THERMAL AND MECHANICAL PROPERTIES OF ALLOYS
AT ELEVATED TEMPERATURES

PROGRESS REPORT # 3

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By

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1. SUMMARY

During this reported period, we have accomplished 4 types of experimental measurements and obtained 5 sets of thermal and mechanical property data of 12 alloys at elevated temperatures. Each type of measurement involved development of a feasible experimental method to enable the small alloy specimens to be tested. The reported properties included thermal expansion coefficient at 225F, thermal conductivity at 150F, tensile elastic modulus at 225F, ultimate tensile strength and elongation-at-break at 482F.

The coefficient of thermal expansion of the alloys was measured using the Instron environmental chamber as a temperature control unit. The linear thermal expansion was monitored by a strain gage bonded onto the sample. The strain gage was connected to a strain indicator for taking readings. By collecting strain gage readings at different temperatures, coefficients of thermal expansion were determined.

The measurement of thermal conductivity involved measurements of the heat flux and temperature difference of 12 different alloys. A comparative cut bar method, which was suitable for the alloys of small dimensions, was developed by CFC for axial thermal conductivity testing. The principles of the measurement lay with passing the heat through a known metal and an unknown alloy sample and comparing the respective thermal gradients. The gradients were inversely proportional to the thermal conductivities of the alloys.

The mechanical characterization of the alloys at high temperatures such as 500F and 1050F posed several difficulties. In our previous report, we developed the required methodology for conducting tensile tests of the alloys at room temperature. However, operational and safety issues at high temperatures concerning the testing system, experimental procedure, and personal safety had to be taken into considerations. Some
technical solutions developed are: (1) specimen loading procedure for high temperature testing, (2) compensation method due to thermal expansion of the testing system, (3) test procedures for tension test at high temperatures, and (4) data correction method due to heating effect on load cell. The ultimate tensile strength and elongation-at-break data at 482F were reported herein for all the 12 alloys. The measurements to determine the ultimate tensile strength and elongation-at-break of alloys at 1050F are being conducted and the results will be reported in the final report.

The elastic modulus measurement at high temperatures was limited to a maximum temperature of 225F because the bonding system used to bond the strain gage onto the specimen would become ineffective above 225F.

The machining of different coupon specimens for the above tests constituted a substantial part of the work. All the test samples (12 alloys of 2 groups, 2 sets) were used to conduct the above tests.

The main results can be summarized as follows:

1). Most of the alloys have a coefficient of thermal expansion of about $2 \times 10^{-6}$ in/in/°F; i.e., these alloys have extremely low values of coefficient of thermal expansion.

2). Thermal conductivity varies from one alloy to another. Sample A5 appears to have the highest thermal conductivity, up to a value of 396 W/mK, but samples B4 and B2 have thermal conductivity as low as about 130 W/mK.

3). Most of the alloys have similar elastic modulus at 225F to those measured at 75F, giving an average value of about 24 mpsi. Samples A3 and B1 have relatively larger elastic modulus at 225F, of a value about 32 mpsi.

4). The alloys have lower ultimate tensile strength (UTS) at 482F as compared to those at room temperature. Sample A2 again has been identified the toughest alloy, giving an UTS of 135 ksi at 482F. Other alloys have UTS varying from 70 ksi to 95 ksi at 482F.
The alloys have nearly identical elongation-at-break at 482F, giving a nominal elongation-at-break of around 100%, except in sample A2. Sample A2 has a nominal elongation-at-break of 160%.

The following sections, in depth, discussed coefficient of thermal expansion, thermal conductivity, elastic modulus at 225F, ultimate tensile strength and elongation-at-break at 482F. This report not only presented the specific magnitudes of thermal and mechanical properties for each alloy, but also described the corresponding experimental methods specially developed by CFC for thermal and mechanical characterization of the alloys of small dimensions at elevated temperatures.

2. CHARACTERIZATION OF THERMAL EXPANSION COEFFICIENT

The coefficient of thermal expansion of a material is generally defined as the fractional increase in length per degree increase in temperature. The exact definition varies, depending on whether it is specified at a precise temperature (true coefficient of thermal expansion) or over a temperature range (mean coefficient of thermal expansion). Here, the coefficient of linear thermal expansion over a temperature range of 75-225F was determined for the alloys.

First, the experimental method and conditions, such as test specimen, temperature, and equipment, were determined. Then the method was verified by testing a known material with a known value of coefficient of thermal expansion. Once the method was verified and consistent for the known sample, the tests were conducted for the samples, provided by US DOE.

2.1 Test Specimens

Measurements of the coefficient of thermal expansion of alloys were made by use of the coupon specimens prior to their use for tensile tests at 482F. These alloys were of dog-bone shape and small in size. Figure 1 is a schematic diagram illustrating coupon specimens used in our testing. The broader ends of the specimen were inserted into the
grips. The test section of these specimens provided enough surface area for bonding the strain gages. The key dimensions of the specimens are reported in Table 1.

![Figure 1. A dog-bone shape alloy coupon specimen](image)

### 2.2 Experimental Method

A new method was developed to measure the coefficient of thermal expansion of the alloys on the Instron machine. The strain gages were bonded on the rectangular part of the sample. One end of the sample was mounted on the grips inside the Instron environmental chamber. The other end was pending in the space so that the test section area of the sample was subjected to uniform thermal expansion in a free space as the specimen gets heated. The degree of the expansion was monitored from the readings of a strain indicator. The coefficient of thermal expansion was calculated from the slope of the curve obtained from plotting the strain readings versus the temperature.

The test temperature ranged from 75\(^{0}\)F to 225\(^{0}\)F. The temperature chamber was allowed to stabilize at a particular temperature and then the readings of the strain indicator were noted. During the test, the temperature intervals chosen were 75\(^{0}\)F, 100\(^{0}\), 125\(^{0}\), 150\(^{0}\), 175\(^{0}\), 200\(^{0}\), 225\(^{0}\)F. At every interval the strain readings were taken. A plot of strains versus temperature was then obtained. The slope of the corresponding curve gave the coefficient of thermal expansion. This process was repeated for all the 12 samples of alloys. The measurements were limited to 225\(^{0}\)F because the bonding system would fail above 225F. It takes 3 hrs to conduct each test.
2.3 Thermal Expansion Coefficient of Alloys

The calculated thermal expansion coefficients for 12 alloys are listed in Table 2 and are shown in the form of bar graph in Figure 2. The value for sample A2 is not reported because the strain gage on the sample A2 got debonded, giving erroneous readings.

Most of the alloys have a coefficient of thermal expansion of about $2 \times 10^{-6}$ in/in/°F. Samples A5 and A6 have relatively higher values while samples A1 and A3 have slightly lower values. Thus, samples A5 and A6 will give rise to relatively larger thermal expansion than samples A1 and A3 under the same thermal history. The alloys in B-group have an average thermal expansion value of $2 \times 10^{-6}$ in/in/°F.

3. CHARACTERIZATION OF THERMAL CONDUCTIVITY

The thermal conductivity is defined as the amount of heat transmitted, due to unit temperature gradient, under steady conditions in a direction normal to a surface of unit area, when the heat transfer is dependent only on the temperature gradient. Its unit is
W/mK. This section reported the experimental method developed by CFC and used to determine the thermal conductivity of the alloys and the results obtained.

3.1 Test Specimens

The test specimens for the measurement of thermal conductivity comprised of a piece of well-machined rectangular alloy block welded to another block having the same shape, which was made of the known metal with a code of 4140H steel. Figure 3 is a schematic diagram illustrating the structure of a small test bar. All the 12 alloys were welded respectively to the reference blocks to form pieces of test bars having 2 different metals on either side. The dimensions of the test bars of all the 12 alloys are reported in Table 3.

![Figure 3. A schematic diagram showing the structure of the sample used for thermal conductivity measurements.](image)

3.2 Experimental Method

Due to small dimensions of the alloy samples, a new method was developed to measure thermal conductivity of the given alloys. A small alloy block was welded to another metal block with a known thermal conductivity and the thermal conductivity of the alloy was determined by using a comparative method. The reference metal used was
4140H steel. At a testing temperature of 150F, the thermal conductivity of 4140H is 42W/mK.

During the measurement, the whole specimen was kept inside a channel of the temperature chamber of the Instron machine. It was placed in such a manner so as to expose one end of the specimen (alloy) to room temperature and the other end (known metal) to a heat resource of constant temperature. The specimen was wrapped in Styrofoam in order to minimize heat loss in the radial direction through the surrounding surfaces. The entire heat flux was confined to axial flow through the reference and the test sample. The temperatures were measured using thermocouples attached to the exposed surface of the unknown alloy and the interface between the alloy and the reference.

In the experiment, the known metal was exposed to the temperature of 150°F provided by the Instron chamber and the alloy was exposed to the room temperature outside the machine. The temperatures at the bottom surface of the alloy sample and the interface were recorded as a function of time. It took about 2 hrs for temperatures to reach equilibrium values. The readings of the temperatures from the 2 thermocouples were taken every 10 minutes for the first 2hrs and then every 20 minutes for another 2 hrs. The measurements were limited to 150°F because the bond system of the thermocouples would fail at higher temperatures.

3.3 Data Analysis Method

Thermal conductivity is defined as

\[ K = \frac{Q/A}{\Delta T / \Delta L} \]

(1)

Where Q is the amount of heat passing through a cross sectional area, A, and causing a temperature difference, \( \Delta T \), over a distance of \( \Delta L \). \( Q/A \) is the therefore the heat flux which is causing the thermal gradient \( \Delta T / \Delta L \).
For the structure shown in Figure 3, where both the unknown sample and reference metal have the same sectional area, A, the heat flux can be determined as

\[
\frac{Q}{A} = K_s \frac{T_2 - T_1}{L_1} = K_r \frac{T_3 - T_2}{L_2}
\]  

(2)

Where \(K_s\) is the thermal conductivity of the unknown sample, \((T_2 - T_1)\) is the temperature difference between two ends of the unknown sample, \(L_1\) is the length of the unknown sample, \(K_r\) is the thermal conductivity of the reference metal, \((T_3 - T_2)\) is the temperature difference between two ends of the reference metal, \(L_2\) is the length of the reference metal. From Equation 2, thermal conductivity of the unknown sample can be calculated as:

\[
K_s = K_r \frac{T_3 - T_2}{T_2 - T_1} \times \frac{L_1}{L_2}
\]  

(3)

If the unknown sample has a slightly different sectional area \((A_1)\) than the reference metal \((A_2)\), thermal conductivity of the unknown sample can be calculated as:

\[
K_s = K_r \frac{A_2}{A_1} \frac{T_3 - T_2}{T_2 - T_1} \times \frac{L_1}{L_2}
\]  

(4)

### 3.4 Thermal Conductivity of Alloys

The calculated thermal conductivity data for the alloys are listed in Table 2 and are shown in the form of bar graph in Figure 4. The thermal conductivity of sample B5 was not obtained because there was a significant heat loss in the radial direction during the measurement due to poor insulation around the sample. The temperature at the bottom surface of the alloy was not reaching an equilibrium value.

The result is first presented in the form of ratios between the alloys’ thermal conductivity and the reference’s in Figure 4a. As seen in Figure 4a, sample A5 gives a ratio of 9.44, much higher than others. In order to make sure it is valid, we examined the following: 1) the thermocouples were mounted correctly on the sample and the temperature differences were properly measured. 2) The size of the test bar of sample A5
was among the large samples (see Table 3) and the measured temperature difference should reflect the temperature gap between two sides of the sample. 3) The dimensions of the sample were correctly measured. 4) The collected temperature data were sufficient and reliable, showing a very good trend. 5) There was no mistake in data analysis. Therefore, after an investigation of possible errors, we believe that sample A5 has the highest thermal conductivity.

Figure 4a. The ratio of the alloys’ thermal conductivity over the reference’ thermal conductivity at 150F

Figure 4a also shows that samples A2, A7, B1, and B3 have relatively higher thermal conductivity as compared with others except A5, while samples A6, B2 and B4 have lower thermal conductivity. Thermal conductivity of the reference metal (Kr) is 42 W/mK, to compute the values of thermal conductivities of the 12 alloys. These values are listed in Table 2 and are plotted in Figure 4b.

During the experiments, it was also observed that the rate of conduction of heat varied with the alloy. Some of the alloys, such as A3, A4, and B2, absorbed heat faster and then reached the equilibrium temperature more quickly than others, such as A1, A6, and B4.
4. CHARACTERIZATION OF TENSILE ELASTIC MODULUS AT 225F

The elastic modulus is determined by calculating the slope of the linear part of the stress-strain curve, which is obtained by taking strain gage readings under different loads. At small strain values (the elastic region), the relationship between stress and strain is nearly linear. Within this region, the slope of the stress-strain curve is defined as the elastic modulus, as schematically shown in Figure 5. This section reported our results on elastic modulus of alloys at 225F and the data are compared with those obtained at 75F.

4.1 Test Specimens

More coupon specimens were machined in the form of dog-bone shape (see Figure 1) for each alloy sample. Due to the irregular dimensions of the alloy samples, each sample was cut into specimens of its own dimensions. The relevant dimensions of the specimens are listed in Table 1.
4.2 Experimental Method

The experiment was carried out using the Instron machine model 8501 at a stretching rate of 0.005in/min. The strain gage was bonded to the sample at the rectangular portion. The sample was mounted in the grips. Figure 6 shows the experimental set up for tension tests. The sample had to be straight without any bend before mounting it into the grips. The mounting of the specimen was done in such a way as to enable maximum surface area to the grips, thus protecting the sample from sliding out of the grips during the experiment.

The test temperature was set at 225°F. Once the temperature inside the chamber was stabilized, the sample was stretched and the strain readings were taken. The readings were taken at steps of 10 pounds from 0 to 150 pounds. The load applied was limited to 150 pounds so as to maintain the readings in the linear region. The test temperature was limited to 225°F because above 225°F, the bond between the strain gage and the sample would fail.
A routine experimental operation consists of the following steps: 1) Calibrate the load cell at room temperature without samples. 2) Load the specimen into the grips. 3) Heat the chamber to the set temperature. 4) Start the test when the chamber reaches the temperature. 5) Take readings from the strain indicator at an increment of 10 lbs. 6) Stop the test when the load is over 150 lbs.

It was noticed that after the test lasted more than 600s, or another test was continuously conducted after one test, the load cell got warm and gave higher load readings than expected. This phenomenon was further observed during the tests at 482F. So precaution was taken during the measurements. Otherwise corrections of load cell readings had to be made.
4.3  Tensile Elastic Modulus of Alloys at 225F

The tensile elastic moduli of the alloys at 225F are listed in Table 2. There are no valid data for samples A5 and B3 because the strain readings were not correctly collected due to loss of bonding of strain gages during the experiments. For a better comparison among the alloys tested, the data are also plotted in the form of bar chart as presented in Figure 7. The result shows that samples A3 and B1 have the larger elastic modulus at 225F while samples A2 and A6 have the smaller elastic modulus.

![Figure 7. The elastic modulus of alloys at 225F](image)

In Figure 8, the elastic modulus data of the alloys at 225F are compared with the data obtained at 75F, which were reported in the project report #2. For most of the alloys, they have similar elastic modulus at 225F and 75F. This is expected because such a temperature difference shouldn’t affect the elastic modulus very much. But the difference between two sets of elastic modulus data may be ascribed to the following reasons:
1). Experimental errors: the system error, reading errors in load cell and strain gage indicator, effectiveness of bonding between the gage and the alloy surface, etc.

2). Measurement errors in dimensions of the specimen to determine cross-sectional area due to its small size.

3). Effects of extra loads caused by the load cell at elevated temperatures as discussed in the next section. For some tests where these effects were noticeable, corrections were already made during calculation.

In fact, multi-run tests demonstrated that the results had a good reproducibility. For example, two runs of tests at 225F gave elastic modulus of values of 33.04 mpsi and 33.06 mpsi to sample A3; 31.52 mpsi and 31.68 mpsi to sample B1. A more appropriate value of elastic modulus of one alloy sample may be obtained by taking average of these two measurements at 225F and 75F. In any means, sample A3 has the highest elastic modulus among the 12 alloys.
5. CHARACTERIZATION OF TENSILE PROPERTIES AT 482F

Tensile tests of the alloys at 482F were conducted on the Instron machine to determine ultimate tensile strength and elongation-at-break properties of the alloy at 482F. The results are reported in the following section and compared with the data obtained at 75F.

5.1 Test Specimens

The same set of coupon specimens of the alloys were used to run tensile tests at 225F for the determination of elastic modulus and at 482F for the determination of ultimate tensile strength. These were two separate batches of measurements. The former was limited to a maximum load of 150 lbs where strain readings were taken at each load increment of 10 lbs beginning from 0 lbs. The later was allowed to stretch the coupon until it broke at a load about 1000 lbs. The relevant dimensions of the specimens tested at 482F can be found also in Table 1.

5.2 Experimental Method

The test method developed previously for tensile testing at 75F was applied to determine tensile yield strength at 482F. However, testing at high temperatures differed from the testing at room temperature in the following aspects:

1) We were not able to bond any strain gage onto the surface of a specimen to measure true strain during extension at such a high temperature as 482F.

2) It would be very difficult and also dangerous to load a specimen in the grips at 482F. Thus the sample was pre-loaded in the grips before the environmental chamber was heated.

3) The sample and grips were going to expand as they were being heated. This might get the sample bend and even damage the machine. Extra precaution was needed in the course of an experiment.
4) The thermal expansion of the grips would generate a compression force in the pre-loaded sample as the sample is being heated and before the test begins. It was necessary to release this compression force properly by adjusting the position of the bottom pull-rod from time to time.

5). Since the grips and pull-rod were all made of metallic materials and heat was gradually conducted through the pull-rod to the load cell, the load cell would get quite hot during the testing. It was found that this would make the load cell misread the load, i.e., higher than true value.

6) We could identify the shift in load cell readings as a function of time, due to heat, by running a test without loading any sample. The shift in load cell readings could also be detected at the end of a test by recording the load after specimen rupture.

7) The load versus strain data needed to be corrected for the load cell shift before calculating the ultimate tensile strength.

8) A 5hr interval was needed between two tests because the load cell and hydraulic system became very hot and had to be cooled before running another test. The calibration of the load cell must be done at room temperature.

All tests reported herein were conducted on Instron 8500 at 482F and at a stretching rate of 0.005in/min, using position control mode.

5.3 Ultimate Tensile Strength of Alloys at 482F

Ultimate tensile strength is the maximum stress a material can withstand before failing. The ability of a material to resist breaking under tensile stress is one of the most important and widely measured properties in structural applications.

The ultimate tensile strength data of the 12 alloys at 482F are listed in Table 2. For a better comparison among alloys, these data are also plotted in the form of bar chart as presented in Figure 9. The results show that A2 sample has the highest ultimate strength at 482F and samples A1, A3, and B5 are among the lowest. Except B5 sample, other alloys in B-group have better ultimate strength than most of alloys in A-group.
Figure 9. The ultimate tensile strength of alloys at 482F

Figure 10. The comparison between the ultimate tensile strength of alloys at 482F and 75F
The ultimate tensile strength data of 12 alloys at 482F are also compared with data obtained at 75F, which were reported in the project report #2. The two sets of data were plotted in Figure 10. The result shows that the ultimate tensile strength values of nearly all the alloys are more or less reduced at 482F from their corresponding values at 75F. The general trends of two sets of data are similar. Sample A2 has the highest ultimate tensile strength at both 482F and 75F. There are two exceptions: 1) The ultimate tensile strength of sample B5 is most significantly reduced from a value of 101.8 ksi at 75F to that of 70.73 ksi at 482F. 2) On the other hand, sample A4 appears to have the least change in its ultimate tensile strength.

5.4 Elongation-at-Break of Alloys at 482F

The nominal elongation-at-break data of the 12 alloys at 482F are listed in Table 2. For a better comparison among alloys, these data are also plotted in the form of bar chart as presented in Figure 11. The result shows that A2 sample has the best extension. A5 sample exhibits the least extension because this sample had been subjected to bending deformation prior to the tensile testing. All others have nearly identical elongation-at-break behaviors.

Figure 11. The elongation-at-break of alloys at 482F
The nominal elongation-at-break data of the 12 alloys at 482F are also compared with the data obtained at 75F, which were reported in the project report #2. Two sets of data are plotted in Figure 12. An obvious feature is that for these alloys, the elongation behaviors are much diverse at 75F, but the elongation behaviors tend to become very similar and identical at 482F. It is interesting to note that most of the alloys show slightly reduced elongation-at-break at 482F as compared with the values at 75F. However, for those whose elongation-at-break at 75F are relatively small, for example, samples A6, B1, and B3, they exhibit larger elongation-at-break at 482F. Sample A2 has significantly larger elongation-at-break than others at 482F and 75F.

Due to the micro-slip phenomenon discussed in the previous report, these elongation-at-break data may not completely represent actual extension abilities of alloys. Since these specimens were tested at the same conditions, these data are reported for comparison purpose. The actual extensions should be a bit smaller.
6. CHARACTERIZATION OF TENSILE PROPERTIES AT 1050F

More tensile tests of alloys are being conducted at a temperature of 1050F. The measurements will determine tensile yield strength and nominal elongation-at-break properties of alloys at 1050F. The results will be reported in the next report and compared with data obtained at 482F and 75F.
Table 1. The dimensions of alloy coupon specimens for tension tests.

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Material</th>
<th>Test section length (in)</th>
<th>Test section width (in)</th>
<th>Thickness (in)</th>
<th>Cross section area (in²)</th>
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<tbody>
<tr>
<td>A-1</td>
<td>Fe3Al Clad Stain</td>
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<td>0.274</td>
<td>0.0378</td>
<td>0.01036</td>
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<td>Thermie Alloy</td>
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<td>A-3</td>
<td>H Mod 80017433</td>
<td>0.210</td>
<td>0.272</td>
<td>0.034</td>
<td>0.009248</td>
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<td>A-4</td>
<td>Incolaid 671</td>
<td>0.096</td>
<td>0.198</td>
<td>0.036</td>
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<tr>
<td>A-5</td>
<td>347-FG</td>
<td>0.200</td>
<td>0.257</td>
<td>0.032</td>
<td>0.008224</td>
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<tr>
<td>A-6</td>
<td>Save 25</td>
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<td>0.2</td>
<td>0.036</td>
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<td>A-7</td>
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<td>0.249</td>
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<td>0.263</td>
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Table 2. Thermal and mechanical properties of alloys at elevated temperatures.

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Material</th>
<th>Coefficient of thermal expansion at 225F (in/in/F)</th>
<th>Thermal conductivity at 150F (W/mK)</th>
<th>Young’s modulus at 225F (mpsi)</th>
<th>Ultimate tensile strength at 482F (ksi)</th>
<th>Nominal elongation-at-break at 482F (%)</th>
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<tbody>
<tr>
<td>A-1</td>
<td>Fe3Al Clad Stain</td>
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<td>272.2652</td>
<td>*</td>
<td>95.16</td>
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<td>310-HCBN</td>
<td>2.12E-6</td>
<td>**</td>
<td>27.39</td>
<td>70.73</td>
<td>96</td>
</tr>
</tbody>
</table>

*No valid data. Strain values are underestimated due to loose bonding of strain gages.

** No valid data. Heat loss occurs in the radial direction due to poor sample insulation.
Table 3. The dimensions of alloy cut bars for thermal conductivity testing.

<table>
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<tr>
<th>S. No.</th>
<th>Alloy</th>
<th>Reference metal</th>
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<td>Width (in)</td>
<td>Thickness (in)</td>
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<td>0.368</td>
<td>0.351</td>
</tr>
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<td>A-2</td>
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<td>0.356</td>
</tr>
<tr>
<td>A-3</td>
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<td>0.366</td>
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<td>A-4</td>
<td>0.24</td>
<td>0.224</td>
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<tr>
<td>A-5</td>
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<td>0.366</td>
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<tr>
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<td>A-7</td>
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<tr>
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<tr>
<td>B-5</td>
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</tr>
</tbody>
</table>
For further information, please contact:

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Fax: 304-293-7109