NIST Technical Note 1687

Spent Fuel Cask Seal Performance Testing: Interim Report

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National Institute of Standards and Technology • U.S. Department of Commerce

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

BACKGROUND

The Nuclear Regulatory Commission (NRC) is collecting data to better characterize the performance envelope of seals used on spent nuclear fuel (SNF) transportation packages, during fire exposures that exceed the hypothetical accident condition (HAC) fire described in 10 CFR Part 71 Section 73 [1]. An example of an accident that could potentially produce an exposure beyond the HAC fire was the Baltimore tunnel fire that occurred in 2001. The performance of package seals is important for determining the potential for release of radioactive material from a package during a beyond-design-basis accident because the seals, in general, have lower temperature limits than other package components.

NUREG/CR-6886, "Spent Fuel Transportation Package Response to the Baltimore Tunnel Fire Scenario," [2] describes in detail an evaluation of the potential release of radioactive materials from three different spent fuel transportation packages. This evaluation used estimates of temperatures resulting from the Baltimore tunnel fire as boundary conditions for finite element models to determine the temperature of various components of the packages, including the seals. For two of the packages evaluated, the model-estimated temperatures of the seals exceeded their continuous-use rated service temperature, meaning a release of radioactive material, such as Cobalt 60 (from CRUD) or Cesium 137 (from fission products), could not be ruled out with available information. However, for both of those packages, the analysis determined by a bounding calculation that the maximum expected release was well below the regulatory limits for a release during the HAC series of events in 10 CFR Part 71.

Testing of package seals to determine their performance in beyond-design-basis fire scenarios can provide physical data needed to understand the likelihood of a release of radioactive materials.

Previous work has mainly focused on elastomeric seals and temperatures well below 800 °C. The test fixture typically consisted of two flanges or two plates with two concentric O-ring grooves, one for the test seal and one for the secondary external seal, and a small cavity for helium tracer gas [e.g., 3,4].

OBJECTIVE

The objective of this work is to provide experimental seal performance data for metallic seals for exposures beyond their rated temperatures.

EXPERIMENTAL APPARATUS

The test fixture consists of a seamless vessel body with a flange machined from a stainless steel (SS 304) cylindrical stock and a removable SS 304 flange (vessel cap) with seal groove machined to O-ring manufacturer specifications. The flange dimensions were made in conformity with the ASME Standard B16.5-2009 [5], Flange Class 2500 with a design pressure rating up to 29.2 bar (Table 2-2.1, ASME Standard B16.5-2009). The vessel cavity had a nominal internal volume[1] of 100 mL. The seal was a Garlock Helicoflex[2] metal O-ring made of

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[1] Actual internal volume was measured using an internal micrometer with a resolution of 0.005 mm (0.0002 in.).

[2] The seal material and manufacturer were selected and specified by the U.S. NRC.

Inconel 718 and silver with an outer diameter of 6.35 cm (2.5 in.) and a cross section of 0.32 cm (0.125 in.). The O-ring groove size and flange surface roughness (minimum 0.80 µm and maximum 1.6 µm) were based on the specifications listed in the Garlock Helicoflex technical literature. The design drawings of the test vessel body and the removable flange (vessel cap) are provided in Appendix I.

The vessel body and the cap were joined together using four bolts (SS 304 1-1/8 in. 7 TPI). Each bolt was tightened with a torque of 416 N·m (307 ft·lb) ± 2 N·m (expanded uncertainty with a coverage factor of 2) using a micrometer torque wrench (KD Tools 2953 ¾ in. drive 100-600 ft·lb; 153-830 N·m, with a resolution of 3.4 N·m). A 24 cm long Stainless steel tubing with an inside diameter of 0.48 cm (0.189 in.) and an outside diameter of 0.953 cm (0.375 in.) was inserted into the bottom of the vessel body flush through a straight-hole with a bevel-groove and was all-around fillet welded to the vessel. The exposed end of the tubing was connected to a union cross equipped with two needle valves (Swagelok SS-1RS4) or bellow valves (Swagelok SS-4BK)[3] for filling and evacuating the test vessel and a tee connection for mounting a pressure transducer (Omegadyne, PX01C1-500A5T) and a thermocouple (Omega K-type CAIN-18G-24) to monitor the vessel pressure and temperature. Figure 1 is a schematic of the experimental apparatus, two photographs of which are shown in Figure 2. Five test fixtures with the same dimensional tolerances were constructed and used for this test series.

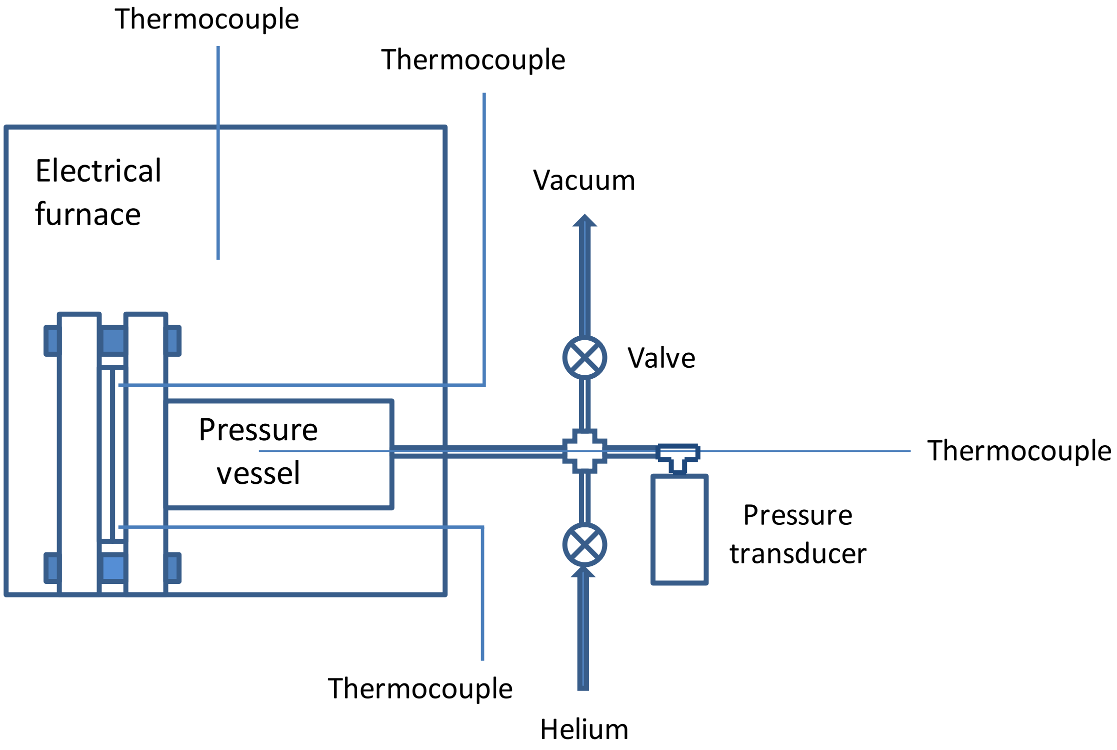

Figure 1. A schematic of the experimental apparatus.

The exposure of the seal to high temperature environment was achieved using a programmable temperature-controlled electrical furnace (Carbolite CWF 1200) with an internal capacity of

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[3] For the first three tests, needle valves were used.

25.4 cm × 25.4 cm × 40.64 cm (10 in. × 10 in. × 16 in.). The electrical furnace has a maximum operating temperature of 1200 °C. Due to the large mass and thermal inertia of the test fixture, it was anticipated that the test fixture temperature might not be totally uniform. Therefore, four K-type grounded thermocouples (Omega K-type CAIN-18G-24)[4] were used to monitor the transient temperature distribution; one for vessel interior temperature, one for furnace temperature, and two for temperatures of the vessel cap at two locations 90° apart near the O-ring groove to monitor the temperature experienced by the metallic seal. Two nominal temperatures, 800 °C and 427 °C, were selected for thermal exposure testing of the metallic cask seals; 10 CFR Part 71 uses an exposure temperature of 800 °C and the published maximum operating temperature of the metallic seal used in this study is 427 °C (800 °F)[5]. The thermal exposure time was 9 h after the seal had attained the test temperature.

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[4] See Appendix II for measurement uncertainty discussion.

[5] Generally seals do not reach the HAC exposure temperature of 800 °C in SNF transportation package designs.

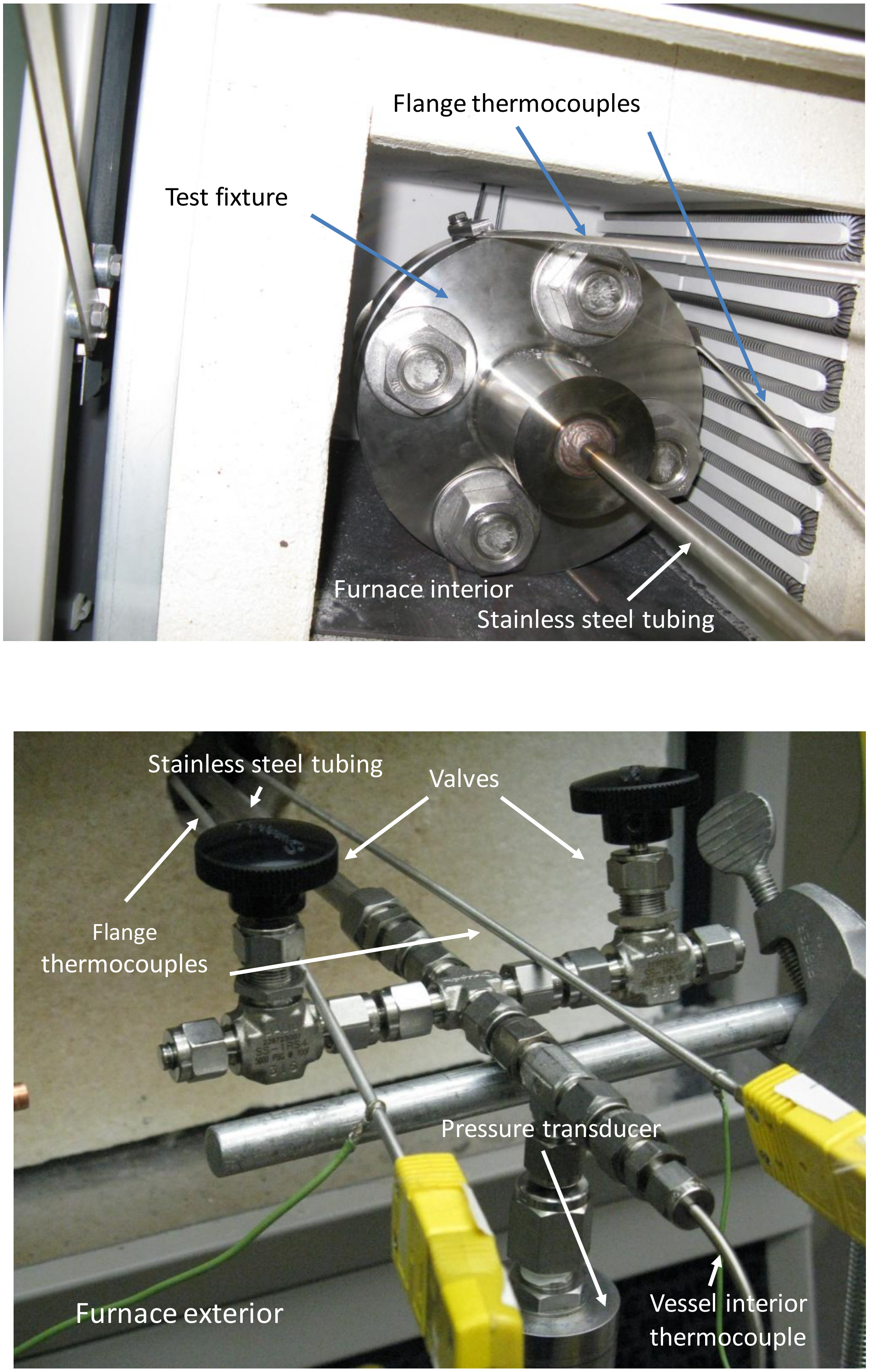

Figure 2. Photographs of the experimental apparatus.

EXPERIMENTAL PROCEDURE

This test series involved the thermal exposure of a test vessel with a metallic seal in an electrical furnace to determine the seal performance at elevated temperatures.

The test vessel with the test seal installed was placed in the electrical furnace, and the tubing system was connected to the vessel. The entire test system was then evacuated and filled with helium at room temperature to a nominal pressure of 5 bar. The tubing connections of the test apparatus were immediately tested for leaks using soapy water. The vessel pressure was then monitored for more than 48 h. If the vessel pressure variations over this period were within the expanded uncertainty of the pressure transducer (0.11 bar)[4], it could be confidently assumed that the vessel pressure was unchanged, and the invariance of vessel pressure was used as an additional "no leak" indicator. With no leaks detected, the electrical furnace was turned on to heat the test vessel from room temperature to either 800 °C or 427 °C. The heating process typically took about 4 h. Once the vessel flange temperature had reached the pre-determined value (800 °C or 427 °C), the vessel was heated for an additional 9 h at this temperature. The furnace was then turned off, and the vessel was allowed to cool to room temperature inside the furnace. The internal temperature and pressure of the vessel, the furnace temperature, and the flange temperatures were recorded during the heat-up and cool-down phases using a LabVIEW-based 16-bit DAQ (Data Acquisition) system (National Instruments NI USB-6251) with an input/output connector block (National Instruments NI SCC-68). The DAQ system sampled at a rate of 100 Hz; however, the data were logged at one minute intervals.

Test Matrix

Table 1 summarizes the test conditions and parameters for this test series.

Table 1 Test conditions and parameters

Test #	Vessel #	Nominal initial vessel conditions	Exposure duration
1*	1	24 °C at 5 bar	Heat-up + 30 min at 800 °C + cool-down
2	2	24 °C at 5 bar	Heat-up + 9 h at 800 °C + cool-down
3	3	24 °C at 5 bar	Heat-up + 9 h at 800 °C + cool-down
4	4	24 °C at 5 bar	Heat-up + 9 h at 800 °C + cool-down
5	5	24 °C at 5 bar	Heat-up + 9 h at 427 °C + cool-down
6	2**	24 °C at 5 bar	Heat-up + 9 h at 427 °C + cool-down
7	1**	24 °C at 5 bar	Heat-up + 9 h at 427 °C + cool-down

*Shakedown test; during this test DAQ malfunctioned, and no temporal data was collected.

**flange and groove surfaces refurbished.

RESULTS AND DISCUSSION

During the heat-up phase of the vessel, the temporal variation of vessel pressure could not readily be used to determine if there was a potential leak unless a catastrophic seal failure occurred causing a significant drop in pressure. As the vessel was heated, the vessel pressure and temperature increased. If there was a very small leak, the reduction in pressure due to the reduction in helium in the vessel from the leak could easily be compensated by the increase in pressure due to increasing temperature. The net effect would still show an increase in pressure, thus masking the leak. The use of the temporal variation of vessel pressure as a means to detect potential leakage is best applied to conditions when the vessel is at a constant temperature.[6] Although the monitoring of pressure drop is not the most sensitive way to detect leaks, the sensitivity to detect a small pressure drop could be greatly enhanced if the vessel pressure is monitored over a very long duration. In addition, the sensitivity of the method can further be improved by using a smaller test volume since the sensitivity of a pressure drop is inversely proportional to the test volume. [6] The implementation of the pressure drop technique in a harsh thermal exposure environment proved to be less demanding than other more sensitive test methods, as described in Reference [6]. This was the approach used in this study to analyze the temporal pressure data to determine the seal performance at elevated temperatures.

Test #1 (Vessel #1)

The thermal exposure test using Vessel #1 was intended to be a shakedown experiment. The entire heating process took about 4.5 h for the flange temperature to reach the equilibrium furnace temperature of 800 ^{o}C. The vessel was then maintained at this temperature for an additional 30 min before turning off the electrical furnace.

During the heat-up phase, the DAQ readings from the pressure transducer and the thermocouples became erratic due to reasons unknown; the problem was later diagnosed to be improper grounding and connection to the DAQ system. The readings from the pressure transducer and thermocouples were recorded manually using a voltmeter and a thermocouple reader, respectively. At 800 ^{o}C, the vessel pressure reached 14.6 bar and was holding at 14.6 bar for an additional 30 min of heating. After the vessel was cooled to room temperature, the vessel pressure recovered its initial pressure of 5.0 bar at room temperature, which indicated the absence of a leak.

A postmortem inspection of the tested vessel revealed that the metallic seal was soldered to the flange of the vessel body and a silver-color coating was imprinted on the surface of the O-ring groove, as shown in Figure 3. The high-temperature exposure also discolored the test fixture.

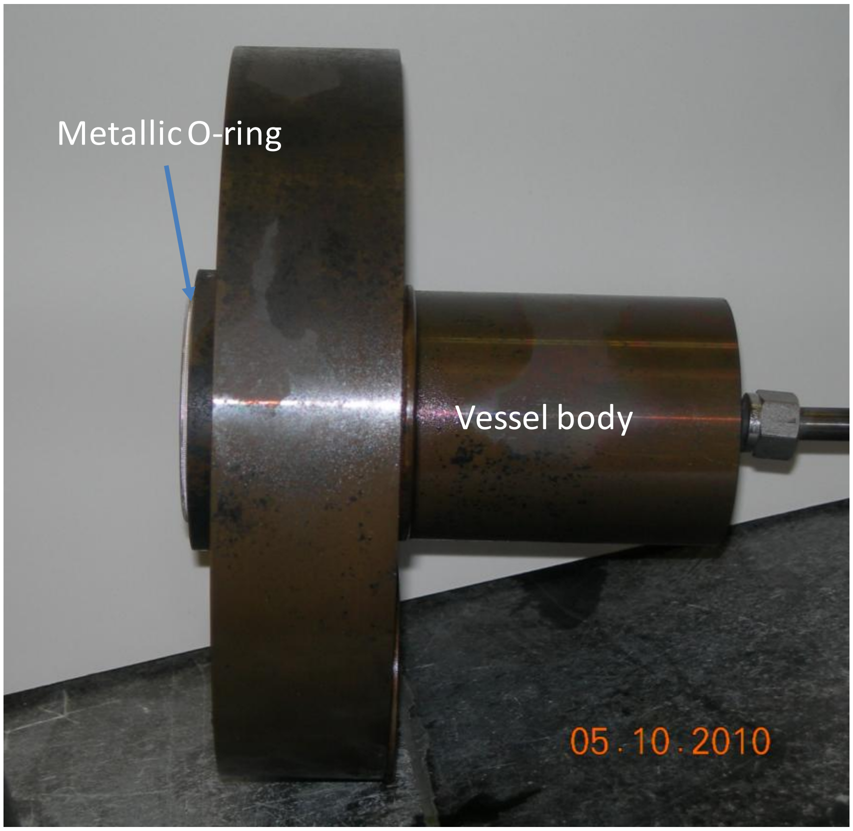

Figure 3. Postmortem photographs showing silver from the metallic O-ring deposited on the O-ring groove and the O-ring bonded to the tested vessel (Vessel #1) body.

Test #2 (Vessel #2)

Figure 4 shows the temporal variations of the vessel internal pressure and temperature, two flange temperatures and furnace temperature during the heat-up, the constant-temperature heating (isothermal), and the cool-down phases for Test #2. The entire cool-down phase is not shown in Figure 4 because it normally takes more than several days to naturally cool the test vessel inside the powered-down furnace from 800 °C to room temperature.

The scale of the ordinate for pressure in Figure 4 is magnified to show that the pressure starts to decrease shortly after the vessel temperature has reached 800 °C and continues to decrease during the rest of the 9 h constant-temperature heating phase. Although the decrease in pressure, which is within the expanded measurement uncertainty of the pressure transducer, is not significant in this test, the continuous downward trend does seem to imply the occurrence of a very small leak.

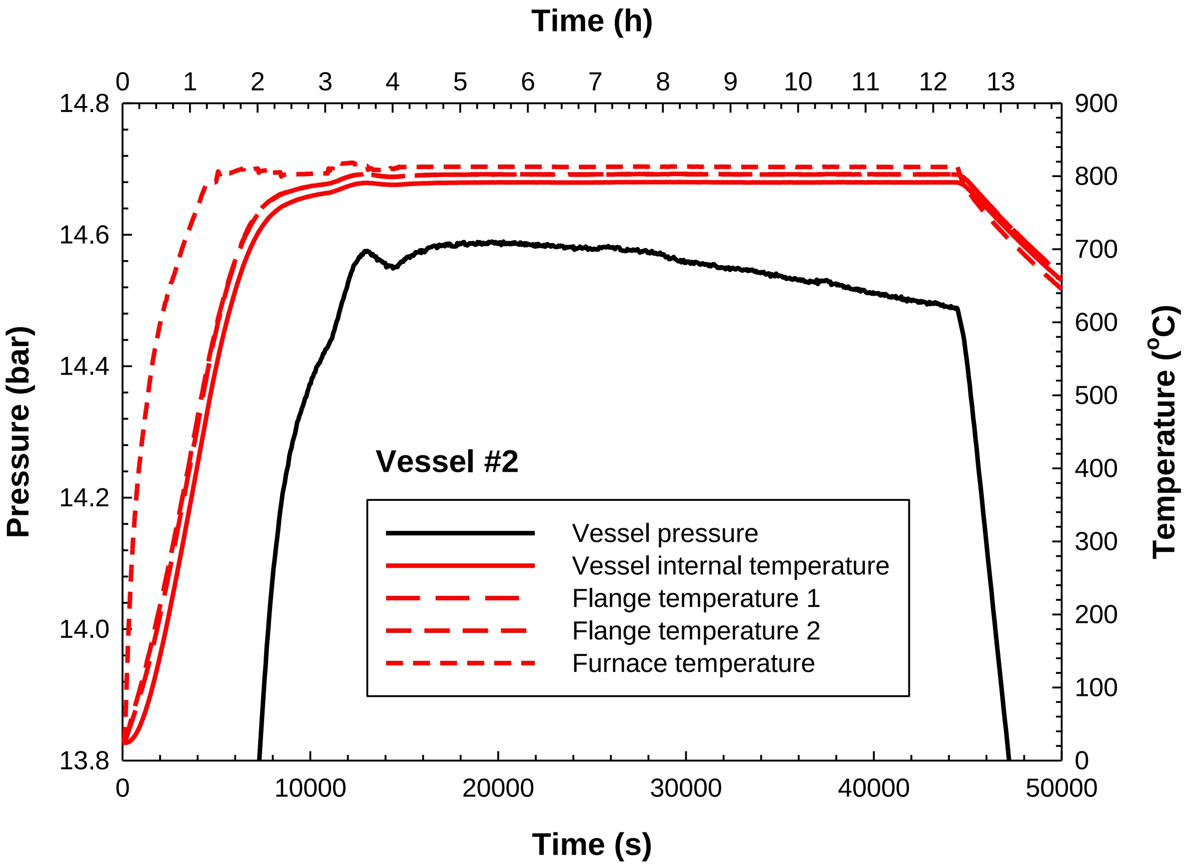

Figure 4. Temporal variations of vessel pressure and temperature in Test #2.

Test #3 (Vessel #3)

Tests #3 is a repeat of Test #2. Figure 5 is the test results and also shows the occurrence of a leak during the 9 h constant-temperature heating phase at 800 °C. In Test #3, the vessel pressure decreases slowly initially and then significantly after about 25 000 s at 800 °C. Since the leakage rate is directly proportional to the time rate of change of pressure, this two-stage decrease in pressure indicates a slower leak rate (small *dP*/*dt*) at first and then a faster leak rate (large *dP*/*dt*) at the end.

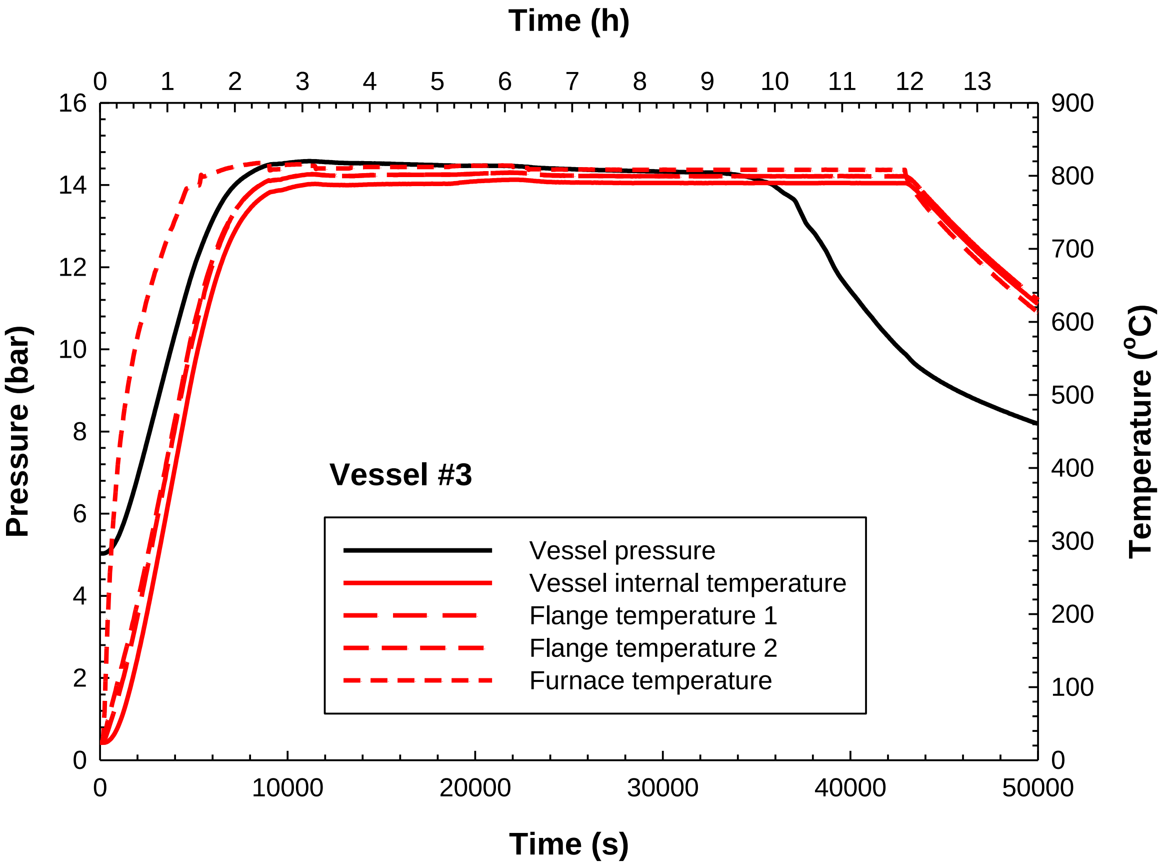

Figure 5. Temporal variations of vessel pressure and temperature in Test #3.

Test #4 (Vessel #4)

Test #4 is another repeat of the thermal exposure condition used in Test #2. Figure 6 shows the test results from Test #4. In this test, the pressure remains relatively constant for about 10 000 s initially during the 9 h constant-temperature heating phase, begins to drop significantly for about 15 000 s (large *dP*/*dt*), and decreases slowly (small *dP*/*dt*) for the remaining duration.

It is interesting to note that despite the consistent occurrence of a leak in these three tests (Tests #2, #3, and #4), the vessel pressure did not equilibrate to atmospheric pressure at the end of the cool-down-phase, which took several days. This observation indicated the possibility that re-sealing could potentially occur as the vessel was cooled down.

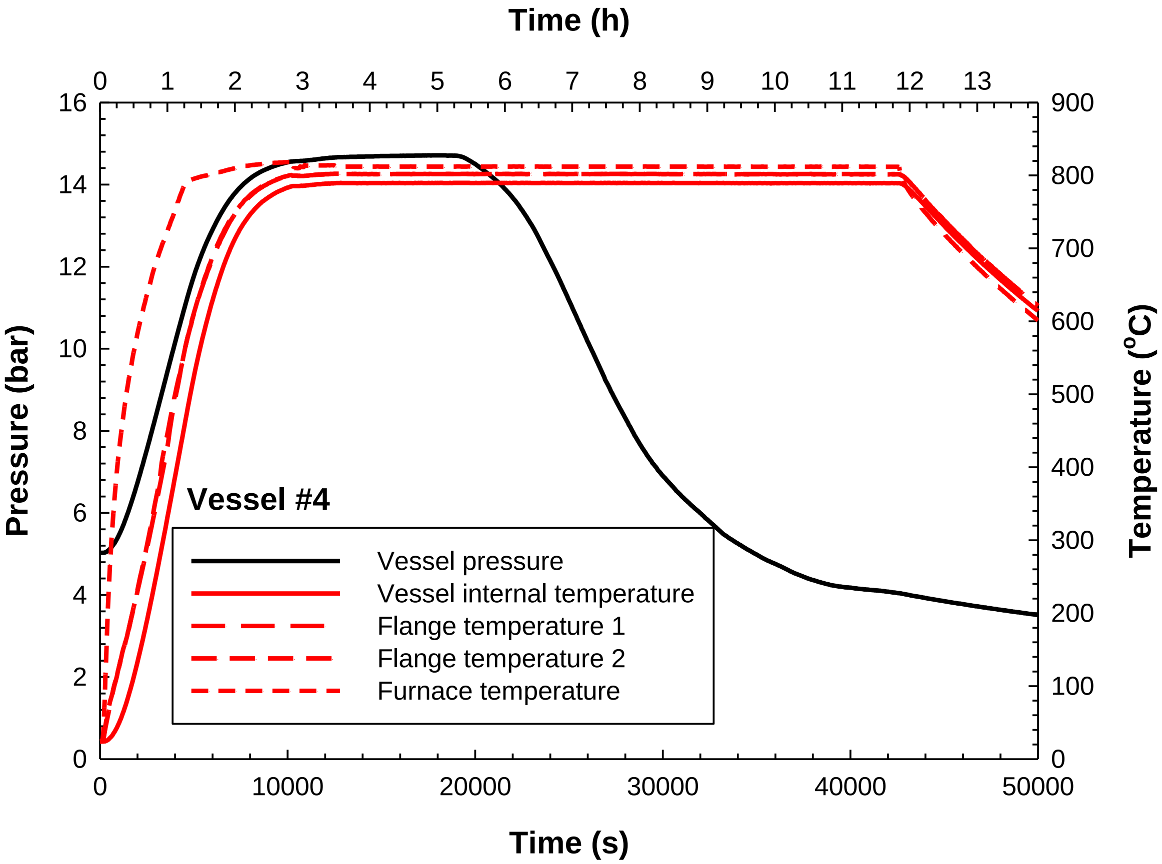

Figure 6. Temporal variations of vessel pressure and temperature in Test #4.

Test #5 (Vessel #5)

The results for Test #5 are shown in Figure 7. The vessel pressure remains constant during the entire 9 h constant-temperature heating period at 427 °C. The seal held vessel pressure. This was further confirmed by the fact that the initial pressure at room temperature was recovered after the cool-down phase. In addition, the pressure remained unchanged for over 140 h after the cool-down. A photograph of the exposed vessel is shown in Figure 8. The extent of discoloring of the vessel depends on the test temperature (Figure 3 *vs*. Figure 8). A postmortem inspection revealed that the metallic O-ring seal was not soldered to either flange surfaces, and no silver coating from the seal was transferred to the O-ring groove.

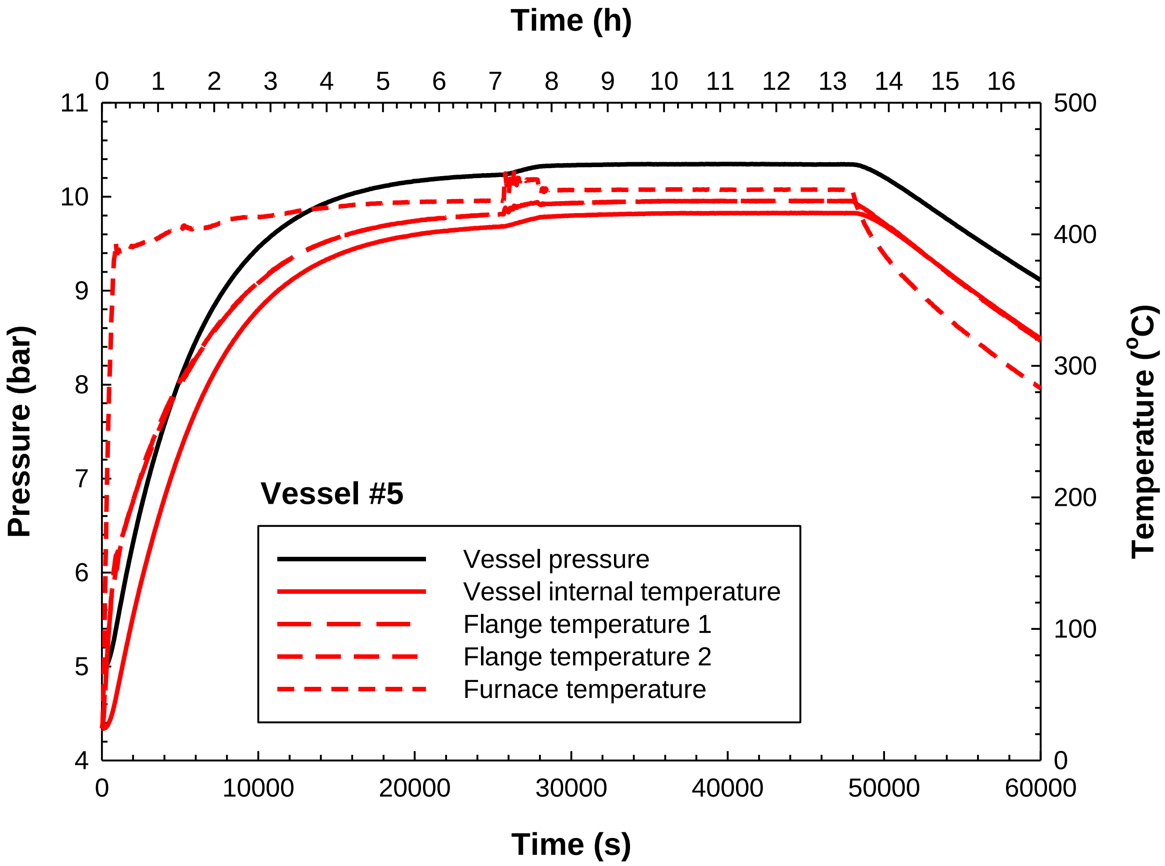

Figure 7. Temporal variations of vessel pressure and temperature in Test #5.

Figure 8. Postmortem photograph of Vessel #5 (vessel body with O-ring removed) after exposure at 427 °C for 9 h.

Test #6 (Vessel #2 Refurbished)

The results for Test #6 using the refurbished Vessel #2 are shown in Figure 9. The refurbishment only involved the re-facing of the surfaces of the flanges and O-ring groove to the specified tolerances. For this test, the vessel pressure started to decrease very slowly during the constant-temperature heating period. The scale of the ordinate is magnified in Figure 9 to elucidate the small pressure drop, which is within the expanded measurement uncertainty of 0.11 bar. However, the continuous decrease in pressure does seem to imply that a very small leak might have occurred during the test.

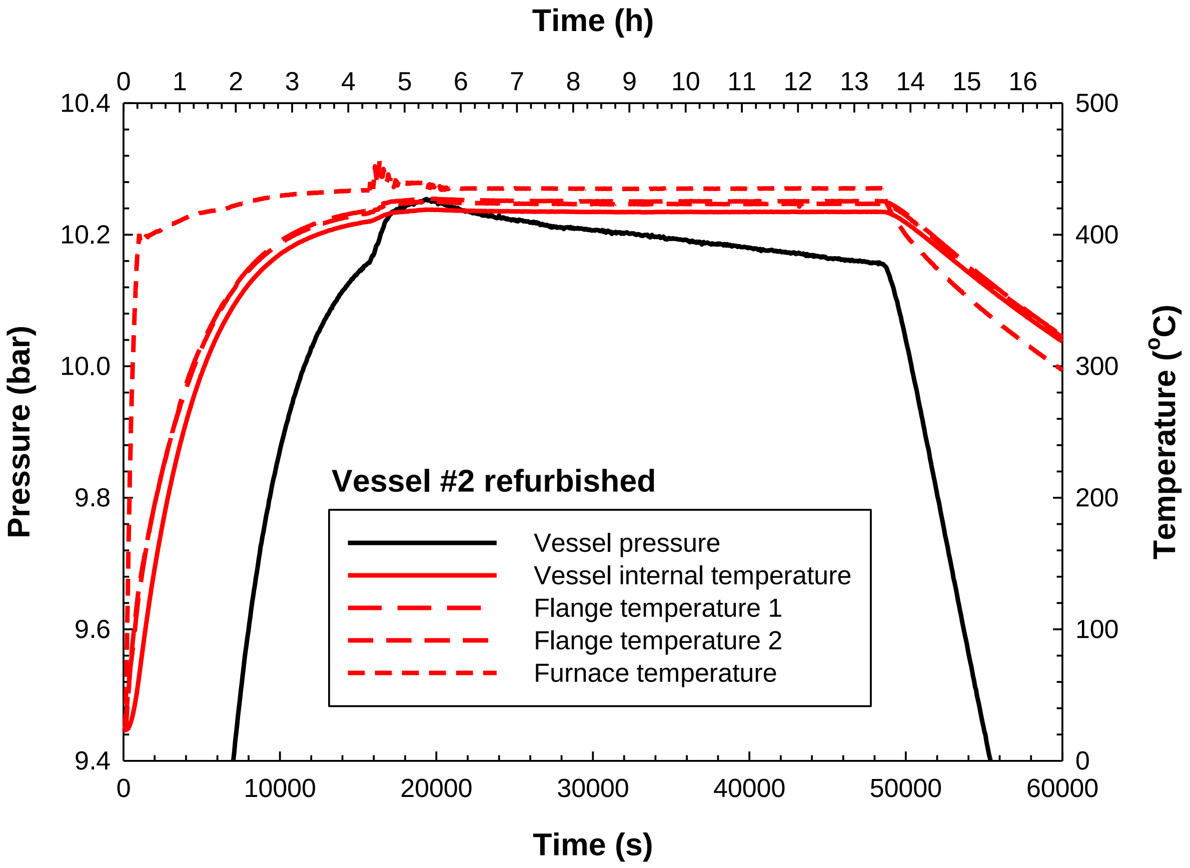

Figure 9. Temporal variations of vessel pressure and temperature in Test #6.

Test #7 (Vessel #1 Refurbished)

Figure 10 shows the results for Test #7 using the refurbished Vessel #1. The same refurbishing process used in Vessel #2 was applied to Vessel #1. As indicated in the figure, the vessel pressure remained unchanged during the 9-hour constant-temperature heating period. No leak was observed during Test #7.

It is not known if the leak in the refurbished Vessel #2 was caused by a different thermal response of the vessel, which had previously been exposed at 800 °C for 9 h. Although Vessel

#1 was also refurbished, the previous exposure time at 800 °C was much shorter than that of Vessel #2 (30 min *vs*. 9 h).

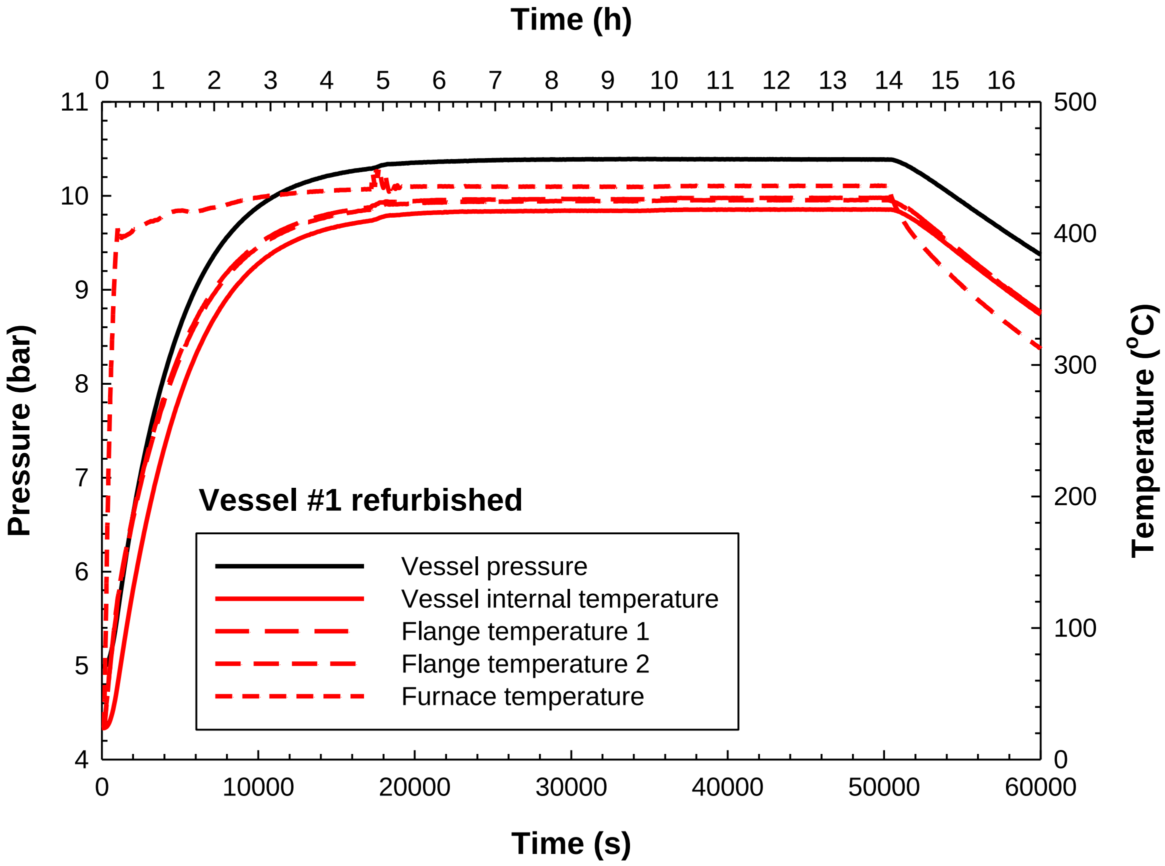

Figure 10. Temporal variations of vessel pressure and temperature in Test #7.

SUMMARY

Seven high-temperature metallic seal performance tests including one shakedown were performed. With the exception of the shakedown test, all tests were conducted exposing the test fixture to 800 °C or 427 °C for 9 h in an electrical furnace. The shakedown test was conducted using a 30 min exposure at 800 °C, and the seal appeared to hold vessel pressure. In the three subsequent repeat tests under similar beyond-design-basis thermal exposure conditions of 9 h heating at nominal 800 °C, the metallic seal did not maintain vessel pressure. The times when the leakage occurred (the vessel pressure started to decrease) varied in the three tests performed. Three repeat tests were also conducted at the seal maximum operating temperature of 427 °C for 9 h. The seal maintained vessel pressure in two tests (one using a brand new test vessel and one using a refurbished vessel which had previously been exposed at 800 °C for 30 min). However, a very slow leak appeared to occur in the third test which used a refurbished vessel previously exposed for 9 h at 800 °C.

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[5] ASME B16.5-2009, Pipe Flanges and Flanged Fittings NPS ½ through NPS 24 Metric/Inch Standard.

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[7] ISO Guide to the Expression of Uncertainty in Measurement, first edition, 1993, International Organization for Standardization, Geneva, Switzerland.

APPENDIX I

Design Drawings of Test Vessel

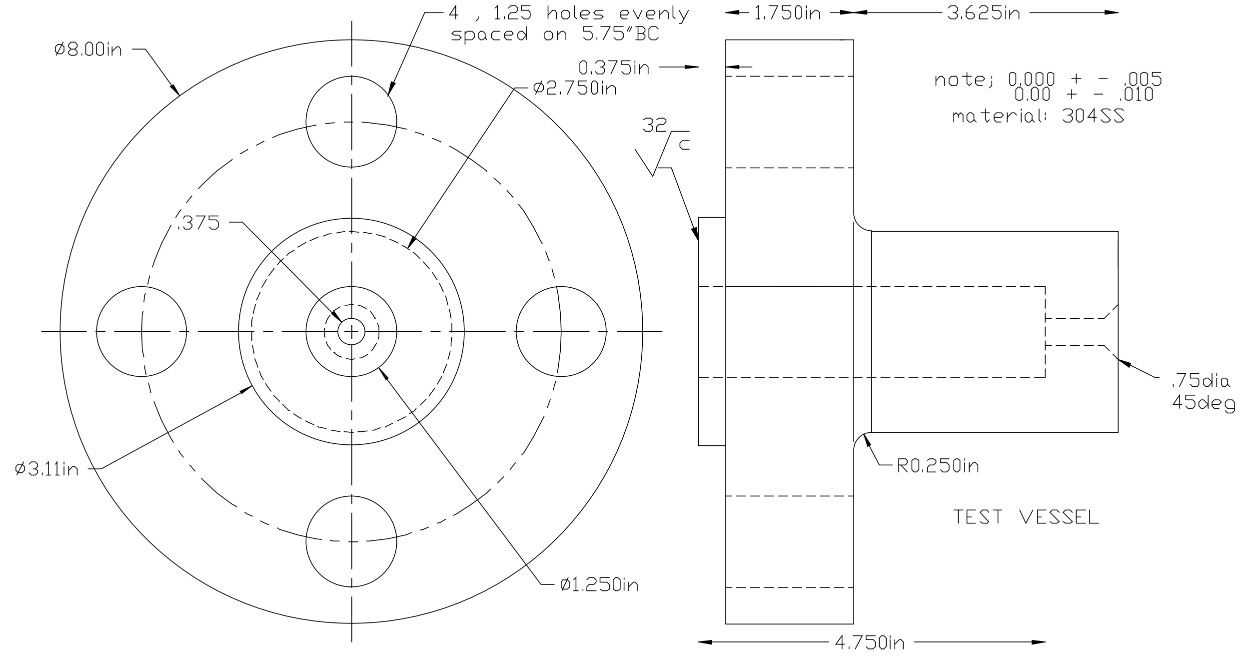

Figure A1 Design drawing of test vessel body.

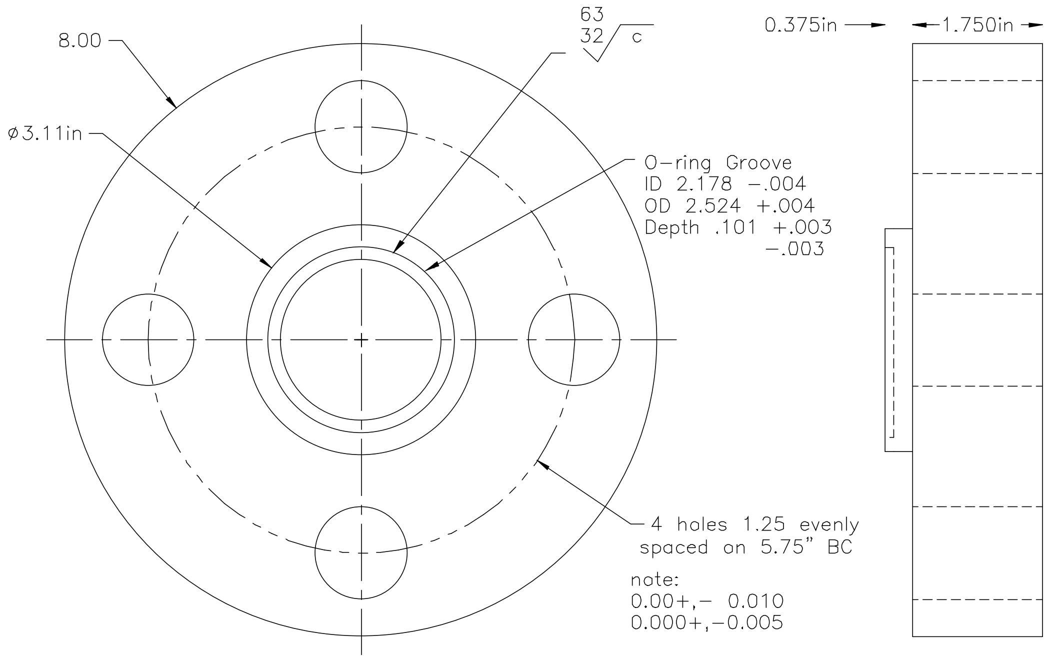

Figure A2. Design drawing of removable flange (vessel cap).

APPENDIX II

Uncertainty Estimate in Thermocouple Measurements

The uncertainty budget associated with the thermocouple measurements is summarized in Table A-1.

Table A-1 Summary of standard uncertainty components in thermocouple measurements

Standard uncertainty component	Source of uncertainty	Value of standard uncertainty	Source and comments
u_{TC}	Type-K thermocouple	0.64 °C	The thermocouples have a limit of error of 2.2 °C. An assumed rectangular probability distribution [7] results in 2.2 °C × 0.29 = 0.64 °C.
u_{ADC}	Analog to digital conversion	0.04 °C	The K-type thermocouples have an output range of ± 50 mV; 100 mV/2^{16} (16-bit) = 0.00153 mV, which corresponds to 0.04 °C using an approximate sensitivity of 40 μV/°C.
u_{CJ}	Cold-junction compensation	1 °C	Software cold-junction compensation using 25 °C as reference temperature.
Combined standard uncertainty (u_c)		$u_c = \sqrt{u_{TC}^2 + u_{ADC}^2 + u_{CJ}^2} = 1.2$ °C	
Expanded uncertainty U (with coverage factor k = 2)		$U = k\,u_C = 2.4$ °C	

Uncertainty Estimate in Pressure Transducer Measurements

The measurement uncertainty of the pressure transducer was estimated based on the specifications of the transducer provided by the manufacturer (see Table A-2). The uncertainty budget is listed in Table A-3.

Table A-2 Manufacturer's specifications of the pressure transducer

Excitation	24 VDC to 32 VDC
Input range	(0 to 500) psia ; (0 to 34.0229825) bar
Output range	(0 to 5) VDC ± 0.03 VDC
Linearity	0.05 % FSO (full scale output)
Hysteresis	0.05 % FSO
Repeatability	± 0.05 % FSO
Thermal zero drift	0.001 % FSO/°F
Operating temperature range	– 46 °C to 121 °C

Table A-3 Summary of standard uncertainty components in pressure measurements

Standard uncertainty component	Source of uncertainty	Value of standard uncertainty	Source and comments
u_P	Transducer output	0.0087 V	A rectangular probability distribution was assumed [7], resulting 0.29 × 0.03 V = 0.0087.
u_L	Linearity	0.0025 V	0.05 % × 5 V
u_H	Hysteresis	0.0025 V	0.05 % × 5 V
u_R	Repeatability	0.0025 V	0.05 % × 5 V
u_T	Thermal zero drift	9×10^{-4} V	18 °F × 0.001 % × 5 V/°F §
u_{ADC}	Analog to digital conversion	7.63×10^{-5} V	5 V/2^{16} (16-bit)

§ Since the pressure transducer was mounted outside the furnace, the temperature of the transducer was close to room temperature, and a 10 °C (18 °F) rise from room temperature was assumed to be the worst case.

The combined standard uncertainty (u_c)

$$u_c = \sqrt{u_P^2 + u_L^2 + u_H^2 + u_L^2 + u_T^2 + u_{ADC}^2} \tag{7}$$

Then u_c = 0.00976 V. Based on the calibration data of the pressure transducer provided by the manufacturer, P (bar) = –0.0147 + 6.8835 × output (V), the combined standard uncertainty, u_c = 0.00976 V, corresponds to u_c = 0.0525 bar. The expanded uncertainty U of the pressure transducer with a coverage factor k of 2 is $U = k \times u_c$ = 0.11 bar.