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DESIGN CONSIDERATIONS FOR HEATED WELLS IN GLOVEBOXES

Abstract: Heated wells in gloveboxes have been used for many years by the Argonne National Laboratory Chemical Technology Division for nuclear-technology, waste-management, chemical-technology, and analytical-chemistry research. These wells allow experiments to be isolated from the main working volume of the glovebox. In addition, wells, when sealed, allow experiments to be conducted under pressurized or vacuum conditions. Until recently, typical maximum operational temperatures were about 500°C. However, more recent research is requiring operational temperatures approaching 900°C. These new requirements pose interesting design challenges that must be resolved. Some problem areas include temperature effects on material properties, maintaining a seal, cooling selected areas, and minimizing stresses. This paper discusses issues related to these design challenges and the ways in which these issues have been resolved.

INTRODUCTION

Heated wells in gloveboxes have been used for many years by the Argonne National Laboratory Chemical Technology Division (ANL CMT) for nuclear-technology, waste-management, chemical-technology, and analytical-chemistry research. The primary function of heated wells is to provide housings for various high-temperature processes. These wells also provide a number of other advantages for glovebox applications. Heated wells

- allow experiments to be isolated from the main working volume of the glovebox
- when sealed, allow experiments to be conducted under pressurized or vacuum conditions
- allow experiments to be run under controlled conditions, other than those of the glovebox environment
- provide containment for liquids, gases, and other materials
- provide overhead clearance for the insertion and removal of equipment

TYPES OF HEATED WELLS

Heated wells are attached to the underside of gloveboxes and are a continuation of glovebox containment. They may have the glovebox environment or contain a unique environment. Typically, the well is the outer containment, and liners and vessels are used to house the actual experimental and processing work. Information on basic well design and installation is given in Reference 1.

There are two types of heated wells, internally heated wells and externally heated wells. Both types offer a variety of advantages.

DISCLAIMER

Internally Heated Wells

In internally heated wells, the heating elements are installed inside the well. An example of an internally heated well is shown in Figure 1. Some advantages of this configuration are the following:

- Higher operational temperatures can be achieved.
- Heating of well wall can be avoided and, thus, material strength issues associated with elevated temperatures can be avoided.

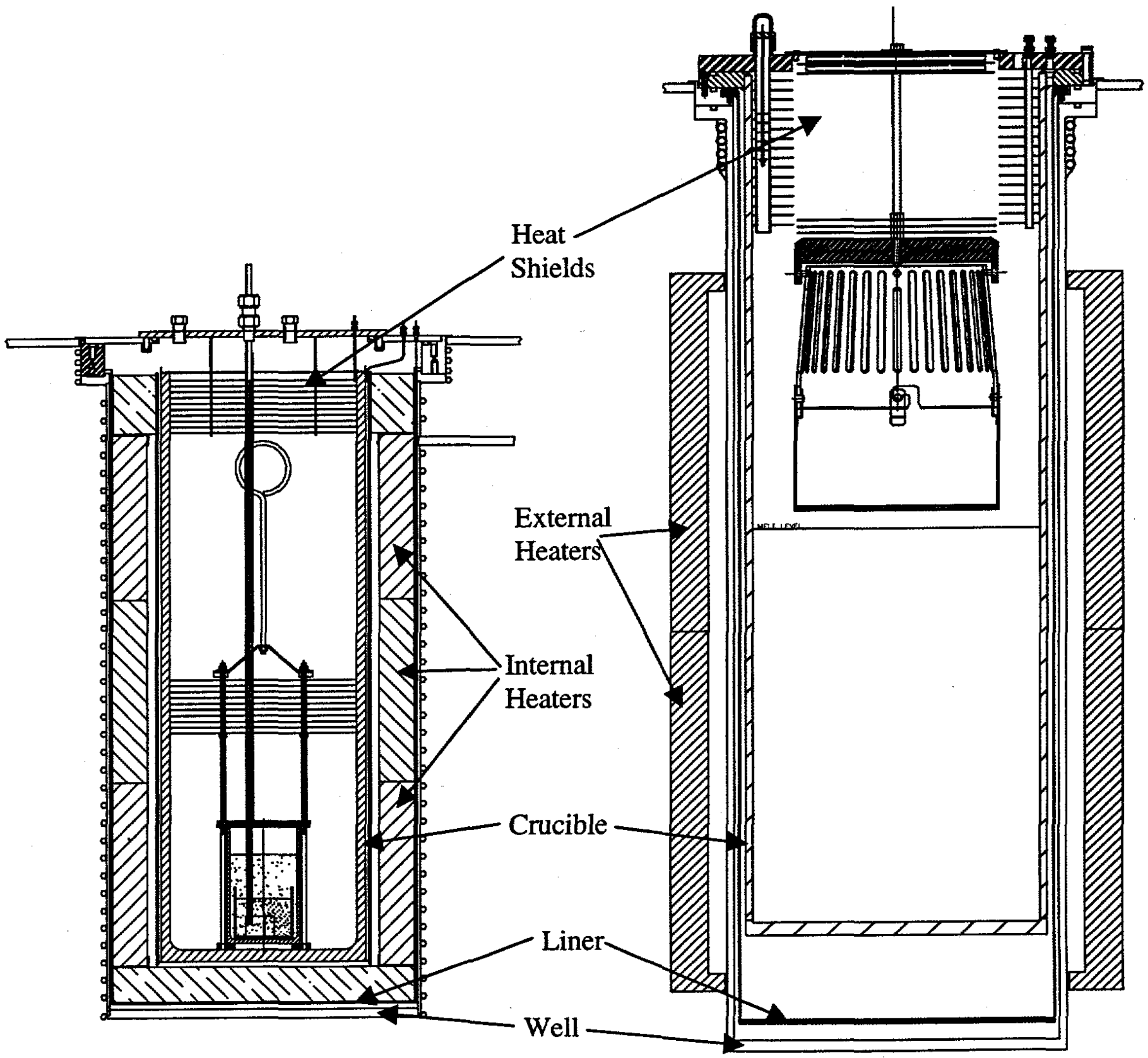

FIGURE 1. INTERNALLY HEATED WELL

FIGURE 2. EXTERNALLY HEATED WELL

Externally Heated Wells

In externally heated wells, the heating elements are installed on the outside of the well. A typical externally heated well is shown in Figure 2. Some advantages of this configuration are the following:

- Heaters do not occupy well volume and, thus, the entire well volume can be utilized.
- Heaters can easily be replaced.
- Off-gassing of ceramic heaters is not a concern.
- Power to the heaters does not require penetrating the glovebox.

In designing a heated well, the design requirements and features should be noted and reviewed so that the type of well most suited to the application can be chosen. The design challenges related to that type of well can then be addressed.

DESIGN CHALLENGES

Advancing research increasingly requires the ability operate experiments at temperatures approaching 900°C. Operating at these higher temperatures poses a number of challenges in designing heated wells. Some common challenges that arise when designing heated wells include selecting materials for their performance under process conditions, maintaining the desired temperatures, maintaining an effective seal, cooling selected areas to protect seals and equipment, and minimizing induced stresses within the well.

Material Selection

Elevated temperatures can have considerable effects on material properties. Most notably, the tensile and ultimate strength, ductility, impact strength, and corrosion resistance of most materials change considerably with temperature. The temperature effects on the yield strength of two materials are shown in Figure 3. At higher temperatures, "creep" also becomes an issue. Creep is the continuing deformation of a material, under constant load, over time at elevated temperature. Some materials may become more sensitive to corrosion at elevated temperatures. For example, stainless steels tend to "sensitize" in certain elevated temperature ranges. They may become embrittled and more susceptible to failure. Also, the properties in heat-affected zones of welds and the properties of weld filler metals may need to be taken into account. These welds may be considerably weaker than the parent material itself.

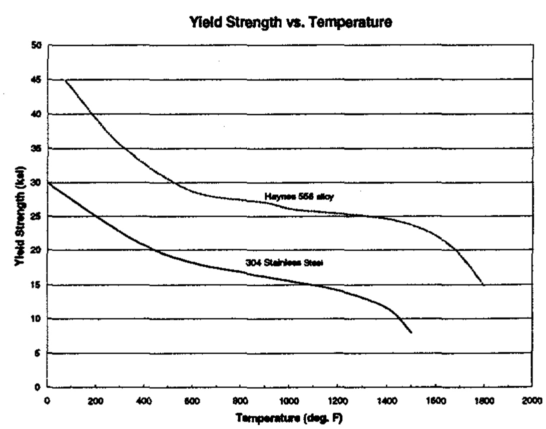

FIGURE 3. TEMPERATURE EFFECT ON YIELD STRENGTH

Some materials can handle these higher temperatures better than others. These materials are commonly referred to as **high-temperature, high-strength alloys**. Some of the materials that are used at ANL CMT for high-temperature wells are Inconel, Hastelloy, and Haynes 556 alloy. For example, Figure 3 compares the yield strengths of Haynes 556 and 304 stainless steel.

It is also very important to consider the environment to which the well will be exposed. Many metals tend to oxidize in air. Materials that the well may be required to contain may also have corrosive effects on the well. These corrosive effects may, on some metals, greatly accelerate the corrosion process. Therefore, material selection should consider the corrosive influences of the environment that the well is exposed to.

Often, liners, vessels, and crucibles are used to protect the well from harmful reactants. However, even when this equipment is sealed from the well, there is still potential for gases and other contaminants to pass the seal and instigate the corrosion process.

Therefore, since the well itself is the primary barrier between the glovebox environment and the outside, proper material selection is a vital design decision. Care should be taken in selecting the construction materials for the well to ensure that they can operate at the desired temperatures and that they are compatible with the materials that they are designed to contain.

Heat Management

Maintaining the desired temperature can also be a problem. If the heated well is not designed properly, too much heat may be lost to the environment and the well will not be able to maintain a steady-state temperature. Therefore, in the design of heated wells, it is most important to minimize the heat-transfer paths to the environment. There are a number of ways to reduce the heat transfer to the environment.

- Use insulation on the outside of the well. High-temperature ceramic insulating materials (blankets and boards) come in a variety of shapes and thicknesses and work very well in reducing heat loss from wells.
- Use heat shields to prevent heat transfer to the glovebox. Heat shields can be described as an arrangement of thin, properly spaced metal sheets that reduce radiation heat transfer. For more information on heat shielding, see Reference 2.
- Minimize the conduction paths to the glovebox. Well and liner walls provide paths for heat to conduct to the glovebox floor. Experimental equipment inserted into the well may also provide heat conduction paths to the glovebox and environment. Here it is important to minimize the cross-sectional areas and increase the length of equipment inserted into the well. This approach will help to provide more resistance to heat transfer.

Along with reducing the heat transfer to the environment, it is very important to provide enough heat to the well to perform the intended process. If too little heat is provided, the desired temperatures will not be achieved, even in a perfectly insulated well. Therefore, the following items concerning heater selection and operations should be considered.

- Properly size the heating elements. Select heaters that are capable of reaching the required operating temperature and can provide enough energy to overcome the heat losses to the environment. It is good practice to operate heaters at under sixty percent of their rated capacity. This technique will increase heater life and reduce the chance of burning out a heater.
- Preheat items before inserting into heated liquids. Quick insertion of cold equipment into a liquid may provide enough of a heat sink to "freeze" some of the liquid and overpower the heaters.

It may also be important to control temperatures in various regions of the well. Some mechanical and glovebox components, such as seals, bearings, and gloves, have maximum operating temperatures. It is important to protect these components, so that they may perform as intended. For safety reasons, it is important to keep regions within reach of the glovebox user at moderate temperatures. Often, for glovebox applications, human factors and glove operating conditions require temperatures of surfaces that can be touched by the user to be below some specified value. A temperature of 70°C is typically considered a safe value.

Overall, the design is important in providing adequate and safe heat management of the heated well. Due care must be taken to assure that all operations can be achieved without jeopardizing the operating requirements and the protection of equipment and user. Appropriate heater sizing and the minimization of heat-transfer paths from the well will help to provide an efficient and safely heated well. See Reference 3 for a program developed to aid in heat-transfer analysis of wells.

Sealing

Typically, heated wells are attached to the underside of glovebox floor flanges. The glovebox floor flanges are welded to glovebox floors. An O-ring is compressed between a well and a glovebox floor flange to provide the seal between the glovebox environment and the outside atmosphere. A typical O-ring seal design is shown in Figure 4.

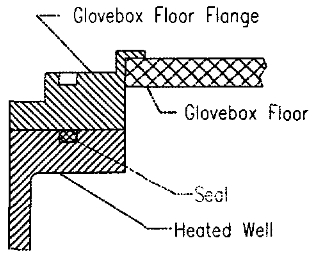

FIGURE 4. TYPICAL SEAL

O-rings are almost always used to provide a seal between the well and the glovebox floor flange. Only one O-ring is necessary to provide a seal, but multiple O-rings can be used to increase confidence in the maintaining of the seal. Some typical O-ring materials are Viton, Silicone, and Buna-N. The effectiveness of these materials to seal is highly temperature dependent. Therefore, the selection of these materials is often based on specified temperature limits. Some typical operating temperature ranges for O-ring materials are given in Table 1.

O-Ring Material	Temperature Range
Viton	-31°F to +400°F (-35°C to +205°C)
Silicone	-175°F to +450°F (-114°C to +232°C)
Buna-N	-65°F to +275°F (-54°C to +135°C)

TABLE 1. SEAL TEMPERATURE LIMITS

When operating temperatures are outside of these temperature limits, it is necessary to provide a means of selectively cooling the O-ring surfaces. This requirement can be achieved by using cooling coils.

Another issue has to do with attaching the well to the glovebox floor flange. Attachment devices, such as screws, should be appropriately numbered, sized, and spaced to be able to provide enough uniform compression of the O-ring to maintain an effective seal. Common practice is to use attachment devices in multiples of four (4) for flanges. Also, screws should be torqued to specified values in order to properly compress the seal, to carry the load of the well and equipment, and to prevent overstressing of the screws. Torquing values must take into account any variations of coefficients of thermal expansion when different metals are used at high temperatures.

Selective Cooling

Cooling coils can be used to cool various regions of the well, most importantly, the O-ring surfaces. An advantage of cooling coils is that they allow the seal region to be water-cooled, yet keep the water separated from the glovebox environment. Cooling coils are usually made of either copper or stainless steel tubing and are wrapped around the outside of the well near the glovebox floor flange. Often, two or three coil wraps are enough to effectively cool the seal. A simple, yet effective, way to attach the coils to the well is to use clips that are welded to the vessel. This method allows the vessel to "grow" without subjecting the coils to any undue stresses. Thermon Heat Transfer Cement is packed around the coils to help transfer heat from the well to the cooling water. The use of cooling coils can significantly reduce the temperature at the seal surfaces. A typical cooling coil design is shown in Figure 5.

In the case of externally heated wells, the heating elements are located on the outside of the well; they operate directly below the cooling coils. If a coil were to fail, the heater would be exposed to water and potentially short out. The possibility of this happening is usually minimal but must be considered.

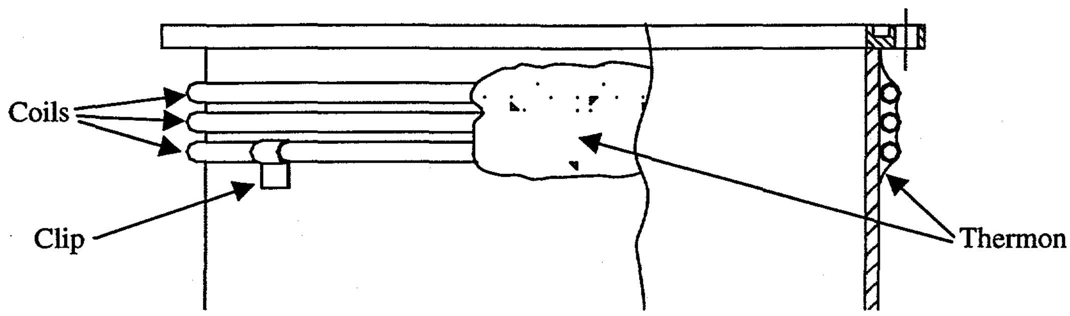

FIGURE 5. COOLING COIL DESIGN

Another benefit of using cooling coils is that other operating equipment may also have temperature limits (e.g., bearings) and require cooling. When this equipment is located close to the floor flange, the cooling coils may be used to cool these components as well. If used properly, cooling coils can often provide a much-needed heat sink for vessel components and operations.

Optimizing Design to Manage Stresses in Heated Wells

Wells can often be classified as pressure vessels because they provide a boundary between the well environment and the atmospheric and/or glovebox environments. The pressure level of this environment can subject the well walls to either internal or external pressure. These pressures will induce stresses within the well walls. The weight of the well will also generate some stress in the walls. These stresses can be classified as primary stresses. In designing a well, primary stresses should always be considered. Wall thickness should be determined to allow for these stresses.

Heated wells present another type of stress. A common trait of metals is that they expand as they increase in temperature. Thus, the greater the

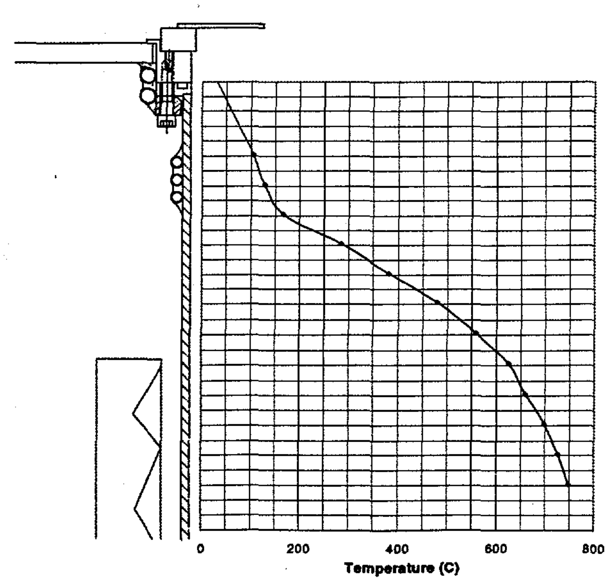

FIGURE 6. TEMPERATURE DISTRIBUTION CURVE

temperature change, the more the material will expand. When a higher-temperature region expands more than the cooler region, stresses are induced in the material. These are called thermal stresses (i.e., due to temperature), and can be classified as secondary stresses.

At ANL, a number of tests and analyses were performed to determine the severity of the effects of thermal stresses in heated wells. Data were collected from new and existing wells. Using a thermocouple, temperatures were taken at various locations along the well wall. These measurements provided a reasonable approximation of the temperature distribution in the wall of the well. Figure 6 shows an example of a temperature distribution along a well wall. Inserting this temperature distribution into a finite element analysis (FEA) program allowed an approximation of the stresses generated due to temperature differences to be calculated. The largest stresses were found to be located in the region between the heated zone and the cooling coils. Typical results from the analysis are shown in Figure 7.

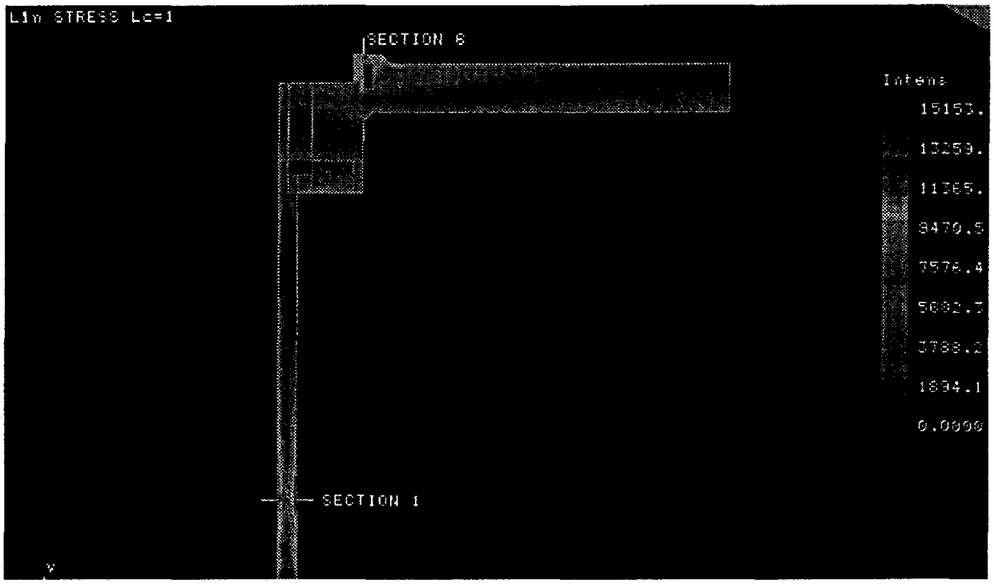

FIGURE 7. FINITE ELEMENT ANALYSIS RESULTS

The most direct cause of large thermal stresses in wells is related to the spacing between the cooling coils and the heated zone of the well. If the cooling coils are located too close to the heated zone, large thermal stresses can be induced into the well walls. Analysis has shown that these stresses, when combined with primary stresses (internal or external pressure, weight of well, etc.), can lead to the eventual failure of the well.

A practical solution to this issue is to take care in spacing the heating elements relative to the cooling coils. There is no simple method to determine what this spacing should be. The issue is highly dependent on the operating temperature of the heated area, the temperature distribution in the well walls, the temperature dependent properties of the well material, and the dimensions of the well (i.e., wall thickness, well diameter, etc.).

An empirical formula, derived from a series of parametric analyses, has been used by ANL CMT to generate preliminary estimates for the spacing. This formula applies to temperature differences along the axis of a cylinder. The equation (Reference 4) is

$$SI = 0.45175 E\alpha (tR)^{1/2} \left(\frac{dT}{dL} \right) \qquad (1)$$

where: SI = Maximum Allowable Secondary Stress Intensity for material at maximum temperature, psi; $SI = 2S_y$

S_y = Yield Stress of material at temperature, psi

E = Modulus of Elasticity at temperature, psi

α = Linear Coefficient of Expansion, in./in./°F

t = Wall thickness of heated well, in.

R = Inside radius of heated well, in.

dT = Temperature difference between cooling coils and heated zone, °F

dL = Axial distance between the cooling coils and the heated zone, in.

This formula assumes a linear temperature distribution between the cooling coils and the heated zone. However, as seen in Figure 6, temperature distributions may not always be linear. Also, the formula assumes that the number of thermal cycles is low. If the well is often subjected to large temperature fluctuations (i.e., between elevated and room temperatures), material deformations may accumulate and weaken the well.

Equation 1 has been used at ANL CMT to determine preliminary spacing between cooling coils and heated zones. However, considerable analysis, including FEA, is also used to ensure that stresses generated in each specific design are limited to acceptable levels. Sections III and VIII of the ASME Boiler & Pressure Vessel Code (Reference 5) have typically been used as a standard for the design of these wells. Two sample problems that demonstrate the application of Equation 1 follow.

Sample Problem 1

Assume that a heated well is to be designed with a sixteen-inch (16 in.) inside diameter and a wall thickness of three-eighths of an inch (3/8 in.). The well is to be fabricated out of 304 stainless steel and operate at 1500°F (816°C). Assume that the cooling coils are able to reduce the wall temperature at the O-ring to 70°F (21°C). The design goal is to minimize the required spacing between the cooling coils and the heating elements. The required spacing can be calculated using Equation 1, where at 1500°F (816°C), the parameters are as follows:

S_y = 8000 psi

SI = $2S_y$ = 16,000 psi

E = 1.83×10^7 psi

α = 10.59×10^{-6} in./in./°F

t = 0.375 in.

R = 8 in.

dT = 1500°F - 70°F = 1430°F

Solving for the length, dL, shows that the minimum spacing between the cooling coils and the top of the heaters is 13.5 inches.

Sample Problem 2

Assume now that the same well is to be designed, but this time the material is Haynes 556 alloy. Again, the required spacing can be calculated using Equation 1, where at 1500°F (816°C)

$S_y = 23,700$ psi
$SI = 2S_y = 47,400$ psi
$E = 2.13 \times 10^7$ psi
$\alpha = 9.3 \times 10^{-6}$ in./in./°F
$t = 0.375$ in.
$R = 8$ in.
$dT = 1500°F - 70°F = 1430°F$

Solving for the length, dL, shows that the minimum spacing between the cooling coils and the top of the heaters is 4.7 inches.

Since the actual temperature distribution and geometry will be much more complex than assumed here, the results of these calculations should be viewed as only estimates.

Overall, it is very important to evaluate the stresses induced in the well, both primary and secondary. If too high, these stresses may lead to the eventual failure of the well. Depending on the application, well failure could have negative effects on the experiment, health, safety, and environment.

CONCLUSIONS

Heated wells provide housings for high-temperature experimental and processing work. Operating temperatures approaching 900°C can create a number of challenges in the design of heated wells. Selection of the right type of heated well (i.e., internally or externally heated) for the application can alleviate a number of the problems encountered in design. The issues of material selection, heat transfer, sealing, cooling, and, primary and secondary stress all can significantly affect the performance of the heated well. Care in addressing these issues will ensure that the heated well will perform as intended.

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