# ULTRA HIGH TEMPERATURE ISOTHERMAL FURNACE LINERS (IFLS) FOR COPPER FREEZE POINT CELLS

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**Abstract.** Primary Laboratories use large fixed-point cells in deep calibration furnaces utilizing heat pipes to eliminate temperature gradients. This combination of furnace, heat pipe, and cell gives the smallest of uncertainties. The heat pipe, also known as an isothermal furnace liner (IFL), has typically been manufactured with Alloy 600/601 as the envelope material since the introduction of high temperature IFLs over 40 years ago. Alloy 600/601 is a widely available high temperature material, which is compatible with Cesium, Potassium, and Sodium and has adequate oxidation resistance and reasonable high temperature strength. Advanced Cooling Technologies, Inc. (ACT) Alloy 600/Sodium IFLs are rated to 1,100°C for approximately 1,000 hours of operation (based on creep strength). Laboratories interested in performing calibrations and studies around the copper freeze-point (1084.62°C) were frustrated by the 1,000 hours at 1,100°C limitation and the fact that expensive freeze-point cells were getting stuck and/or crushed inside the IFL. Because of this growing frustration/need, ACT developed an Ultra High Temperature IFL to take advantage of the exceptional high temperature strength properties of Haynes 230.

**Keywords:** Heat Pipe, Haynes 230, Isothermal Furnace Liner, IFL, Copper Freeze Point Cell, Alloy 600, High Temperature, Calibration

## INTRODUCTION

Primary Laboratories use large fixed-point cells in deep calibration furnaces to eliminate temperature gradients. Heat pipes, also known as Isothermal Furnace Liners (IFLs), have been used for decades as part of the calibration furnace to achieve the smallest of uncertainties. The various heat pipes, operating temperatures, and fixed-point elements follow:

- Water (50 to 270°C); Gallium and Indium
- Cesium (300 to 600°C); Tin and Zinc
- Potassium (400 to 1000°C); Aluminum
- Sodium (500 to 1100°C); Silver and Copper

The choice of heat pipe working fluid depends on the desired operating temperature. The properties of the working fluid must allow for optimum heat transfer. Another concern is the internal pressure of the heat pipe. The working fluid inside of a heat pipe is at saturation conditions. Therefore, the internal pressure of the heat pipe is the vapor pressure of the working fluid, which is a function of the operating temperature. As the temperature increases the internal pressure increases.

As the temperature increases, the stress load on the heat pipe envelope also increases. Unfortunately, as the temperature increases, the strength of the envelope material is decreasing. The standard practice for nearly 40 years of IFL manufacturing has been to use Alloy 600/601 as the heat pipe envelope material and to accept a short life when operating at the high end of the temperature range. For example, sodium / Alloy 600 IFLs operated at close to 1100°C often show measurable deformation after as little as a few hundred hours and significant deformation at about 1000 hours.

Over the past decade, the interest in copper freeze point cells has increased and more and more researchers are operating their sodium IFLs near 1100°C. As reports of IFL deformation and trapped and broken fixed point cells increased, it became clear that the standard Alloy 600/601 / sodium IFLs were not strong enough to meet the needs of the researchers that wanted to do work at the copper point.

Heat pipe developers in other industries, such as the solar thermal industry, which also requires heat pipes to withstand high temperature and high stress levels, were investigating alternative superalloy materials. Haynes 230 was one of the most promising alloys identified. Haynes® 230® alloy (57Ni-22Cr-14W-2Mo-La) is a more highly alloyed nickel base alloy than alloy 600 (72Ni-15Cr-8Fe). Therefore, it is expected to have greater solid solution strengthening. Alloy 230 was designed specifically for high temperature strength, while Alloy 600 was designed for corrosion resistance at lower temperatures.

Haynes 230 has a set of properties and characteristics that make it one of the best candidates for high temperature heat pipes. It has high yield strength and high creep strength. It is relatively easy to fabricate complex shapes and to join using conventional welding techniques such as gas tungsten arc welding (GTAW). Several Haynes 230/sodium heat pipes had been successfully demonstrated for other applications, proving compatibility. Based on these promising material properties, the ultra-high temperature IFL for copper freeze point cells utilizing Haynes 230 as the envelope material was developed.

## ISOTHERMAL FURNACE LINERS AND FREEZE POINT CELLS

Figure 1 is a schematic representation of a freeze point cell inside of an isothermal furnace liner. The freeze point cell contains ultra-pure copper and has a well to accept a high temperature platinum resistance thermometer (HTPRT). The freeze-point cell and HTPRT are lowered into the IFL. The IFL is heated on the exterior by electrical resistance heaters. The entire package is well insulated.

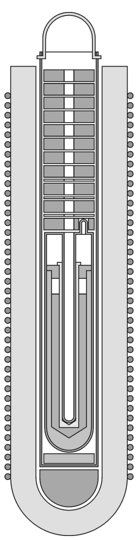

**FIGURE 1.** Schematic representation of freeze-point cell inside of an Isothermal Furnace Liner (IFL).

In operation, the electrical resistance heaters are powered and controlled to warm up the freeze-point cell to the melting point of copper. Once melting begins, the additional energy input changes the copper from solid to liquid phase. During this period, the temperature of the cell should remain constant at the melting point. Once all of the copper has melted, the temperature will begin to rise again. At this point, the power is reduced to allow for cooling and the temperature will again be constant during the liquid to solid phase transition. It is during these transition or plateau periods that the temperature of the HTPRT is calibrated to the melting point of copper. The HTPRT can then be used to calibrate other sensors by comparison. By extending the melting and freezing plateau time, the uncertainty of the measurements is minimized. A plot of a typical melt plateau is shown in Figure 2. Melt and freeze plateaus of five (5) to twenty (20) hours are typical.

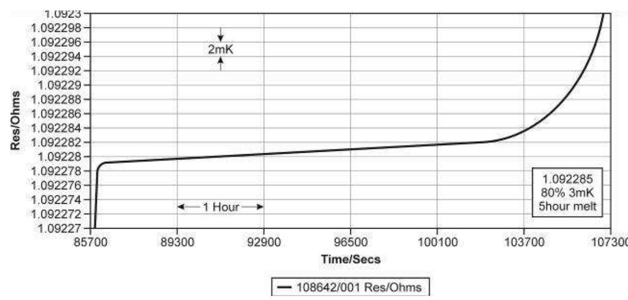


**FIGURE 2.** Typical melt plateau plot. The resistance (Ohms) of the HTPRT is directly proportional to temperature. In this chart, one milli-Kelvin is 0.000001 Ohms.

## HEAT PIPE STRESS LEVELS

As discussed above in the introduction, the internal pressure of the heat pipe is set by the vapor pressure of the working fluid. Typical high temperature working fluids include cesium, potassium, sodium, and lithium. The vapor pressure curves for each of these working fluids is shown in Figure 3 below.

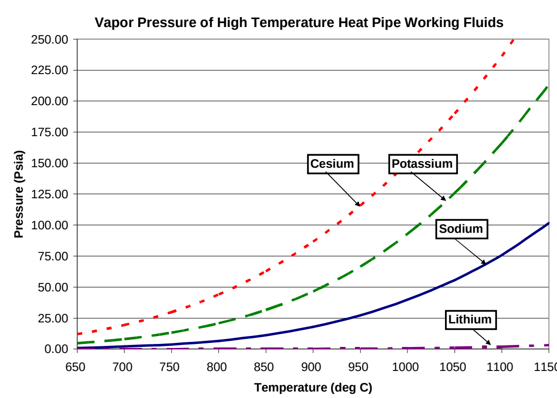


**FIGURE 3.** Vapor pressure curves for typical high temperature heat pipe working fluids.

In the case of copper freeze point cells that operate at about 1085°C, sodium is the working fluid of choice. While the selection of heat pipe working fluid for a particular operating temperature is based on all of the thermal and physical properties, a good rule of thumb for vapor pressure is that it should be above 0.1 atmospheres (1.5 psia) on the low end and not so high that the envelope is under high stress at the high end. Compared to sodium, the vapor pressure of potassium is significantly higher, which would result in higher stresses at the hot end. While lithium is near the lower limit of vapor pressure at 1100°C, it is much more corrosive than sodium and is not compatible with nickel-based superalloys. In other words, the life of the lithium heat pipe would be limited by corrosion through the envelope wall or welds.

The vapor pressure for sodium, as with most materials, increases exponentially with temperature. The temperature at which the internal pressure is equal to the external atmospheric pressure is approximately 893°C (normal boiling point). Above this temperature, the internal pressure is greater than the external pressure and this applies a hoop stress in the outer cylindrical envelope wall.

The hoop stress ($\sigma_{hoop}$) is a function of the internal pressure (P) and the envelope inner radius (r) and wall thickness (t). In the most simplified case, an infinitely long cylinder with internal pressure, the hoop stress can be calculated by the following equation;

$$\sigma_{hoop} = Pr/t$$

Isothermal furnace liners have typically been designed using Schedule 40 pipe for the inner and outer envelope walls. Common sizes are in the 1½ to 6 inch range. Tables 1 and 2 show the hoop stress for IFLs operating at 1100°C and 1150°C. At 1100°C, the hoop stress is on the order of 400 to 500 psi or 0.4 to 0.5ksi. Table 2 is included to demonstrate how quickly the stress level increases over the next 50°C (42% higher).

**Table 1.** Hoop Stress for Sch 40 IFL Envelope at 1100C

| **Sodium at 1100C (Vapor Pressure = 61.2 psi)** | | | |
|---|---|---|---|
| **Pipe Size (Sch 40)** | **ID (in.)** | **Wall t (in.)** | **Hoop Stress (psi)** |
| | | | |
| 1 1/2 | 1.61 | 0.145 | **340** |
| 2 | 2.07 | 0.154 | **411** |
| 2 1/2 | 2.47 | 0.203 | **372** |
| 3 | 3.07 | 0.216 | **435** |
| 3 1/2 | 3.55 | 0.226 | **480** |
| 4 | 4.03 | 0.237 | **520** |
| 5 | 5.05 | 0.258 | **599** |
| 6 | 6.07 | 0.28 | **663** |

**Table 2.** Hoop Stress for Sch 40 IFL Envelope at 1150C

| **Sodium at 1150C (Vapor Pressure = 86.9 psi)** | | | |
|---|---|---|---|
| **Pipe Size (Sch 40)** | **ID (in.)** | **Wall t (in.)** | **Hoop Stress (psi)** |
| | | | |
| 1 1/2 | 1.61 | 0.145 | **482** |
| 2 | 2.07 | 0.154 | **583** |
| 2 1/2 | 2.47 | 0.203 | **528** |
| 3 | 3.07 | 0.216 | **617** |
| 3 1/2 | 3.55 | 0.226 | **682** |
| 4 | 4.03 | 0.237 | **738** |
| 5 | 5.05 | 0.258 | **850** |
| 6 | 6.07 | 0.28 | **941** |

## ALLOY 600 VERSUS HAYNES 230

Isothermal furnace liners, used for the freeze point calibration application, operate for extended periods at high temperatures. Over time, the constant stress resulting from the internal pressure will cause the envelope material to strain. Therefore, in addition to evaluating the pressure induced stress against the yield strength, it is also important to evaluate against the creep strength.

As mentioned previously, the typical stress level from internal pressure is on the order of 500psi. Table 3 shows the yield strength comparison and Table 4 shows the creep strength comparison between Alloy 600 and Haynes 230. Figure 4 is a creep strength comparison that Haynes International uses to show the improvement versus various common superalloys including Alloy 600.

**Table 3.** Yield Strength - Alloy 600 versus Haynes 230

| **Yield Strength @ 0.2% Offset (ksi)** | | |
|---|---|---|
| **Temp (°F/°C)** | **Alloy 600** | **Haynes 230** |
| 1800 / 982 | 4.0 | 17.0 |
| 2000 / 1093 | not reported | 9.1 |

**Table 4.** Creep Strength - Alloy 600 versus Haynes 230

| | **0.5% Creep Strength (ksi) in 1000Hours** | |
|---|---|---|
| **Temp (°F/°C)** | **Alloy 600** | **Haynes 230** |
| 1700 / 927 | 1.50 | 3.40 |
| 1800 / 982 | 0.68 | 1.60 |
| 2000 / 1093 | 0.40 | 0.55 |

Table 3 shows that the Haynes 230 alloy has over four times the yield strength of Inconel 600. Table 4 shows the creep strength advantage. The other interesting thing to note is that the creep strength of Alloy 600 operating near 1100°C is less than or equal to the calculated hoop stress levels, which is consistent with the creep deformation that has been observed. While the Haynes 230 is tougher, the rapid decline in strength above 1100°C highlights the importance of precise temperature controllers and over-temperature safety cutouts in the case of primary controller failure for all IFLs.

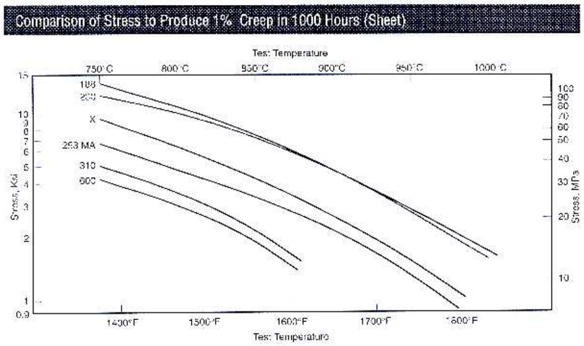


**FIGURE 4.** Stress to produce 1% creep in 1000 hours. Reprinted from Haynes International's Haynes 230 Product Brochure (www.haynesintl.com)

The data clearly shows that the Haynes 230 IFL will be stronger than the Alloy 600 version. However, the amount of data and the number of samples that have been run at these temperatures is small. Therefore, it is not simply a linear extrapolation to estimate expected life. The life of the IFL is still a complex function of the time and temperature history and the amount of deformation that can be tolerated.

With regards to field experience, the ultra high temperature Haynes 230/Sodum IFL was first prototyped in 2005 and regular production shipments started in 2007. These units are more expensive than Alloy 600 units; and therefore, they are typically only purchased by researchers working at the highest temperatures, in other words, at the copper point. To date, no field failures or negative feedbacks have been received.

## EXAMPLE AND EXPERIENCE WITH A PRODUCTION CALIBRATION DEVICE

This section of the paper will present the experiences of a first prototype ultra-high temperature IFL in a production-like calibration device. Isothermal Technology Ltd. has patented the integration of the furnace, heat pipe, and freeze-point cell in their Isothermal Towers, where the fixed-point cell and heat pipe have been combined to produce the ideal realizations for calibrating standard thermometers. The Isothermal Tower will be described and results from the initial copper fixed-point cell feasibility studies will be presented.

The Isothermal Tower, shown below in Figure 5, combines the fixed-point cell and the heat pipe into one assembly by building the copper fixed-point device directly inside of the heat pipe versus the standard practice of sliding a fixed-point subassembly into the heat pipe. The new configuration is expected to achieve very low uncertainties, be very repeatable, and less expensive. A cross sectional view of the Isothermal Tower is shown in Figure 6.

**FIGURE 5.** High temperature Isothermal Tower

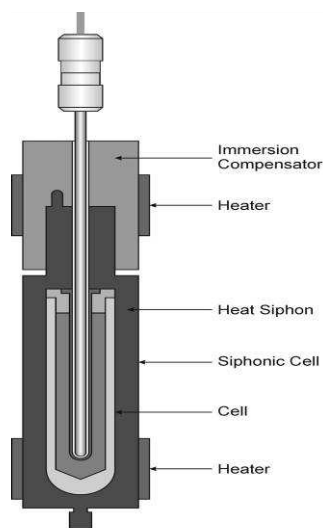


**FIGURE 6.** Cross-sectional view of the Isothermal Tower with integral copper fixed-point cell.

The first Haynes 230 IFL / copper fixed point Isothermal Tower was assembled as follows:

1. A pre-assembled ingot of pure copper surrounded by a pure graphite crucible was placed inside the IFL

2. An assembly made of Haynes 230 comprising of a re-entrant tube, a lid and an extension tube that allowed the copper cell to be flushed with pure argon was lowered into IFL.
3. After flushing, the re-entrant tube was permanently sealed.
4. A heated block was placed around the upper neck of the IFL to compensate for top-end losses.

The main heater heated the IFL while a secondary controller, with feedback from the primary controller, kept the area above the IFL at the same temperature. The purpose of the assembly was to melt and freeze the copper cell for calibration purposes.

To evaluate the assembly it was soaked at 1070°C for over 2,000 hours after which time the IFL was separated and compared dimensionally with an unused IFL. No measureable deformation or corrosion had taken place. A previous Alloy 600 design demonstrated measurable deformation after as few as 200 to 500 hours. A photograph of the ultra-high temperature IFL after 2000 hours of operation is shown in Figure 7. The IFL to the right is a twin that has not been operated yet.

**FIGURE 7.** There is no sign of deformation or corrosion on the ultra-high temperature IFL (left side) after being operated for over 2000 hrs at 1070°C. For comparison, a second ultra-high temperature IFL is shown (right side) with zero hours of operation.

Next, the assembly was subjected to another 800 hours at the copper point while a number of melts and freezes were performed.

Before testing the IFL and top heater were profiled as follows:

1. A PR thermometer (especially designed for the purpose) was used to measure the temperature inside the cell. The temperature of the immersion compensator (top heater) was set to 5°C lower than the copper melt temperature of 1084.62°C.
2. The thermometer was raised 100mm and the top heater was adjusted until the temperature was the same as the fully immersed temperature.
3. The gradient along the bottom 100mm could be adjusted to be just a few mK.

Next a series of melts and freezes were performed to assess the copper cell, the thermometer and the IFL. The IFL/top heater combination behaved perfectly and the copper cell could be melted and frozen over a long period of time. Typical copper cell melt and freeze curves are shown in Figures 8 and 9. The IFL was able to keep such a small temperature profile that the slope of the melt and freeze could be measured within a milliKelvin accuracy.

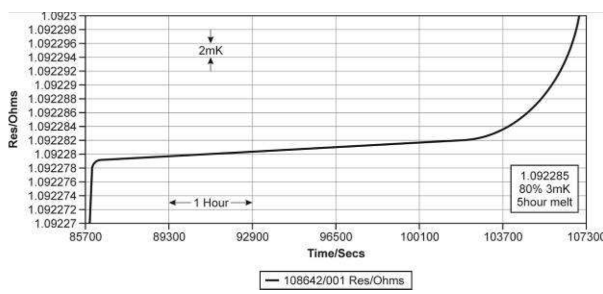


**FIGURE 8.** Copper IsoTower Melt Plateau – 02-24-2010

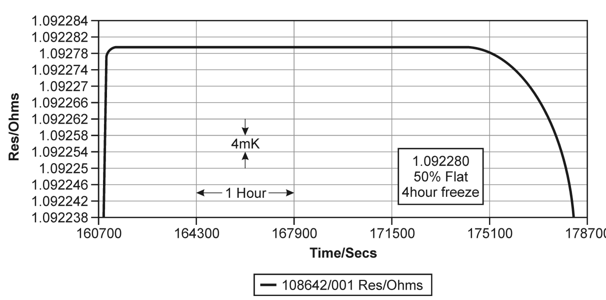


**FIGURE 9.** Copper IsoTower Freeze Plateau – 02-25-2010

After a total of 3000 hours operation at or about the copper point, the heat pipe was damaged by an electrical arc from the heating element to the heat pipe envelope. The heat pipe was inspected and there was no sign of mechanical deformation.

## CONCLUSIONS

A Haynes 230 / Sodium heat pipe, isothermal furnace liner (IFL), has been developed to operate at the copper point. Haynes 230 is significantly stronger at high temperatures than the typical Alloy 600 material that has been used for nearly four decades. Production units have been in the field since 2007 with no field failures or negative feedbacks. Alloy 600 pipes have failed or shown signs of significant deformation after several hundred hours of operation at the copper point. The Haynes 230 / Sodium heat pipe highlighted in this paper, operated for over 3000 hours with no sign of deformation.

Researchers that are doing work at the copper point should use the ultra-high temperature Haynes 230 / Sodium IFL heat pipe to achieve long, trouble-free operation.

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