ASSESSMENT OF MOLTEN-SALT SOLAR CENTRAL-RECEIVER FREEZE-UP AND RECOVERY EVENTS

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ABSTRACT

Molten salt used as a heat transfer fluid in central-receiver solar power plants has a high freezing point (430°F (221°C)). It is very likely during the life of the plant that the receiver will accidentally freeze up due to equipment malfunction or operator error.

Experiments were conducted to measure the effects of a molten salt receiver freeze-up and recovery event and methods to thaw the receiver. In addition, simulated freezethaw experiments were conducted to determine what happens when salt freezes and is thawed in receiver tubes and to quantify the damage caused to candidate receiver tube materials. Fourteen tube samples of various materials, diameters and wall thicknesses were tested to destruction. Results of these tests are presented in this paper.

BACKGROUND

In a molten salt solar central receiver, large tracking mirrors focus sunlight onto a receiver on top of a centrally located tower. Molten salt at 288–550°F (288°C) is pumped from a storage tank to the receiver where it is heated to 1050°F (565°C) in the receiver by the absorbed solar energy. The “hot” salt is stored in a tank where it can be withdrawn and pumped through a steam generator to produce steam to power a turbine and make electrical power [Chavez, Kolb, and Tyner, 1995]. Molten salt was chosen as the heat transfer fluid because of its high heat capacitance, low cost, and chemical stability at the upper operating temperature. Its disadvantage is its high freezing point (approximately, 430°F (221°C) [Cook and McMordie, 1989].

In the receiver there are multiple drain valves which allow the salt to drain back the storage tanks. During the nightly shut down of the receiver, there exists the possibility that a drain valve will fail to actuate. Based on a valve failure rates, a valve could fail to actuate more than once in a thousand times [Brookhaven National Laboratory], a receiver - which has 14 drain valves (such as the Solar Two 10MW, solar central receiver located in Daggett, CA [Chavez, Kolb, and Tyner, 1995]) - will fail to drain approximately once every two and a half months. That does not mean a panel will freeze that often. Only if this failure is not detected in time and corrective action (such as manually opening the valve) is not taken will the salt trapped in the associated panels freeze. Since the volume of salt increases when it goes from the solid to the liquid state (approximately 4.6%), damage can occur to the receiver tubing if the salt is thawed in a section of tubing or piping which is constrained at both ends.

Considering the multiple drain valves in the receiver, the likelihood that a panel could fail to drain during nightly shut down is high over the life of the receiver. In addition, a panel could freeze up during morning startup if areas of the receiver are below the salt freezing temperature. In previous receiver experiments, detailed assessments of freezing and thawing of the panel tubes had not been conducted. The Martin Marietta molten salt receiver, tested at Sandia National Laboratories, became frozen with salt during morning start up and was successfully thawed, though no data on tube deformation was available [Tracey, 1995]. In addition, during a test to determine the potential of cold filling a molten salt receiver in the external configuration, the receiver was partially frozen [Bergan, 1986].

In this paper we describe freeze/thaw experiments conducted with a receiver panel where we intentionally let salt freeze in the receiver panels. The objectives of these tests were to understand the freezing phenomenon under non-flowing (stagnant) conditions and quantify the deformation that takes place in an actual receiver panel. We also wanted to determine the procedure required to thaw a receiver panel if it became frozen with salt and to determine what amount of damage was done to the receiver tubes during the thawing process.

In addition to the panel freeze/thaw tests, we assessed the damage induced in various potential receiver tube materials under simulated freeze/thaw conditions in an oven. We conducted four series of these tests to understand the freezing/thawing process. We froze and melted salt sequentially in tube samples using a two-chamber oven to determine if the tubing can be thawed without damage. A fifth series of tests was conducted to quantify the damage that occurred to 14 samples of tubing (5 materials, 3 diameters and 3 wall thicknesses - not every combination was tried) under the most severe freeze/thaw
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PANEL EXPERIMENTS

The two receiver panels each consisted of two flow passes connected in series. Each pass has six 1-inch (2.54 cm) diameter tubes connected to a common 6-inch (15.2 cm) diameter manifold. The tubes are made of 304 stainless steel. The height of the panel is 16 feet (4.9 m). Figure 1 shows a schematic and photograph of the panels. Prior to installation of the panels, all the tubes' outside diameters were measured at various locations along its length so we could determine the permanent strain induced during the freeze/thaw tests. The freeze/thaw test procedures we used are described here.

First, we established flow in all tubes of the panels to allow the panels to reach thermal equilibrium. After flowing salt through the panels for several minutes, we shut off all drain and outlet valves to prevent salt from draining out of the panels. Then we allowed the panels to cool so their temperatures dropped below the salt freezing point, but kept the header heat trace on. Figure 2 shows panel and header temperatures as they cooled. Note how the slopes of the curves changed at the salt freezing point. When the salt became solid, the slopes again changed. The header temperature was maintained above 460°F (238°C) by heat trace. The amount of time the panels took to cool to the salt's freezing point was only 25 minutes from the time the pump stopped. An exposed external central receiver will likely cool much faster.

After the average temperature of the panels was less than 280°F (138°C), we opened the drain and outlet valves to empty the lower header of salt. Once the headers had drained, we initiated thawing with heliostats. The only way for salt to leave the panels as it thaws is through the drain. Therefore, we started thawing from the bottom by putting on one heliostat and allowing it to heat that area of the panel to >500°F (260°C). We continued to add heliostats, one at a time above the previous one, raising the panel temperature. One test was interrupted by weather and had to be continued on the next sunny day.

Once all thermocouple readings on the panel were above the salt freezing point, we tried to establish flow through the panel. On the first attempt, we were could not flow though the majority of the panel because there were frozen salt plugs in the sections of tubes covered by insulation. In these areas, the heat trace could not conduct very much heat to it. Since it was under the insulation we could not heat it directly with solar. Once we achieved flow through part of a panel we continued to flow salt which helped thaw the frozen areas. After several hours we were able to clear all tubes.

After two freeze/thaw cycles we measured the tube diameters. Figures 3 and 4 show the plastic (permanent) strain as a function of the
Figure 4. Permanent strain as a function of the width (tube number) for the west panel. Each line represents data at a vertical height.

horizontal location (tube number). The permanent strain is the change in diameter divided by the original diameter. The maximum permanent strain induced in the tubes was over 4%. Tubes 3, 4, and 5 in the east panel have much lower permanent strains than the other tubes in the panel. The west panel does not show this behavior. Some observations and conclusions regarding these tests are:

1) Thawing a frozen panel can require several hours and could result in significant down time.

2) The major problem with thawing the panel was a lack of sufficient heat, particularly where the header oven ended and the panel started under the insulation. We could not get enough heat into that region with the oven heaters or by conduction with the solar heated panels. It will most likely be necessary to install additional heat trace in the regions where the insulation meets the panel to ensure those areas can heat up above the salt melting point.

3) Although we had over 41 thermocouples on the 24 receiver tubes and 4 headers, we were unable to determine where the blockages were. Even when all the thermocouples indicated that the temperatures were above the salt melting point, we still had plugs of salt. In the Solar Two and commercial receivers there will be even less instrumentation. If a panel becomes frozen, temporary thermocouples should be installed to monitor the panel temperatures more thoroughly. Another option is to monitor the temperatures with an IR camera.

SIMULATED FREEZE/THAW EXPERIMENTS

A freeze/thaw oven was built from a section of 12 inch diameter schedule 10 steel pipe and external band heaters, covered externally with insulation. The oven was divided into two chambers by an insulated baffle that had holes drilled in it so tube samples could fit through. Each chamber had separate heaters and a controller to provide independent temperature control of that chamber. The lower chamber contained a blower to enhance cooling. The lid to the upper chamber was removable to speed the cooling in that chamber. Figure 5 shows photographs of the oven. Five-foot-long tube samples fit through baffle into the oven. Half of each tube was in the upper section (0 to 30 inches) and the other half in the lower (30 to 60 inches). Each tube had a welded plug at the bottom.

Test Series 1 to 4 - Understanding Freezing Phenomenon

Four series of tests were conducted to understand the freezing and thawing phenomenon. In each series, the sequence in which the upper and lower sections were frozen and thawed was different. These sequences are shown in Table 1. Two samples of tubes were tested for each of these four test series: one with a metal stop in the upper section to prevent the frozen salt from sliding and another without a stop.

In the first series, we investigated the freezing/thawing sequence thought to be most damaging. This sequence is where there is liquid salt in the upper portion of a tube when the lower portion freezes. Liquid salt can seep into the voids that form in the lower section as the salt contracts upon freezing. If the upper section freezes, then the lower section is thawed, the salt will expand but is restrained from sliding by the frozen salt in the upper portion. The stresses developed by the expanding liquid are higher than the yield strength so the metal deforms plastically. This is a process where the strain is somewhat controlled.

<table>
<thead>
<tr>
<th>Series</th>
<th>Freeze 1st</th>
<th>Freeze 2nd</th>
<th>Thaw 1st</th>
<th>Thaw 2nd</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Lower</td>
<td>Upper</td>
<td>Lower</td>
<td>Upper</td>
</tr>
<tr>
<td>2</td>
<td>Upper</td>
<td>Lower</td>
<td>Lower</td>
<td>Upper</td>
</tr>
<tr>
<td>3</td>
<td>Lower</td>
<td>Keep upper hot</td>
<td>Lower</td>
<td>Keep upper hot</td>
</tr>
<tr>
<td>4</td>
<td>Upper</td>
<td>Keep lower hot</td>
<td>Upper</td>
<td>Keep lower hot</td>
</tr>
</tbody>
</table>

The second series simulated a sequence that was thought to be less damaging than the first. If the salt is frozen in the upper section first, then the lower section freezes, the salt must contract in the lower section. Since the volume is restricted by the frozen salt in the upper section, voids will form. If the lower section is melted first, less damage is expected to occur because the liquid salt will fill the voids giving a place for the salt to go.
The third and fourth series simulated freezing in sections of tube near the header ovens. Keeping the lower section hot while freezing the upper section simulates the lower header. Likewise, freezing the lower section while keeping the upper section hot simulates the upper header.

For these experiments, 316 SS tubing (1.0 inch OD x 0.049 inch wall) was chosen as a representative material. Two tubes for each test series were used, one with a metal stop in the upper section and one without at stop. The tube diameters were measured every six inches along the tube before and after each cycle. Also salt was added to maintain the salt level.

Twelve freeze/thaw cycles were conducted in each series. The most damaging freeze/thaw sequence was the first series where the salt was frozen in the lower section, then the upper. See Table 2. The tube with the metal stop ruptured in the lower section after twelve cycles. Figure 6 shows a photograph of a ruptured tube sample. The permanent strain in this tube increased nearly linearly with the number of cycles. The tube without the stop experience only minor yielding indicating the frozen salt in the upper section probably slid, relieving the pressure built up in the lower section.

Table 2. Maximum Strain in Lower and Upper Sections for Test Series 1 to 4.

<table>
<thead>
<tr>
<th>Test #</th>
<th>Cycles tested</th>
<th>Max. ( \varepsilon_p ), %</th>
<th>Max. ( \varepsilon_f ), %</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>12</td>
<td>20.2(^{\circ}), 0.6(^{\circ})</td>
<td>3.9(^{\circ}), 0(^{\circ})</td>
<td>Rupture of tube with stop after 12 cycles</td>
</tr>
<tr>
<td>2</td>
<td>12</td>
<td>5.6(^{\circ}), 0(^{\circ})</td>
<td>0(^{\circ}), 0(^{\circ})</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>12</td>
<td>0(^{\circ}), 0(^{\circ})</td>
<td>0(^{\circ}), 0(^{\circ})</td>
<td>No damage.</td>
</tr>
<tr>
<td>4</td>
<td>12</td>
<td>0(^{\circ}), 0(^{\circ})</td>
<td>0(^{\circ}), 0(^{\circ})</td>
<td>No damage.</td>
</tr>
</tbody>
</table>

1 Tube with stop. 2 Tube without stop.

In the second series there was much less damage. Only the tube with the stop had permanent strain. The tube ratcheted in a linear fashion similar to the strain in the first series but at a lower strain per cycle. The lower strain per cycle is consistent with the idea that there were voids formed during the freezing process which accommodated the expanding molten salt. In the third and fourth series there was no measurable permanent strain indicating that if there is a free salt boundary, the salt could easily expand when it undergoes phase change.

The conclusion from these four series of tests is that if the frozen salt cannot freely slide in a tube when the salt melts, then damage to the tube will result. The freeze/thaw sequence is also critical in determining the extent of damage induced in a tube. A restrained frozen plug which has liquid salt above it that eventually freezes will experience more damage when thawed than a plug formed by liquid salt restrained by two plugs of salt at each end.

Test Series 5 - Quantifying the Damage in Various Materials

Considering results from the above tests, we selected the freeze/thaw sequence which caused the most damage (freeze lower section, then upper, thaw lower, then upper) and continuously cycled the samples to rupture. Several five-foot-long receiver-tube samples were repeatedly cycled to determine the strain per cycle and how many freeze/thaw cycles the materials could handle. The materials, tube diameters, and wall thicknesses tested in this series are listed in Table 3. In these tests, two measurements were made in the lower section (36 and 48 inches from the top of the tube) and one in the upper (18 inches from the top). In addition the tube length was measured.

Table 3. Materials tested to rupture in fifth series of freeze/thaw test.

<table>
<thead>
<tr>
<th>Material</th>
<th>Diameter, inches</th>
<th>Wall Thickness, inches</th>
</tr>
</thead>
<tbody>
<tr>
<td>304H SS</td>
<td>0.75</td>
<td>0.049</td>
</tr>
<tr>
<td>316H SS</td>
<td>0.75</td>
<td>0.049</td>
</tr>
<tr>
<td>304H SS</td>
<td>0.75</td>
<td>0.065</td>
</tr>
<tr>
<td>316L SS</td>
<td>0.75</td>
<td>0.065</td>
</tr>
<tr>
<td>316H SS</td>
<td>0.75</td>
<td>0.065</td>
</tr>
<tr>
<td>Alloy 800H</td>
<td>0.75</td>
<td>0.065</td>
</tr>
<tr>
<td>304H SS</td>
<td>1.0</td>
<td>0.049</td>
</tr>
<tr>
<td>316L SS</td>
<td>1.0</td>
<td>0.049</td>
</tr>
<tr>
<td>304H SS</td>
<td>1.5</td>
<td>0.049</td>
</tr>
<tr>
<td>316H SS</td>
<td>1.5</td>
<td>0.049</td>
</tr>
<tr>
<td>304H SS</td>
<td>1.5</td>
<td>0.065</td>
</tr>
<tr>
<td>316H SS</td>
<td>1.5</td>
<td>0.065</td>
</tr>
<tr>
<td>Alloy 800H</td>
<td>1.5</td>
<td>0.065</td>
</tr>
</tbody>
</table>

Measured Strain per Cycle

Figures 7 and 8 show typical results of the permanent strain (in the lower section of the tube) as a function of the number of freeze/thaw cycles for four tubes of 316H SS and five samples of 304H SS. In every case the permanent strain increased linearly with cycle, except for the 0.75 in OD x 0.065 in wall (0.019 m OD x 1.7 mm wall) between 13 and 17 cycles. There was no evidence of significant strain hardening since the slope of each line was nearly constant throughout the test, except in the previously mentioned sample. In this sample the strain did not change for the last four cycles before rupture. Also, there was no significant change in the length of any of the samples.

Correlation of Tube Properties to Strain per Cycle

Since the thawing process is a strain controlled phenomenon, the permanent strain induced in each cycle is the difference between the total strain and the elastic strain of the tube:

\[
\varepsilon_{\text{perm}} = \varepsilon_{\text{total}} - \varepsilon_{\text{elastic}}
\]
The elastic strain is a function of the material properties. If one considers that the work done by the salt to the tube material per cycle is constant for a given diameter, some of the work is reversible and the rest irreversible.

\[
W_{\text{salt}} = W_{\text{tube, rev}} + W_{\text{tube, irrev}}
\]  

The reversible work corresponds to the work done in the elastic range of the material and the irreversible to the plastic range:

\[
W_{\text{tube, rev}} = \int_{e_{\text{elastic}}}^{e_{\text{yield}}} \sigma_{\text{elastic}} A_{\text{tube wall}} D_{\text{tube}} de
\]

\[
W_{\text{tube, irrev}} = \int_{e_{\text{yield}}}^{e_{\text{plastic}}} \sigma_{\text{plastic}} A_{\text{tube wall}} D_{\text{tube}} de
\]

where \(A_{\text{tube wall}}\) is the cross sectional area of the tube wall and \(D_{\text{tube}}\) is the tube mean diameter. In the elastic range, the stress-strain relation for a thick-walled cylinder is [Young, 1989]:

\[
\sigma_{\text{elastic}} \equiv \frac{E \varepsilon_{\text{elastic}} (\frac{D_{\text{tube}}}{D_{\text{wall}}} + 1)}{2(1-v)}
\]

A rough estimate of the strain in the plastic range can be approximated as a constant equal to the tensile strength, \(\sigma_{\text{tensile}}\). Carrying out the integration the permanent strain per cycle is:

\[
\varepsilon_{\text{perm}} = \frac{W_{\text{salt}}}{4 \sigma_{\text{tensile}} L_{\text{wall}} r_0} - \frac{\sigma_{\text{yield}} \varepsilon_{\text{elastic}}}{2 \sigma_{\text{tensile}}}
\]

Assuming \(W_{\text{salt}}\) scales with the diameter, Eq. (6) gives us a way to extrapolate the results from one sample size to another. Table 4 shows a comparison between the measured strain per cycle and the predicted strain per cycle for 304H SS based on the measured values for the 0.75 inch diameter by 0.049 inch wall sample and for constant material properties. The values were based on a 240 MPa (35ksi) yield strength (at 0.2% strain) and a tensile strength of 560 MPa (81 ksi) as referenced from the data sheet provided by the supplier of the tubing. The material was cold worked.

For the 0.75 and 1.0 inch diameter tubes the equation gives a good estimate of the strain per cycle. Variations in tube material samples may result in the greater variation for the larger diameter tube samples.

Table 4. Measure and Predicted Strain per Cycle for Samples of 304H SS.

<table>
<thead>
<tr>
<th>Outside Diam., in.</th>
<th>Wall, in.</th>
<th>Measured % Strain/Cycle</th>
<th>Predicted % Strain/Cycle</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.75</td>
<td>0.049</td>
<td>1.54</td>
<td>-- (basis for rest)</td>
</tr>
<tr>
<td>0.75</td>
<td>0.065</td>
<td>1.10</td>
<td>1.09</td>
</tr>
<tr>
<td>1.0</td>
<td>0.049</td>
<td>1.60</td>
<td>1.60</td>
</tr>
<tr>
<td>1.5</td>
<td>0.049</td>
<td>1.76</td>
<td>1.66</td>
</tr>
<tr>
<td>1.5</td>
<td>0.065</td>
<td>1.79</td>
<td>1.21</td>
</tr>
</tbody>
</table>

Cycles to Failure and Permanent Strain at Failure

Figure 9 shows the number of cycles to failure for each sample. All the 0.049 inch walled steel samples failed at 11 to 13 cycles without any significant effect of sample diameter. The 0.065 inch walled steel samples failed at 10 to 16 cycles with a slight trend of enduring more cycles at the smaller diameter than the larger diameter. For the Alloy 800H, the samples both failed at 16 cycles. The high yield and tensile strength are the reasons for the greater number of cycles the Alloy 800H can handle.

One would expect all the tubes of a same material to fail at the same level of strain. In general, though for the stainless steel samples, the thinner walled tubes tended to rupture at slightly higher permanent strain levels. See Figure 10. The permanent strain at failure ranged between 13.1 and 19.4% for the steel samples and 19.9 and 25.6 for
the Alloy 800H samples. Generally, the highest strains at failure were measured at the location of failure (90° from the tear). The strain farthest away from the failure point (but in the lower section where the yielding took place) exhibited the least amount of permanent strain.

Implications on Receiver Fatigue Life

We believe the fraction of the life of the receiver used up in a single receiver panel freeze up could be at least \( \frac{1}{N_f} \), where \( N_f \) is the number of cycles to failure for that strain range based on the cumulative damage from the linear damage rule [Smith, 1992]. It is likely that the effect will be greater than this since the strain is so high that there could be non-linear effects. In any case the design of a receiver should take into account at least one freeze/thaw cycle in the life of the receiver assuming corrective action is not taken once every 150 times a valve fails to actuate.

CONCLUSIONS

These tests helped understand the freezing phenomenon and quantify the damage to material samples. The method by which a receiver freezes and thaws will dictate how much damage the material will experience. The most effective method to thaw a receiver panel will be one where the panel is thawed from a known free salt boundary into which the salt can easily expand (e.g., thawed from the bottom of the panel starting at the lower header after the lower header has been completely thawed and drained of salt and its temperature is above 550°F.) This will ensure there are no frozen salt plugs restraining the expansion of the salt as it melts. If the salt is restrained upon thawing, the receiver tubes could be strained beyond the elastic limit causing significant permanent damage and shortening the life of the receiver.

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Tracey, 1995, personal communications.

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