EFFECTS OF CRYOGENIC PROCESSING ON RAPID PROTOTYPING MATERIALS (DSM SOMOS-8110 AND DURAFORM PA)

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ABSTRACT

This research investigates the effects of cryogenic processing on the properties of rapid prototyped materials. Not much research has been done on the post-processing (aging) of rapid prototyped (RP) polymers at temperatures below 159K (–173˚F). Test specimens of RP thermoplastic resin DSM-Somos 8110 and DuraformPA Nylon were fabricated and cryogenically aged from 5-30 hours. The tensile strength and impact toughness were measured. The goal of this work was to study the effect of cryogenic aging on yield strength and ductility. This research investigated (1) the cryogenic aging of DSM-Somos 8110 and DuraformPA Nylon, (2) the effects of controlled ramp-downs/ups on the ultimate and tensile strengths of samples, (3) the experimental methods, and (4) the analysis and interpretation of the data.

INTRODUCTION

The primary objective of this research project is to apply cryogenic processing to Rapid Prototyped materials. Cryogenics is the science and art of producing cold. It started in 1877 when two scientists, Cailletet in Paris and Picet in Geneva, developed a procedure to liquefy oxygen in a laboratory [1-2]. Nowadays, nitrogen and helium are the most common cooling media. Since the normal boiling points of nitrogen and other permanent gases such as helium, oxygen and argon are about 120 K (approx. –244 ^{o}F), the cryogenic temperature is generally considered 120 K or below [3-4].

Cryogenic processing is one of the most important fields in industry today [4]. It helps to reduce costs for industry and increase industrial efficiency. For example, industrial application has reported 195% to 817% increase of wear resistance for standard steel that was cryogenically treated [2]. The cryogenic process consists of three stages based on time and temperature variables. This process starts with gradual ramping down of temperature to a specific point, the temperature is held at that point for a period of time, then the temperature is brought up to room temperature. As a result of this deep cooling and heating cycle, molecular changes occur, binding the atoms in the metal together [4].

Over the last 10-20 years, relatively little research has been conducted on cryogenic processing. Most research in cryogenics has been preformed on metals. Rapid Prototyping (RP) is a new technology the takes information from a computer-aided design file and makes a 3D part by building it one layer at a time [5]. When RP was first introduced in the late 80's, the materials used to produce the parts had low yield strength. This experiment attempts to show that the strengths of RP materials can be increased by cryogenically processing these parts before industrial application. As mentioned before, in all the cryogenic work the scientists and engineers lowered the temperature of the sample to cryogenic condition very fast, held it to the temperature for a few hours and then ramped up the temperature as fast as possible [4]. Two years ago, the authors did research on the cryogenic processing of both ABS plastic using Fused Deposition Modeling (FDM) and DSM SOMOS 8110 from Stereolithography Apparatus (SLA)-250 machines. While the cryogenic processing did not have much effect on ABS plastic, the yield strength of DSM SOMOS was increased between 25-50% [6]. The authors believe the ramp-down and ramp–up conditions might make a significant contribution to the increase in strength of the polymer samples.

The authors have recently developed a Data Acquisition System (DAS) using LabVIEW from National Instruments that provided the programming power for the ramp-down and ramp-up conditions. The conventional and proposed cryogenic processes are shown in **Figures 1** and **2**.

RAMP DOWN/UP PROCESS

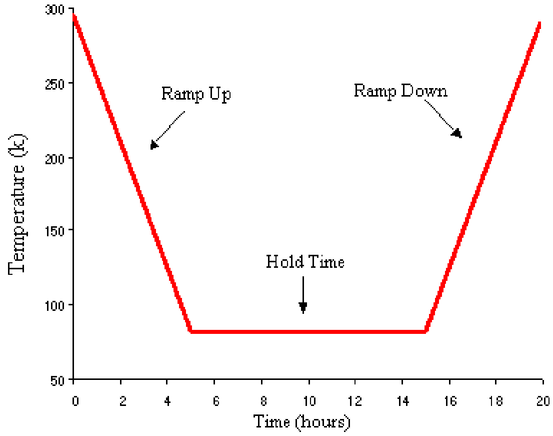


Figure 1. Original Procedure

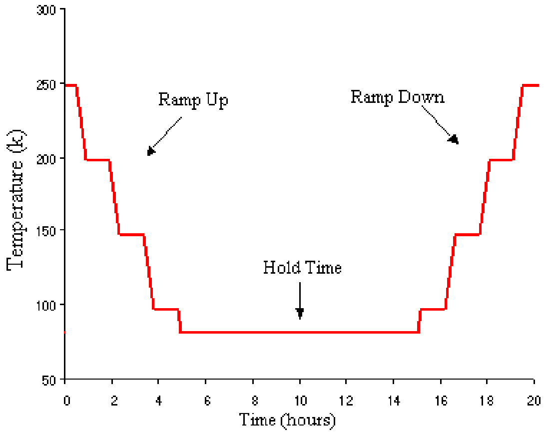


Figure 2. Proposed Procedure

EXPERIMENTAL PROCEDURE

Equipment and Process

The following major components were used in this experiment:

1. Northrop Grumman SLA-250 RP Machine
2. Cryogenic Treatment Equipment, **Figure 3**
3. Instron Tensile Testing Machine
4. Izod Impact Tester
5. Scanning Electron Microscope (SEM)

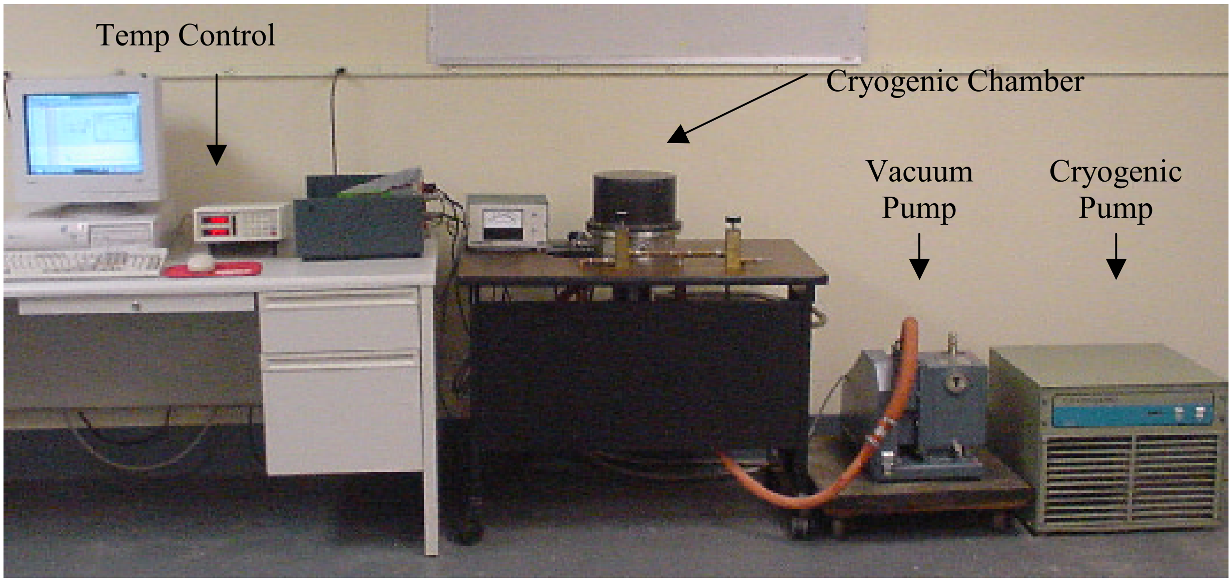


Figure 3. Cryogenic Equipment

The following **Figure 4** outlines the experimental process that was used to design, fabricate, test, and analyze the samples.

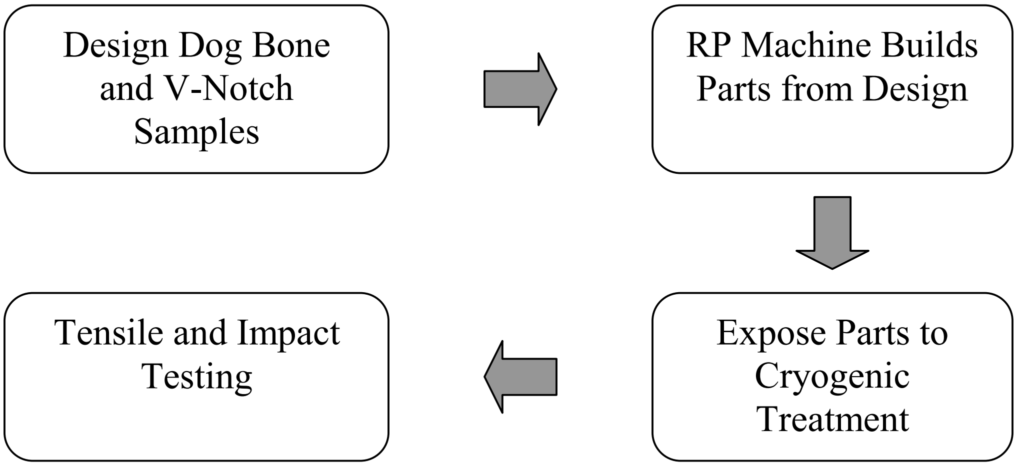
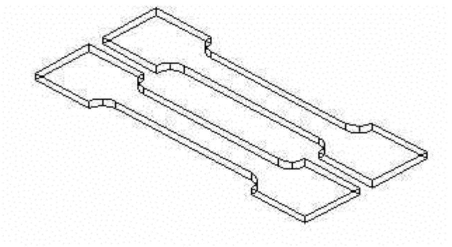


Figure 4. Outlines of Steps for the Experiment

Designing and Prototyping the Samples

Drawings of the dog bone and v-notch shaped samples were created using AutoCAD (**Figs. 5A and 5B**) and then were saved as separate .DWG files. These files were then converted into .STL format for use with QuickSlice software. The SLA-250 (Northrop Grumman) RP machine was then used to rapid prototype the parts.



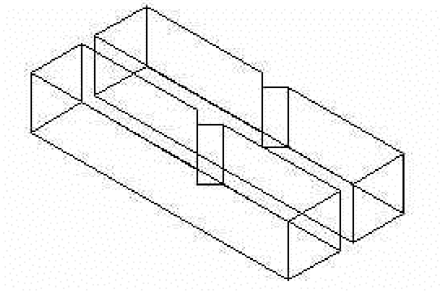


Figure 5A. Two dog bone-shaped samples. **Figure 5B.** Two v-notch-shaped samples.

Cryogenic Treatment

The following procedure was used to cryogenically treat each sample.

1. All samples except the baseline went through cryogenic treatment before testing. The samples were prepared at Northrop Grumman and processed at Loyola Marymount University.
2. The cryogenic process is characterized by three parameters: ramping down time from room temperature to 82K (–312^{o}F), holding time at 82K, and ramping up time from 82K to room temperature.
3. Preliminary experiments are then performed on the samples with the ramping cryogenic treatment process. Ramp-down times of 5 hours are used. Holding times of 5, 8, 10, 12 and 20 hours, and ramp up times of 5 hours are used.
4. Samples are labeled as follows: XX-XX-XX. The numbers represent the ramp-down time, holding time and ramp-up time, respectively, in hours. (e.g: 05-10-05 means that the samples were ramped-down in 5 hours, held in a cryogenic state for 10 hours, and then ramped-up in 5 hours.)

Tensile Testing

The tensile strength of the samples was measured using the Instron Universal Testing Instrument 4500. The cross-head speed of the test machine was 0.0212 mm/s. The machine was interfaced using its front panel and a software program running on a desktop computer. The test specimens were created in the shape of dog-bones, as shown in **Figure 5A**. The dog-bone shaped samples were held in place by the two opposing grips of the testing platform. A computerized load cell located inside the frame unit measured the force applied to the dog-bone shaped samples. Strain was measured with an extensometer, and the stress vs. strain curves were plotted while the specimen was gradually loaded. The yield stress was measured at 0.2% offset strain, and the ultimate strength was measured at the maximum stress the material could withstand.

Four to six samples were tested for each cryogenic aging treatment (0-20 hours). The tensile strength data was statistically analyzed to determine the effects of aging treatment on yield and ultimate strength.

Izod Impact Testing

Izod impact testing was performed to determine the toughness of the treated material. The samples were made with a centered v-shaped notch. During the impact testing, the samples were subjected to an impulsive blow by a hammer pendulum. The impact test evaluates the material's resistance to crack propagation.

RESULTS AND DISCUSSION

Tensile and Impact Testing

The results of the yield strength, ultimate strength and impact energy per unit area vs. cryogenic treatment time (0-30 hours) are shown in **Figures 6A, 6B, 6C, 6D, 6E and 6F.** For the yield strength of the Somos 8110, it appears to peak at a hold time of 10 hours, which is consistent with the results of the previous year, though the results show too much variance to determine a definite trend. Three of the four points of the 10 hour hold time are above the base line, so an increase is inferred, **Figure 6A**. On the other hand, the 10 hour hold time appears to be a low point of the ultimate tensile strength graph for the Somos 8110. Again, the scatter of data points makes it difficult to discern a trend, **Figure 6B**.

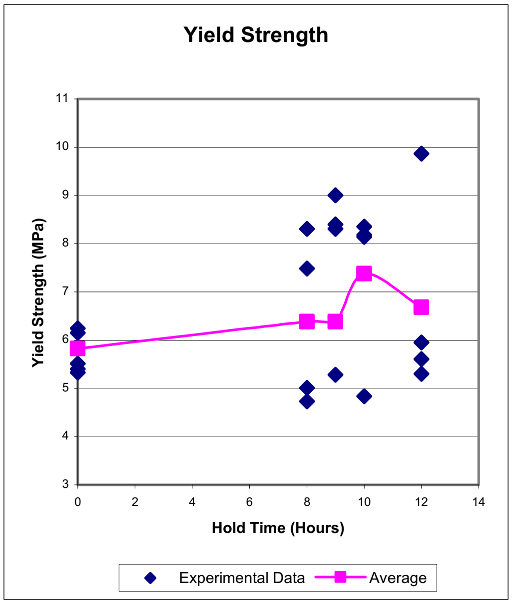


Figure 6A. Somos 8110 Yield Strength.

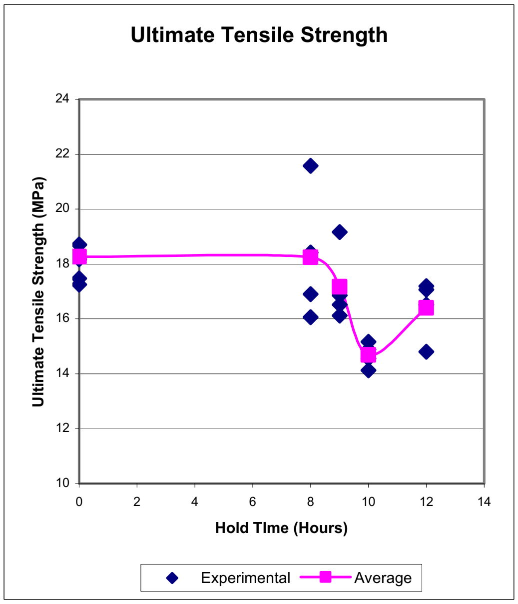


Figure 6B. Somos 8110 Ultimate Strength.

The impact energy of the Somos 8110 appears to decrease as the holding time increases. The trend, again, is complicated to find because of the erratic nature of the data -- even the baseline samples have a large degree of scatter, **Figure 6C**. As for the yield strength of the DuraformPA nylon, there is a significant drop for the hold time of 12 hours, **Figure 6D**. Further testing will need to be done to determine a reason for this effect.

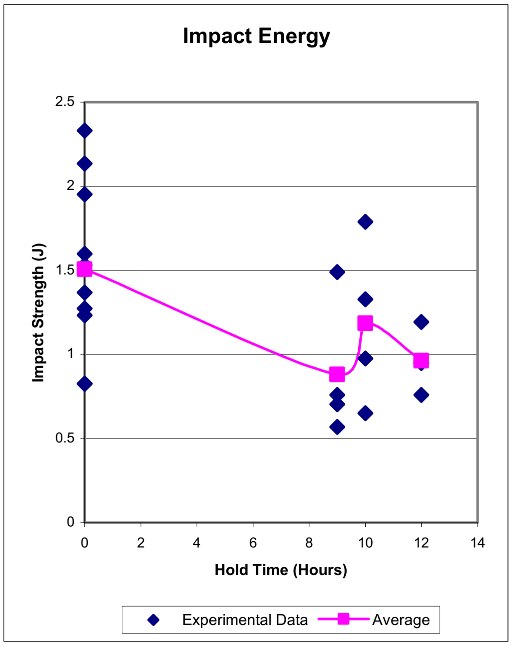


Figure 6C. Somos 8110 Impact Energy.

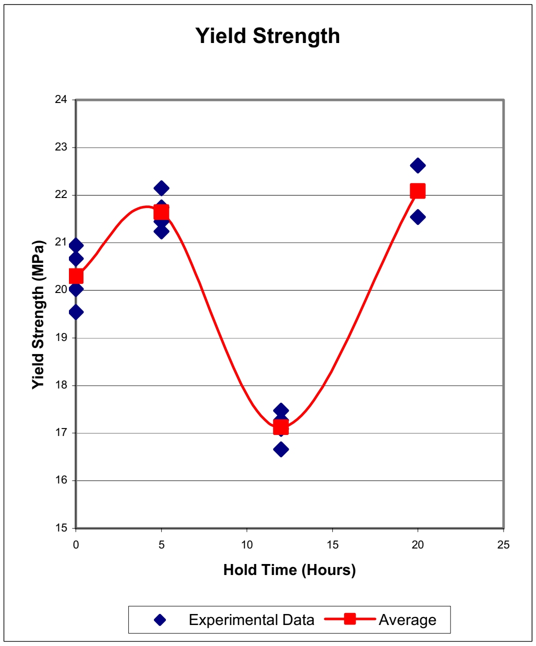


Figure 6D. DuraformPA Yield Strength.

The ultimate strength appears to increase with the increase in hold time, **Figure 6E.** Once again there is a significant scatter of data and the true trend is unclear. There is a significant increase in the impact energy at the 5 hour hold time, **Figure 6F**, with a variance of the data points as in the previous samples. The v-notch samples varied based on their position in the copper carrier. The v-notch samples were arranged four in a row. The middle samples had lower impact energies than the outside samples.

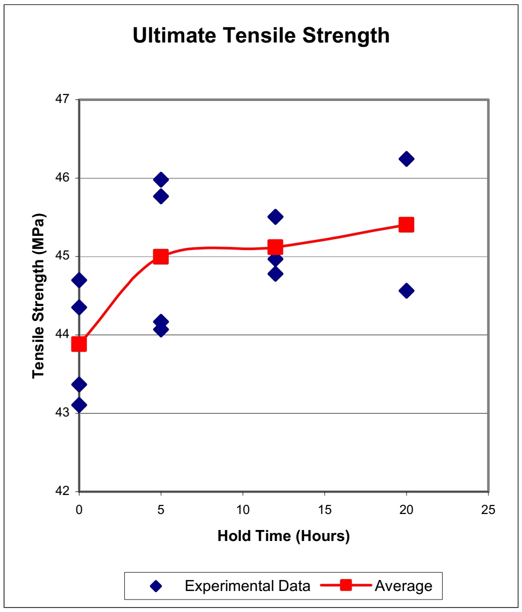


Figure 6E. Duraform Ultimate Strength.

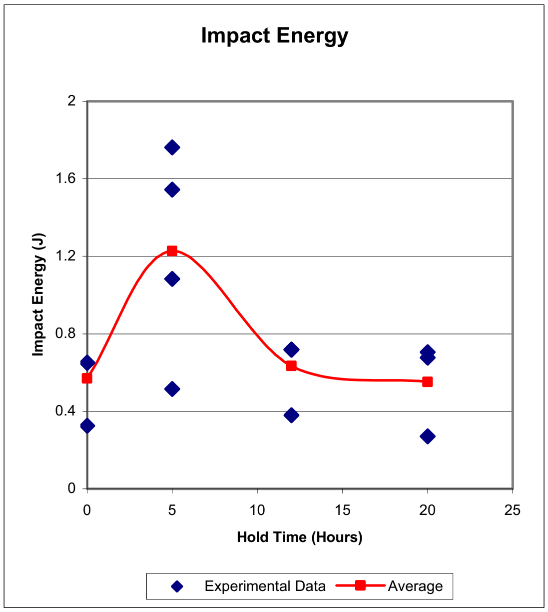


Figure 6F. Duraform Impact Energy.

The effects of ramp-down times on the yield strength and ultimate strength are shown in **Figures 7A and 7B**. In both cases, the strengths appear to be negatively affected.

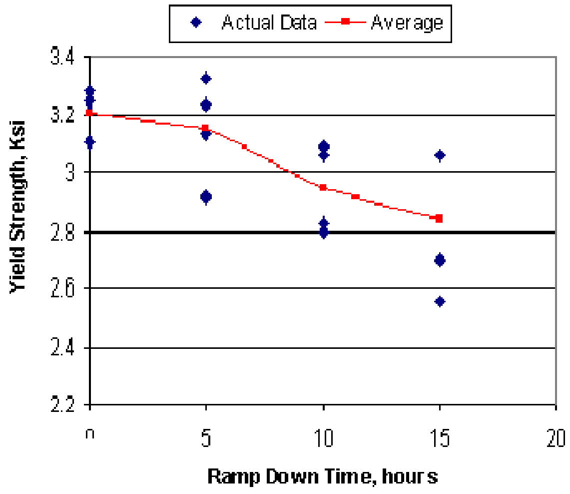


Figure 7A. Yield Strength vs. Ramp-up Time

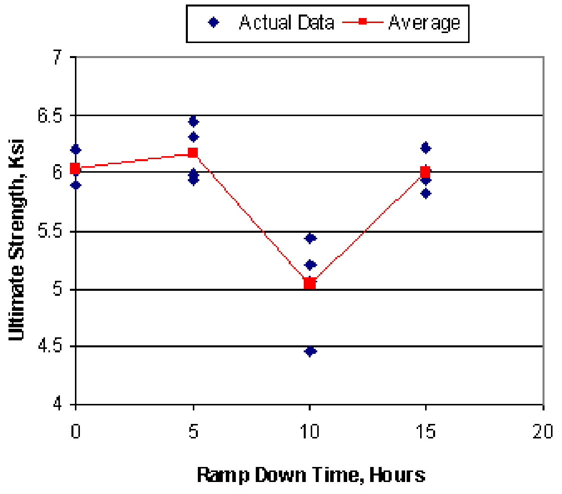


Figure 7B. Ultimate Strength vs. Ramp-up Time

Fractography

The DuraformPA Nylon samples were too ductile to produce a flat fracture surface. The plasticity was demonstrated by the necking of the sample as shown in **Figure 8A,** which also illustrates the rough surface of the break.

The Somos 8110 samples broke cleanly, producing a clear mirror which can be seen in **Figure 8B.** All Somos 8110 samples contained an array of dimples along one side, which were a byproduct of the rapid prototyping process. The dimples introduced weak points and can be seen in **Figure 8C**. The parts always broke along the dimples and this can be seen in **Figure 8D**.

Figure 8A. Duraform PA Nylon Necking

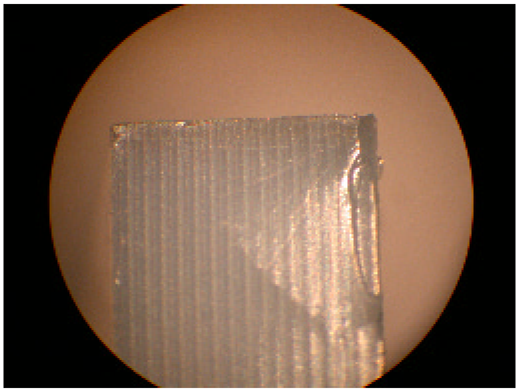


Figure 8B. 36x SEM micrograph of DSM-Somos 8110

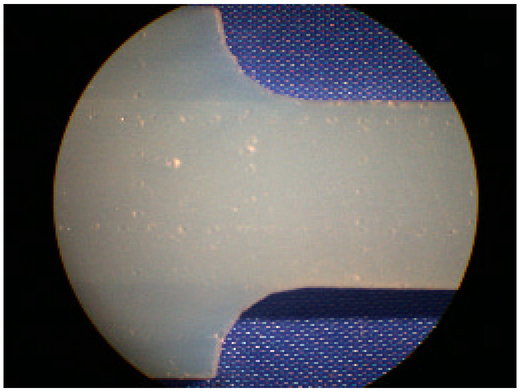


Figure 8C. Top view of DSM-Somos

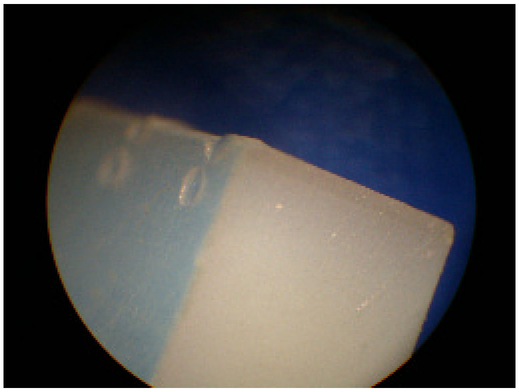


Figure 8D. 36x Somos 8110 Dimples and fracture surface

CONCLUSIONS AND RECOMMENDATIONS

Based on the findings from this experiment, the following conclusions and recommendations can be made:

1. Due to large data scatter it is difficult to determine the trend of the data.
2. The defects in the Somos 8110 samples from the previous year were corrected resulting in improved data, but the Somos 8110 contained an array of dimples that created weak points. If these dimples could be removed from the sample the results may improve.
3. The v-notch parts of both the Somos 8110 and the Duraform 8110 should be arranged around the perimeter of the copper carrier of the cryogenic chamber to ensure a uniform heat distribution to each sample during the ramp-up phase.
4. Cryogenic processing of prototypes samples has not significantly and consistently impacted the strengths of the samples. As the strengths of the rapid prototyping materials are increased through research and development, cryogenic processing of rapid prototyped samples may not be necessary.

ACKNOWLEDGEMENTS

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