UNION CARBIDE CORPORATION
LINDE DIVISION

QUARTERLY PROGRESS REPORT OF RESEARCH AND DEVELOPMENT
IN A THERMAL INSULATION STUDY

September - November, 1968

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September - November, 1968

Prepared By:
W. E. Grunert

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# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>ABSTRACT</strong></td>
<td><strong>Page</strong></td>
</tr>
<tr>
<td>1.0 SUMMARY</td>
<td>1</td>
</tr>
<tr>
<td>1.1 Thermal Conductivity Testing - Cylindrical Tests</td>
<td>1</td>
</tr>
<tr>
<td>1.2 Thermal Conductivity Testing - Flat Plate Tests</td>
<td>1</td>
</tr>
<tr>
<td>1.3 Longitudinal Thermal Conductivity Testing</td>
<td>2</td>
</tr>
<tr>
<td>1.4 Insulation Interfaces - Exposed Edge Effects</td>
<td>2</td>
</tr>
<tr>
<td>1.5 Vacuum Enclosure - Hastelloy-X Specimen Analysis</td>
<td>3</td>
</tr>
</tbody>
</table>

## 2.0 TECHNICAL REPORT

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1 Thermal Conductivity Testing - Cylindrical Tests</td>
<td>4</td>
</tr>
<tr>
<td>2.1.1 Summary</td>
<td>4</td>
</tr>
<tr>
<td>2.1.2 Current Long-term Performance Testing</td>
<td>5</td>
</tr>
<tr>
<td>2.1.3 Nickel Multi-foil Systems - 1800°F Behavior</td>
<td>5</td>
</tr>
<tr>
<td>2.2 Thermal Conductivity Testing - Flat Plate Tests</td>
<td>16</td>
</tr>
<tr>
<td>2.2.1 Nickel Multi-foil Insulations</td>
<td>16</td>
</tr>
<tr>
<td>2.2.2 Nickel Opacified Quartz Multi-layer Insulation</td>
<td>24</td>
</tr>
<tr>
<td>2.3 Longitudinal Thermal Conductivity Testing</td>
<td>32</td>
</tr>
<tr>
<td>2.3.1 Test Apparatus and Procedure</td>
<td>32</td>
</tr>
<tr>
<td>2.3.2 Results</td>
<td>34</td>
</tr>
<tr>
<td>2.4 Insulation Interfaces - Exposed Edge Effects</td>
<td>36</td>
</tr>
<tr>
<td>2.4.1 Analytical and Experimental Approaches</td>
<td>36</td>
</tr>
<tr>
<td>2.4.2 Exposed Edge Heat Loss Results</td>
<td>39</td>
</tr>
<tr>
<td>2.5 Vacuum Enclosure - Hastelloy-X Specimen Analysis</td>
<td>42</td>
</tr>
<tr>
<td>2.5.1 Sample Description - Test Procedure</td>
<td>42</td>
</tr>
<tr>
<td>2.5.2 Results - Interim and Final Evaluations</td>
<td>43</td>
</tr>
<tr>
<td>2.5.2.1 Vacuum Integrity and Materials Stability</td>
<td>43</td>
</tr>
<tr>
<td>2.5.2.2 Mechanical Properties</td>
<td>50</td>
</tr>
<tr>
<td>2.5.2.3 Hastelloy-X Photomicrographs</td>
<td>56</td>
</tr>
</tbody>
</table>

### 3.0 ACTIVITIES DURING NEXT REPORTING PERIOD | 60

**REFERENCES** | 61
ILLUSTRATIONS

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1.1</td>
<td>Thermal Conductivity Versus Time - Long-Term Tests</td>
<td>6</td>
</tr>
<tr>
<td>2.1.2</td>
<td>Cylindrical Test Results - Nickel-Nickel Opacified Quartz</td>
<td>7</td>
</tr>
<tr>
<td>2.1.3</td>
<td>Nickel Foil Samples - 1800°F Composite</td>
<td>9</td>
</tr>
<tr>
<td>2.1.4</td>
<td>Nickel 1800°F Compatibility Samples</td>
<td>12</td>
</tr>
<tr>
<td>2.1.5</td>
<td>Molybdenum 1800°F Compatibility Samples</td>
<td>14</td>
</tr>
<tr>
<td>2.2.1</td>
<td>Apparent Thermal Conductivity -- Nickel Refrasil Quartz Multi-layer Insulation</td>
<td>17</td>
</tr>
<tr>
<td>2.2.2</td>
<td>Apparent Thermal Conductivity - Nickel-Nickel Opacified Quartz Multi-layer Insulation</td>
<td>18</td>
</tr>
<tr>
<td>2.2.3</td>
<td>Apparent Thermal Conductivity - Nickel-Astroquartz Multi-layer Insulation</td>
<td>19</td>
</tr>
<tr>
<td>2.2.4</td>
<td>Apparent Emissivity Relationship - Nickel-Nickel Opacified Quartz Multi-layer Insulation</td>
<td>23</td>
</tr>
<tr>
<td>2.2.5</td>
<td>Backscattering Cross-section - Nickel Opacified Quartz Multi-layer Insulation</td>
<td>27</td>
</tr>
<tr>
<td>2.2.6</td>
<td>Solid Conductivity Coefficient - Nickel Opacified Quartz Multi-layer Insulation</td>
<td>28</td>
</tr>
<tr>
<td>2.2.7</td>
<td>Apparent Thermal Conductivity as a Function of Thickness</td>
<td>29</td>
</tr>
<tr>
<td>2.2.8</td>
<td>Scattering Cross-section for Fibrous Papers at 900°F Temperature</td>
<td>31</td>
</tr>
<tr>
<td>2.3.1</td>
<td>Longitudinal Thermal Conductivity Test Configuration</td>
<td>33</td>
</tr>
<tr>
<td>2.3.2</td>
<td>Longitudinal Thermal Conductivity - Aluminum Opacified Glass Multi-layer Insulation</td>
<td>35</td>
</tr>
<tr>
<td>2.4.1</td>
<td>Exposed Edge Heat Loss - Analytical Model</td>
<td>37</td>
</tr>
<tr>
<td>2.4.2</td>
<td>Exposed Edge Test Schematic</td>
<td>38</td>
</tr>
</tbody>
</table>
ILLUSTRATIONS (cont'd.)

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.5.1</td>
<td>Vacuum Enclosure Material Tester</td>
<td>44</td>
</tr>
<tr>
<td>2.5.2</td>
<td>Hastelloy-X Vacuum Enclosure Specimen After 10,000-hour, 1400°F Exposure</td>
<td>46</td>
</tr>
<tr>
<td>2.5.3</td>
<td>Surface Photographs - Hastelloy-X Base Metal</td>
<td>48</td>
</tr>
<tr>
<td>2.5.4</td>
<td>Surface Photographs - Hastelloy-X Weld Areas</td>
<td>49</td>
</tr>
<tr>
<td>2.5.5</td>
<td>Average One Percent Total Elongation, Hastelloy-X Sheet</td>
<td>51</td>
</tr>
<tr>
<td>2.5.6</td>
<td>Room Temperature Strength of Aged Hastelloy-X</td>
<td>54</td>
</tr>
<tr>
<td>2.5.7</td>
<td>Room Temperature Tensile Elongation of Aged Hastelloy-X</td>
<td>55</td>
</tr>
<tr>
<td>2.5.8</td>
<td>Photomicrographs of Hastelloy-X Base Metal Cross-section</td>
<td>57</td>
</tr>
<tr>
<td>2.5.9</td>
<td>Photomicrographs of Hastelloy-X Weld Area Cross-section</td>
<td>58</td>
</tr>
<tr>
<td>Number</td>
<td>Table Title</td>
<td>Page</td>
</tr>
<tr>
<td>--------</td>
<td>-----------------------------------------------------------------------------</td>
<td>------</td>
</tr>
<tr>
<td>2.2.1</td>
<td>Empirical Thermal Conductivity Parameters for Nickel Multi-foil Insulations</td>
<td>22</td>
</tr>
<tr>
<td>2.2.2</td>
<td>Flat Plate Data on Nickel Opacified Quartz Paper Insulation</td>
<td>25</td>
</tr>
<tr>
<td>2.4.1</td>
<td>Exposed Edge Sample Characteristics</td>
<td>39</td>
</tr>
<tr>
<td>2.4.2</td>
<td>Exposed Edge Heat Loss Results</td>
<td>41</td>
</tr>
<tr>
<td>2.5.1</td>
<td>Hastelloy-X Enclosure - Vacuum Integrity</td>
<td>45</td>
</tr>
<tr>
<td>2.5.2</td>
<td>Room Temperature Strength of Hastelloy-X Enclosure Material</td>
<td>53</td>
</tr>
</tbody>
</table>
Results of long-term thermal conductivity tests on copper opacified and nickel opacified multi-layer insulations are up-dated. The 1800°F nickel and Refrasil quartz instability problems are discussed in detail. Molybdenum and woven quartz cloth are recommended as alternate materials at this temperature.

Apparent thermal conductivity data (flat plate tests) are presented for three nickel multi-foil insulations and the nickel opacified fibrous multi-layer system. Thermal performance is correlated to temperature and compressive load (layer density). The resultant empirical thermal conductivity expressions are presented and applied.

Longitudinal thermal conductivity data for aluminum opacified glass multi-layer insulation are given. Experimental technique and sample design are described.

Results of the exposed edge effects analytical and experimental program are presented and discussed. Two multi-foil systems --- aluminum-106 glass and nickel-Refrasil --- were evaluated.

The vacuum enclosure program has been completed. The Hastelloy-X specimen was analyzed in as-received condition and after 1400°F, 10,000-hour exposure. Pertinent results on vacuum integrity and material strength are given. Magnified surface photographs and photomicrographs are presented and discussed.
1.0 SUMMARY

1.1 Thermal Conductivity Testing - Cylindrical Tests

Long-term tests continue on the copper opacified and nickel opacified quartz multi-layer insulations, with 9,000 hours and 1,500 hours test times, respectively. Apparent thermal conductivities, presented in Figure 2.1.1, remain stable.

Analysis of the 1800°F instability and performance deterioration problems with nickel foil and Refrasil quartz paper has indicated the following:

(1.) Thermal etching and recrystallization of the nickel foil surface at 1800°F in vacuum result in an increased foil emissivity (higher radiation heat transfer).

(2.) Phase change in Refrasil quartz from amorphous to crystalline, along with embrittlement and increased fiber-to-fiber contact area, at 1800°F yield an increased spacer solid conductance.

Replacing the above foil-spacer materials with molybdenum and woven quartz (Astroquartz) appears to minimize these instabilities. A modified composite insulation system, using the new materials in the 1800°F zone, will be tested.

1.2 Thermal Conductivity Testing - Flat Plate Tests

Data are presented on three nickel multi-foil insulations and a nickel opacified fibrous multi-layer system. The data indicate insulation performance as a function of temperature and layer density over specified operating ranges.

Apparent thermal conductivity of the multi-foil systems are correlated by an expression of the form:

\[ K_a = K_s + K_r = aT^{1/2} + bT^3 \]

The empirically-derived coefficients satisfying this expression are found in Table 2.2.1. For the nickel foil systems, an "apparent" emissivity is expressed in terms of temperature and layer density. Previously described (2) methods for correlating the data are applied. Regenerated apparent thermal conductivities, calculated from the derived expressions, generally agree with measured values to within 10-20%.

Apparent thermal conductivity expression for the nickel opacified fibrous material includes the backscattering cross-section as well as insulation thickness and boundary effects. Figure 2.2.7 illustrates the influence of all these parameters, particularly when dealing with small insulation thicknesses.
Ranges of performance achieved by the nickel multi-foil and nickel opacified insulations are tabulated for the limiting conditions (low temperature, low density and high temperature, high density).

<table>
<thead>
<tr>
<th>Insulation</th>
<th>$T_h$ °F</th>
<th>$T_c$ °F</th>
<th>Layer Density l/inch</th>
<th>Apparent Thermal Conductivity $10^{-3}$ Btu/hr-ft-°F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni-Refrasil</td>
<td>550</td>
<td>120</td>
<td>85</td>
<td>0.14</td>
</tr>
<tr>
<td></td>
<td>1800</td>
<td>380</td>
<td>175</td>
<td>2.60</td>
</tr>
<tr>
<td>Ni-Ni Opacified</td>
<td>500</td>
<td>120</td>
<td>56</td>
<td>0.29</td>
</tr>
<tr>
<td>Refrasil</td>
<td>1500</td>
<td>350</td>
<td>110</td>
<td>2.60</td>
</tr>
<tr>
<td>Ni-Astroquartz</td>
<td>550</td>
<td>110</td>
<td>108</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>1540</td>
<td>250</td>
<td>152</td>
<td>5.60</td>
</tr>
<tr>
<td>Ni Opacified</td>
<td>500</td>
<td>150</td>
<td>53</td>
<td>1.40</td>
</tr>
<tr>
<td>Refrasil</td>
<td>1500</td>
<td>356</td>
<td>108</td>
<td>4.90</td>
</tr>
</tbody>
</table>

### 1.3 Longitudinal Thermal Conductivity Testing

The longitudinal or lateral apparent thermal conductivities of opacified fibrous multi-layer insulations are determined experimentally over the full operating temperature range specified for each material. A flat plate test configuration is used with a cylindrical sample 6" diameter x 1-1/2" high. One-dimensional heat flow laterally across the 1-1/2" thickness is metered over a 2" x 2" center "core" section.

Results are presented for aluminum opacified glass insulation over the range 250-900°F. The longitudinal conductivity ($K_L$) was ~ 15 times higher than corresponding $K$-values in the normal direction. It is felt that this increase is due largely to high solid conduction in the longitudinal direction.

### 1.4 Insulation Interfaces - Exposed Edge Effects

The heat losses from exposed edges radiating to an ambient environment were evaluated both analytically and experimentally for two multi-foil insulations --- aluminum-106 glass to 900°F and nickel-Refrasil to 1700°F. A flat, planar sample was considered with two-dimensional heat flow (normally across insulation thickness and laterally to the exposed edge). The sample is shown schematically in Figures 2.4.1 and 2.4.2.

Analytical and experimental exposed edge heat loss data are summarized in Table 2.4.2. Presence of the exposed edge caused a 4-7 times increase in heat flux for the specified sample panels. Agreement between analytical and experimental results was generally within 10-30%, with the analytical data being consistently conservative (higher).
1.5 Vacuum Enclosure - Hastelloy-X Specimen Analysis

The 10,000-hour, 1400°F exposure testing of the Hastelloy-X vacuum enclosure specimen has been completed. The cylindrical sample had dimensions of 3-1/4" O.D. x 12" long, and was fabricated from .012" thick sheet. The exterior surface was exposed to vacuum environment and the interior to pressurized air.

The Hastelloy-X enclosure, in general, withstood the 1400°F, 10,000-hour exposure quite well. Specific observations from the post-test analyses are as follows:

(1.) Vacuum integrity was maintained throughout, with measured leak rates less than 1 x 10^{-8} STD cc air/second (reference Table 2.5.1).

(2.) Total specimen creep was < 1% in the circumferential direction corresponding to an applied hoop stress of 5000 psi.

(3.) Tensile and yield strengths are given in Table 2.5.2. Measured values for Hastelloy-X base metal are in agreement with published data. Strength of weld areas had deteriorated significantly due to porosity in the "heat affected zone" of the weld.

(4.) Photomicrographs indicated minimal surface penetration on both vacuum side and air side, with the exception of the "heat affected zone" mentioned above (reference Figures 2.5.8 and 2.5.9).
2.1 Thermal Conductivity Testing - Cylindrical Tests

2.1.1 Summary

During the reporting period, three new long-term stability tests were initiated on nickel foil and nickel opacified multi-layer systems. The spirally wrapped, cylindrical test technique was used in these extended evaluations. The test samples, including the copper opacified multi-layer system which continues on test, are described briefly.

<table>
<thead>
<tr>
<th>Insulation System</th>
<th>Layer Density</th>
<th>Hot-side Test Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Copper opacified quartz</td>
<td>37</td>
<td>1220</td>
</tr>
<tr>
<td>B. Nickel opacified quartz</td>
<td>65</td>
<td>1485</td>
</tr>
<tr>
<td>C. Nickel-nickel opacified quartz</td>
<td>56</td>
<td>1800</td>
</tr>
<tr>
<td>D. Composite</td>
<td>96</td>
<td>1800</td>
</tr>
<tr>
<td>Nickel-Refrasil quartz</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Copper-Refrasil quartz</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Aluminum-Aluminum opacified glass</td>
<td></td>
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</tbody>
</table>

Detailed description of the insulation systems and basic thermal conductivity data were previously reported (2).

The primary objectives of the test program are:

(1.) Obtain a first indication of long-term stability of the insulation systems at their maximum specified operating temperatures. Test duration is from 8000-10,000 hours.

(2.) Obtain preliminary assessment of effects on thermal conductivity in vacuum due to elevated temperature exposure of multi-foil insulations to air.

Current status of the long-term test program is as follows:

(a.) Copper and nickel opacified multi-layer insulation

Long-term testing continues on the copper (9000 hours) and nickel (1500 hours) systems with no evidence of instability.

(b.) Nickel - nickel opacified and composite multi-foil insulations

Instability and permanent performance deterioration were encountered in the 1800°F hot-side temperature range. The deterioration (40% or
greater) was attributed to (1.) emissivity increase (i.e. higher radiation heat transfer) due to thermal etching and recrystallization occurring on the nickel foil surface at 1800°F, and (2.) higher solid conduction due to quartz recrystallization and "softening" of the fibrous paper structure at 1800°F.

Subsequent investigation has led to the recommendation of alternate shield and spacer materials for 1800°F operation. These materials, which exhibit improved stability at 1800°F, are molybdenum foil and woven quartz (Astroquartz). They will be used as part of a modified composite insulation system, to be tested to 1800°F.

2.1.2 Current Long-term Performance Testing

The two fibrous quartz multi-layer systems -- copper opacified and nickel opacified paper -- remain on test. System performance is measured at regular intervals; apparent thermal conductivity has remained stable over the testing duration. Data on multi-layer insulation systems in long-term operation are summarized in Figure 2.1.1.

2.1.3 Nickel Multi-foil Systems - 1800°F Behavior

2.1.3.1 Definition of the 1800°F Material Problem

Long-term tests on the nickel-nickel opacified quartz and composite multi-foil systems were interrupted due to material instability problems at 1800°F temperature. This instability was discovered during measurement of apparent thermal conductivities (K_a) for the two systems at hot-side temperatures of ~1800°F. Erratic behavior of the samples and inability to achieve thermal equilibrium at the 1800°F level indicated a deteriorating K_a. As a check, thermal conductivity at a lower, previously measured level was re-determined and compared to the original K_a-value. Results for the nickel-nickel opacified quartz multi-foil system are illustrated in Figure 2.1.2. The 38% increase in this system's performance is attributed to deterioration of the nickel and/or quartz during 1800°F exposure.

The composite system was dismantled and close inspection of the 1800°F materials showed a visible dullness to the nickel shields and an embrittlement of the Refrasil quartz spacer. These apparent changes in the materials pointed to (1) loss of fibrous characteristics in the Refrasil (increased solid conduction) or (2) higher emissivity of the nickel foil (increased radiation) as contributors to the poor performance.

Several samples of nickel foil and Refrasil quartz from the composite insulation system were analyzed. Observations are summarized below:
THERMAL CONDUCTIVITY VS. TEST TIME

- Copper Opacified, $K_a = 122^\circ F$
- Aluminum Opacified, $K_a = 890^\circ F$
- Aluminum Foil-Aluminum Opacified, $K_a = 887^\circ F$
- Nickel Foil-Refrasil, $K_a = 1625^\circ F$
- Nickel Opacified, $K_a = 1485^\circ F$

Bands indicate estimated test point error due to:
1. Calibration and readability on voltmeter, ammeter, and temperature recorder
2. Ambient temperature fluctuations

FIGURE 2.1.1
Figure 2.1.2

NI - NI OPACIFIED CYLINDRICAL TEST RESULTS

APPARENT THERMAL CONDUCTIVITY ($K_a$) - $10^{-3} \text{ Btu/hr-ft-\textdegree F}$

HOT-SIDE TEMPERATURE - °F

INITIAL $K_a$ DATA

$K_a$ RECHECK AFTER 1800°F EXPOSURE

$T_h = 1208°F$

$T_c = 275°F$
(a.) The nickel foil underwent thermal etching* and recrystal-
lization* as a result of the 1800°F exposure. The surface effects are
severe at 1800°F, and quite mild at 1600°F.

(b.) Photomicrographs (Figure 2.1.3) comparing as-received and
1800°F exposed nickel foil show a high degree of surface roughness on the
latter samples as a result of (a). The as-received surface is "pinch-rolled"
to give high reflectivity.

(c.) No evidence of chemical interaction between the Refrasil quartz
spacer and nickel foil. Electron probe studies did not indicate silicide,
carbide or oxide formations.

(d.) At 1800°F, Refrasil quartz underwent a change of form from
amorphous to crystalline. The change was accompanied by severe embrittlement
and loss of fibrous characteristics. The condition was not observed at lower
temperatures.

As previously indicated, the thermal etching and recrystallization
bring about an increase in foil surface emissivity and, ultimately, a de-
terioration in multi-foil system performance. The emissivity increase may be
explained as follows:

(1.) Thermal etching, in general, creates a more diffusely reflecting
surface as illustrated by the highly irregular grain surfaces and grooved
boundaries of Figure 2.1.3. Optical characteristics are probably more closely
described by hemispherical emissivity ($\varepsilon_h$) than by the normal emissivity ($\varepsilon_n$).
The former is usually 20-30% higher than the latter.

(2.) Increase in high emissivity boundary area per unit area of
surface through thermal etching (widening and deepening of existing boundaries)
and recrystallization (formation and growth of new grains). Both phenomena
are illustrated in Figure 2.1.3.

*Thermal Etching* (1) -- Surface grooving at the grain boundaries where boundaries
meet the metal foil surface; brought about through re-equilibration of grain
boundary and surface free energies. Plastic deformation and surface vaporization
are thought to be the mechanisms by which thermal etching occurs. High tempera-
ture and a vacuum environment are sufficient conditions for occurrence. Nature of
the process suggests a finite time (possibly quite short) to re-stabilize at new
equilibrium state.

*Recrystallization* (1) -- Formation and growth of grain nuclei in high temperature
environment. Process brings about new grain orientation, proceeding from original
cold-worked state (typical condition of as-rolled foils). Grains formed from
the nuclei combine and enlarge at the expense of others (grain growth).
Thermal etching and recrystallization are illustrated in Figure 2.1.3.
FIGURE 2.1.3

NICKEL FOIL SAMPLES

1800° COMPOSITE TEST SYSTEM

AS-RECEIVED NICKEL

NICKEL - HOT-SIDE LAYER (1800°F)
REFRASIL CONTACT

ALL SAMPLES - 1000X

NICKEL - HOT-SIDE LAYER (1800°F)
REFRASIL CONTACT
During the photomicrograph work, a gross optical reflectivity comparison was made between "thermally etched" and as-received nickel foil. The reflectivity of the former was estimated to be a factor of 2 lower than the as-received material. Current effort is directed toward getting reflectivity comparisons for incident energy with wave length of 1-2 \mu, which is more typical of the wave lengths associated with high-temperature thermal emission.

Recrystallization at 1800°F was evident with both the plain Refrasil quartz* and nickel opacified quartz papers. The materials changed from the amorphous phase to the alpha-cristobalite phase. Although the latter has a somewhat higher parent thermal conductivity, the significant increase in spacer conduction is attributed to the destruction of the characteristic fibrous matrix; i.e. a severe increase in fiber-to-fiber contact area. In addition, the material becomes quite brittle with the phase change and may be unstable in a mechanical load environment.

2.1.3.2 1800°F Compatibility Testing - Recommendations

Compatibility tests at 1800°F were carried out in an effort to more thoroughly define the problems and to investigate some alternate materials for potential 1800°F service. Nickel and molybdenum foils, in combination with Refrasil fibrous quartz paper and woven quartz (Astroquartz) cloth were considered.

Sample descriptions and photomicrographs of the nickel and molybdenum foils are given in Figures 2.1.4 and 2.1.5. Based on these 1800°F compatibility tests, the following conclusions are made:

(1.) From a surface stability standpoint, molybdenum is preferred over nickel for 1800°F service. Thermal etching and recrystallization are much less severe with the molybdenum. Grooving at the grain boundaries is light.

(2.) Based on its stability in the amorphous state and its apparent minimal effect on the foils at 1800°F, woven, high purity quartz (Astroquartz) is preferred over Refrasil quartz paper.

The superior stability of molybdenum foil at 1800°F is expected because of its high melting point and low vapor pressure in comparison to nickel. The molybdenum appears to be an excellent substitute for nickel from the performance standpoint in high temperature, vacuum multi-foil insulation applications; it also has potential for higher temperature environments (e.g. 2200-2500°F). It is available commercially in .0005" thickness and 12" width.

The compatibility samples indicate that the Refrasil quartz paper, in contact with the metals, promotes the roughness and dull appearance of the

* This problem had not occurred in previous long-term nickel-Refrasil testing at 1600°F (2000 hours).
FIGURE 2.1.4a NICKEL COMPATIBILITY SAMPLES

1. Nickel Properties
   Melting Point 2650°F
   Vapor Pressure 1x10^-6 mm Hg @ 1800°F
   Emissivity $\varepsilon = .18$ @ 1800°F

2. Sample Description
   N-1 Ni (.0006") -- no spacer (control)
   N-2 Ni (.0006") -- woven Astroquartz (.005")
   N-3 Ni (.0006") -- Refrasil quartz (.005")

3. Test Conditions
   Wrapped samples with foil and spacer in light contact; 1 week continuous exposure to 1800°F in vacuum (P ≤ 1μ Hg).

4. Results
   Micrographs indicate moderate etching on samples N-1 and N-2. Strong etching and recrystallization sites are indicated on N-3. Visually, N-1 and N-2 had not appeared to lose reflectivity. Sample N-3, on the other hand, was noticeably dulled.

5. Observations
   (a.) Refrasil quartz appears to promote Ni foil surface changes; Astroquartz role does not appear to be significant.
   (b.) Possible reasons for this are:
       (1) impurities in Refrasil, (2) greater contact between Refrasil mat and foil, (3) form change in Refrasil, (4) slow decomposition of SiO$_2$ liberating O$_2$.
   (c.) Astroquartz remained in the amorphous state; Refrasil changed from amorphous to crystalline.
FIGURE 2.1.4b
NICKEL 1800°F COMPATIBILITY SAMPLES

AS-RECEIVED NICKEL

N-1 NICKEL CONTROL (NO SPACER)

N-2 NICKEL - ASTROQUARTZ

N-3 NICKEL - REFRASIL

ALL SAMPLES - 1000X
FIGURE 2.1.5a MOLYBDENUM COMPATIBILITY SAMPLES

1. Molybdenum Properties
   - Melting Point: 4750°F
   - Vapor Pressure: < 1x10^-10 mm Hg @ 1800°F
   - Emissivity: $\varepsilon = 0.15$ @ 1800°F

2. Sample Description
   - M-1: Mo (.0003") -- no spacer (control)
   - M-2: Mo (.0003") -- woven Astroquartz (.005")
   - M-3: Mo (.0003") -- Refrasil quartz (.005")

3. Test Conditions
   - rectangular, alternate-layer samples with foil and spacer in light contact; 1 week continuous exposure to 1800°F in vacuum ($P < 1\mu$ Hg).

4. Results:
   - Very light thermal etching on samples M-1 and M-2; large grains and shallow boundary grooves.
   - Sample M-3 showed a somewhat higher degree of thermal etching; light boundaries and much smaller grain size than in M-1 and M-2. Visually, all three samples appeared to have retained high reflectivity.

5. Observations
   - (a) Surface change of the molybdenum foil was minimal, and appeared to cause less emissivity deterioration than in the Ni foil case.
   - (b) Again, the Refrasil quartz promoted surface changes but the Astroquartz exerted no apparent influence.
FIGURE 2.1.5b
MOLYBDENUM 1800°F COMPATIBILITY SAMPLES

AS-RECEIVED MOLYBDENUM

M-1 MOLYBDENUM CONTROL (NO SPACER)

M-2 MOLYBDENUM - ASTROQUARTZ

M-3 MOLYBDENUM - REFRASIL

ALL SAMPLES - 1000X
foil surface. This is particularly evident in the composite samples (Figure 2.1.3), where the Refrasil was in excellent contact, under mild loading, with the nickel during 1800°F exposure.

This effect is not readily explained but may be due, in part, to a physical embedding of the fine (1μm diameter) Refrasil fibers in the foil grain boundaries. On the other hand, the 9μm diameter Astroquartz fibers in a woven matrix do not permit the intimate contact attained with the Refrasil. A second possibility is that the silica, gradually decomposing at 1800°F, liberates some oxygen which reacts with the nickel. If this decomposition occurs at all*, it would be more pronounced in the fine fiber, Refrasil paper. Accurate definition of any physical interaction between the quartz spacer and metal foil would require considerable further study.

* Not substantiated in our analysis to date.
2.2 Thermal Conductivity Testing - Flat Plate Tests

The objective of this task has been to develop methods for accurate measurement of thermal performance of multi-layer insulations at temperatures to 1800°F and layer densities over a 0-15 psi compressive loading range. From the experimental data, the apparent thermal conductivity of each system is expressed and correlated to temperature and layer density.

During the reporting period, four nickel multi-layer insulations were investigated --- three nickel multi-foil systems and a nickel opacified quartz fiber system. The four samples are listed below, along with their maximum test temperatures.

<table>
<thead>
<tr>
<th>Insulation Systems</th>
<th>Test Temperature Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel-Refrasil quartz paper</td>
<td>1800°F</td>
</tr>
<tr>
<td>Nickel-Nickel opacified quartz paper</td>
<td>1500°F</td>
</tr>
<tr>
<td>Nickel-woven quartz (Astroquartz)</td>
<td>1500°F</td>
</tr>
<tr>
<td>Nickel opacified quartz paper</td>
<td>1500°F</td>
</tr>
</tbody>
</table>

Operating in a vacuum environment \((P \leq 1 \times 10^{-3} \text{ mm Hg})\), heat transfer through the multi-layer insulations is by solid conduction and radiation. An effective or apparent thermal conductivity \((K_a)\) is then defined approximately as the sum of the conduction \((K_s)\) and radiation \((K_r)\) terms. The temperature polynomial, as developed by Wang, expresses the \(K_a\) parameter in the following form:

\[
K_a = a T^{1/2} + b T^3
\]  

(2.2.1)

where \(aT^{1/2}\) and \(bT^3\) represent the \(K_s\) and \(K_r\) terms respectively.

The empirical methods used in obtaining the \(a\) and \(b\) coefficients pertaining to a particular insulation system have been well documented in previous reports \((2)\). The ensuing sections will discuss the measured thermal performance data for the nickel multi-foil and nickel opacified insulations, and present the empirical correlations for each based on Equation 2.2.1.

2.2.1 Nickel Multi-foil Insulations

Flat plate test data for the three nickel multi-foil samples are presented in Figures 2.2.1 to 2.2.3. Apparent thermal conductivities are plotted against layer density (compressive load) at various hot-side temperature levels from 500°F to 1800°F*. All systems were tested in a vacuum

* Corresponding cold-side (sink) temperatures ranged from 100°F to 400°F for these tests.
FIGURE 2.2.3 APPARENT THERMAL CONDUCTIVITY OF NICKEL-ASTROQUARTZ
MULTI-LAYER INSULATION

APPEARANT THERMAL CONDUCTIVITY ($K_a$) $= 10^{-3}$ Btu/hr-ft-$^\circ$F

$T_1$ (hot) $\quad T_2$ (cold)
550°F $\quad$ 110°F
900°F $\quad$ 150°F
1200°F $\quad$ 190°F
1375°F $\quad$ 220°F
1540°F $\quad$ 250°F

- $\bigcirc$, $\bigtriangleup$, $\times$ - Denote calculated values based on correlation of Table 2.2.1

LAYER DENSITY ($n$) - $\ell$/in

\[ 19 \]
environment of $\leq 1 \times 10^{-3}$ mm Hg.

The characteristic behavior of $K_a$ over the full 0-15 psi load range is typical of what would be expected of the multi-layer systems, and is comparable to that of the aluminum and copper systems previously described (2).

Apparent thermal conductivity increases rapidly over the initial 0-.5 psi load range, reflecting a significant increase in solid conductance due to a breakdown in interface resistance. At zero load, the fibrous spacer and foil layers are in minimal contact. The interface or contact resistance between adjacent layers is instrumental in maintaining a low solid conductance. With even mild loading, the interface contact increases significantly with the result that $K_a$ rises sharply.

In the loading range .5 - 15.0 psi, the interface effects are not predominant. The $K_S$ increase here is mainly governed by the further compression of the fibrous matrix (increase in contact points between fibers) and the physical construction and parent material conductivity of the fibrous spacer itself. At the same time, the layer density is increasing steadily which reduces the $K_T$ contribution. Consequently, the net effect is a smaller rate of increase in total conductivity ($K_a$) due to the compensating behavior of the $K_S$ and $K_T$ terms. This compensating effect is noted particularly at the higher temperatures, where the $K_T$ term is of greater significance.

The higher basic solid conductance of the woven quartz (Astroquartz) spacer compared with the Refrasil quartz fibrous paper is noted in comparing the $K_a$ - load relationships of Figures 2.2.1 and 2.2.3. In the .5 - 15.0 psi loading range, the $K_a$ curves for the nickel-Astroquartz system increase at a significantly faster rate than those for the nickel-Refrasil system, implying a greater solid conduction ($K_S$) contribution. This is due to the higher density and "parallel-fiber" construction of the woven quartz, which results in substantially more solid conduction heat transfer through the spacer.

Examination of 0 - .5 psi data for these two systems further illustrates the importance of interface resistance. At essentially zero-load, the $K_a$ values for the nickel-Refrasil and nickel-Astroquartz insulations are virtually identical. Only when this interface resistance is overcome by increasing load does the higher solid conductivity of Astroquartz become influential. It can be concluded, then, that under conditions of zero load the interface resistance, not the inherent properties of the spacer, govern the solid conductance contribution to total system performance.
The nickel multi-foil systems are correlated by means of Equation 2.2.1. In integrated form, the apparent thermal conductivity \( (K_a) \) between boundary temperatures \( T_1 \) and \( T_2 \) is expressed as:

\[
K_a = \left( \frac{T_1}{T_2} \right)^{3/2} \left( \frac{T_1^{3/2} - T_2^{3/2}}{T_1 - T_2} \right) + \frac{b}{4} \cdot \left( \frac{T_1^4 - T_2^4}{T_1 - T_2} \right)
\] (2.2.2)

Using experimental data at several temperatures and loads, the solid conduction \((2a/3)\) and radiative \(\left( \frac{b}{4} \right)\) coefficients of Equation 2.2.2 were determined by means of the empirical graphical techniques previously described (2). These coefficients, and the temperature and layer density ranges for which they are valid, are given in Table 2.2.1.

The forms of the \(K_s\) and \(K_r\) coefficients are interpreted as follows:

**Solid conduction \((2a/3)\)** - As was found in analyzing the copper multi-foil systems, the \((2a/3)\) coefficient is closely approximated by a log function. The interface resistance effect is reflected in that the coefficient approaches zero for layer density \((n)\) equal to the zero load value. The high solid conductivity of the woven quartz in comparison with the fibrous quartz paper is indicated by the order of magnitude higher \((2a/3)\) coefficient for the woven material.

**Radiative \((\frac{b}{4})\)** - The radiative coefficient varies inversely as the layer density \(\left( \frac{b}{4} = \frac{\sigma_e}{24n} \right)\). For the nickel systems, the "apparent" emissivity of the nickel shield material is defined as a function of temperature \((T)\) and layer density \((n)\). The temperature dependency is substantiated by the literature. The layer density dependency was determined in the correlation and is illustrated for the nickel-nickel opacified quartz system in Figure 2.2.4.

As shown, the "apparent" emissivity increases in approximately linear fashion with both temperature and layer density (load). Actually, the effect of increasing load is to reduce the attenuating surface area of the nickel foils due to the increased contact between spacers and foil. This attenuating area reduction is merely represented in the correlation as an increase in the "apparent" emissivity of the nickel reflective shields.

The empirical \(K_a\) expressions as presented in Table 2.2.1 were used to regenerate apparent thermal conductivity values for the nickel multi-foil systems. The calculated data are included in Figures 2.2.1 to 2.2.3. Comparison of calculated and measured \(K_a\) values provide a check on the ability of the empirical correlations to fit the data over the full load and temperature range.
<table>
<thead>
<tr>
<th>Multi-foil Systems</th>
<th>Hot-side Temperature $T_1$ Range $^\circ R$</th>
<th>Layer Density (n) $\ell$/in.</th>
<th>K. Coefficient $\left(\frac{2}{3}\right)$ Btu/hr-ft-$^\circ R^{1/2}$</th>
<th>K. Coefficient $\left(\frac{5}{4}\right)$ Btu/hr-ft-$^\circ R^{3}$</th>
<th>Effective Emissivity Function</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel-Refrasil quartz</td>
<td>900 - 2200</td>
<td>85 - 180*</td>
<td>[.6410gn - 1.22]$\times$10$^{-4}$</td>
<td>72.1$\times$10$^{-12}$ $\frac{e}{n}$</td>
<td>$e = 0.00008T_1 + 0.0013n - 0.14$</td>
</tr>
<tr>
<td>Nickel-Nickel Opacified Refrasil quartz</td>
<td>900 - 2000</td>
<td>53 - 110</td>
<td>[.5110gn - 0.842]$\times$10$^{-4}$</td>
<td>72.1$\times$10$^{-12}$ $\frac{e}{n}$</td>
<td>$e = 0.00008T_1 + 0.002n - 0.15$</td>
</tr>
<tr>
<td>Nickel-woven quartz (Astroquartz)</td>
<td>900 - 2000</td>
<td>108 - 152</td>
<td>[.51310gn - 1.04]$\times$10$^{-3}$</td>
<td>72.1$\times$10$^{-12}$ $\frac{e}{n}$</td>
<td>$e = 0.00126T_1 - 0.03$</td>
</tr>
</tbody>
</table>

* 180 $\ell$/inch corresponding to 6.2 psi compressive load.
EXTRAPOLATED TO $T_i = 0$ IN ORDER TO DEFINE LINEAR EXPRESSION FOR $\varepsilon(T)$.

\[ \varepsilon = 0.00008 T_i + 0.002 \quad \text{for} \quad T_i \leq 1500 \]

HOT-SIDE TEMPERATURE ($T_i$) = °C.
With the exception of the zero-load, high temperature operating condition, the calculated and measured apparent thermal conductivities agreed to within 10-20%. Calculated $K_a$ values were conservative (i.e. higher than measured) in all cases. For the excepted condition mentioned above, the calculated $K_a$ values were 30-50% higher than measured data. The general tendency for maximum deviations to occur at the higher temperatures is attributed to an over-estimation of the radiative ($K_r$) term, and more specifically, the emissivity of the nickel foil. The hot-side temperature ($T_1$) was used in the $\varepsilon(T, n)$ function for computing the foil emissivity. An estimated average temperature across the insulation thickness would be more appropriate here. Some efforts are being made to predict and incorporate an average temperature in this computation.

### 2.2.2 Nickel Opacified Quartz Multi-layer Insulation

Experimental thermal performance data on nickel opacified multi-layer insulation, at temperatures ranging from 500-1500°F and compressive loads of 0-15 psi, are presented in Table 2.2.2.

As with the previously discussed fibrous multi-layer systems, the nickel opacified insulation is considered to be partially transparent, and to attenuate thermal radiation primarily by backscattering. As such, an "apparent" thermal conductivity for this system is described by the modified Larkin-Churchill equation:

$$K_a = \frac{(2a/3) \frac{T_1^{3/2} - T_2^{3/2}}{T_1 - T_2}}{\sigma \frac{(T_1^4 - T_2^4) \Delta X}{12 \left( \frac{1}{\varepsilon_1} + \frac{1}{\varepsilon_2} - 1 + N\Delta X \right)(T_1 - T_2)}$$

\[ (2.2.3) \]

The solid conduction ($K_s$) term is in a form similar to that of the multi-foil insulations. The radiative term ($K_r$) is controlled by the backscattering cross-section ($N$) for the fibrous material, the material thickness ($\Delta X$) and the boundary surface optical properties ($\frac{1}{\varepsilon_1} + \frac{1}{\varepsilon_2} - 1$).

It is evident from Equation 2.2.3 that the "apparent" thermal conductivity as defined cannot be considered as an intrinsic property of the fibrous material. Use of this performance parameter must, in many cases, be restricted to specific boundary conditions and insulation geometry. The effects of these parameters are illustrated in Figure 2.2.7.

In empirically correlating the nickel opacified multi-layer system, the backscattering cross-section ($N$) is first computed at various temperature levels and loads. The maximum-minimum method was used, in which upper and lower limits on $N$ are determined from experimental performance data. Details of this technique, and the limiting assumptions associated with it, were discussed previously.  

24
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<th>$T_c$ °F</th>
<th>$T_g$ °F</th>
<th>$\rho$ L/in</th>
<th>Load (PSI)</th>
<th>$K \times 10^{-3}$ Btu/hr-ft-°F</th>
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<td>280</td>
<td></td>
</tr>
<tr>
<td>28</td>
<td>1200</td>
<td>290</td>
<td>223</td>
<td>108.7</td>
<td>15</td>
<td>3.38</td>
<td>201</td>
<td></td>
</tr>
<tr>
<td>29</td>
<td>900</td>
<td>248</td>
<td>183</td>
<td>109.3</td>
<td>15</td>
<td>2.42</td>
<td>103.2</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>500</td>
<td>168</td>
<td>122</td>
<td>110.0</td>
<td>15</td>
<td>1.515</td>
<td>33.2</td>
<td></td>
</tr>
</tbody>
</table>
The $N_{\text{max}}$ and $N_{\text{min}}$ data are fit by inspection to generate an appropriate expression for the backscattering cross-section. As in the plain Refrasil and copper opacified cases, the $N$ expression for nickel opacified quartz was approximated by a linearly increasing function of layer density ($n$), and a linearly decreasing function of hot-side temperature ($T_i$). The generated plots of $N$ versus $n$ for nickel opacified multi-layer insulation are presented in Figure 2.2.5.

The corresponding solid conduction coefficient ($2a/3$) as expressed in Equation 2.2.3 is given in Figure 2.2.6. The $K_S$ term is quantitatively estimated simply by subtraction of the radiative contribution $K_R$ (computed using the $N$ expression of Figure 2.2.5) from the total measured $K_a$. The layer density dependency is approximated by a log function as noted in Figure 2.2.6. This agrees with the usual form of the solid conduction coefficient for multi-foil systems (Table 2.2.1). In general, the log $n$ functionality appears to give a more accurate representation of the interface effects in multi-layer insulations.

The empirically-determined backscattering cross-section ($N$) and solid conduction coefficient ($2a/3$) are used in defining the apparent thermal conductivity ($K_a$) of the nickel opacified multi-layer system. In the form of Equation 2.2.3, the specific expression is:

$$K_a = [0.50 \log n - 0.84] \times 10^{-4} \frac{T_1^{3/2} - T_2^{3/2}}{T_1 - T_2} + \frac{\sigma \lambda}{12n} \left[ \frac{1}{n} \left( \frac{T_1^4 - T_2^4}{T_1 - T_2} - \frac{T_1^4 - T_2^4}{T_1 - T_2} \right) \right],$$

(2.2.4)

Where:

- $\lambda = \text{total layers in sample}$
- $n = \text{layer density}$
- $\frac{\lambda}{n} = \Delta X = \text{sample thickness}$
- $\epsilon = \frac{1}{\epsilon_1} + \frac{1}{\epsilon_2} - 1$

The use of Equation 2.2.4 as a design tool in predicting apparent thermal conductivity is illustrated in Figure 2.2.7. Several $K_a$ versus sample thickness plots are generated for boundary temperatures of 1200°F and 70°F, and for compressive loads of .04 psi (68 l/inch) and 15 psi (110 l/inch). The effects on apparent conductivity of boundary optical properties ($\epsilon_b = 0.05$ and $\epsilon_b = 1.0$), insulation thickness ($\Delta X$) and compressive load (i.e. layer density) are clearly shown. The plots indicate that $K_a$ dependency on thickness exists up to $\Delta X = .1-.2"$ for the $\epsilon_b = 1.0$ case, and to $\Delta X > 1"$ for the $\epsilon_b = .05$ case.
FIGURE 2.2.5 BACKSCATTERING CROSS-SECTION
NIQUEL OPACIFIED QUARTZ MULTI-LAYER INSULATION

\[ N = 37.0 + 4.48n - 0.02T_1 \]

BACKSCATTERING CROSS-SECTION (\(N\)) - IN\(^{-1}\)

LAYER DENSITY (\(n\), \(\ell/\text{in.}\))
Figure 2.2.6 Solid Conductivity Coefficient
Nickel Opacified Quartz Multi-Layer Insulation
FIGURE 2.2.7  APPARENT THERMAL CONDUCTIVITY AS A FUNCTION OF THICKNESS
NICKEL OPAFICIED "REFRASIL" QUARTZ MULTI-LAYER

LOW LOAD -- .04 PSI (68 lbf/in.)
HIGH LOAD -- 15.0 PSI (110 lbf/in.)

MINIMUM THICKNESS AT WHICH BOUNDARY EMISSIVITY ($\varepsilon_b$) EFFECT IS NEGLIGIBLE.
This substantiates the earlier observation concerning the strong dependency of $K_\alpha$ for nickel opacified fibrous multi-layer insulation on boundary conditions and thickness as well as temperature and compressive load, when considering total insulation thicknesses of 1" or less. The same observation was brought out, in some detail, in previous discussion on performance of other fibrous and opacified fibrous multi-layer insulations (2).

Figure 2.2.8 compares the observed backscattering cross-sections (at 900°F and as a function of compressive load) for the various fibrous and opacified fibrous multi-layers. The behavior of the $N$ value for nickel opacified quartz is characteristic of the "Refrasil" fibrous quartz systems (i.e. plain Refrasil and copper opacified Refrasil). For the nickel opacified quartz (5) and plain quartz (4), $N$ increases at such a rate that the $NAX$ coefficient is essentially constant over the layer density range corresponding to 0-15 psi. This behavior has been observed in previous studies (Larkin and Churchill)(3). The corresponding $N$ value for copper opacified quartz increases with load at a somewhat slower rate.

As in the copper opacified quartz case, the nickel opacified system has an $N$ value which is approximately a factor of 3 greater than that of the plain quartz fibrous multi-layer. The superior radiation attenuation capability obtained by including metal flake opacification is clearly demonstrated.

The superiority of the nickel opacified system over the copper opacified material is not readily explained. By design, the two materials are to be virtually identical in all physical properties (e.g., weight, volume of flake, paper thickness, etc.) except the metal used as the opacifier. Under this assumption, and further expecting that some attenuation by the flakes may be by absorption-re-emission mechanism, one might expect the copper* opacified system to be the better performer. The fact that the converse is true may indicate that:

1. The fabricated copper opacified paper may deviate from the initial design specifications and properties, giving a poorer than expected performance (e.g., poor copper flake retention in the finished paper; deteriorated emissivity of the copper flake).

2. Presence of the flake opacifier serves primarily to increase backscattering, with very little absorption-re-emission taking place. This would minimize the effect of different emissivity of the metal flakes.

The copper and nickel opacified quartz materials are to be analyzed further in an effort to explain these performance characteristics.

* Copper has lower emissivity than nickel.
FIGURE 2.2.8 SCATTERING CROSS-SECTION FOR FIBROUS PAPERS AT 900°F TEMPERATURE

1. ALUMINUM OPAQUE GLASS FIBER PAPER
   .003" THK.; IN RANGE (0-15 psi) = 150-420 l/in.

2. GLASS FIBER PAPER; .003" THK.
   IN RANGE (0-15 psi) = 150-380 l/in.

3. COPPER OPAQUE QUARTZ FIBER PAPER
   .010" THK.; IN RANGE (0-15 psi) = 53-115 l/in.

4. QUARTZ FIBER PAPER; .005" THK.
   IN RANGE (0-15 psi) = 100-220 l/in.

5. COPPER OPAQUE QUARTZ FIBER PAPER
   .010" THK.; IN RANGE (0-15 psi) = 53-115 l/inch.

SCATTERING CROSS-SECTION (N) - IN^(-1)

COMPRESSIVE LOAD - PSI

800
700
600
500
400
300
200
100

.02 .1 1.0 10.0 20.
2.3 Longitudinal Thermal Conductivity Testing

The test objective is to determine experimentally the longitudinal or lateral apparent thermal conductivity of opacified fibrous insulations. The longitudinal $K_a$-values are of significance since, in many multi-layer insulation configurations, lateral as well as normal heat flow has a bearing on system performance.

Thermal conductivity data are obtained over the full temperature ranges associated with the opacified fibrous materials. Samples are constructed with a layer density, in the normal direction, which approximates the design layer density at essentially zero load. The three opacified fibrous multi-layer systems to be tested are described below:

<table>
<thead>
<tr>
<th>Multi-layer Sample</th>
<th>Hot-side Temperature Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum opacified glass</td>
<td>250 - 900°F</td>
</tr>
<tr>
<td>$n \approx 186$ ft/inch</td>
<td></td>
</tr>
<tr>
<td>Copper opacified quartz</td>
<td>500 - 1400°F</td>
</tr>
<tr>
<td>$n \approx 68$ ft/inch</td>
<td></td>
</tr>
<tr>
<td>Nickel opacified quartz</td>
<td>500 - 1600°F</td>
</tr>
<tr>
<td>$n \approx 72$ ft/inch</td>
<td></td>
</tr>
</tbody>
</table>

Samples of non-opacified 106 glass paper and/or Refrasil quartz are to be tested, primarily as an aid in data reduction and evaluation of the opacified materials.

2.3.1 Test Apparatus - Sample Design

The basic flat plate test configuration and procedures are used in longitudinal thermal conductivity measurement. A flat, planar sample is designed to provide essentially one-dimensional heat flow between hot and cold boundaries over a central portion of the sample. These measured fluxes, along with temperatures and sample dimensions, are sufficient to compute an apparent thermal conductivity.

The test apparatus, with installed multi-layer insulation sample, is shown in Figure 2.3.1. In testing, hot-side temperature is maintained by a woven graphite resistance heater. Heat is supplied to the sole plate, which is matched to the 6" diameter of the opacified paper sample. The lateral heat flow across the central portion of the sample is measured by a 2" x 2" flux meter, centrally located at the cold-face. The sink plate is water cooled (not shown) to maintain cold side temperatures in the 100-300°F range throughout testing. Text fixtures are contained within a bell jar enclosure, under vacuum ($P \leq 1 \times 10^{-3}$ mm Hg) during all tests.
FIGURE 2.3.1 LONGITUDINAL THERMAL CONDUCTIVITY TEST CONFIGURATION
The opacified paper samples are of cylindrical geometry with 6" diameter and 1-1/2" height. Samples are constructed by spirally-wrapping the fibrous paper (1-1/2" width) from the center outward, to a layer density approximating the specified "zero load" condition. Preliminary analysis indicated that lateral heat flow across the 1-1/2" sample is essentially one-dimensional in the 2" x 2" test "core" section. This condition is attainable because of the "self-guarding", in the radial direction, which is achieved in the 6" diameter sample design.

2.3.2 Results

During the reporting period, longitudinal thermal conductivity data were obtained for aluminum opacified glass material, over the temperature range 250-900°F. Results are plotted in Figure 2.3.2; corresponding data on normal apparent thermal conductivity are included for comparison.

Longitudinal thermal conductivity values are 14-16 times higher than the corresponding normal thermal conductivities. The major contribution to the high $K_s/K_n$ ratio is tentatively attributed to solid conduction for the following reasons:

1. Lack of interface resistance which is significant in minimizing low-load $K_s$ in the normal multi-layer orientation.

2. Fibrous matrix where the fibers are preferentially oriented in the lateral direction (parallel to heat flow).

3. Possibility of the large volume of aluminum flake opacifier providing additional heat paths in the longitudinal direction.

4. The longitudinal conductivity - temperature relationship is much less severe than the ($b/t^3$) function which would be expected if performance was radiation-dominated.

The above observations are tentative at this point. Subsequent data on opacified and plain fibrous materials should permit more in-depth study of the longitudinal heat flow mechanisms.
FIGURE 2.3.2 APPARENT THERMAL CONDUCTIVITY - LONGITUDINAL DIRECTION
ALUMINUM OPAZIFIED GLASS MULTI-LAYER INSULATION

\[ n = 180 \text{ g/in LONGITUDINAL } "K_{g}\text{,}" \]

\[ n = 180 \text{ g/in NORMAL } "K_{g}\text{,}" \]

COLD-FACE TEMPERATURES RANGED FROM 140 - 390°F

HOT-FACE TEMPERATURE \((T_1)\) - °F
2.4 Insulation Interfaces - Exposed Edge Effects

In multi-foil insulation system design, insulation panels may be oriented such that edges are exposed. These edges, which may present a temperature gradient close to the full "hot-side to cold-side" gradient across the insulation proper, radiate freely to the surrounding environment. In evaluating total system performance, it is necessary to determine this exposed edge heat loss. The edge effects study has included analytical prediction and experimental verification of these losses.

2.4.1 Analytical and Experimental Approaches

The exposed edge effects analysis considers a rectangular multi-layer insulation panel, shown schematically in Figure 2.4.1. The conditions of the analysis, as interpreted from the boundary conditions and heat flow equation of Figure 2.4.1 are given below:

1. Panel is of finite width and thickness and infinite length, implying two-dimensional heat flow (normal across T_h and T_c lateral from center to edge).

2. Hot-face and cold-face boundaries are maintained at approximately uniform temperatures.

3. Exposed edges, with an apparent emissivity of 1.0, radiate to an ambient (530°R) environment. Total heat loss from an insulation panel operating at the above conditions is computed and compared with heat flux for an ideal (no edge loss) panel of identical size, to determine the edge effect.

The experimental test set-up is shown schematically in Figure 2.4.2. The dual-sample configuration duplicates the size and two-dimensional heat flow characteristics of the analytical model. Hot-face surface temperatures are maintained by a two-stage, ceramic-encased woven graphite resistance heater. Power can be controlled independently to a central element and two narrow edge elements as a means of approximating a uniform, hot surface temperature. This temperature is monitored by thermocouples located at intervals from center to edge of the sample. Cold boundaries are maintained at a uniform, near-ambient temperature. The aluminum boundary plates stabilize the multi-layer wrap at a specified layer density.

Heat flux is determined by a total power measurement for the system operating at fixed boundary temperatures. Extraneous losses (i.e. heater leads) are subtracted to obtain the net heat loss from the exposed edge sample. Combined radiative and conductive heat loss mechanisms are considered in calculating losses associated with the four stranded molybdenum wire leads. At point of exit from the multi-layer sample, the leads are assumed to be at hot-side temperature.
HEAT BALANCE:
\[
\frac{\delta}{\delta x} \left( k_x \frac{\delta T}{\delta x} \right) + \frac{\delta}{\delta y} \left( k_y \frac{\delta T}{\delta y} \right) = 0
\]

\( k_x \) = longitudinal thermal conductivity

\( k_y \) = normal thermal conductivity
ADIA BATIC CENTER LINE
\( \frac{dT}{\partial x} = 0 \)

HOT-FACE T/C LOCATIONS

ALUMINUM COLD-SIDE PLATES

GRAPHITE RESISTANCE HEATER
MULTI-LAYER INSULATION, 4" THICK

TWO-STAGE GRAPHITE HEATER .1" OVERALL THICKNESS
(2 EDGE STRIPS; CENTER STRIP)

AL PLATES

\( T_H = 1000^\circ F \)
\( T_C = 100^\circ F \)

SECTION A-A

FIGURE 2.4.2 EXPOSED EDGE TEST SCHEMATIC
Testing capability, in vacuum, is to 1800°F. Aluminum-106 glass and nickel-Refrasil quartz multi-layer insulations were evaluated over a range of hot-side temperatures up to 1000°F and 1800°F, respectively. The experimental configuration, with two-stage resistance heater, provided relatively uniform temperatures over the entire hot face. Center-to-edge temperature decrease was less than 10% in all tests.

2.4.2 Exposed Edge Heat Loss Results

Two multi-layer insulation systems have been evaluated — aluminum-106 glass to 900°F and nickel-Refrasil quartz to 1700°F. Pertinent physical and thermal properties of the two insulations are listed below:

<table>
<thead>
<tr>
<th>Exposed Edge Sample Characteristics</th>
<th>Aluminum-106 Glass</th>
<th>Nickel-Refrasil quartz</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Materials</td>
<td>aluminum foil</td>
<td>nickel foil</td>
</tr>
<tr>
<td></td>
<td>(thickness = .00025&quot;)</td>
<td>(thickness = .0006&quot;)</td>
</tr>
<tr>
<td></td>
<td>106 glass paper</td>
<td>Refrasil quartz paper</td>
</tr>
<tr>
<td></td>
<td>(thickness = .003&quot;)</td>
<td>(thickness = .005&quot;)</td>
</tr>
<tr>
<td>2. Temperature Limit</td>
<td>900°F</td>
<td>1800°F</td>
</tr>
<tr>
<td>3. Layer Density</td>
<td>111 g/inch</td>
<td>100 g/inch</td>
</tr>
<tr>
<td>4. Sample Dimensions</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Length</td>
<td>unit</td>
<td></td>
</tr>
<tr>
<td>Width</td>
<td>9.5&quot;</td>
<td>9.5&quot;</td>
</tr>
<tr>
<td>Thickness</td>
<td>0.4&quot;</td>
<td>0.4&quot;</td>
</tr>
<tr>
<td>5. Estimated normal thermal conductivity (Btu/hr-ft-°R)</td>
<td>(K(T) = 10^{-3}(T)\times[.106+.171T^2])</td>
<td>(K(T) = 10^{-3}T\times[.4+.472T^2])</td>
</tr>
<tr>
<td>6. Estimated lateral thermal conductivity (Btu/hr-ft-°R)</td>
<td>(K(T) = 3.9 - .76T)</td>
<td>(K(T) = .344-2.08T; T \leq .7)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>= 2.71-1.04T; .7 &lt; T \leq 1.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>= .97+ .54T; T &gt; 1.1</td>
</tr>
</tbody>
</table>

\[ T = \frac{T_{act}}{1000} \] in the above expressions
Items 5 and 6 are the normal and lateral conductivity expressions, as derived from cylindrical test data and materials properties for the two insulations. These expressions are used in the analysis.

Exposed edge experimental and analytical results are presented and compared in Table 2.4.2. In all cases, the exposed edges are radiating to an ambient environment sink (70-100°F), in a vacuum of $< 1 \times 10^{-3}$ mm Hg. The $Q_e$ flux values are net heat losses, obtained by subtracting the estimated lead losses from the total measured flux. The experimental $Q_e$ values were obtained using flat plate thermal conductivity data. The analytical $Q_o$ values were based on the normal $K_a$ values of Table 2.4.1.

Based on the exposed edge results, it is observed that:

1. Heat loss penalty for an insulation system with exposed edges is significant. Panel configuration (i.e. exposed edge length to surface area) is critical. The rather severe experimental geometry resulted in $Q_e/Q_o$ ratios of ~7.0 and ~4.0 for the aluminum and nickel multi-layer insulations, respectively.

2. Exposed edge heat loss increases rapidly with temperature due to the radiative loss mechanism at the edges.

3. Agreement between analytical and experimental results, as denoted by the $(Q_e/Q_o)_{cal}/(Q_e/Q_o)_{exp}$ ratio, was good for the aluminum system (estimated within 10%). Agreement for nickel-Refrasil quartz data varied from 0-30% (to 1000°F) to 40-55% (1000-1700°F). In all cases, the analytical results were conservative.

4. The analytical $Q_e$ and $Q_o$ values were substantially higher, in most cases, than the corresponding experimental values. This is partially the result of using somewhat conservative* thermal conductivities for the multi-layer insulations.

The reasonably good comparisons between experimental and calculated results indicate that the analytical program can be quite useful in giving approximate performance predictions for multi-layer insulation systems with exposed edges. Based on the comparative results to date, the analysis is conservative and will over-predict the edge loss to a moderate extent.

As indicated in observation (1), the sample size tested (4.75" center-to-edge width; 12" (unit) length; and 0.4" thickness) constituted a severe edge loss geometry. For multi-layer insulation panels with a high surface area to exposed edge area ratio (e.g. thinner panels, wider panels), the $Q_e/Q_o$ ratio would be substantially reduced.

* Based on cylindrical test data, which may be high by 25%.
### TABLE 2.4.2

**EXPOSED EDGE HEAT LOSS RESULTS**

<table>
<thead>
<tr>
<th>Multi-layer Insulation</th>
<th>$T_h$ (average) °F</th>
<th>$T_c$ °F</th>
<th>Analytical $Q_e$ Btu/hr-ft</th>
<th>$Q_o$ Btu/hr-ft</th>
<th>$Q_e/Q_o$</th>
<th>Experimental $Q_e$ Btu/hr-ft</th>
<th>$Q_o$ Btu/hr-ft</th>
<th>$Q_e/Q_o$</th>
<th>$(Q_e/Q_o)_{calc.}$</th>
<th>$(Q_e/Q_o)_{exp.}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum-106 Glass</td>
<td>685</td>
<td>93</td>
<td>11.5</td>
<td>1.6</td>
<td>7.2</td>
<td>7.5</td>
<td>1.1</td>
<td>7.0</td>
<td>1.03</td>
<td></td>
</tr>
<tr>
<td>$n = 114$ ft/in.</td>
<td>935</td>
<td>97</td>
<td>23.7</td>
<td>3.09</td>
<td>7.7</td>
<td>17.0</td>
<td>2.4</td>
<td>7.1</td>
<td>1.08</td>
<td></td>
</tr>
<tr>
<td>Nickel-Refraisil quartz</td>
<td>665</td>
<td>95</td>
<td>17.4</td>
<td>3.93</td>
<td>4.4</td>
<td>8.0</td>
<td>2.7</td>
<td>3.3</td>
<td>1.33</td>
<td></td>
</tr>
<tr>
<td>$n = 100$ ft/in.</td>
<td>950</td>
<td>110</td>
<td>37.3</td>
<td>9.0</td>
<td>4.2</td>
<td>23.0</td>
<td>5.3</td>
<td>4.2</td>
<td>1.00</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1475</td>
<td>155</td>
<td>114.8</td>
<td>25.1</td>
<td>4.6</td>
<td>73.0</td>
<td>21.8</td>
<td>3.3</td>
<td>1.39</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1675</td>
<td>180</td>
<td>161.0</td>
<td>35.6</td>
<td>4.5</td>
<td>100.0</td>
<td>35.0</td>
<td>2.9</td>
<td>1.55</td>
<td></td>
</tr>
</tbody>
</table>

$T_h$ - average hot-side temperature

$T_c$ - cold-side temperature

$Q_e$ - exposed edge heat loss

$Q_o$ - heat loss through corresponding insulation panel with no edge loss.
### 2.5 Vacuum Enclosure - Hastelloy-X Specimen Analysis

Analysis of the Hastelloy-X vacuum enclosure specimen, exposed to simultaneous air and vacuum environments at 1400°F for 10,000 hours continuous testing, has been completed. Effects of this high temperature environment on mechanical properties and overall materials stability and compatibility are discussed.

The primary objectives of the vacuum enclosure test program were to:

1. Get some first-order indications as to suitability of Hastelloy-X as a vacuum enclosure material in 1400°F environment.

2. More specifically, to determine effects on vacuum integrity and mechanical properties due to long-term exposure at 1400°F, under combined conditions of vaporization (vacuum side), oxidation (air side), and internal stress (5000 psi hoop stress).

3. Obtain a first order indication of compatibility between Hastelloy-X and (a) selected 1400°F insulation materials (foils and spacers) and (b) Hastelloy-C and beryllium (potential power capsule materials).

4. Compare resultant mechanical strength data (creep and tensile) for the thin, .012" Hastelloy-X enclosure material with corresponding literature data, to determine potential suitability of the latter in predicting Hastelloy-X material behavior in this special environment.

#### 2.5.1 Sample Description - Test Procedure

The cylindrical Hastelloy-X test specimen had dimensions 3-1/4" O.D. x 12" length with a wall thickness of 0.012". The base sheet stock from which the specimen was fabricated was cold-rolled and annealed (2150°F). The specimen included 3 welds -- a longitudinal seam weld and two circumferential edge welds at the end plates. Tungsten inert gas welding was utilized in fabrication.

Test coupons were also included to provide an indication of compatibility between the Hastelloy-X specimen and typical radioisotope power system enclosure materials; i.e. beryllium and Hastelloy-C. Individual compatibility coupons were located on the air side of the enclosure test specimen.

Test conditions of the 10,000-hour run consisted of maintaining the Hastelloy-X specimen at 1400°F with an external vacuum and internal air pressurization of 37.2 psia. This internal air pressure corresponded to a 5000 psi hoop stress condition in the cylindrical specimen wall.
Samples of each of the following candidate insulation materials were included in the vacuum chamber tube: aluminum foil, copper foil, nickel foil, glass fiber paper, Refrasil quartz fiber paper, and Astroquartz cloth. These materials were placed in the vacuum chamber tube in such a manner that they were at temperature levels within their operational limits. The insulation materials provided a vacuum environment typical of actual service conditions for the test specimen.

The test apparatus is shown schematically in Figure 2.5.1. Basically, the apparatus consists of an electrically heated tube furnace housing a vacuum chamber tube which is fitted with thermocouple and pressure gauge feedthroughs. The Hastelloy-X specimen was enclosed within the heated, tubular vacuum chamber during testing.

The 10,000-hour long-term test run was interrupted at intervals of 2,000, 4,000 and 6,000 hours in order to: (1) check gas leak rate through the specimen, (2) analyze residual gas in vacuum space, (3) measure circumferential creep and (4) observe any vacuum-side physical changes in base metal surface or weld areas. In all cases, these data were compared to initial, time-zero values.

After completing the 10,000-hour cycle, a final set of the above were taken. In addition, an overall evaluation of the exposed Hastelloy-X specimen was made, including tensile strength testing, compatibility sample evaluation, and photomicrograph studies of cross-sections of selected base metal and weld area samples.

Background information and detailed description of test procedures and equipment is given in the Phase I preliminary final report (4).

2.5.2 Results - Interim and Final Evaluations

2.5.2.1 Vacuum Integrity and Materials Stability

The stability of the Hastelloy-X specimen in a vacuum environment is indicated by the results summarized in Table 2.5.1. The specimen remained virtually leak-tight throughout the test period, with the measured rate (helium mass spectrometer testing) less than $1 \times 10^{-8}$ STD cc air/second. The consistency of the residual gas analysis of the vacuum space gave further indication of specimen tightness at 1400°F.

The carbide coating on the exterior surface of the Hastelloy-X enclosure specimen is clearly shown on Figure 2.5.2. Observations at interim periods established that the coating formed in the period between the 6,000 and 10,000-hour inspections. Preliminary analysis of the coating indicated predominately chromium carbide. The chromium, an alloy constituent of the Hastelloy-X, could have been available at the vacuum surface by virtue of vaporization of this constituent at 1400°F. Speculation on the presence of carbon and/or chromium carbide is as follows:
FIGURE 2.5.1 Vacuum Enclosure Material Tester
### TABLE 2.5.1

**HASTELLOY-X ENCLOSURE - VACUUM INTEGRITY**

<table>
<thead>
<tr>
<th>Testing Time</th>
<th>Leak Rate* (\text{cc air (STP)/sec.})</th>
<th>Vacuum Space*</th>
<th>Observations</th>
</tr>
</thead>
</table>
| Initial (0 time) | \(< 1 \times 10^{-8}\)              | 58% - \(\text{H}_2\)  
21% - \(\text{H}_2\text{O}\)  
18% - \(\text{CO}\)  
Trace - \(\text{N}_2 \& \text{CO}_2\) | Hastelloy-X specimen clean; in as-received condition. |
| 2000 hours      | \(< 1 \times 10^{-8}\)              | 78% - \(\text{H}_2\)  
13% - \(\text{CO}\)  
7.5% - \(\text{N}_2\)  
1.2% - \(\text{CH}_x\)  
[Air diffusion through specimen not indicated] | Vacuum side surface of Hastelloy-X specimen slightly tarnished; possibly a light oxide coat developed during slightly higher vacuum pressure periods in early pumpdown stages. Surface photographs (18X) show no visible change in base metal or weld surfaces. Vaporization does not appear to be significant. |
| 45              | \(< 1 \times 10^{-8}\)              | 49% - \(\text{H}_2\)  
24% - \(\text{CO} + \text{N}_2\)  
14% - \(\text{CH}_x\)  
13% - \(\text{CO}_2\)  
[No air diffusion indicated] | Observations at 2000 hours apply. |
| 4000 hours      | \(< 1 \times 10^{-8}\)              | ------        | Observations at 2000 hours apply.                                             |
| 6000 hours      | \(< 1 \times 10^{-8}\)              | ------        | Observations at 2000 hours apply.                                             |
| 10,000 hours    | \(< 1 \times 10^{-8}\)              | 81% - \(\text{H}_2\)  
9% - \(\text{N}_2 + \text{CO}\)  
7.4% - \(\text{CH}_x\)  
2.6% - \(\text{CO}_2\)  
[No air diffusion indicated] | The Hastelloy-X tube specimen was covered entirely by a light coating, loosely held in the central (1400°F) portion of the tube and tightly bonded in the end regions (see Figure 2.5.2). Analysis indicated a carbide coating, most likely chromium-carbide. In areas where coating was removed, the Hastelloy-X surface appeared normal. |

* Leak test and gas analysis performed with specimen at 1400°F
FIGURE 2.5.2
HASTELLOY-X VACUUM ENCLOSURE
SPECIMEN - AFTER 10,000-HOUR,
1400°F EXPOSURE
(1.) Migration from the Inconel furnace tube (100-200°F higher than the sample) and deposition on the 1400°F Hastelloy-X surface.

(2.) Presence of hydrocarbons, due to back-streaming* of pump oil into the furnace chamber, which could have reacted with the free chromium.

The fact that the coating formed between 6,000 and 10,000 hours intervals would support the second possibility. In any event, the carbide coating appears to have had no effect on the behavior of the Hastelloy-X specimen.

Photographs (18 power magnification) of the base metal and weld surfaces are given in Figures 2.5.3 and 2.5.4. Vacuum surfaces are pictured at 0, 4,000, and 10,000-hour exposure times. The oxidized surface (air side) is shown after 10,000-hour exposure. In general, no obvious detrimental effects in either the weld area or base metal could be detected at any of the exposure conditions. The carbide coating can be noted on the 10,000-hour, vacuum-side photographs of base metal and weld.

The 10,000-hour inspection of the Hastelloy-X/Hastelloy-C and Hastelloy-X/Beryllium compatibility samples, which had been air exposed at 1400°F, revealed the following:

(1.) The Hastelloy-X/Hastelloy-C sample indicated no interaction or individual deterioration at the interface.

(2.) No interaction between the Hastelloy-X and beryllium. The beryllium, however, had severely oxidized.

In the 10,000-hour, 1400°F exposure, essentially all the beryllium had oxidized (BeO) and turned to powder. This occurrence is supported by outside data on oxidation of beryllium (5). This reference reports that "the thin oxide that forms on beryllium on exposure to air at ambient temperature is highly protective up to about 1100°F. Above 1400°F, the oxide film becomes unprotective and, in time, the metal can turn to powder". Apparently, under the pressurized air environment (37.2 psia), the critical temperature was exceeded at the indicated 1400°F level.

* The cold-trap on the pumping system, which had been operational up to 6000+ hours, was inadvertently allowed to warm to room temperature.
FIGURE 2.5.3 SURFACE PHOTOGRAPHS (18X)—HASTELLOY-X BASE METAL

AS-RECEIVED - VACUUM SIDE

4,000 HOURS, 1400°F EXPOSURE
VACUUM SIDE

10,000 HOURS, 1400°F EXPOSURE
VACUUM SIDE

10,000 HOURS, 1400°F EXPOSURE
AIR SIDE
FIGURE 2.5.4 SURFACE PHOTOGRAPHS (18X) - HASTELLOY-X WELD AREAS

LONGITUDINAL WELD - NO EXPOSURE

LONGITUDINAL WELD AFTER 4000 HOURS, 1400°F VACUUM SIDE

LONGITUDINAL WELD AFTER 10,000 HOURS, 1400°F VACUUM SIDE

LONGITUDINAL WELD AFTER 10,000 HOURS, 1400°F AIR SIDE
2.5.2.2 Mechanical Properties

Tensile strength, yield strength, and total specimen creep were measured for the as-received Hastelloy-X specimen and the 10,000-hour, 1400°F exposed specimen. The results were compared and referenced to manufacturer's published data (6) showing effects of 1400°F aging on strength of Hastelloy-X sheet.

Total Specimen Creep

Total elongation of the Hastelloy-X specimen, measured in the circumferential direction (loaded under equivalent 5000 psi hoop stress), was obtained at intervals throughout the long-term test. Results are as follows:

<table>
<thead>
<tr>
<th>Test Time</th>
<th>Temperature</th>
<th>Stress</th>
<th>Total Elongation</th>
</tr>
</thead>
<tbody>
<tr>
<td>hrs.</td>
<td>°F</td>
<td>psi</td>
<td>%</td>
</tr>
<tr>
<td>0</td>
<td>1400</td>
<td>5000</td>
<td>0</td>
</tr>
<tr>
<td>2000*</td>
<td>1400</td>
<td>5000</td>
<td>0.31</td>
</tr>
<tr>
<td>4000</td>
<td>1400</td>
<td>5000</td>
<td>0.62</td>
</tr>
<tr>
<td>6000</td>
<td>1400</td>
<td>5000</td>
<td>0.62</td>
</tr>
<tr>
<td>10,000</td>
<td>1400</td>
<td>5000</td>
<td>0.79</td>
</tr>
</tbody>
</table>

As indicated, total specimen elongation was less than 1% for the 10,000-hour exposure.

Manufacturer's data giving stress required for 1% total elongation in Hastelloy-X sheet for various temperatures and times, are given in Figure 2.5.5. The 1400°F line has been estimated and included. In designing the total creep test, it was assumed** that Hastelloy-X in thin section (.010 - .020") would exhibit reduced creep strength in comparison to the published data on thicker sheet. As a result, the test stress level was chosen as one-half the corresponding published value for a particular temperature, time condition. The selected "target" condition is indicated on Figure 2.5.5; i.e. for 1% total elongation in 2000 hours at 1400°F, a stress level of 5000 psi was selected.

The actual results indicate that the one-half strength assumption for the .012" Hastelloy-X sheet was quite conservative. In fact, the preliminary indication is that the published data may be quite suitable for predicting creep behavior of thin Hastelloy-X sheet.

* "Target" condition
** Assumption based on a study of the published data and discussions with manufacturer's technical personnel.
FIGURE 2.5.5  AVERAGE ONE PERCENT TOTAL ELONGATION, HASTELLOY-X SHEET

- **1200°F**
- **1350°F**
- **1500°F**
- **1650°F**
- **1700°F**
- **1800°F**

**Stress - KSI**

**Time - Hours**
Comparison of published and actual data, in this case, may be somewhat imprecise since the actual cylindrical specimen was subjected to two-dimensional creep stress (longitudinal as well as circumferential). However, the longitudinal stress level (estimated at 2500 psi) is sufficiently low so that its effect at 1400°F is assumed small. No change in the longitudinal dimension was observed after 10,000 hours.

**Tensile and Yield Strength**

Ambient temperature strengths of Hastelloy-X base metal and weld zone samples were determined. Table 2.5.2 gives data on ultimate and yield strength and percent elongation for as-received and 10,000-hour, 1400°F aged materials. The samples were prepared such that the tensile loading was applied in the circumferential direction, and perpendicular to the longitudinal weld.

Based on the results of Table 2.5.2, the following observations are made:

1. A noticeable deterioration in ultimate tensile strength was brought about by the 1400°F exposure. Reduction of strength was particularly significant in the weld areas.

2. Marked increase in brittleness (loss of elongation) in both the base metal and weld samples.

3. The reduction of properties has been attributed to general precipitation of carbides, which has been described as characteristic of Hastelloy-X in 1400°F exposure (7).

4. Properties deterioration due to grain boundary attack at the vacuum and oxidized surfaces is considered minor.

5. Substantial deterioration in properties at the weld areas is attributed to porosity of the weld "heat affected zone" (HAZ). This porosity, which occurred under 1400°F exposure, did not extend to the weld nugget. All 1400°F weld samples failed in the HAZ.

Further discussion on causes of properties deterioration for the 1400°F samples will be presented in Section 2.5.2.3, in conjunction with the photomicrographs.

Published data (7) on room temperature strength and elongation of 1400°F aged Hastelloy-X are presented in Figures 2.5.6 and 2.5.7. Data are extrapolated from 1000 hours to 10,000 hours (as indicated by the dashed line). Measured strength data on as-received and 1400°F base metal and weld samples are included for comparison.
### TABLE 2.5.2

**ROOM TEMPERATURE STRENGTH OF HASTELLOY-X VACUUM ENCLOSURE MATERIAL**

<table>
<thead>
<tr>
<th></th>
<th>As-Received Material</th>
<th>10,000-Hr, 1400°F Aged Material</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Base Metal A</td>
<td>Weld Zone Aw</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>B_w</td>
</tr>
<tr>
<td>Ultimate Strength (KSI)</td>
<td>118.8</td>
<td>109.8</td>
</tr>
<tr>
<td>Yield Strength .2% Offset (KSI)</td>
<td>55.0</td>
<td>57.3</td>
</tr>
<tr>
<td>Elongation (%)</td>
<td>42.</td>
<td>24.</td>
</tr>
<tr>
<td>Fracture Location</td>
<td>B</td>
<td>B Haz W.I.</td>
</tr>
</tbody>
</table>

* W.I. -- weld interface
  B -- base material
  HAZ -- heat-affected zone

* Fracture below yield
FIGURE 2.5.7  ROOM TEMPERATURE TENSILE ELONGATION OF AGED HASTELLOY-X

AGING TEMPERATURE
1400°F

ELONGATION -- %

HAYNES-STALLITE DATA
LINDE MEASURED -- BASE MATERIAL
LINDE MEASURED -- WELD

AGING TIME - HRS.
The strength and elongation data for Hastelloy-X base material are in good agreement (~10%) with the extrapolated literature data. This implies that the Hastelloy-X, in thin (.012") section, behaves in characteristic fashion when exposed to long-term 1400°F aging. The surface effects due to the combination vacuum and oxidizing environments do not significantly alter this behavior. This appears to lend further credence to the utility of literature data in estimating the effects of these rather specialized high temperature environments on Hastelloy-X mechanical properties.

Strength data for the as-received weld sections were also in good agreement with the literature data. Elongation values were substantially lower, possibly due to embrittlement of the HAZ during welding. The very low 10,000-hour ultimate strengths, in comparison with the extrapolated literature values, again point up the significant effect of the severe porosity observed in the HAZ.

2.5.2.3 Hastelloy-X Photomicrographs

Photomicrographs (200X magnification) of typical Hastelloy-X base metal sections, at time zero and after 10,000 hours, 1400°F exposure, are presented in Figure 2.5.8. Samples A_e and B_e are identical to A and B, the former having been etched to show grain structure.

The vaporization of alloy constituents and/or impurities in the grain boundaries is indicated at the vacuum-side surface. Some mild porosity is also apparent in the Hastelloy-X cross-section, close to the vacuum surface. This porosity is quite random and does not appear to have a significant effect on strength properties. Surface penetration due to oxidation at the "air-side" surface is minimal. The chemically etched photomicrographs clearly illustrate the general carbide precipitation at 1400°F, which results in loss of strength and ductility as discussed in Section 2.5.2.2.

Photomicrographs of the weld "heat-affected zone" (HAZ) after 10,000 hours, 1400°F exposure are shown in Figure 2.5.9. The acute porosity, which appears to be responsible for the sharp reduction in weld zone strength, is clearly illustrated. The following observations are made:

(1.) In opposition to what was seen on the base metal photographs, the porosity in the HAZ is concentrated nearer the oxidized side of the Hastelloy-X sample.

(2.) Surface oxidation effects are more pronounced than on the base metal samples.

(3.) The weld nugget is not affected.
FIGURE 2.5.8 PHOTOMICROGRAPHS (200X) OF HASTELLOY-X BASE METAL CROSS-SECTION

A. AS-RECEIVED

B. 10,000-HOUR, 1400°F EXPOSURE

A_e AS-RECEIVED

B_e 10,000-HOUR, 1400°F EXPOSURE
FIGURE 2.5.9 PHOTOMICROGRAPHS - HASTELLOY-X
WELD AREA CROSS-SECTION

VACUUM SIDE

WELD NUGGET

AIR SIDE

(200X) WELD HEAT-AFFECTED ZONE

VACUUM SIDE

WELD NUGGET

AIR SIDE

(100X; ETCHED) WELD HEAT-AFFECTED ZONE
Cross-sections of typical original (before 1400°F exposure) weld areas, fabricated with materials and techniques identical to those used in making the welds shown in Figure 2.5.9, were also studied under 100X magnification. No evidence of porosity in the heat affected zone was noted on these original welds. This implies, at least, that the porosity in some way results from the 1400°F, long-term exposure and is not caused by the original welding operation.

The porosity problem and its adverse effect on strength are well illustrated. Its cause is not readily explained. It is felt that in-depth metallurgical investigation is required to gain the necessary insight into this phenomenon. This type of study would be beyond the scope of our current contractual efforts.
3.0 ACTIVITIES DURING NEXT REPORTING PERIOD

- Long-term tests on opacified multi-layer systems to be continued. The 10,000-hour testing on the copper opacified sample will be complete.

- Materials ordered and preparations made for modified composite tests using molybdenum and woven quartz materials in the 1800°F zone.

- Refinements to the empirical data correlation procedures for multi-foil and opacified multi-layer insulations will be completed.

- Test program on longitudinal thermal conductivity of opacified fibrous multi-layer insulations will be completed, and data will be correlated.

- Results from modified analytical program for "filled-gap" penetrations will be evaluated and compared to original data.
REFERENCES


5. DMIC Report 242, Defense Metals Information Center, Battelle Memorial Institute, Columbus, Ohio.
