result may be an accumulation of fine-grained layers. Such an ice cell would have lower creep resistance than a cell that is completely filled before freezing, in which the grain size would be much larger.

The ice that formed outside of the bag during the low-pressure filling test indicates what such a microstructure might look like. Fig. 9 shows a section of this ice, which formed in the bottom of the cold chamber. The ice within the Tedlar cell, where the vapor pressure of water is assumed to have raised the ambient pressure slightly, was much coarser (Fig. 3). By comparison, the ice kept at near-vacuum during freezing shows evidence of rapid freezing of successively deposited layers of water; the thickness of these layers is ~0.2 cm.

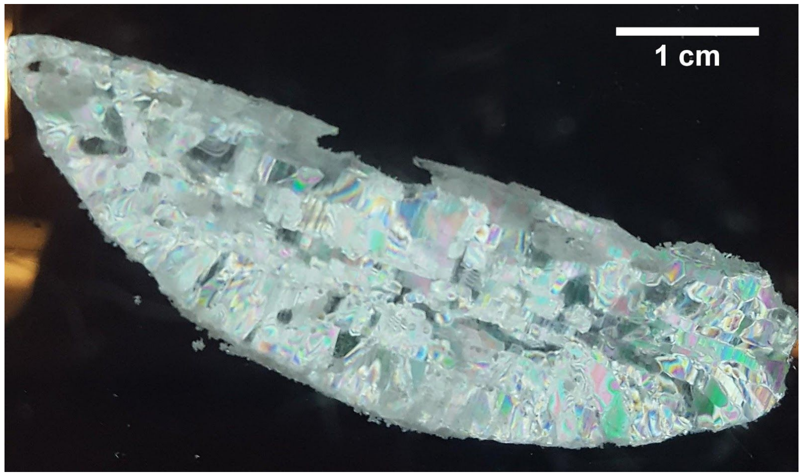


Figure 9: Fine-grained, layered ice formed in the bottom of the low-pressure chamber due to a leak during the “<1 psi” filling test. Fine-grained layers of ice have accumulated to form the overall ice mass. The curved surface of the specimen was in contact with the curved surface of the chamber interior, and layering is parallel to the local horizontal during freezing.

It is important to note that in addition to the ambient pressure during freezing, the rate of cooling strongly affects the rate of nucleation of ice crystals and, hence, the grain size in an ice cell. While these tests provide a qualitative description of the microstructures of ice formed in an Ice Home cell at low pressure, a more precise estimate of grain size (and therefore creep strength) in an Ice Home cell requires freezing tests conducted under the expected conditions for an Ice Home cell. It is recommended that the temperature and pressure environment of the Ice Home cells be defined prior to further ice microstructure tests. The shape of the cells, the expected temperature of water that is introduced, and the thermal environment of the cells should be constrained to better define future ice microstructure tests.

4.1 Future Work

While better constraints on the operational environment will be most useful in improving the usefulness of future filling/freezing tests, improvements could be made to the existing apparatus in order to produce more realistic microstructures. For example, addition of heating elements to the water lines to prevent freezing would allow cooler water to be introduced to the bags, more closely reflecting the actual conditions in an eventual Ice Home cell.

The very high relative transmissivity of the ice is probably due to the fact that the sections evaluated were, in general, thinner than the mean grain size. As a result, few intercrystalline boundaries were present to scatter light. Future evaluations of the optical properties of the ice should be made on thicker specimens once larger prototype cells are available.

Without further testing, annealing models could be run to estimate the amount of static grain growth over time based on variations in Ice Home cell environment. Such models could be used to anticipate how the strength of ice in the cells would evolve over time. These models could be quite simple - i.e., assuming a constant temperature within the cell, then determining grain size vs. time from simple grain growth kinetics laws - or complex, incorporating temporal and spatial variability in the temperature of the cells. The latter would require better definition of cell shape, which would influence the thermal mass of the cells.

Mechanical tests could also be performed on simulated ice cells. The cell could be filled and frozen within a fixture that would then be installed into a mechanical testing apparatus outfitted with a cold chamber (e.g., McCarthy & Cooper (2016), Caswell et al. (2015)). Such an apparatus could apply uniaxial compressive loading to simulate dIce Home cells in order to determine whether the cell material imparts extra creep strength to the structure.

5. References Cited

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