Water-Based Phase Change Material Heat Exchanger Development

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In a cyclical heat load environment such as low Lunar orbit, a spacecraft’s radiators are not sized to reject the full heat load requirement. Traditionally, a supplemental heat rejection device (SHReD) such as an evaporator or sublimator is used to act as a “topper” to meet the additional heat rejection demands. Utilizing a Phase Change Material (PCM) heat exchanger (HX) as a SHReD provides an attractive alternative to evaporators and sublimators as PCM HXs do not use a consumable, thereby leading to reduced launch mass and volume requirements. Studies conducted in this paper investigate utilizing water’s high latent heat of formation as a PCM, as opposed to traditional waxes, and corresponding complications surrounding freezing water in an enclosed volume. Work highlighted in this study is primarily visual and includes understanding ice formation, freeze front propagation, and the solidification process of water/ice. Various test coupons were constructed of copper to emulate the interstitial pin configuration (to aid in conduction) of the proposed water PCM HX design. Construction of a prototypic HX was also completed in which a flexible bladder material and interstitial pin configurations were tested. Additionally, a microgravity flight was conducted where three copper test articles were frozen continuously during microgravity and 2-g periods and individual water droplets were frozen during microgravity.

Nomenclature

\begin{itemize}
  \item $^\circ$C = degree Celsius
  \item DRM = design reference mission
  \item ESLI = energy sciences laboratory
  \item HX = heat exchanger
  \item IDC = Innovation Development Center
  \item JSC = Johnson space center
  \item $kJ$ = kilojoule
  \item $km$ = kilometer
\end{itemize}

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lbs/hr = pounds per hour
LLO = low lunar orbit
LN\textsubscript{2} = liquid nitrogen
PC = phase change
PCM = phase change material
PVC = polyvinyl chloride
RIP = replicative ice phase change material
RoHS = replicative heat sink
SHReD = supplemental heat rejection device
SHRIMP = small heat sinks of replicative ice material for phase change

\section{Introduction}

NASA's current Design Reference Missions (DRM) pushes the boundaries of current spacecraft technology, including the thermal control systems. Specifically, these DRM's require a spacecraft to operate under cyclical thermal environments, such as experienced in low Lunar orbit (LLO). As shown in Figure 1, the lunar surface temperature varies from approximately 400 Kelvin to less than 100 Kelvin. The hottest portion of the orbit corresponds to the subsolar point; i.e., the area directly aligned with the sun. Similarly, the coldest portion corresponds to the area on the opposite side of the moon. Because of the large variations in the temperature, the vehicle will experience large changes in radiative sink temperatures. Therefore, robust spacecraft thermal control systems must be developed to provide adequate heat rejection demands for both the hot portion and the cold portion of an orbit. Figure 2 plots an example of the variability of a vehicle’s heat rejection capability using only body mounted radiators for a 100 km circular orbit with a beta angle of zero degrees, representing the worst-case hot LLO environment. The radiators are capable of rejecting the full vehicle heat load for the majority of the orbit period. However, when the vehicle is orbiting at or near the subsolar point (0 to 0.4 hours and 1.6 to 2 hours), the radiators do not meet the full heat rejection demands of the spacecraft. Thus, some type of Supplemental Heat Rejection Device (SHReD) is required to meet the vehicle’s heat rejection requirement. SHReDs typically employed in thermal control systems include evaporators, sublimators, or Phase Change Material (PCM) heat exchangers (HXs). Using a PCM HX as a SHReD can be advantageous for long mission durations because it does not require a consumable as is required in an evaporator or sublimator.

PCM HXs act as a thermal battery and store excess thermal energy during periods of high heat loads (hot thermal environments) by melting the PCM within the heat exchanger. The PCM is then refrozen during periods of low heat loads (cold thermal environments). PCMs have been used in spacecraft since the Apollo era. The Apollo Lunar Rover utilizes two wax-based PCM heat sinks to cool the rover’s batteries and electronics. Skylab also utilized a wax PCM.\textsuperscript{1} More recently, Energy Sciences Laboratory partnered with Goddard Space Flight Center to fly a paraffin wax carbon composite heat sink on Space Transportation System (STS) STS-95 in a

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure1.png}
\caption{Lunar surface temperatures.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure2.png}
\caption{SHReD requirements.}
\end{figure}
system called Cryogenic Thermal Storage Unit.² Although paraffin wax heat sinks have been used since the 1960s, extensive on-orbit testing has not been conducted and only wax-based HXs have been used. Water PCMs have neither been flown on spacecraft nor tested in microgravity.

Water is advantageous for use in a HX due to water’s large heat of fusion. When compared to n-pentadecane, the baseline wax for Orion, NASA’s new manned spacecraft, a given mass of water is capable of storing about 60% more energy than that of wax. The heat of fusion for n-pentadecane is 200 kJ/kg, whereas the heat of fusion for water is 333 kJ/kg. Thus, by increasing the amount of energy storage per unit mass, water has potential to significantly reduce a HXs mass and volume requirements.

Utilizing water has one particular disadvantage. Unlike most materials, water expands when frozen, thereby leading to concerns regarding structural integrity of the HX, especially when enclosed in a ridged structure. This report summarizes previous efforts to develop a water-based PCM HX, current testing efforts and outcomes, and provides future direction to develop a functional water-based PCM HX.

## II. Prior Phase Change Material Development and Testing

### A. Small Heat Sinks of Replicative Ice Material for Phase Change/Replicative Ice Material Phase Change Material Testing

A total of 17 PCM test articles have been built and tested in conjunction with Energy Sciences Laboratory (ESL). These studies included a life test of four wax PCM HXs, testing of eight Small Heat Sinks of Replicative Ice Material for Phase Change (SHRIMP), and testing of five Replicative Ice PCMs (RIPs).³ ⁴ ⁵ Testing of these articles occurred between 2009 and 2013. A summary of testing can be found in Table 1. Specific care was given to the interstitial material and void space distribution in the test article during the designing and testing of these articles. All test articles utilized some type of aluminum fin and carbon fiber interstitial material (Figure 3). Interstitial material was used to act as a sponge and hold water in a specific location independent of orientation. Additionally, as water expands 10% when frozen, a generous 20% void space was given to the test articles to allow the water to expand. Four generations of interstitial material configurations were cycled through freeze/thawing environments in both favorable (freezing in normal, bottom-upwards freezing) and unfavorable (freezing from top-down) orientations.

During the course of testing, all water-based RIP and SHRIMP test articles failed. Failure typically occurred due to mechanical pressure from ice spike formation within a test article. A picture of failure can be seen in Figure 4, and the hypothesized process is highlighted in Figure 5. This is identical to why ice cubes have a small bulge in the center of the cube.

![Figure 4. Failure in SHRIMP test article.](image)

It is also important to note that significant manufacturing problems were present in the machining of the groves cut into the test article, as can be seen in Figure 3. As these grooves were cut, chips of un-anodized aluminum were pressed into the carbon fibers. Although particular care was given to remove these chips and anodize any exposed aluminum area, two test articles developed bows due to increased pressure (as opposed to bulges caused from ice spike formation) from the formation of hydrogen gas, a by-product of non-anodized aluminum in water.

### B. Replicative Heat Sink (RoHS)/Integrated Replicative Ice PCM (IRIP)

The Replicative Heat Sink (RoHS) and the Integrated Replicative Ice PCM

![Figure 5. Ice spike formation in a SHRIMP.](image)

<table>
<thead>
<tr>
<th>Test Article Phase Change Material</th>
<th>Energy Storage (kJ)</th>
<th>Articles Tested</th>
<th>Cycles Tested</th>
<th>Failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wax (Life Test)</td>
<td>Wax</td>
<td>450</td>
<td>4</td>
<td>700</td>
</tr>
<tr>
<td>SHRIMP</td>
<td>Water</td>
<td>45</td>
<td>8</td>
<td>524</td>
</tr>
<tr>
<td>RIP</td>
<td>Water</td>
<td>450</td>
<td>4</td>
<td>140</td>
</tr>
<tr>
<td>1-Sided RIP</td>
<td>Water</td>
<td>225</td>
<td>1</td>
<td>50</td>
</tr>
</tbody>
</table>
(IRIP) were test articles constructed to develop a suitable water-based PCM HX for use on the lunar electric rover (Chariot). RoHS evaluated several types of interstitial material including six finned and four foam interstitial materials as well as copper and aluminum fins. Carbon fibers were not used. These materials were evaluated to determine which material was best for use in a PCM heat exchanger. An interstitial material was selected from these tests, and a full-scale IRIP was constructed.

IRIP was tested in summer 2012 in Chamber E at Johnson Space Center (JSC), and it combined a PCM HX with a radiator surface. It used folded aluminum fins brazed to a top facesheet as interstitial material for phase change. Approximately 38 kg (20% void space) of water was stored in the HX. During day 4 of the 5-day test, the test article developed a 2” long tear in the facesheet near the exit of the coldplate seen in Figure 6. One possible cause of failure indicates that as water was freezing, it was forced near the exit (relative warmest location of water) of the coldplate through the course of several freeze/thaw cycles. As this local area of warm water began to freeze, hydraulically locked water expanded and eventually the face sheet yielded to the pressures induced by freezing water.

C. Foam Insert Testing

A PCM Supercooled Ice Pack was developed by Paragon Space Development Corporation for use in thermal control of an astronaut in a space suit. This ice pack was designed to be removable and reusable, and was allowed to be supercooled between extravehicular activities. In particular, this design utilized a flexible membrane (polyvinyl fluoride) material coupled with a flexible foam material to accommodate expansion and contraction of water through freeze and thaw cycles. Testing results of this device are not reported.

A water-based PCM with a foam core was developed and tested in development of a Small Payload Quick Return vehicle. In this study, requirements for a foam, or crushable, media were identified as being able to compress under a relatively low load, water resistant, closed cell, resilient, sturdy, low density, and insulative. The foam selected but was able to compress to 50% of its original volume and meets the previously stated criteria. Initial testing of their foam inserts in developmental test articles resulted in failure. However, the flight-like design with foam insert was successful.

D. Mezzo Technologies Heat Exchanger Testing

A Mezzo Technologies Microtube HX was tested during summer 2013. This HX was manufactured by Mezzo Technologies and contains approximately 5,000 tubes positioned in a 4” x 4” area and is capable of storing 315 kJ of latent energy storage. A total of four favorable, ten neutral, and five unfavorable tests (19 total cycles) were completed with no visible signs of failure of the test article. However, it was observed that in adverse orientation, the ice level before and after freezing was higher when compared to favorable orientation as seen in Figure 7.

In addition to the 19 cycles completed, consideration was given to controlling freeze front propagation by utilizing outside-in and inside-out freezing in this test article. By controlling how water freezes within the test article and flow distribution, one could control freeze front propagation, thereby leading to a HX that could freeze repeatedly in a predictable manner. To mimic inside-out freezing, a manifold flow restriction plate was three-dimensionally printed and tested (Figure 8). Two favorable cycles and

Figure 6. IRIP failure.

Figure 7. Comparison of favorably and adversely frozen Mezzo HX.

Figure 8. Inside-out flow restriction plate.

Figure 9. Mezzo HX midplate deflection.
one unfavorable cycle were completed with this plate. Although no major structural damage occurred with these test articles, the midplate (used for structural support and positioned halfway between the length of the tubes) of the test article experienced deformation during the two favorable freeze/thaw cycles and is pictured in Figure 9. The max deflection is about 0.2”.

With the successful testing of this test article, it was concluded that utilizing a microtube HX should be further investigated. However, a basic understanding of ice spike formations, midplate locations, and freeze front propagation should be completed to aid in developing a water-based PCM HX.

III. Copper Heat Exchanger Coupon Design and Testing

To understand freeze front propagation, ice spike formation, and other phase change phenomenon to a greater extent, four small copper HX with varying interstitial material patterns were constructed at JSC’s Innovation Development Center (IDC). The purpose in building these test articles was three-fold:

1. Notionally understand basic freeze front propagation and ice spike formation
2. Understand uniform freezing as well as inside-out and outside-in freezing
3. Apply outcomes of test to a future Mezzo HX designs

The Gen 1.0 coupon was consisted of a flat copper plate in which several copper rods were thermal epoxied into holes that had been drilled into the surface. A Lexan case was also built to enclose the coupon. The Generation 2.0 test article was constructed in a similar manner to the Gen 1.0 article; however, copper plates were used as sides instead of Lexan. Unique pin spacing was utilized, allowing for outside-in freezing (Figure 10). Gen 2.0 was constructed as a cube of ~2.0”. Generations 2.1 and 2.2 were essentially 3” cubed scaled versions of the Gen 2.0 test article and used to compare and contrast inside-out versus outside-in freeze front propagation (Figure 11).

As expected, ice spikes were formed in all test articles and occurred in a similar manner as were formed in the SHRIMP test articles (Figure 5). However, ice spike formation, size, and location varied between the test articles. In the Gen 1 test article (uniform pitch), several small ice spikes (~0.12”) formed at the intersections between copper rods at the location of the red dotted line in Figure 12. A single, large ice spike (~0.25”) formed at the middle of the test article in the Gen 2.1 test article (Figure 13). In the Gen 2.2 test article, an ice spike “ring” was formed around the two pin perimeters (Figure 14). The height of this ring was approximately 0.18”. The results of this test indicate that, for a given volume of water, the more locations liquid water has to expand into, the smaller the ice spike will be formed.

An additional liquid nitrogen (LN₂) test was completed to understand how a fast freeze affects ice spike formation. For this test, the Gen 2.0 test article was placed into a bath of LN₂ and allowed to freeze (Figure 15). This...
ice spike was approximately 0.5” compared to the 0.25” when frozen at a slow rate (2 hours). This test suggests that for a faster freeze time, ice has greater expansion than when compared to a slow rate. This leads to an interesting hypothesis. Typically, when water is frozen at a slow rate, dissolved gasses within the water escape from the freeze front (ice/water boundary) and, due to buoyancy forces, float to the surface. This may not be occurring within a quick freeze because as the freeze front propagates rapidly, dissolved gasses do not have sufficient time to escape and float to the surface. Instead, they become trapped in the ice as the freeze front quickly propagates. This extra gas within the ice could account for the added volume and height of the ice spike.

This could also account for the increased ice volume in unfavorable (adverse) testing in the Mezzo Technologies HX (Figure 7). As the freeze front propagates downward, dissolved gases escape from the ice/water boundary. Due to buoyancy, these air bubbles would float to the surface. However, since the surface is a ridged layer of ice, the air bubbles are pressed against the freeze front. As the front propagates, these bubbles become trapped in the ice, thereby increasing the overall volume of ice. This process is captured in Figure 16. It is hypothesized that this process would also occur in microgravity where buoyancy forces are not present. Future testing should be completed to confirm this hypothesis.

As a result of these tests, it was concluded that ice spikes form where water freezes last. Therefore, most void space should be situated at this location. Additionally, “ice spike distribution” can be utilized to reduce the overall size of ice spike formation. This would thereby reduce the mechanical pressures induced by an ice spike on a solid surface.

IV. Microgravity Flight Experiment

In fall 2013, a microgravity freezing experiment was flown aboard NASA’s Reduced Gravity Aircraft. This experiment was developed as part of NASA’s Minority University Research and Education Program (MUREP) in which Scott Hansen, Principal Investigator, was paired with students from the University of Houston. The experiment consisted of an individual water droplet study and three copper coupon studies. The copper coupons used in this experiment were constructed in a similar manner as the coupons reported in Section III but utilized Lexan sides to visually record how water freezes in microgravity conditions. Each study utilized a Thermoelectric Cooler (TEC) to freeze each test article of about 20g of water. The test articles were allowed to freeze continually throughout the various gravity levels in the aircraft (1g, 0g, and 1.5g). The water droplet study yielded inconclusive results while the copper coupon studies yielded exciting results when frozen in microgravity. In favorable, bottom-up, 1g freezing, as the freezing process occurs dissolved gasses escape from the ice/water interface and float to the surface. However, it was observed that during 0g, gas bubbles escaping from the ice/water interface did not float to the surface. Rather, the gas bubbles floated in place due to neutral buoyancy, with some gas bubbles forming on the surface of the ice/water front (Figure 17). Once the 0g parabola was completed, the bubbles floated to the surface of the water. If continued to freeze while in microgravity, it is hypothesized that the gas bubbles forming on the surface of the freeze front would either become trapped in the ice formation or be pushed, by the freeze front, to the surface forming what are known as “ice worms”. Either process...
would lead to less dense ice and visually cloudier ice than ice formed in a 1-g environment. It is also hypothesized that because air is trapped in the ice during zero-g, a greater volume of ice will be formed, as was seen in unfavorable freezing of the mezzo technologies HX. It is uncertain how much more ice will be formed.

V. Future Water-Based PCM HX Designs

After ice spike formation and freeze front propagation was notionally understood through copper coupon testing, the next step was to develop a coupon test that mimicked the Mezzo Technology HX. This coupon mimicked the microtubes of the Mezzo Technologies HX and also utilized a flexible membrane. The purpose of this test article was to evaluate the membrane manufacturing techniques and membrane interface with the HX core and to understand freezing within the membrane HX. Additionally various cores for the HX were to developed to determine the ideal freezing mode. The test article was tested in several configurations which all resulted in successful testing.

References