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NUREG/CR-7115

Performance of Metal and Polymeric O-Ring Seals in Beyond-Design-Basis Temperature Excursions

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ABSTRACT

This report documents the beyond-design-basis thermal exposure test results of the performance of one type of metallic seal and two different polymeric compound (ethylene-propylene and polytetrafluoroethylene) seal designs typically used in spent fuel transportation packages. Fifteen tests were conducted using a small scale-model package made of stainless steel SS 304 filled with helium initially pressurized at either 5 bar or 2 bar at room temperature. The test package was then exposed in an electric furnace to temperatures equal to or over the specified maximum operating temperatures of these seals for a pre-determined period (typically 9 h). The pressure drop technique was used to determine if leakage occurred during thermal exposure. A total of fifteen tests, twelve used metallic seals, two used ethylene-propylene compound, and one used polytetrafluoroethylene, were performed. Leakage was observed in some of the thermal exposure tests. The time when leakage occurred varied. The overall goal of the project is to provide insights to the performance of these seals when exposed to beyond-design-basis temperature conditions that could result due to a severe fire.

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EXECUTIVE SUMMARY

The objective of this work is to provide experimental seal performance data for one type of metallic seal and two different polymeric compound seals typically used in spent fuel transportation packages for beyond-design-basis temperature excursions (beyond the seal manufacturer rated operating temperatures).

Fifteen tests were conducted using a scale-model package (a pressure vessel) made of stainless steel SS 304 filled with helium initially at either 5 bar (for metallic seal) or 2 bar (for polymeric seals) at room temperature. Since it is difficult to control a real fire environment for testing, a programmable electric furnace was used to provide various controlled thermal environments to heat the vessel to a specified temperature for a pre-determined duration, which varied from several hours to one or two days. The vessel pressure was monitored to determine if a leak occurred at the exposed temperature.

Fifteen tests including two shakedowns were performed to establish seal performance at beyond-design-basis thermal exposure conditions. Of the fifteen tests conducted, twelve tests used metallic seals, two used ethylene-propylene compound seals, and one used a polytetrafluoroethylene (PTFE) seal.

Of the five repeat metallic-seal tests, leakage was observed in three of the tests during the 9 h 800 °C (1472 °F) exposure. The times when the leakage occurred (the vessel pressure started to decrease) varied in the three tests performed. In one test, measurable leakage occurred approximately 6.9 h after the test temperature of 800 °C (1472 °F) had been reached. In another test, measurable leakage occurred about 2.8 h into the 800 °C (1472 °F) exposure. Yet, in another test, leakage was observed roughly 3 h into the test. In the two shakedown tests (Tests #1 and 9) using metallic seals, leakage was not observed during the respective 30 min and 4 h exposure to 800 °C (1472 °F). No leakage was observed for the remaining tests using metallic seals exposed to temperatures just below 750 °C (1382 °F) for 9 h.

No leakage was observed in one ethylene-propylene compound seal tested at 300 °C (572 °F) for more than 20 h. Leakage was observed immediately after the vessel had attained the nominal target temperature of 450 °C (842 °F) in another ethylene-propylene compound seal test. Leakage was also observed during the cooling phase in the test that used a PTFE seal *after* it had been subjected to 300 °C (572 °F) exposure for 22 h.

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DISCLAIMER

ABBREVIATIONS

ANSI	American National Standards Institute
ASME	American Society of Mechanical Engineers
CFR	Codes of Federal Regulations
CRUD	Chalk River Unidentified Deposits
DAQ	Data Acquisition
FDS	Fire Dynamics Simulator
HAC	Hypothetical Accident Conditions
ISO	International Organization for Standardization
NIST	National Institute of Standards and Technology
NRC	United States Nuclear Regulatory Commission
RES	NRC Office of Nuclear Regulatory Research
SNF	Spent Nuclear Fuel
PTFE	Polytetrafluoroethylene

1 BACKGROUND

1.1 Introduction

The U.S. Nuclear Regulatory Commission (US 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 conditions (HAC) fire described in 10 CFR Part 71 Section 73. Examples of an accident that could potentially produce an exposure beyond the HAC fire were the Caldecott Tunnel fire in 1982 (Adkins *et al*., 2007a), the Baltimore Tunnel fire that occurred in 2001 (Adkins *et al.*, 2007b), and the MacArthur Maze fire in 2007 (Dunn *et al*., 2009). 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," describes in detail an evaluation of the potential release of radioactive materials from three different spent fuel transportation packages (Adkins *et al.*, 2007b). This evaluation used estimates of temperatures from a simulation of the Baltimore Tunnel fire using the NIST Fire Dynamics Simulator (FDS) (McGrattan and Hamins, 2003) 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 the release allowed during the HAC series of events in 10 CFR Part 71.

Previous work has mainly focused on elastomeric seals and temperatures well below 800 °C (1472 °F). The test fixtures in previous work 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., Bronowski, 2000, Marlier, 2010). A similar experimental configuration was used to examine the performance of elastomeric seal at temperatures below 0 °C (32 °F) (Jaunich *et al*., 2011). 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.

The objective of this work is to provide experimental seal performance data for metallic and polymeric seals for thermal exposures beyond their rated temperatures using a scale-model package (a pressure vessel) to house a test seal. Since it is difficult to control a real fire environment for testing, an electric furnace was used to provide various controlled thermal environments.

The pressure drop method was used in this study to examine the seal performance at elevated temperatures. 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

ANSI N14.5-1997. Since the use of the temporal variation of vessel pressure as a means to detect potential leakage is best applied to isothermal conditions (ANSI N14.5-1997), all the tests were conducted by maintaining the pressure vessel at a constant elevated temperature in the furnace. 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 (ANSI N14.5-1997).

2 TECHNICAL APPROACH

2.1 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, Flange Class 2500 with a design pressure rating up to 29.2 bar (424 psi) at 800 °C (1472 °F) (Table 2-2.1, ASME Standard B16.5-2009). The vessel cavity had a nominal internal volume[1] of 100 mL. The design drawings of the test vessel body and the removable flange (vessel cap) are provided in Appendix A. Nine test vessels with the same dimensional tolerances were constructed and used for this test series.

The metallic seal was a Garlock Helicoflex[2] metal O-ring made of 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 metallic 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 polymeric seals were ethylene-propylene compound O-ring (DBR Industries, 2-228 E0740-75, Lot # 2Q060080040952) and PTFE O-ring (DBR Industries, 228 PTFE; Lot # 04/07 12730-1).

The vessel body and the cap were joined together using four bolts (SS 304 1-1/8 in. 7 TPI). Each bolt was tightened 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 torque of 416 N·m (307 ft·lb) ± 2 N·m (expanded uncertainty with a coverage factor of 2 [ISO, 1993]) was used for the metallic seals and 271 N·m (200 ft·lb) for the ethylene-propylene compound and PTFE O-rings.[3] In addition, a silicone-base O-ring lubricant (Parker Super O-Lube® from Parker Seal, Lexington, Kentucky) was applied to the ethylene-propylene compound O-ring before the two flanges were bolted together. 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.[4] The exposed end of the tubing was connected to one arm of a union cross (Swagelok SS-400-4) via a reducing union (Swagelok SS-600-6-4) and a port connector (Swagelok SS-401-PC). Two needle valves (Swagelok SS-1RS4) or bellow valves (Swagelok SS-4BK)[5] for filling and evacuating the test vessel were mounted on the other two respective arms of the union cross using two port connectors (Swagelok SS-401-PC). A union tee (Swagelok SS-400-3) was connected to the

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1 Actual internal volume (see Appendix B) was measured using an internal micrometer with a resolution of 0.005 mm).

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

3 The torque requirement for the polymeric O-ring used as a face seal was recommended by the manufacturer to be metal-to-metal fit.

4 Three vessels (Vessels #1, #6, and #7) were tested for surface flaws in the welds using the ASME liquid penetration test (ASME, BVPC-VIII-1-2007) by a local testing laboratory. Vessels #6 and #7 were tested brand new while Vessel #1 was tested after two thermal exposures, one at 800 °C (1472 °F) for 0.5 h and one at 427 °C (800 °F) for 9 h. The test results showed no surface flaws in the welds in all three vessels. Note that performing x-ray of this type weld is difficult at best and with the tube passing through it makes obtaining a good x-ray of the entire weld impossible.

5 For the first three tests, needle valves were used.

fourth arm of the union cross via a port connector (Swagelok SS-401-PC). A pressure transducer (Omegadyne, PX01C1-500A5T)[6] was attached to one arm of the union tee via a tube adapter (Swagelok SS-4-TA-7-4) to measure the vessel pressure. A grounded thermocouple (Omega K-type CAIN-18G-24)[6] to monitor the vessel internal temperature was inserted into the vessel interior through a reducing union (Swagelok SS-400-6-2) which was connected to the other arm of the union tee via a port connector (Swagelok SS-401-PC), the union cross, and the stainless steel tubing.

Due to the large mass and thermal inertia of the test vessel, it was anticipated that the vessel temperature might not be completely uniform when the vessel was heated. Two additional thermocouples (Omega K-type CAIN-18G-24) were placed near the O-ring groove to monitor the temperatures experienced by the seal. In order to securely attach the two thermocouples to their desired locations, two divots 1.25 mm deep and 3 mm in diameter were drilled 90 ° apart in the side of the flange that contained the O-ring groove. The thermocouple tips were placed in these divots, and the thermocouples were clamped onto the sides of the vessel.

Figure 2-1 is a schematic illustration of the experimental apparatus, two photographs of which are shown in Figure 2-2. The upper photograph of Figure 2-2 shows the test vessel placed inside the furnace before testing, and the lower photograph shows the furnace opening was covered with a thermal insulation board during testing. Note that all the connection fittings, the two valves, and the pressure transducer were located outside the furnace.

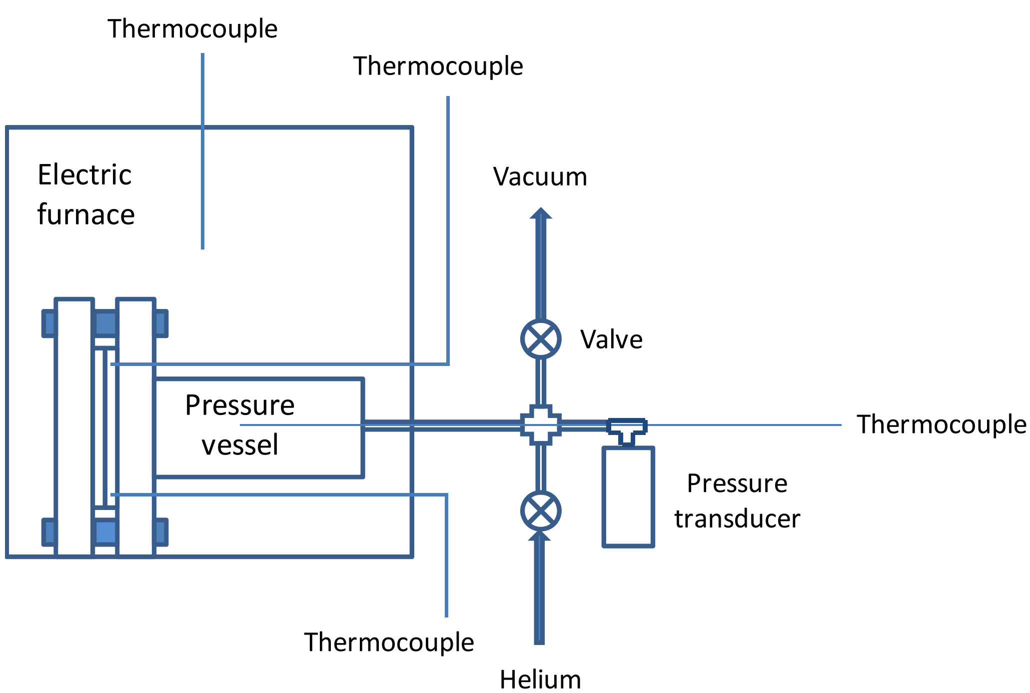

Figure 2-1. A schematic illustration of the experimental apparatus.

6 See Appendix B for measurement uncertainty discussion.

The exposure of the seal to high temperature environment was achieved using a programmable temperature-controlled electric furnace (Carbolite CWF 1200) with an internal cavity of 25.4 cm × 25.4 cm × 40.64 cm (10 in. × 10 in. × 16 in.). The electric furnace has a maximum operating temperature of 1200 °C (2192 °F). Although the furnace was equipped with a digital temperature display for internal furnace temperature, a grounded thermocouple (Omega K-type CAIN-18G-24) was placed inside the furnace interior to monitor the furnace temperature for crosscheck. The differences between these two readings were less than 10 °C.

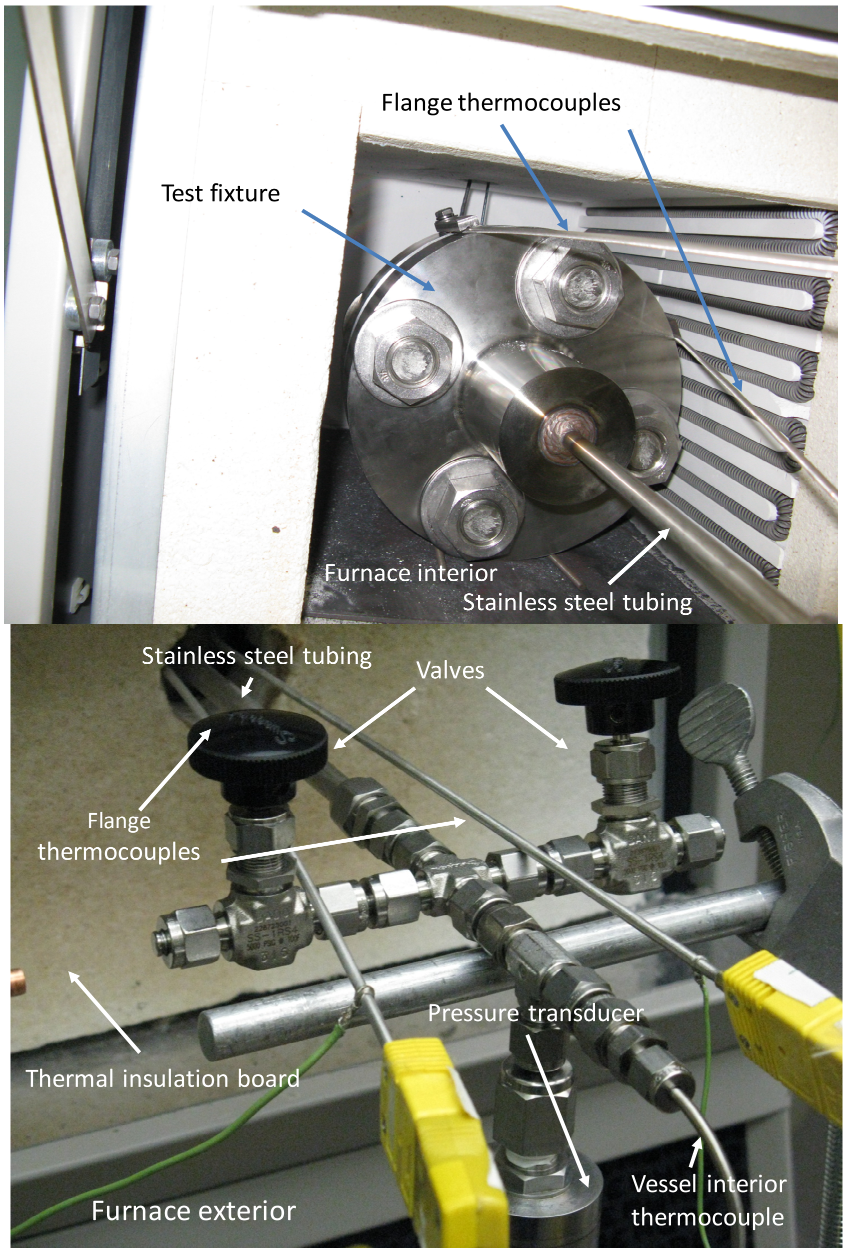

Figure 2-2. Photographs of the experimental apparatus.

2.2 Experimental Procedure

This test series involved the thermal exposure of a test vessel with a metallic or polymeric seal in an electric furnace to determine the seal performance at elevated temperatures. Two nominal temperatures, 800 °C (1472 °F) and 427 °C (800 °F), were selected for thermal exposure testing of the metallic cask seals; 10 CFR Part 71 uses a fire environment thermal exposure temperature of 800 °C (1472 °F) and the published maximum operating temperature of the metallic seal used in this study is 427 °C (800 °F)[7]. For most of the metallic seal tests, the thermal exposure time was 9 h after the flange with the O-ring groove had attained the test temperature. Other thermal exposure protocols were used for some tests (see Table 2-1 below). For the polymeric seals, the test seal was exposed to a flange temperature of 150 °C (302 °F) for 1 h, 200 °C (392 °F) for 1 h, 250 °C (482 °F) for 1 h, and 300 °C (572 °F) for 22 h. The maximum operating temperatures of the ethylene-propylene compound and PTFE O-rings are 149 °C (300 °F) and 260 °C (500 °F), respectively.

The assembled test vessel with the test seal installed was placed in the electric furnace, and the tubing system was connected to the vessel. A vacuum line was attached to one valve of the tubing system, and a helium supply line from a 1A cylinder of compressed helium (analytical grade) was connected to the other valve. With the two valves opened and the pressure regulator of the cylinder closed, the entire test system was then evacuated for 60 s using an oil-less rocking piston vacuum pump (GAST Manufacturing Corporation Model 71R645-V110-UAP). The valve that was connected to the vacuum line was closed, separated from the vacuum line, and capped using a plug (Swagelok SS-400-P). The pressure regulator of the compressed helium cylinder was then slowly opened to fill the test vessel with helium at room temperature to a nominal pressure of 5 bar. The helium supply line was disconnected from the closed fill valve, and the valve was capped using a plug (Swagelok SS-400-P). 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)[6], 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 electric furnace was turned on to heat the test vessel from room temperature to the target temperature. Depending on the specific target temperature, the heating process could take more than 4 h. Once the vessel flange temperature had reached the target value, the vessel was heated according to the pre-determined exposure duration (see Table 2-1 below). After the thermal exposure, 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 entire 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 1 min intervals.

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7 Generally, in HAC analyses using computer codes, SNF Transportation package design seals do not reach the HAC temperature of 800 °C (1472 °F).

2.3 Test Matrix

Table 2-1 summarizes the test conditions and parameters for this test series.

Table 2-1. Nominal test conditions and parameters

Test #	Vessel #	Nominal initial vessel conditions	Exposure duration
1*	1	24 °C at 5 bar (Metallic seal)	Heat-up + 30 min at 800 °C + cool-down
2	2	24 °C at 5 bar (Metallic seal)	Heat-up + 9 h at 800 °C + cool-down
3	3	24 °C at 5 bar (Metallic seal)	Heat-up + 9 h at 800 °C + cool-down
4	4	24 °C at 5 bar (Metallic seal)	Heat-up + 9 h at 800 °C + cool-down
5	5	24 °C at 5 bar (Metallic seal)	Heat-up + 9 h at 427 °C + cool-down
6	2**	24 °C at 5 bar (Metallic seal)	Heat-up + 9 h at 427 °C + cool-down
7	1**	24 °C at 5 bar (Metallic seal)	Heat-up + 9 h at 427 °C + cool-down
8	6	24 °C at 5 bar (Metallic seal)	Heat-up + 9 h at 800 °C + cool-down
9	1***	24 °C at 5 bar (Metallic seal)	Heat-up to 427 °C and then to 800 °C for about 4 h + cool-down
10	7	24 °C at 5 bar (Metallic seal)	Incremental heating from 427 °C to 627 ºC with 100 ºC increment§ + cool-down
11	3**	24 °C at 2 bar (ethylene-propylene seal)	Incremental heating from 150 ºC to 300 ºC with 50 ºC increment§§ + cool-down
12	3**	24 °C at 2 bar (PTFE seal)	Incremental heating from 150 ºC to 300 ºC with 50 ºC increment§§ + cool-down
13	8	24 °C at 5 bar (Metallic seal)	Incremental heating from 427 °C to 727 ºC with 100 °C increment§ + cool-down
14	9	24 °C at 5 bar (Metallic seal)	Heat-up + 9 h at 800 °C + cool-down
15	3**	24 °C at 2 bar (ethylene-propylene seal)	Heat-up + more than 24 h at 450 ºC

*Shakedown test; during this test DAQ malfunctioned, and no temporal data was collected.
**flange and groove surfaces refurbished.
***flange and groove surfaces refurbished for second time.
§Vessel was heated at each set temperature for 9 h or more; exact exposure duration for each test is described in Section 3.
§§Vessel was heated at 150 °C (302 °F) for 1 h, 200 °C (392 °F) for 1 h, 250 °C (482 °F) for 1 h, and 300 °C (572 °F) for more than 20 h.

3 RESULTS AND DISCUSSION

In this section, test results are presented and discussed. It should be noted that the pressure drop method was applied under isothermal conditions. During the transient 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. The temporal volumetric thermal expansion of the pressure vessel also introduced an additional parameter. 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 might still show an increase in pressure, thus masking the leak. Therefore, only the vessel pressure data at an elevated constant temperature were used to properly detect potential leak. Another indicator for leak was to determine whether the vessel pressure could be restored to the initial vessel pressure after the thermal exposure test. The restoration of initial vessel pressure indicated no leak had occurred during a test.

3.1 Test #1 (Vessel #1)

The thermal exposure test using Vessel #1 was intended to be a shakedown of the experimental apparatus. The entire heating process took about 4.5 h for the flange temperature to reach the equilibrium furnace temperature of 800 °C (1472 °F). The vessel was then maintained at this temperature for an additional 30 min before turning off the electric furnace.

During the heat-up phase, the DAQ readings from the pressure transducer and the thermocouples became erratic due to unknown reasons; 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 ºC (1472 °F), 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 that no leakage had occurred.

A post-test inspection of the test 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 3-1. The high-temperature exposure also discolored the test fixture.

Figure 3-1. Post-test photographs showing silver from the metallic O-ring deposited on the O-ring groove (above) and the O-ring bonded to the test vessel body (below).

3.2 Test #2 (Vessel #2)

Before Test #2 was conducted, an exploratory test was performed to examine the severity of the leak without using a metallic O-ring. The vessel was charged with helium to 5.62 bar at room temperature, and the vessel pressure was monitored at room temperature. Figure 3-2 is the test result, which indicates that the vessel pressure began to drop and leakage occurred immediately at the start of the test and continued slowly over the entire experimental run-time (26.43 h). The reference helium leakage rates $L_{R,He}$ (ref·cm^3/s) were estimated based on the method described in Appendix D. The time-averaged leakage rate is estimated to be 2.4×10^{-3} ref·cm^3/s.

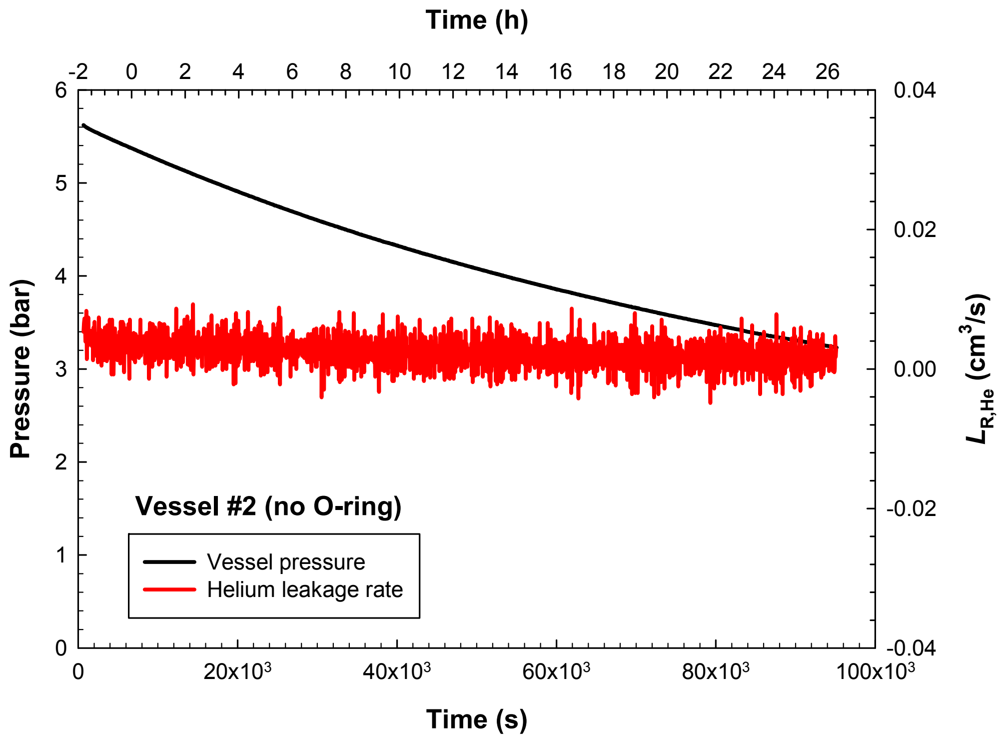

Figure 3-2. Temporal variations of vessel pressure at 24 °C without metallic O-ring.

The vessel was disassembled and assembled again using a metallic seal to start Test #2. Figure 3-3 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 a portion of the cool-down phases for Test #2. The rapid decrease in the furnace temperature signifies turning-off the furnace and the beginning of the cool-down phase. Figure 3-4 shows a re-plot of Figure 3-3 with the time scale extended to include the entire cool-down phase, which normally takes more than several days to naturally cool the test vessel inside the powered-down furnace from 800 °C (1472 °F) to room temperature.

The scale of the ordinate for pressure in Figure 3-3 is magnified to show that the pressure starts to decrease shortly after the vessel temperature has reached 800 °C (1472 °F) and continues to decrease very slowly during the rest of the 9 h constant-temperature heating phase. Although the continuous downward trend does seem to imply the occurrence of a very tiny leak, the decrease in pressure is within the expanded measurement uncertainty of the pressure transducer (0.11 bar) and is not considered significant in this test. However, the complete thermal exposure history, shown in Figure 3-4, indicates that the vessel pressure is restored to its original charge pressure (≈ 5 bar) after the thermal exposure, indicating no leakage.

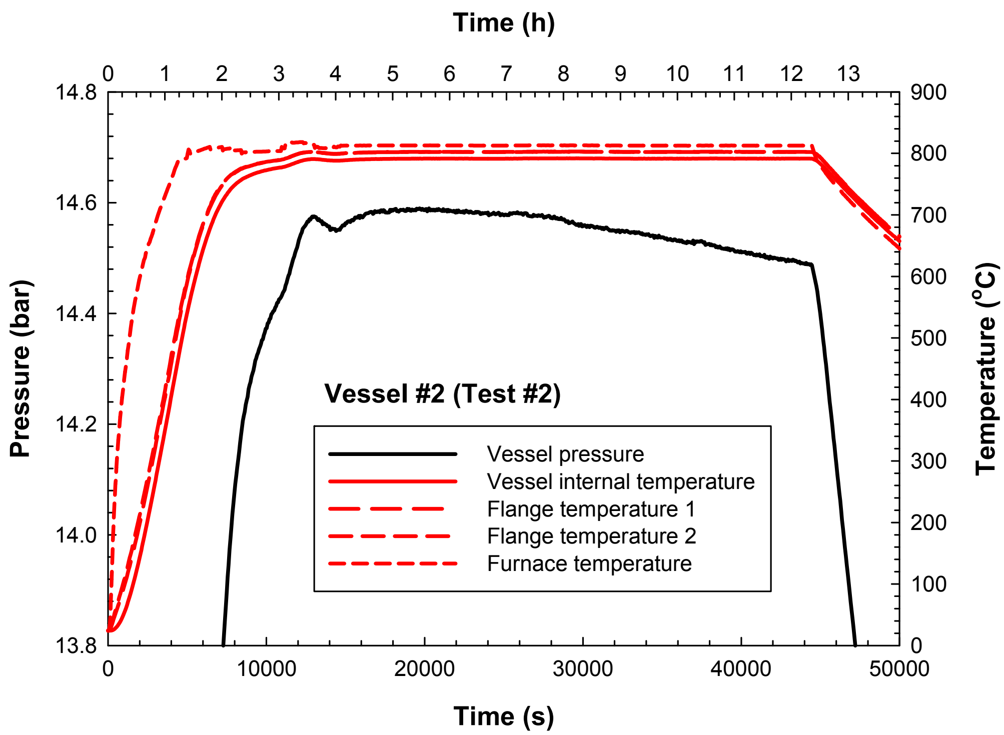

Figure 3-3. Temporal variations of vessel pressure and temperature in Test #2.

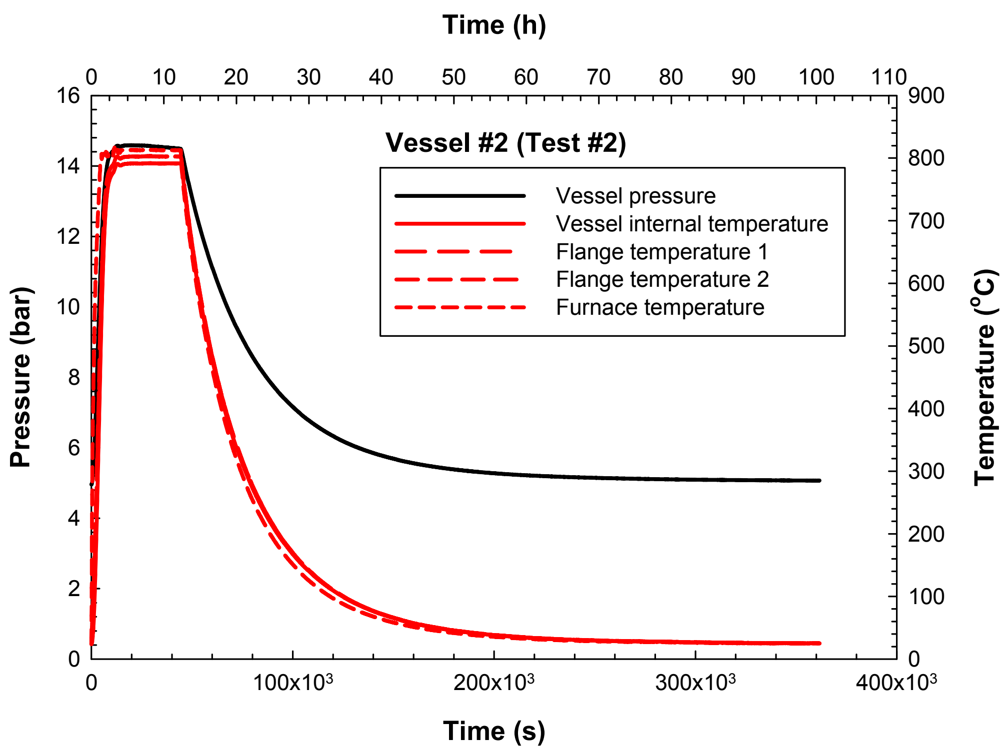

Figure 3-4. Temporal variations of vessel pressure and temperature in Test #2 (time scale extended, including the complete cool-down phase).

3.3 Test #3 (Vessel #3)

Tests #3 is a repeat of Test #2. Figure 3-5 presents the test results which show the occurrence of a leak during the 9 h constant-temperature heating phase at 800 °C (1472 °F). In Test #3, the vessel pressure decreases slowly initially and then significantly after about 25 000 s (6.9 h) at 800 °C (1472 °F). 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. Another leak indicator is revealed in Figure 3-6, which shows the entire time history of the test. After cooling to room temperature, the vessel does not recover its initial charge pressure of approximately 5 bar because of the leak. Isothermal reference helium leakage rates ($L_{R,He}$) during the 9 h heating in this test are shown in Figure 3-7.

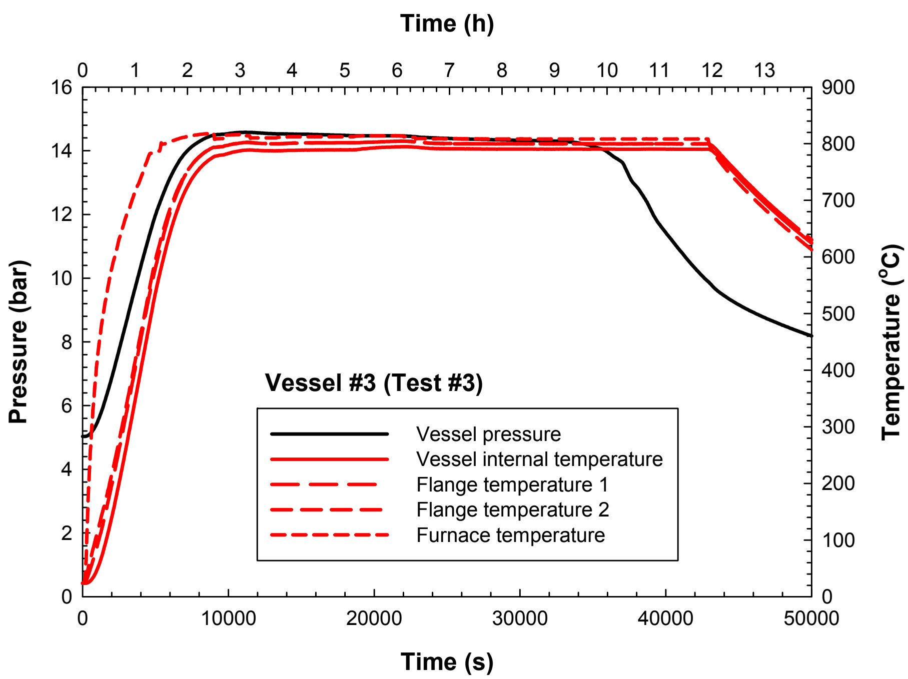

Figure 3-5. Temporal variations of vessel pressure and temperature in Test #3.

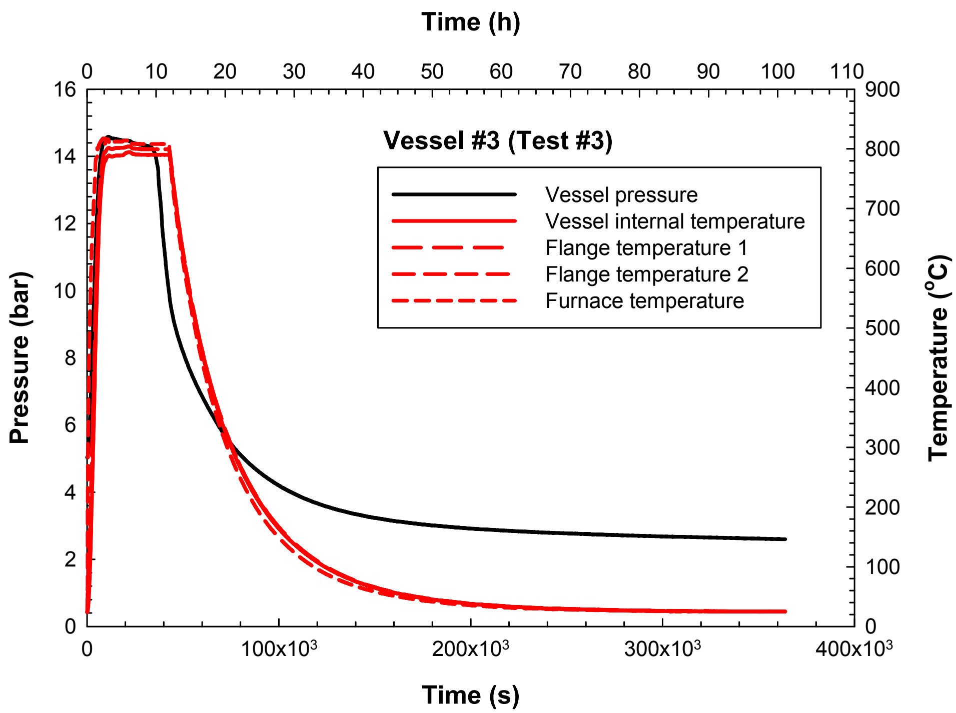

Figure 3-6. Temporal variations of vessel pressure and temperature in Test #3 (time scale extended, including the complete cool-down phase).

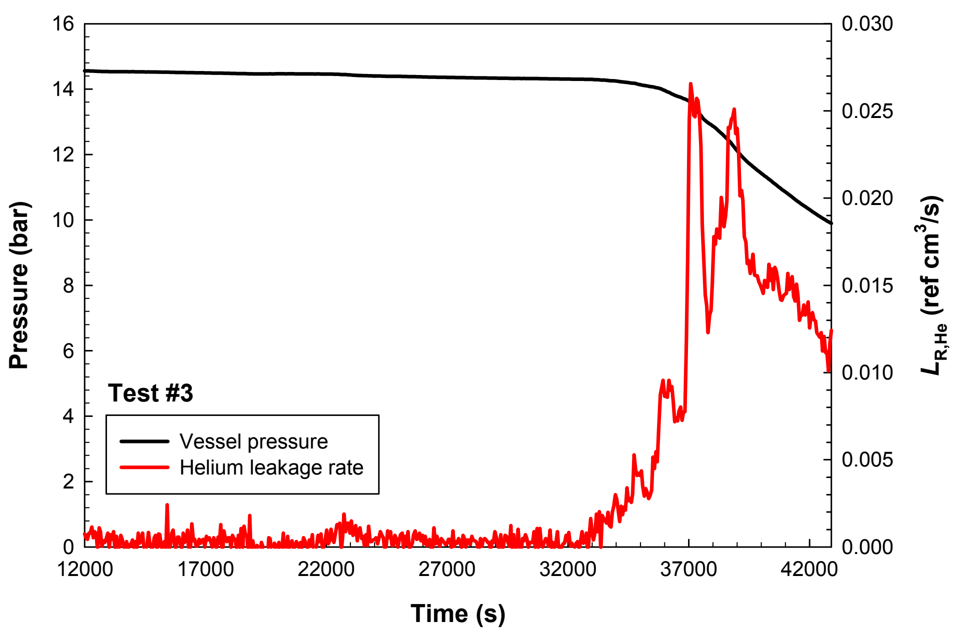

Figure 3-7. Isothermal reference helium leakage rate during the 9 h heating in Test #3.

3.4 Test #4 (Vessel #4)

Test #4 is another repeat of the thermal exposure condition used in Test #2. Figure 3-8 shows the test results from Test #4. In this test, the pressure remained relatively constant for about 10 000 s (2.8 h) initially during the 9 h constant-temperature heating phase, begins to drop significantly for about 15 000 s (4.2 h) with large *dP*/*dt*, and decreases slowly with small *dP*/*dt* for the remaining duration. Figure 3-9 shows the complete time-pressure and time-temperature histories of the test. The calculated reference helium leakage rates are shown in Figure 3-10.

It is interesting to note that despite the occurrence of a leak in these two tests (Tests #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 that re-sealing could potentially occur as the vessel was cooled down.

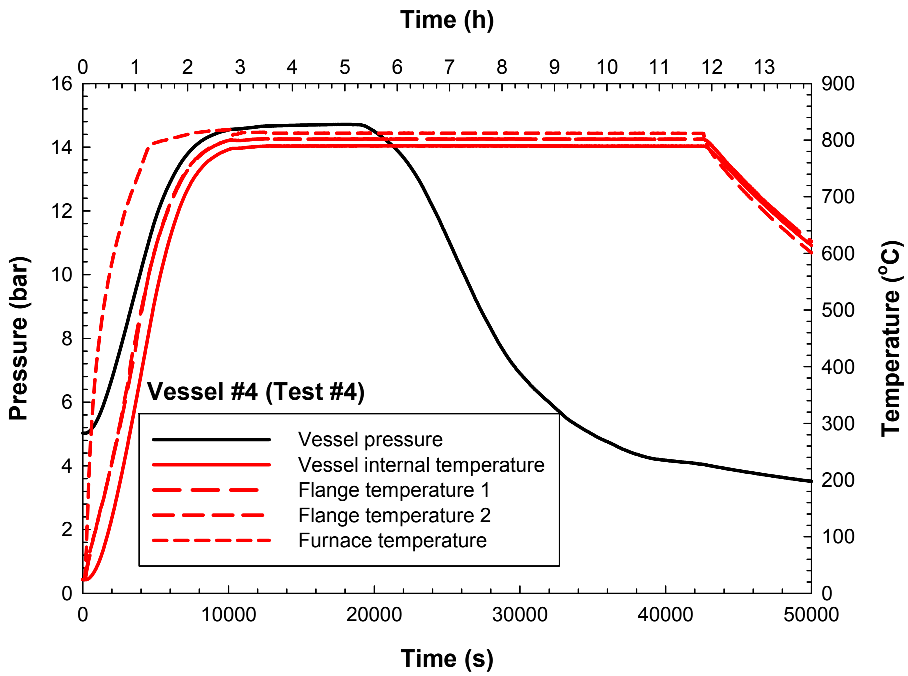

Figure 3-8. Temporal variations of vessel pressure and temperature in Test #4.

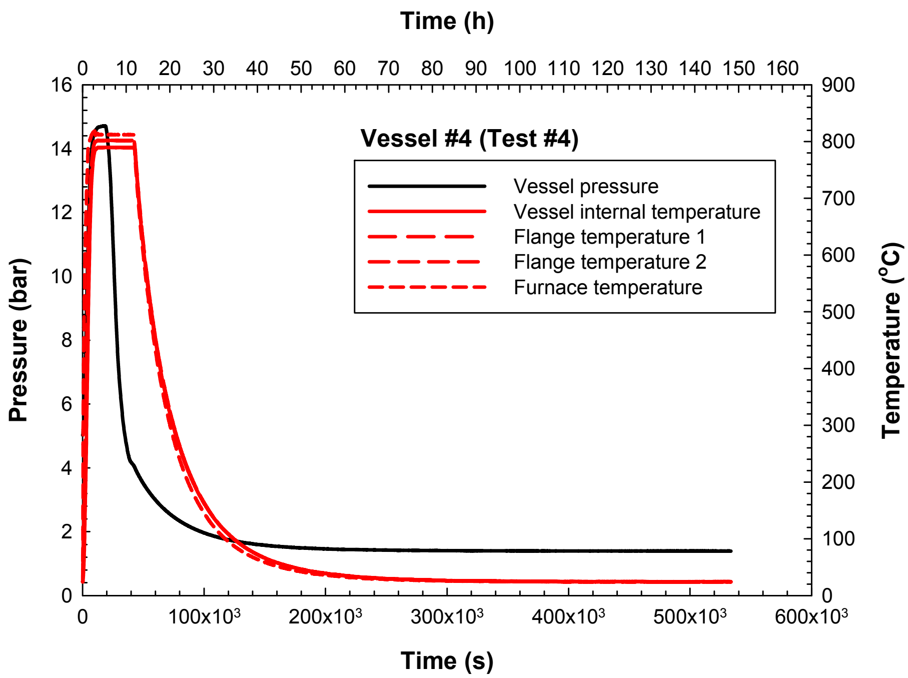

Figure 3-9. Temporal variations of vessel pressure and temperature in Test #4 (time scale extended, including the complete cool-down phase).

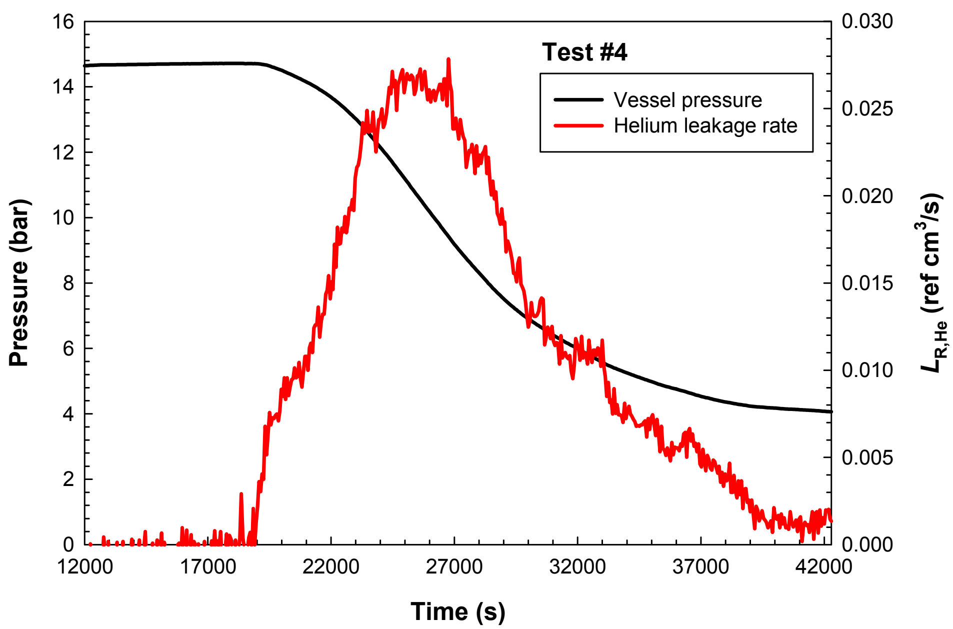

Figure 3-10. Isothermal reference helium leakage rate during the 9 h heating in Test #4.

3.5 Test #5 (Vessel #5)

The results for Test #5 are shown in Figure 3-11. The vessel pressure remained constant during the entire 9 h constant-temperature heating period at 427 °C (800 °F). The seal held vessel pressure. This was confirmed as 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. Figure 3-12 shows the complete thermal exposure cycle of the test.

A photograph of the exposed vessel is shown in Figure 3-13. The extent of discoloration of the vessel depends on the test temperature (Figure 3-1 *vs*. Figure 3-13). A post-test 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.

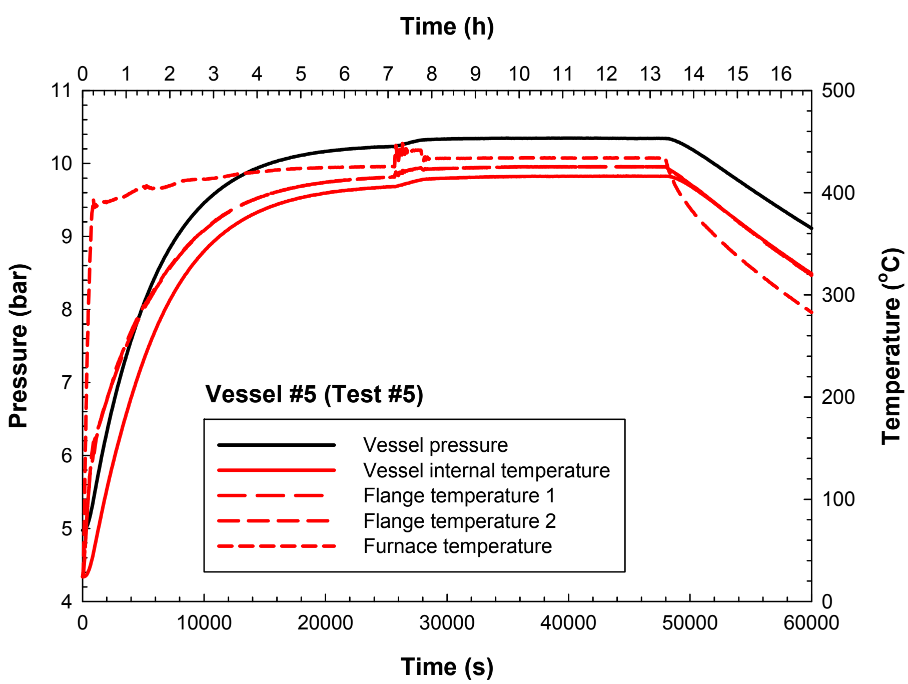

Figure 3-11. Temporal variations of vessel pressure and temperature in Test #5.

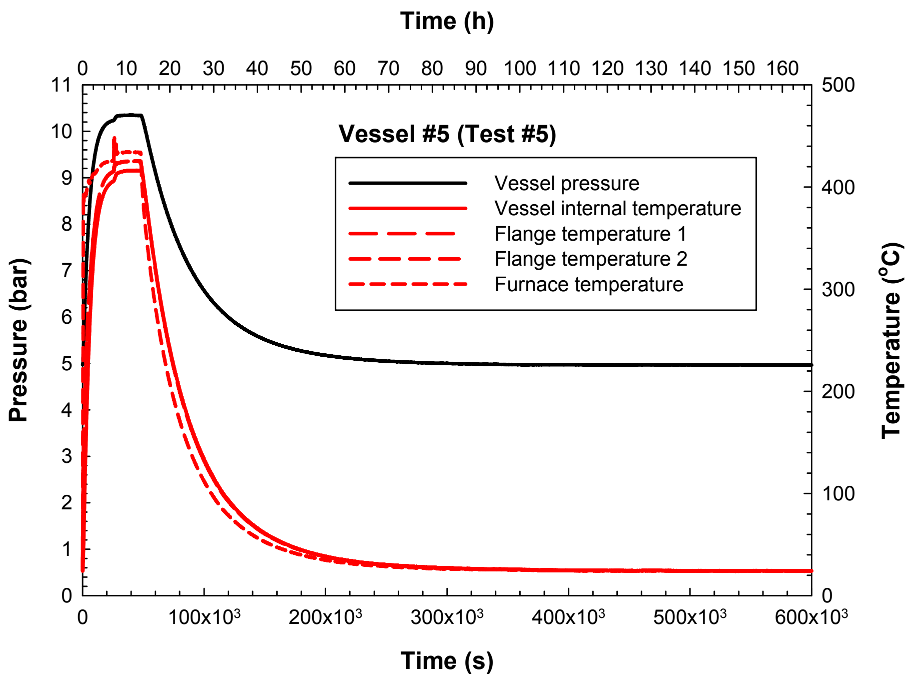

Figure 3-12. Temporal variations of vessel pressure and temperature in Test #5 (time scale extended, including the complete cool-down phase).

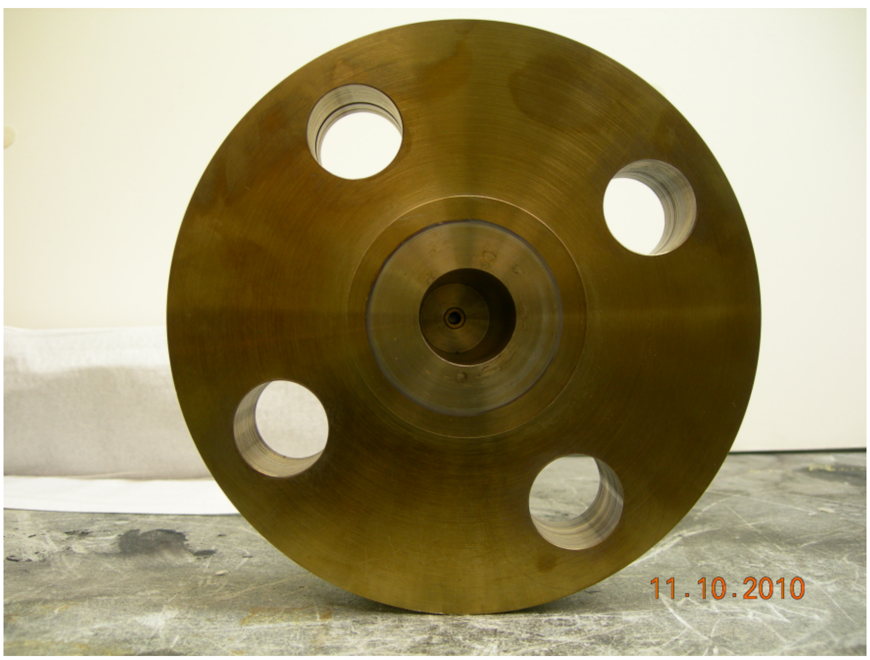

Figure 3-13. Post-test photograph of Vessel #5 (vessel body with O-ring removed) after exposure at 427 °C (800 °F) for 9 h.

3.6 Test #6 (Vessel #2 Refurbished)

The results for Test #6 using the refurbished Vessel #2 are shown in Figure 3-14. 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 3-14 to elucidate the continuous decrease in pressure. However, the small pressure drop is within the pressure measurement uncertainty of 0.11 bar. Although the continuous decrease in pressure does seem to imply that a very small leak might have occurred during the test, the initial charge pressure was restored when the vessel was cooled to room temperature after the thermal exposure (as shown in Figure 3-15), an indication of no leakage.

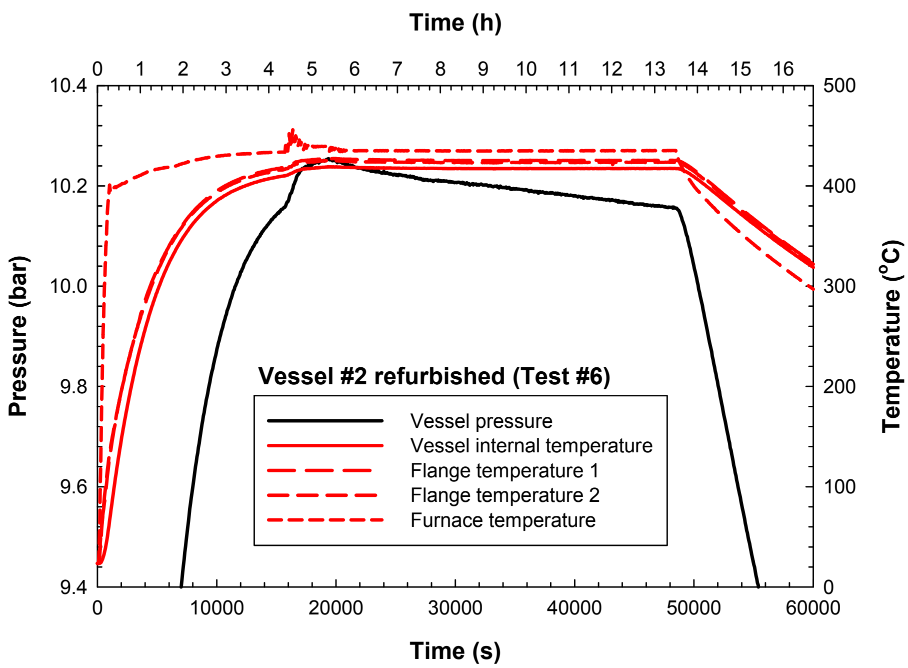

Figure 3-14. Temporal variations of vessel pressure and temperature in Test #6.

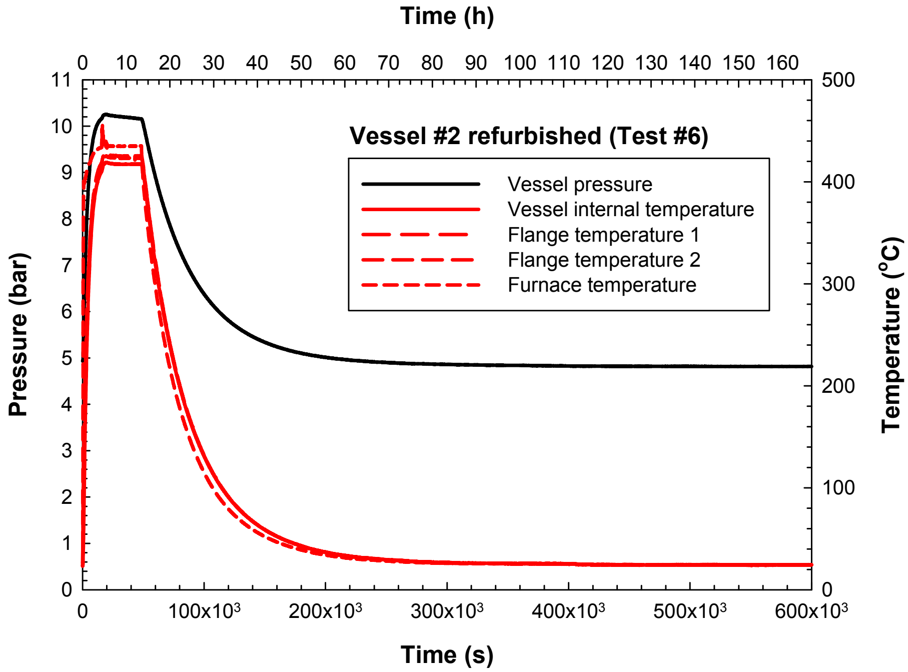

Figure 3-15. Temporal variations of vessel pressure and temperature in Test #6 (time scale extended, including the complete cool-down phase).

3.7 Test #7 (Vessel #1 Refurbished)

Figure 3-16 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 leakage was observed during Test #7. Figure 3-17 is the complete thermal exposure history of Test #7.

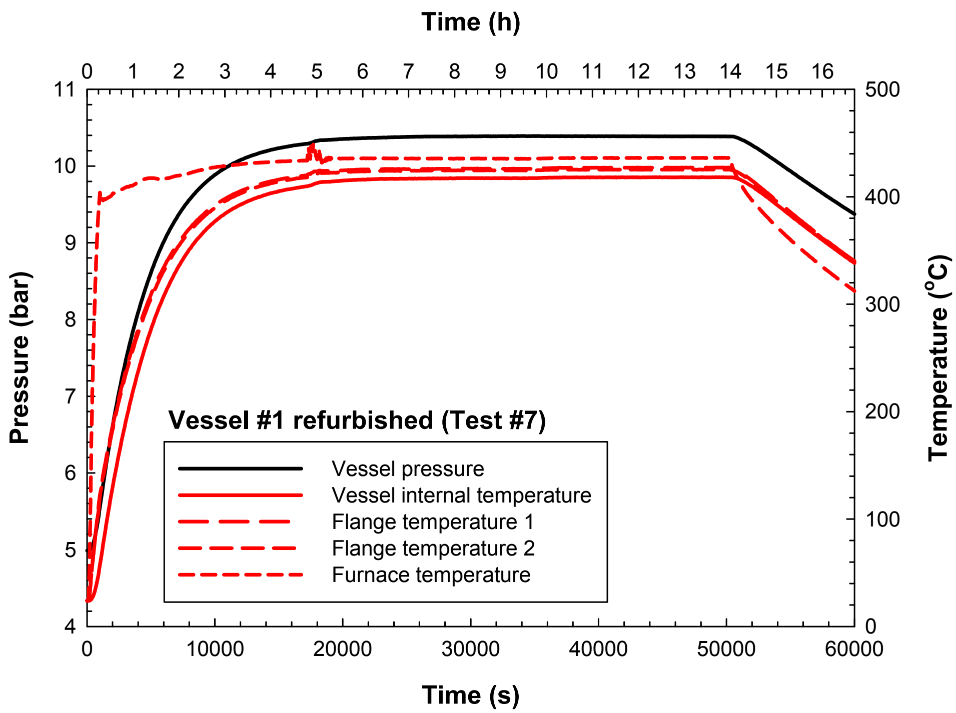

Figure 3-16. Temporal variations of vessel pressure and temperature in Test #7.

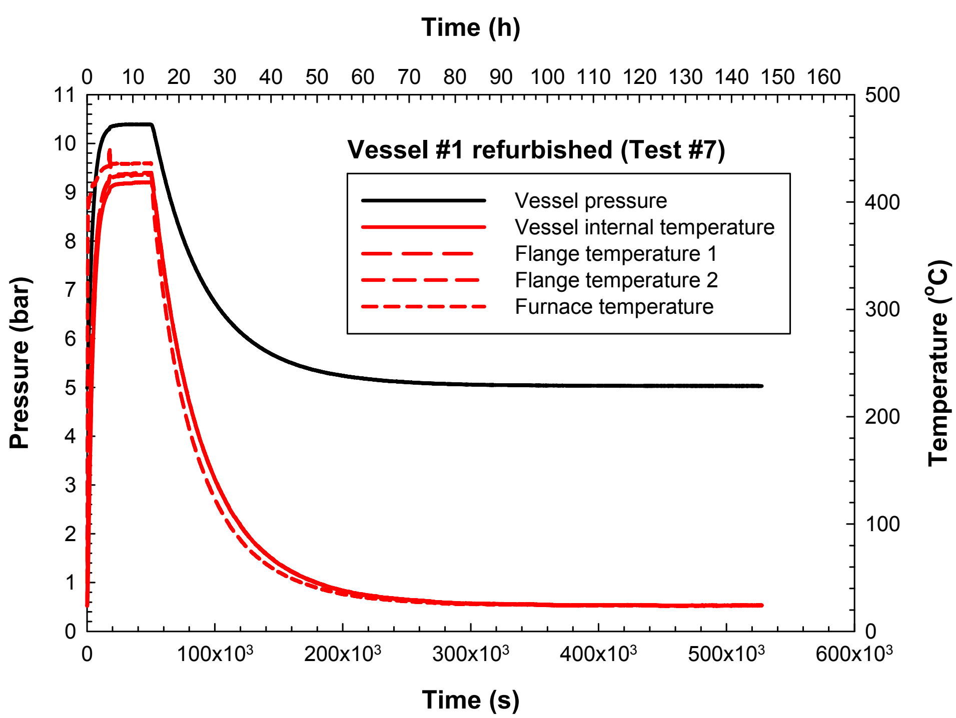

Figure 3-17. Temporal variations of vessel pressure and temperature in Test #7 (time scale extended, including the complete cool-down phase).

3.8 Test #8 (Vessel #6)

The test conditions were similar to Tests #2, 3, and 4. Figure 3-18 shows the thermal exposure results for Vessel #6. The pressure trace indicates that leakage occurs after about 3 h exposure to 800 °C (1472 °F). For this test, the pressure initially decreases very rapidly, followed by a gradual decrease in vessel pressure. However, the pressure never dropped below 2 bar, indicating that the seal had some residual sealing capacity. Figure 3-19 is the complete time-pressure and time-temperature histories of Test #8. Figure 3-20 shows the calculated isothermal reference helium leakage rates during the 9 h heating.

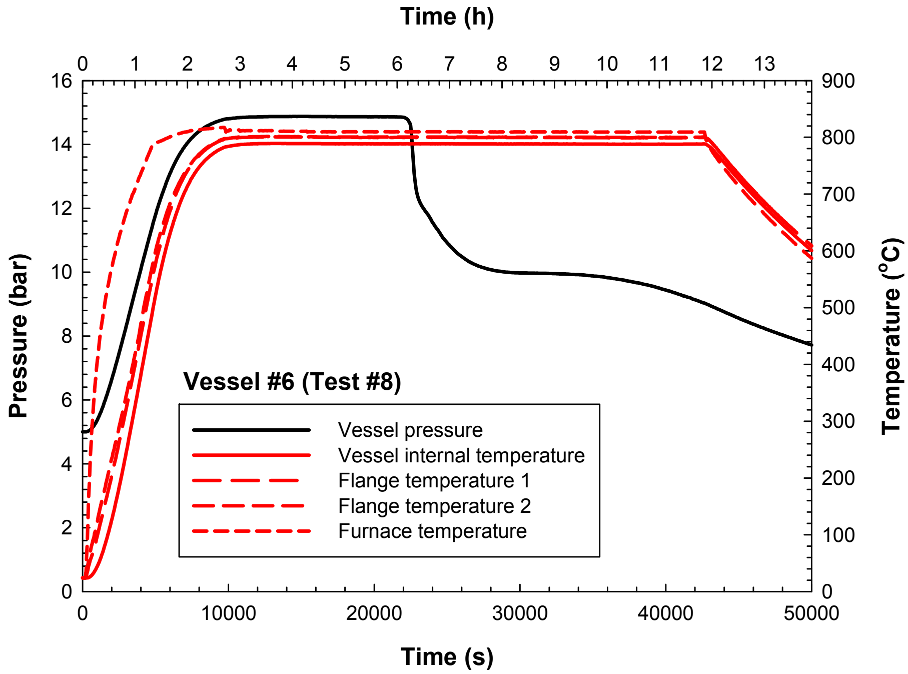

Figure 3-18. Temporal variations of vessel pressure and temperature in Test #8.

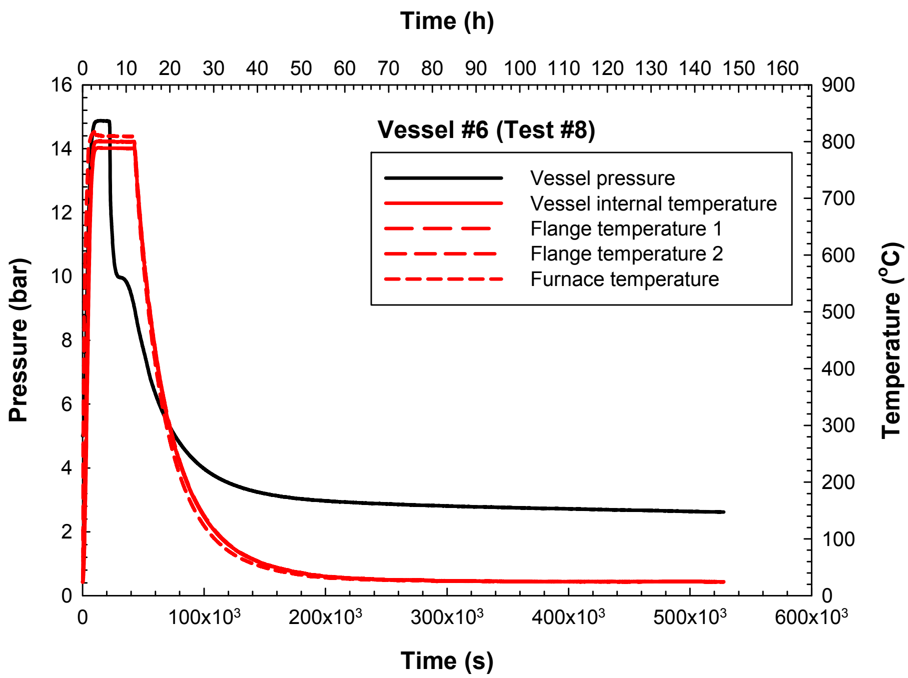

Figure 3-19. Temporal variations of vessel pressure and temperature in Test #8 (time scale extended, including the complete cool-down phase).

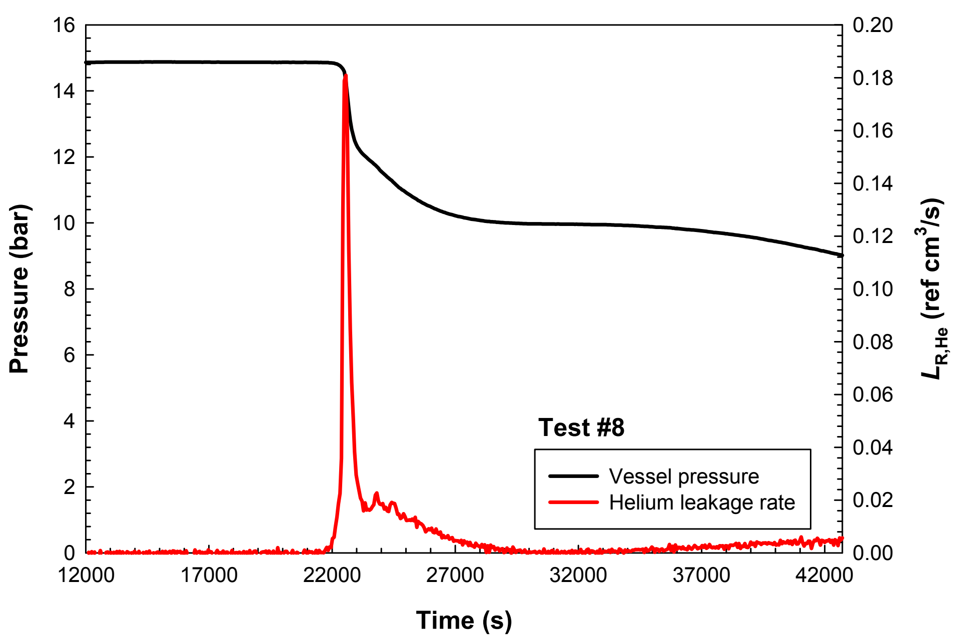

Figure 3-20. Isothermal reference helium leakage rate during the 9 h heating in Test #8.

3.9 Test #9 (Vessel #1 Re-Refurbished)

Test #9 was performed using Vessel #1, which had been used once at 800 °C (1472 °F) for 30 min and once at 427 °C (800 °F) for 9 h and refurbished twice. The sole intent of this test was to check the performance of the pressure transducer against a pressure gauge[8] to ensure that the pressure transducer functioned properly. Figure 3-21 shows the set-up used for this comparative test. The only difference between this arrangement and the original was the addition of a pressure gauge.

The test protocol involved the heating of the vessel to 427 °C (800 °F). Once the vessel reached 427 °C (800 °F), the furnace temperature was raised to 800 °C (1472 °F), and the vessel was heated at 800 °C (1472 °F) for a little more than 3 h. Figure 3-22 and Figure 3-23 show the pressure and temperature data using different time scales respectively. No leak is indicated in the figures since the attainable pressure at 800 °C (1472 °F) remained constant. However, the pressure at 800 °C (1472 °F) was lower than those recorded in previous tests under similar conditions (≈ 12 bar *vs.* ≈ 14.5 bar). This is due to the increase of the measurement volume with the addition of the pressure gauge which used a C-shape Bourdon tube with a surprisingly large volume and was located outside the furnace at room temperature (see Appendix D for theoretical volume and pressure calculation) as shown in Figure 3-21. The gauge was dissembled and the internal volume of the Bourdon tube, which had a nearly elliptical cross section, was estimated to be more than 12 mL. Figure 3-24 correlates the pressure readings from the pressure transducer with those from the pressure gauge at different times during the heating phase. The pressure transducer clearly functioned properly, and the transducer readings correspond, within the measurement uncertainties, to those obtained from the pressure gauge.

Figure 3-21. A photograph showing the pressure gauge used to check the pressure transducer performance.

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8 WIKA model with a range of 0 bar (0 psig) to 13.79 bar (200 psig) and a resolution of 0.14 bar (2 psig).

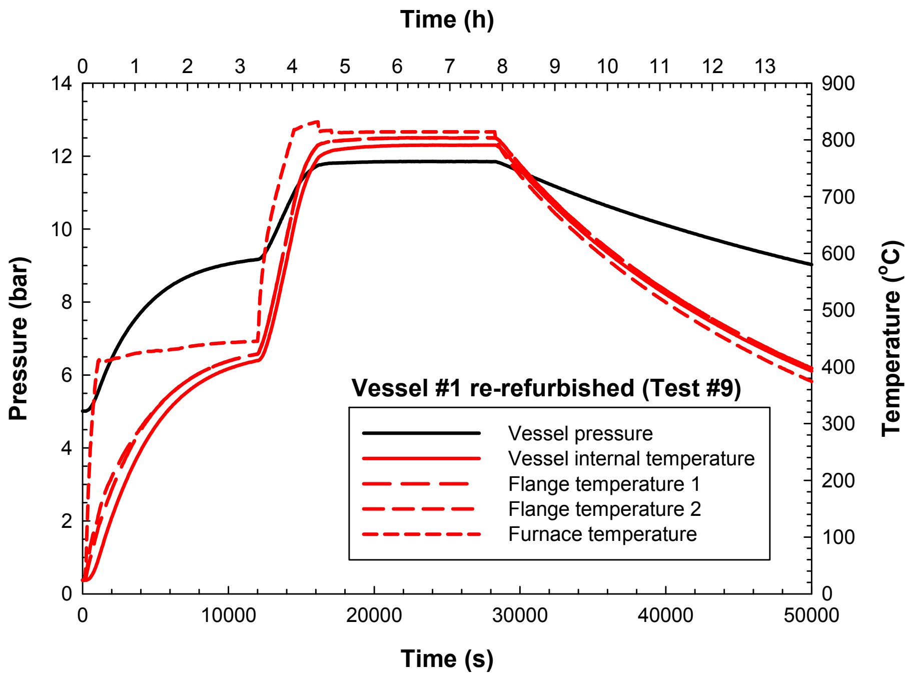

Figure 3-22. Temporal variations of vessel pressure and temperature in Test #9.

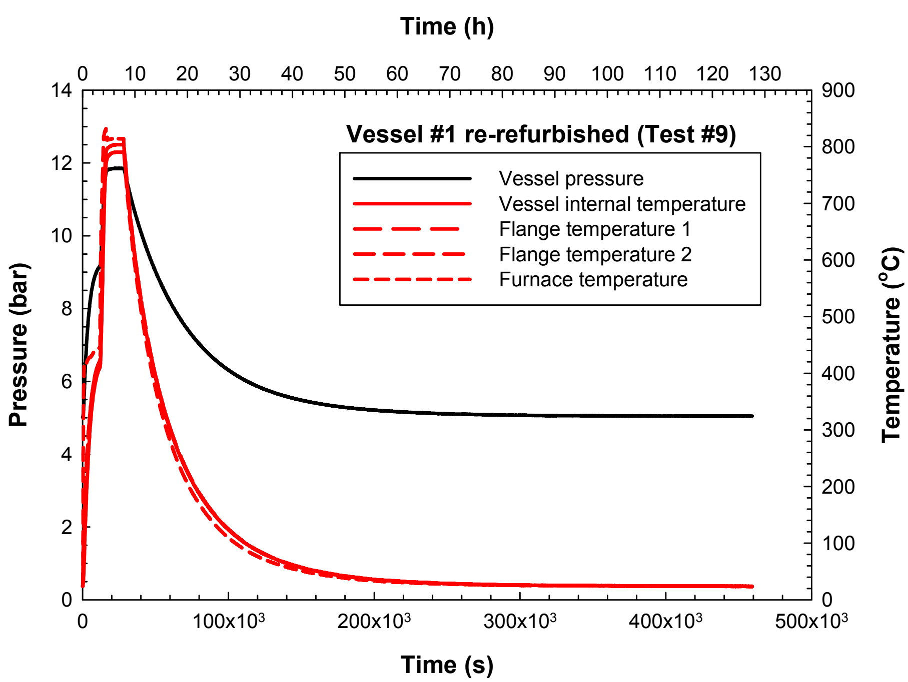

Figure 3-23. Temporal variations of vessel pressure and temperature in Test #9 (time scale extended, including the complete cool-down phase).

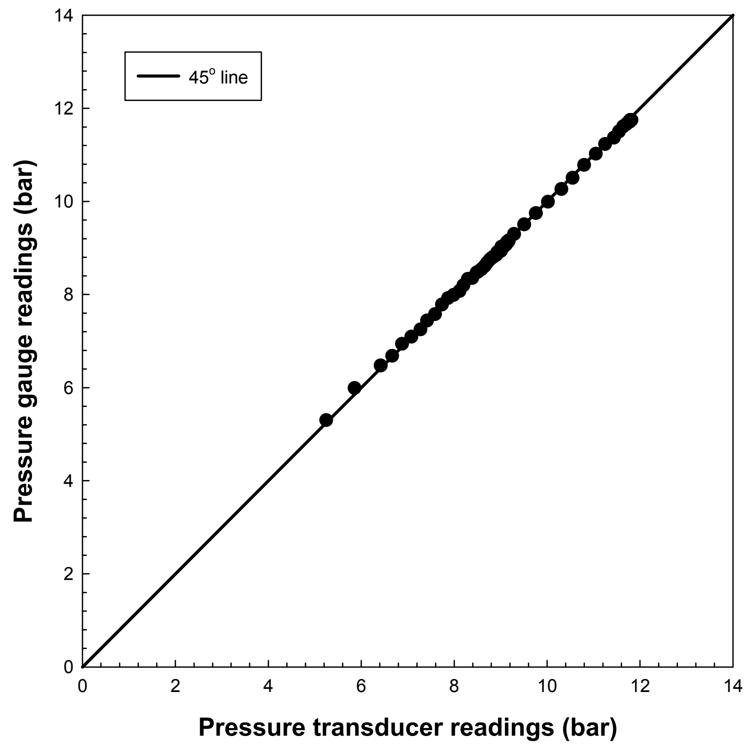

Figure 3-24. Comparison of pressure measurements from pressure transducer and pressure gauge.

3.10 Test #10 (Vessel #7)

In Test #10, an incremental heating protocol was used to assess the metallic seal performance. The vessel was initially heated to 427 °C (800 °F) and held for 9 h at this temperature. Then, the furnace temperature was raised to 527 °C (981 °F), and the vessel was heated at 527 °C (981 °F) overnight (more than 9 h). The vessel was then heated to 627 °C (1161 °F) for 9 h. Figure 3-25 and Figure 3-26 show the test results. No leak was observed based on the pressure traces at the test temperatures, 427 °C (800 °F), 527 °C (981 °F), and 627 °C (1161 °F), and the recovery of the initial pressure at room temperature. Post-test removal of the O-ring from the groove was very easy. A post-test inspection of the vessel indicated that the metallic seal did not bond onto the O-ring groove.

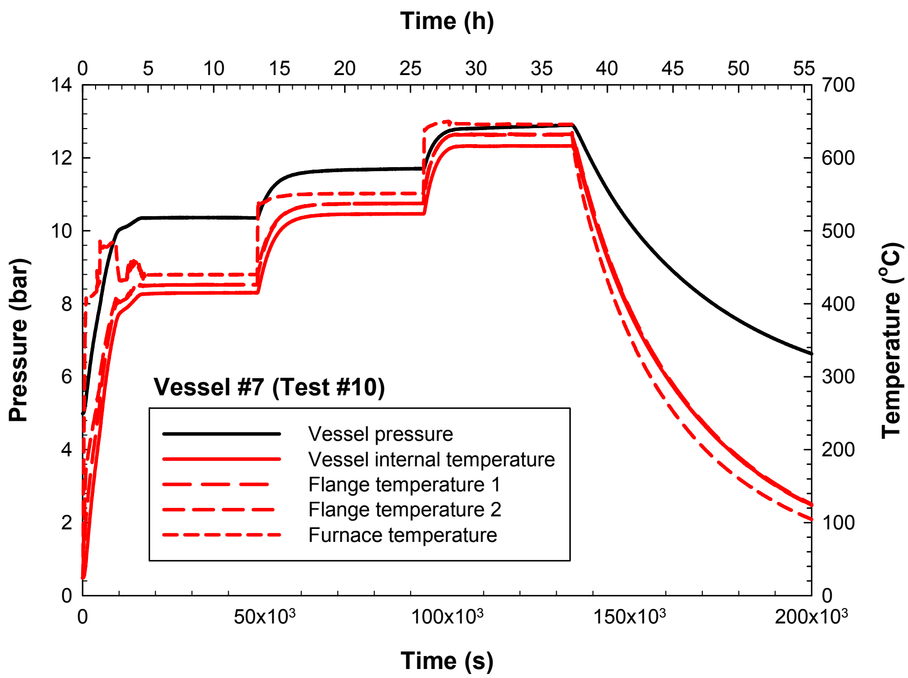

Figure 3-25. Temporal variations of vessel pressure and temperature in Test #10.

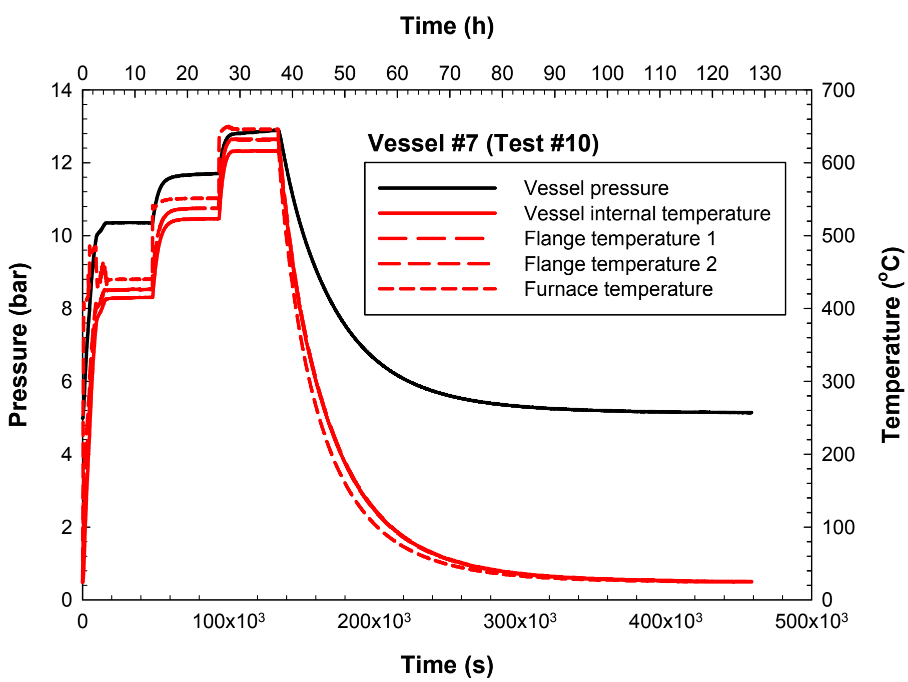

Figure 3-26. Temporal variations of vessel pressure and temperature in Test #10 (time scale extended, including the complete cool-down phase).

3.11 Test #11 (Vessel #3 Refurbished)

An ethylene-propylene compound O-ring (DBR Industries 2-228 E740-75, Lot # 2Q060080040952) with an outer diameter of 6.35 cm (2.5 in.) and a cross section of 0.32 cm (0.125 in.) and refurbished Vessel #3 were used in this test. Vessel #3 was refurbished by re-facing the flange to remove the O-ring groove originally machined for the metallic seal and re-machining a new O-ring groove for the ethylene-propylene compound O-ring which has different groove dimensions from its metallic counterpart.

Figure 3-27 and Figure 3-28 are the test results plotted using different time scales. The spikes (overshoots) in the furnace temperature measurements reflect the way the furnace temperature was intentionally ramped and manipulated to accelerate the incremental heating of the vessel to the set temperatures. During the first 1 h heating at 150 °C (302 °F), the vessel pressure remained constant. The subsequent 1 h heating at 200 °C (392 °F) and the following 1 h heating at 250 °C (482 °F) also indicated the maintenance of vessel pressure. The final heating at 300 °C (572 °F) for more than 20 h shows a slow decrease in pressure, albeit within the pressure measurement uncertainty. However, the vessel pressure was restored to its initial value after the thermal exposure test, as shown in Figure 3-28. Post-test inspection of the exposed vessel revealed that the O-ring was still pliable and could be easily removed from the O-ring groove.

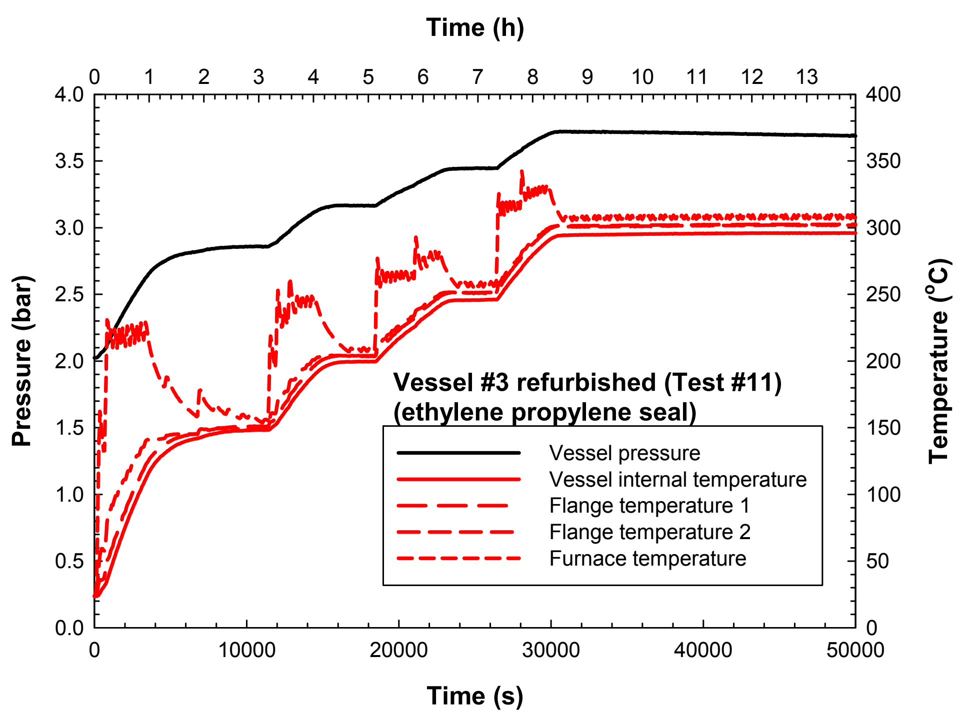

Figure 3-27. Temporal variations of vessel pressure and temperature in Test #11.

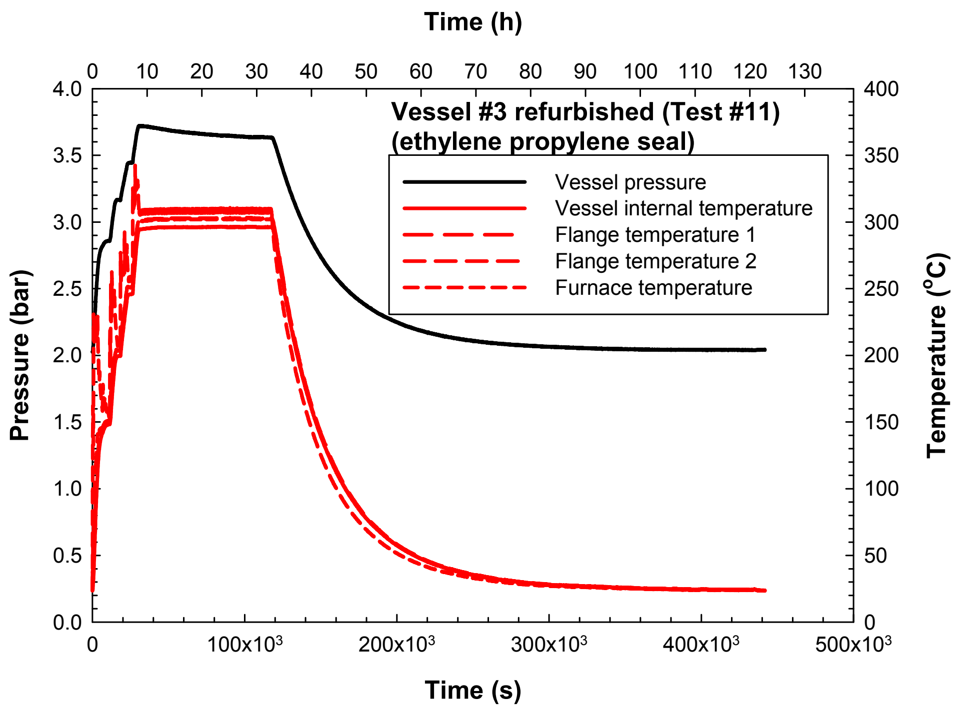

Figure 3-28. Temporal variations of vessel pressure and temperature in Test #11 (time scale extended, including the complete cool-down phase).

3.12 Test #12 (Vessel #3 Refurbished)

A PTFE O-ring (DBR Industries 228 PTFE, Lot #04/07 12730-1) with an outer diameter of 6.35 cm (2.5 in.) and a cross section of 0.32 cm (0.125 in.) and refurbished Vessel #3 was used for this test. The refurbishment of the vessel simply consisted of cleaning of the O-ring groove previously used for the ethylene propylene seal test (Test #11). Figure 3-29 and Figure 3-30 show the results during the heating phase and the complete heating-cooling history respectively. During the 22 h heating at 300 °C (572 °F), there was a slight drop in vessel pressure at the end of the isothermal heating phase; however, the pressure decrease was within the measurement uncertainty of the pressure transducer. Figure 3-31, which shows the calculated reference helium leakage rates during the 22 h heating period, indicates a time-averaged reference leakage rate of 4.5×10^{-5} ref·cm^3/s. The fluctuation in the leakage rate data was due to taking of numerical time derivatives of the unsmoothed pressure-time data. As indicated in the pressure trace, leakage was observed in this test during the cooling phase. The original pressure (≈ 2 bar) did not recover, and the vessel pressure dropped below the original pressure at room temperature. Post-test inspection of the exposed vessel revealed that the compressed O-ring was still intact and could be easily removed from the O-ring groove.

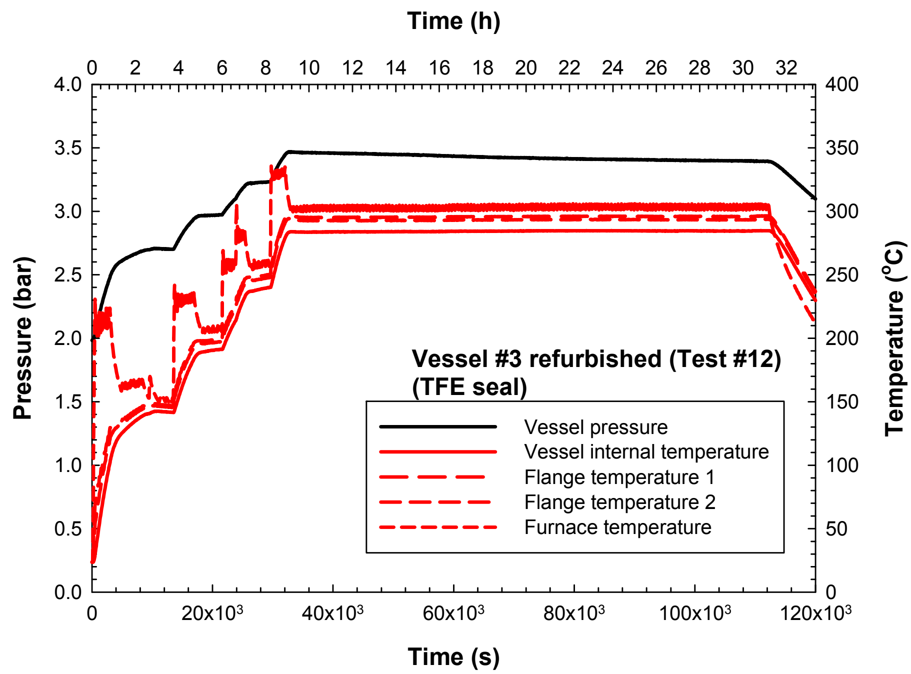

Figure 3-29. Temporal variations of vessel pressure and temperature in Test #12.

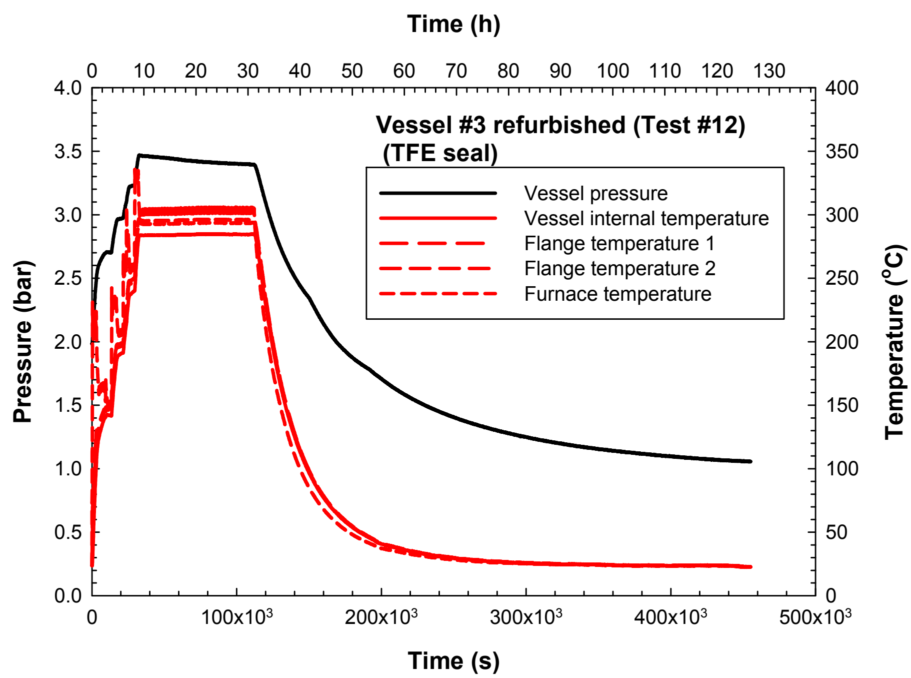

Figure 3-30. Temporal variations of vessel pressure and temperature in Test #12 (time scale extended, including the complete cool-down phase).

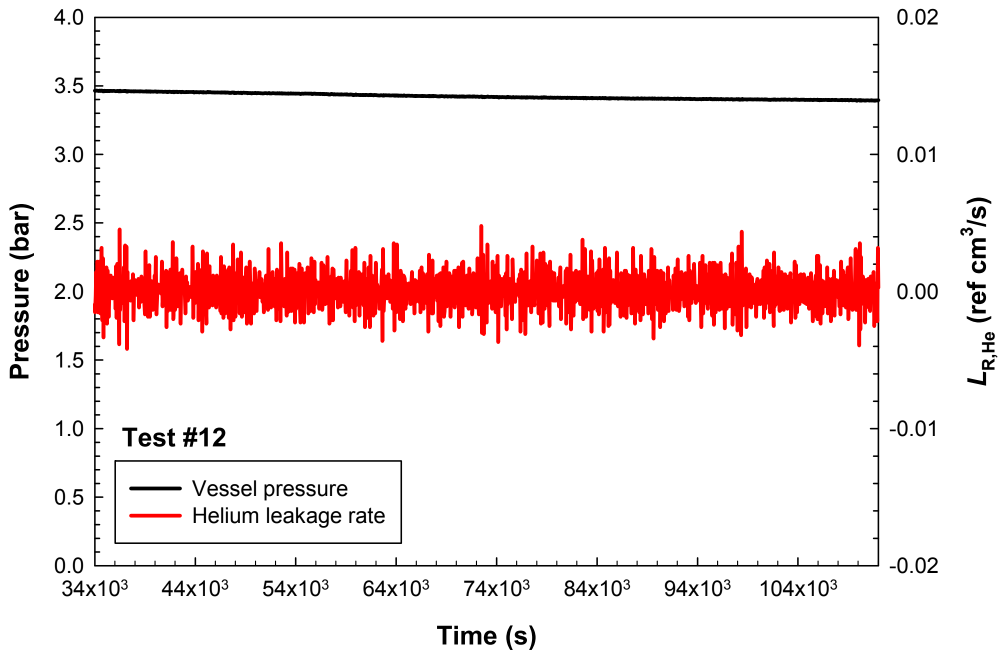

Figure 3-31. Isothermal reference helium leakage rate during the 22 h heating in Test #12.

3.13 Test #13 (Vessel #8)

For Test #13, a metallic seal and a new vessel were used. The vessel was evacuated (for 1 min) and filled with helium (to 5 bar) and then evacuated and filled again twice. Contrary to the previous vessel filling process used in Tests #1 to #12, two additional evacuating and filling cycles were applied in order to determine if the evacuation process had any effect on the results.[9] In this test, an incremental heating protocol was used. The test vessel was first heated at 427 °C for more than 10 h, then at 527 °C for 9 h, then at 627 °C for more than 10 h, and finally at 727 °C for 9 h. Figure 3-32 and Figure 3-33 show the results. No leakage was observed during the entire incremental heating processes. However, after the vessel was cooled down to room temperature, the vessel pressure did not recover to its original (starting) value (≈ 5 bar) but remained at a slightly higher constant value of ≈ 5.2 bar over a duration of more than 50 h, albeit within the uncertainty of the transducer. This was not due to the drift of the pressure transducer because when the vessel content was vented to the atmosphere, the pressure transducer showed an atmospheric pressure reading.

9 Whether the test vessel was evacuated once or three times, no discernible difference in the final attainable vessel pressure was observed under the same test conditions.

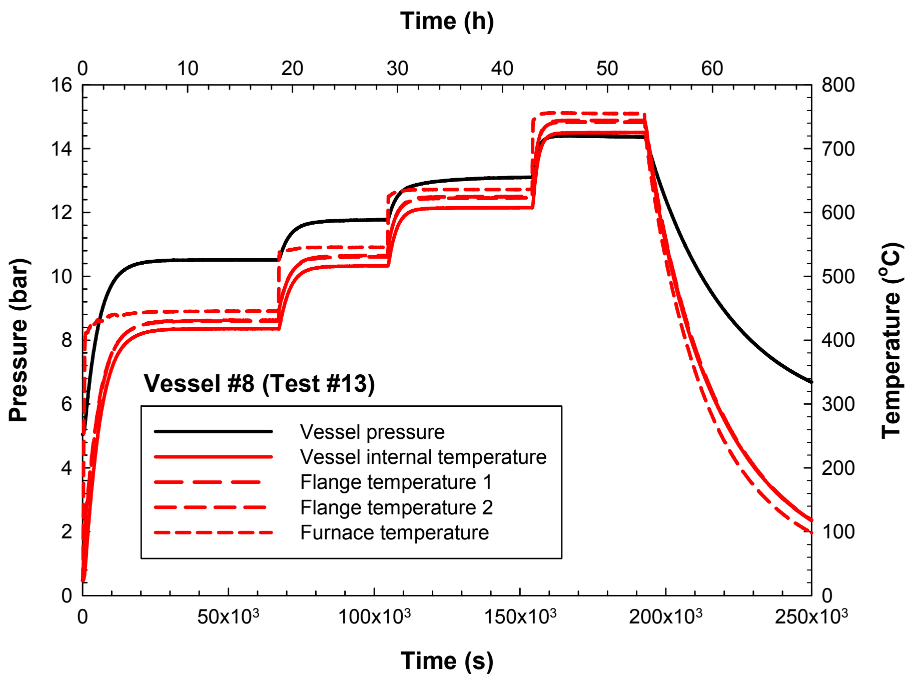

Figure 3-32. Temporal variations of vessel pressure and temperature in Test #13.

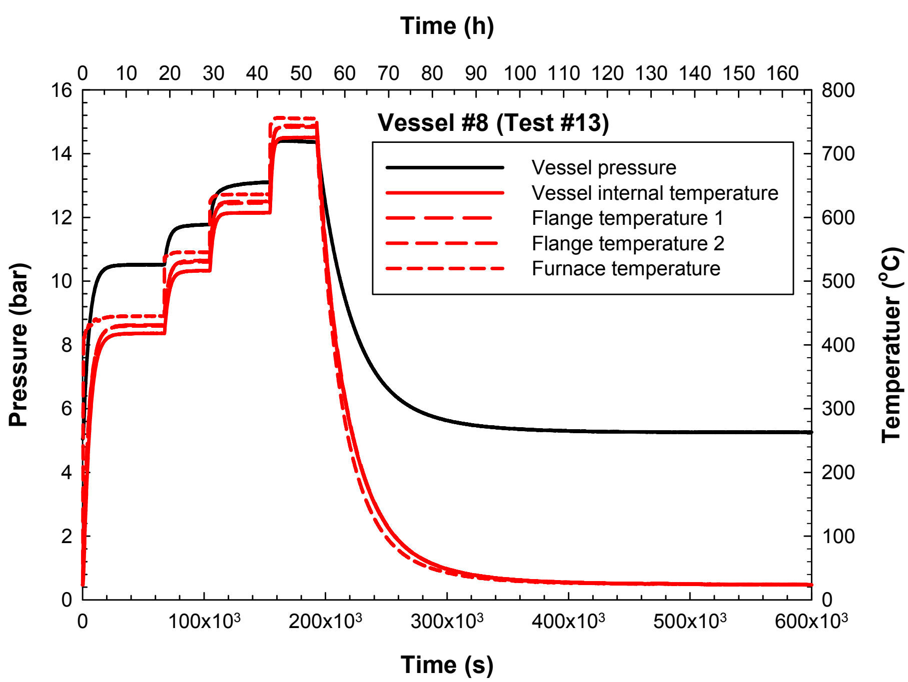

Figure 3-33. Temporal variations of vessel pressure and temperature in Test #13 (time scale extended, including the complete cool-down phase).

3.14 Test #14 (Vessel #9)

In Test #14, a new vessel with a metallic seal was used. The vessel was evacuated and charged with helium three times to prepare for the test. This test was a repeat of Tests #2, #3, and #4. Figure 3-34 shows the first 50 000 s (≈ 14 h) of the pressure-time and temperature-time histories to illustrate the absence of a leak (constant pressure at constant temperature). The pressure-time and temperature-histories during the complete heating and cooling cycle is shown in Figure 3-35. However, similar to the observation in Test #13, after the test vessel was cooled down to room temperature, the vessel pressure did not recover to its starting value (≈ 5 bar) but remained at a slightly higher constant value of ≈ 5.2 bar over a duration of more than 50 h. Again, this was not due to the drift of the pressure transducer because when the vessel content was released to the atmosphere, the pressure transducer gave an atmospheric pressure reading.

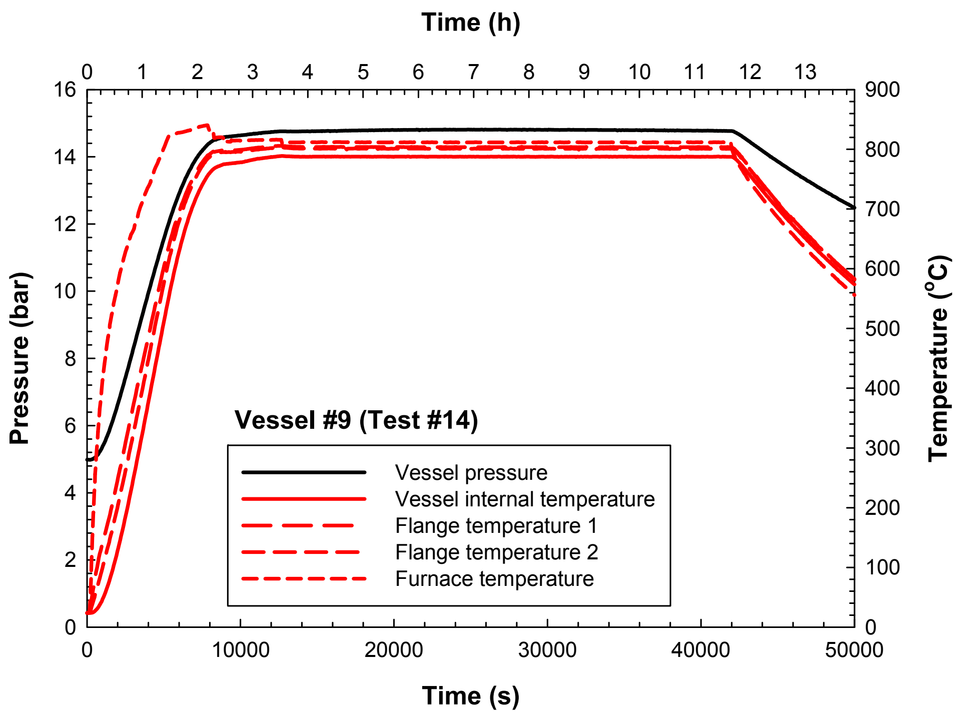

Figure 3-34. Temporal variations of vessel pressure and temperature in Test #14.

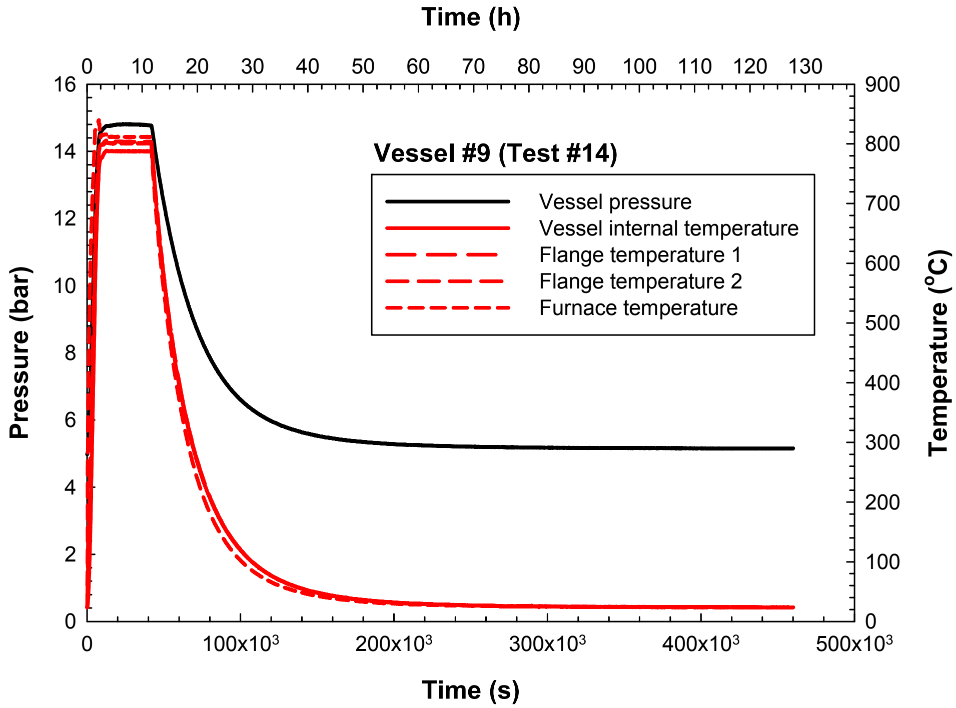

Figure 3-35. Temporal variations of vessel pressure and temperature in Test #14 (time scale extended, including the complete cool-down phase).

3.15 Test #15 (Vessel #3 Refurbished)

In Test #15, an ethylene-propylene compound seal together with the refurbished Vessel #3 (used in Test #11) was employed. The refurbishment simply involved cleaning of the O-ring groove and the cavity of the used vessel with ethanol. The vessel was evacuated and charged with helium three times to prepare for the test. The difference between this test and Test #11 was the temperature used for the thermal exposure test. Instead of 300 °C (572 °F) as used in Test #11, a higher temperature, 450 °C (842 °F) was used.

Figure 3-36 shows the first 39 h of the pressure-time and temperature-time histories obtained from this test. The pressure and temperature histories for the complete heating and cooling cycle are given in Figure 3-37. The pressure trace in Figure 3-36 clearly indicates a leak occurred soon after the vessel had attained the nominal target temperature of 450 °C (842 °F). Figure 3-38 shows the calculated reference helium leakage rates over the 25 h isothermal heating period at 450 °C (842 °F) with a time-averaged reference leakage rate of 9.2×10^{-4} ref·cm^3/s.

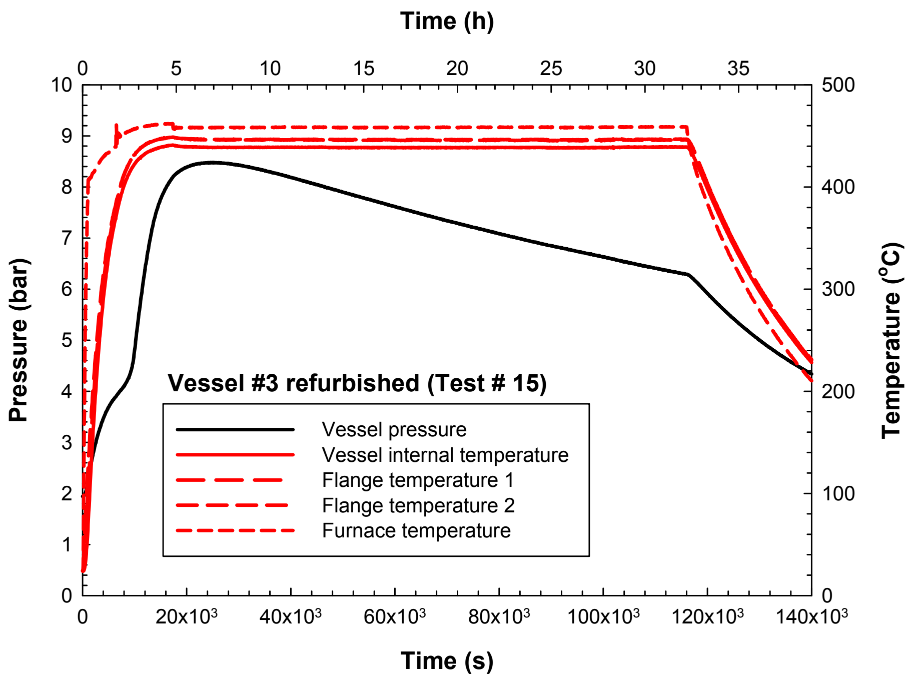

Figure 3-36. Temporal variations of vessel pressure and temperature in Test #15.

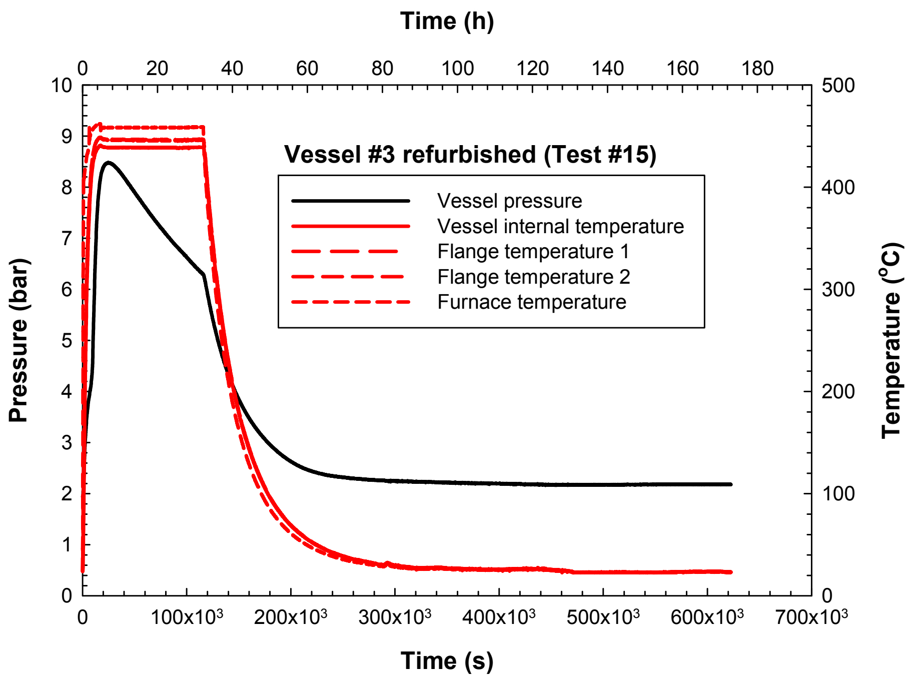

Figure 3-37. Temporal variations of vessel pressure and temperature in Test #15 (time scale extended, including the complete cool-down phase).

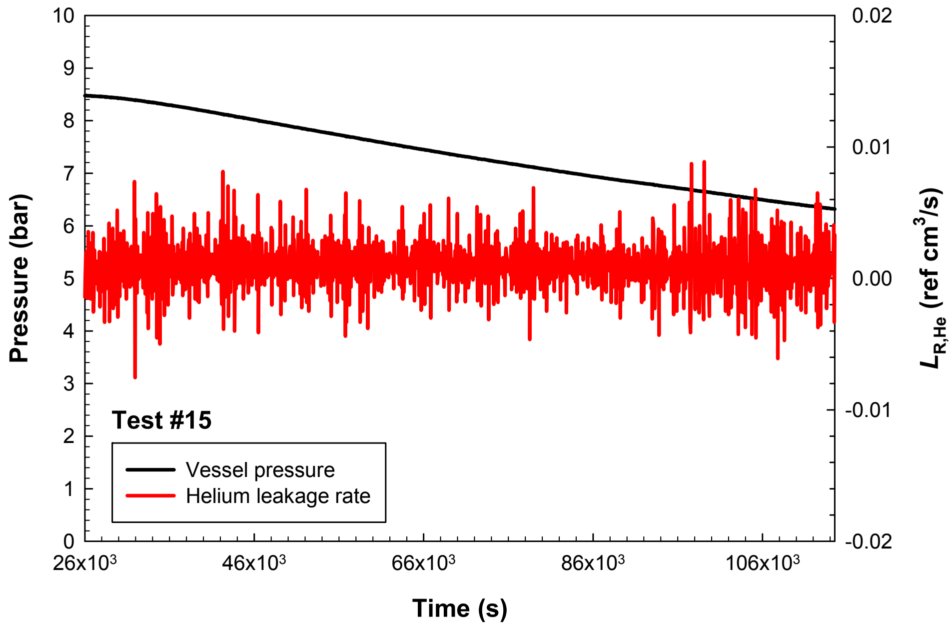

Figure 3-38. Isothermal reference helium leakage rate during the 25 h heating in Test #15.

From Figure 3-36 and Figure 3-37, a peculiarity is noted. The measured attainable pressure (≈ 8.5 bar) at 450 °C (842 °F) before the leak occurred is noticeably higher than the pressure calculated using the ideal gas law (≈ 4.9 bar), even assuming no thermal expansion of the vessel volume. The addition of vapor mass from the potential thermal decomposition and off-gas of the O-ring and the lubricant used at 450 °C (842 °F) is conjectured to be the cause for this unusually high pressure. Another abnormality is that after the vessel was cooled down to room temperature, the pressure never recovered to its original value (≈ 2 bar) but remained relatively constant at ≈ 2.16 bar over a duration of more than 100 h, implying the O-ring still possessed some residual sealing capability. A post-test inspection of the tested vessel revealed that the interior vessel wall and the thermocouple inserted into the vessel interior were coated with a thin layer of black substance. Although the O-ring was still properly seated in the groove (see Figure 3-39), it had turned into a packed layer of powdery charred material and could not be removed from the groove without destroying its structural integrity (see Figure 3-40). Additional tests would be needed to further examine this finding.

Figure 3-39. Photograph of the ethylene-propylene compound O-ring after thermal exposure.

Figure 3-40. Photograph showing the disintegration of the tested ethylene-propylene compound O-ring when an attempt was made to remove the O-ring from the groove.

4 SUMMARY

Fifteen small-scale metallic and polymeric seal performance tests including two “shakedown” tests were performed to explore seal performance during beyond-design-basis thermal exposure conditions. Twelve tests used metallic seals, two used ethylene-propylene compound seals, and one used a PTFE seal.

Of the five repeat metallic-seal tests (Tests #2, #3, #4, #8, and #9), leakage (decreasing vessel pressure) was observed in three of the tests (Tests #3, #4, and #8) during the 9 h 800 °C (1472 °F) exposure. The times when the leakage occurred (the vessel pressure started to decrease) varied in the three tests performed. In Test #3, measurable leakage occurred approximately 6.9 h after the test temperature 800 °C (1472 °F) had been reached. In Test #4, measurable leakage occurred about 2.8 h into the 800 °C (1472 °F) exposure. In Test #8, leakage was observed roughly 3 h into the test. The two shakedown tests were conducted using a 30 min and 4 h exposure to 800 °C (1472 °F), respectively, and the seal appeared to hold vessel pressure. No leakage (unchanged vessel pressure within the pressure measurement uncertainty) was also observed in the two metallic seal tests that used 100 °C (212 °F) incremental heating from 427 °C (800 °F) to 627 °C (1161 °F) and from 427 °C (800 °F) to 727 °C (1341 °F), respectively, with at least 9 h exposure at each temperature increment. Three repeat metallic seal tests were also conducted at the seal maximum operating temperature of 427 °C (800 °F) for 9 h. The seal maintained vessel pressure within the measurement uncertainty of the pressure transducer in all three tests.

No leakage was observed in one ethylene-propylene compound seal tested at 300 °C (572 °F) for more than 20 h; however, leakage was observed immediately after the vessel had attained the nominal target temperature of 450 °C (842 °F) in another ethylene-propylene compound seal test (Test #15). Leakage was also observed in the test (Test #12) that used a PTFE seal *after* it had been subjected to 300 °C (572 °F) exposure for 22 h during the cooling phase.

Since only a very limited number of tests were conducted using polymeric seals, further testing is recommended to verify the observations. In addition, the effect of scale on seal performance under beyond-design-basis thermal exposure should also be explored to ensure that the results are not dependent on the size of the test fixture.

5 REFERENCES

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APPENDIX A

A.1 Design Drawings of the Test Vessel

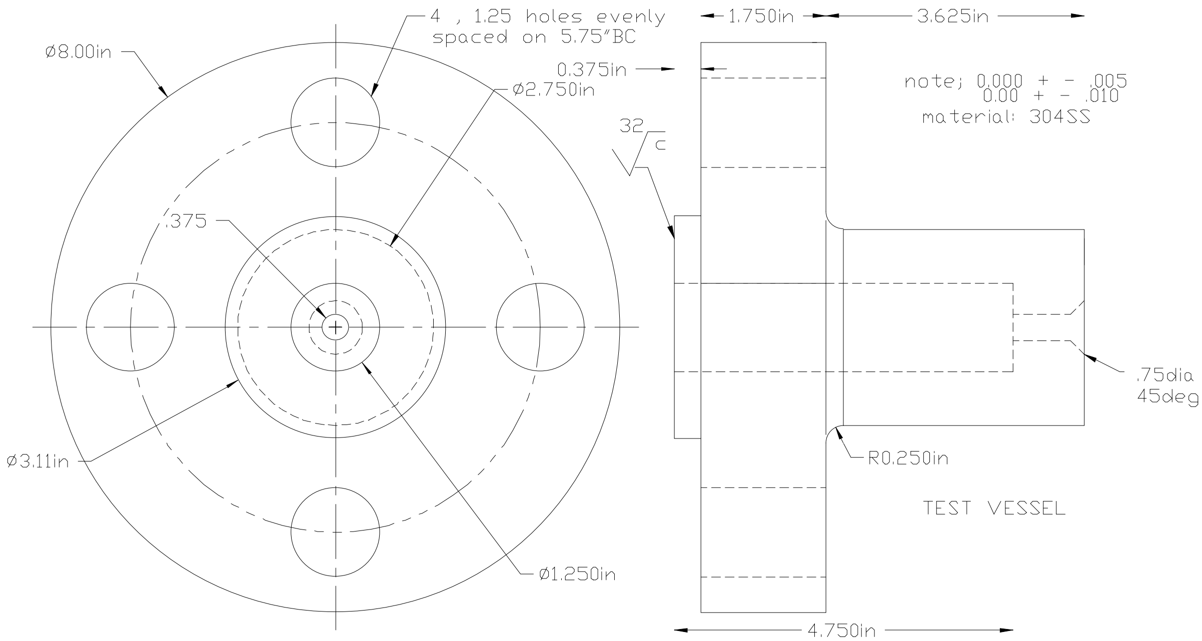

Figure A-1. Design drawing of test vessel body.

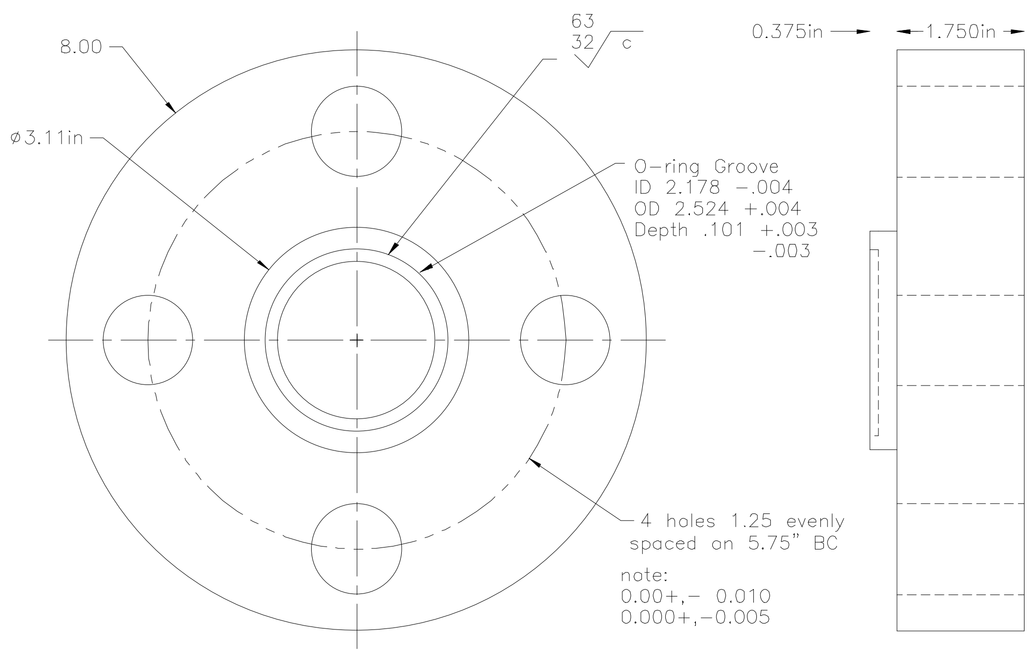

Figure A-2. Design drawing of removable flange for metallic seal (vessel cap).

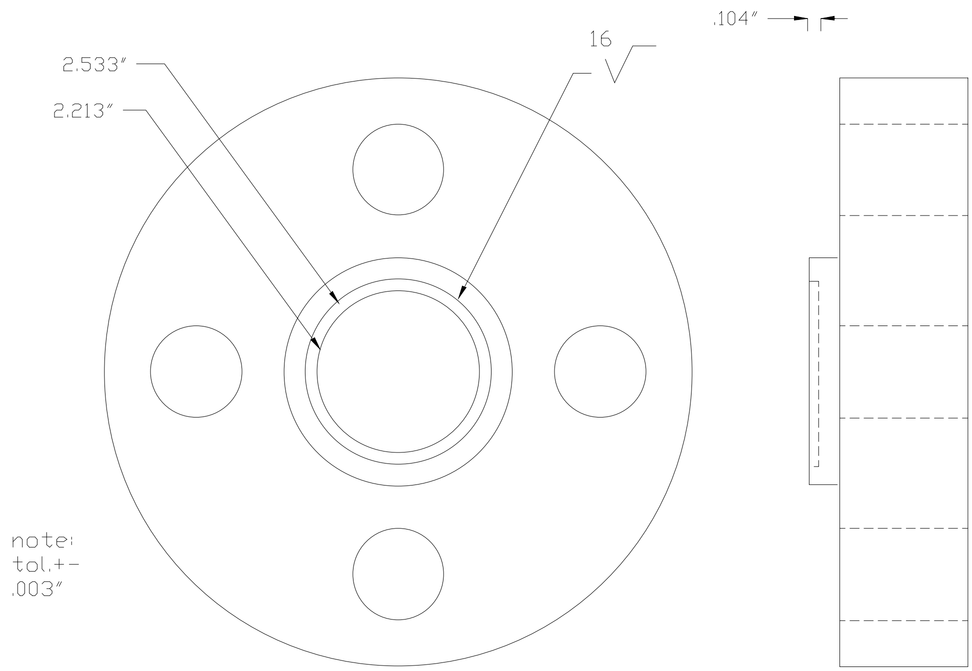

Figure A-3. Design drawing of removable flange for polymeric seal (vessel cap).

APPENDIX B

B.1 Vessel Internal Volume

Vessel internal volume (excluding the volume of the through hole for welding the tubing flush to the vessel interior, see Figure A-1) was measured at room temperature using an internal micrometer with a resolution of 0.005 mm (0.0002 in.). The inside diameter of the vessel was the averaged value from measurements made at several (at least five) depth locations of the vessel cavity.

Table B-1. Measured internal volumes of pressure vessels at room temperature

	Internal volume (m^3)
Vessel #1	9.56×10^{-5}
Vessel #2	9.52×10^{-5}
Vessel #3	9.56×10^{-5}
Vessel #3 (refurbished)	9.53×10^{-5}
Vessel #4	9.55×10^{-5}
Vessel #5	9.53×10^{-5}
Vessel #6*	9.46×10^{-5}
Vessel #7	9.57×10^{-5}
Vessel #8*	9.55×10^{-5}
Vessel #9	9.53×10^{-5}

*post-test measurements

APPENDIX C

C.1 Uncertainty Estimate in Thermocouple Measurements

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

Table C-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 (from manufacturer specifications). An assumed rectangular probability distribution (ISO, 1993) 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	

C.2 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 C-2). The uncertainty budget is listed in Table C-3.

Table C-2. Manufacturer's specifications of the pressure transducer

Excitation	24 VDC to 32 VDC
Input range	(0 to 500) psia ; (0 to 34.02) 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 C-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 (ISO, 1993), resulting 0.29 × 0.03 V = 0.0087 V.
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)

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

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.

APPENDIX D

D.1 Vessel Volume Estimation due to Thermal Expansion

The total measurement system is idealized and treated as two systems, the pressure vessel (inside the electric furnace) and the rest (outside the furnace) which contains tubing, connections, the pressure transducer, and two valves. The pressure vessel is designated as System 1 using subscript 1, and the rest System 2 using subscript 2.

Figure D-1 shows the initial state of the two systems. They are at the same initial temperature T_o and pressure P_o. The initial amount of substance in System 1 is N_{1o}, and System 2, N_{2o}. The initial volume of the pressure vessel is V_{1o}, and the rest is V_{2o}. N is the total amount of substance in the two systems. Figure D-1 also shows the final state of the two systems. If there is no leak in the total system, then

$$N_1 + N_2 = N_{1o} + N_{2o} = N$$

Using the ideal gas law,

$$\frac{PV_1}{RT_1} + \frac{PV_2}{RT_2} = \frac{P_o(V_{1o} + V_{2o})}{RT_o}$$

It is assumed that during the thermal exposure the heat loss to the surrounding from System 2 is sufficient enough to keep System 2 at its initial temperature T_o. This assumption is reasonable since the tubing system remains cold to touch during the thermal exposure experiments. With $T_2 = T_o$ and $V_2 = V_{2o}$ (no volumetric thermal expansion in System 2), then the above equation can be written as

$$\frac{PV_1}{T_1} + \frac{PV_{2o}}{T_o} = \frac{P_o(V_{1o} + V_{2o})}{T_o}$$

Note that due to volumetric thermal expansion in System 1 during the thermal exposure, $V_1 \neq V_{1o}$. The final vessel volume V_1 can be expressed in terms of P as

$$V_1 = \frac{P_oT_1(V_{1o} + V_{2o})}{PT_o} - \frac{T_1V_{2o}}{T_o}$$

Since leakage was not observed in Tests #2, #5, #6, #7, and #14, the test parameters and the experimental results from these five thorough tests were used in the calculations of V_1. The time-averaged values of T_1 (average of the four thermocouple readings) and P over the constant heating period were used. A V_{2o} value of 7 mL was estimated based on the internal dimensions of all the piping and fittings. Table D-1 tabulates the calculated V_1 and the percent volume expansion, $(V_1\text{-}V_{1o}) \times 100/V_{1o}$. The volume expansion is less than 6 % at the maximum temperature (800 °C) used in this test series. Compared to Test #2, the calculated percent volume expansion from Test #14 is a bit low. This could be due to the assumptions used in analysis and the difficulty in delineating exactly Systems 1 and 2 from test to test since the test fixture was placed inside the furnace at a slightly different location in each test.

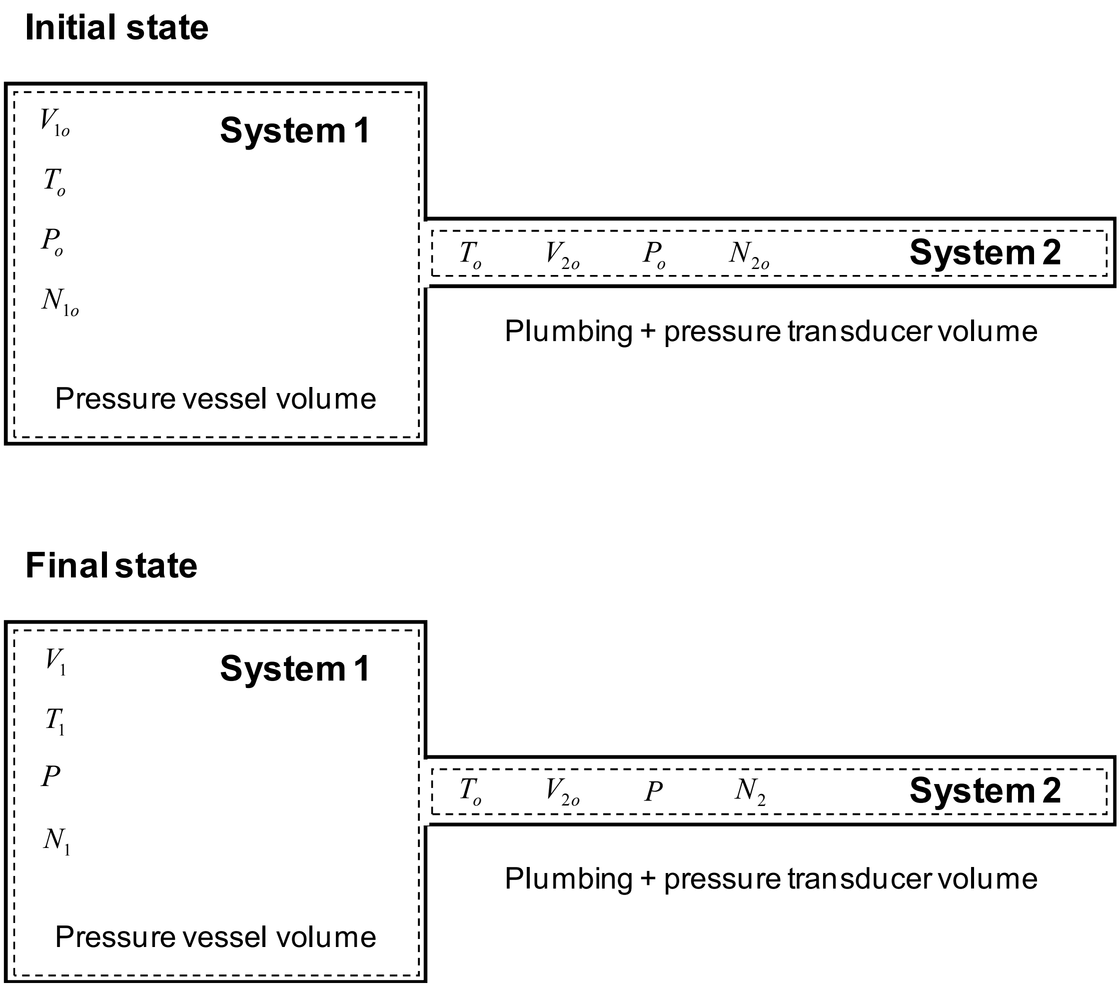

Figure D-1. Schematics showing the systems used in the thermodynamic analysis.

VARIABLES:

N_1	Amount of substance in system 1
N_2	Amount of substance in system 2
N_{1o}	Initial amount of substance in system 1
N_{2o}	Initial amount of substance in system 2
P	Pressure of the system
P_o	Initial Pressure of the system
T_o	Initial Temperature of the system
T_1	Temperature of system 1
V_1	Volume of system 1
V_{1o}	Initial Volume of system 1
V_{2o}	Initial Volume of system 2

Table D-1. Calculations of vessel volume expansion at high temperatures

	T_o (°C)	P_o (bar)	V_{1o} (m^3)	T_1 (°C)	P (bar)	V_1 (m^3)	Volume expansion (%)
Test #2	25	4.96	9.52×10^{-5}	802	14.55	1.01×10^{-4}	5.8
Test #5	25	4.98	9.53×10^{-5}	425	10.34	9.91×10^{-5}	4.0
Test #6	25	4.94	9.52×10^{-5}	425	10.20	9.97×10^{-5}	4.7
Test #7	24	4.99	9.57×10^{-5}	426	10.39	9.97×10^{-5}	4.1
Test #14	24	4.98	9.53×10^{-5}	801	14.79	9.92×10^{-5}	4.1

D.2 Isothermal Leakage Rate Estimation

If there is a leak after the final state is attained, then the leakage rate at the final-state temperature T_1 can be estimated by

$$-\frac{dN}{dt} = -\left(\frac{dN_1}{dt} + \frac{dN_2}{dt}\right) = -\frac{dP}{dt}\left(\frac{V_1}{RT_1} + \frac{V_{2o}}{RT_o}\right)$$

Since V_1/RT_1 is an order of magnitude larger than V_{2o}/RT_o, the above equation can be approximated by

$$-\frac{dN}{dt} \cong -\frac{dP}{dt}\left(\frac{V_1}{RT_1}\right)$$

Knowing the pressure-time history at the final-state temperature T_1 and the final-state volume V_1 due to thermal expansion, the leakage rate can be estimated using the above equation. In the formula described in ANSI N14.5-1997 and ISO 12807 for leakage rate estimation using pressure drop technique, no correction is made for the thermal expansion of vessel volume at high temperatures. For the conditions used in this test series, the thermal expansion of the vessel volume at the highest T_1 (800 °C) was estimated to be less than ≈ 6 % of the initial volume at room temperature, as described in Section 9.1. In conforming to the ANSI and ISO practices and without the introduction of additional uncertainty associated with the estimation of V_1 at different values of T_1, V_1 is approximated in the above equation using V_{1o} with less than 6 % reduction in the leakage rate estimation.

$$-\frac{dN}{dt} \cong -\frac{dP}{dt}\left(\frac{V_{1o}}{RT_1}\right)$$

The time derivatives of vessel pressure (dP/dt) can be computed numerically from the pressure-time history curve using the scientific graphing package SigmaPlot® macro with a running average length of 1. Following ANSI N14.5-1997, the reference helium leakage rates $L_{R,He}$ (ref·cm^3/s) can be obtained by dividing – dN/dt by 4.09×10^{-5} mol/cm^3, which is the amount of substance (ideal gas) contained in 1×10^{-6} m^3 (1 cm^3) at 1.01×10^5 Pa (1 atm) and 298 K (25 °C).

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F. Gonzalez, NRC Project Manager

11. ABSTRACT (200 words or less)

This report documents the beyond-design-basis thermal exposure test results of the performance of one type of metallic seal and two different polymeric compound (ethylene-propylene and Teflon) seal designs typically used in spent fuel transportation packages. Fifteen tests were conducted using a small scale-model package made of stainless steel SS 304 filled with helium initially pressurized at either 5 bar or 2 bar at room temperature. The test package was then exposed in an electric furnace to temperatures equal to or over the specified maximum operating temperatures of these seals for a pre-determined period (typically 9 h). The pressure drop technique was used to determine if leakage occurred during thermal exposure. A total of fifteen tests, twelve used metallic seals, two used ethylene-propylene compound, and one used Teflon, were performed. Leakage was observed in some of the thermal exposure tests. The time when leakage occurred varied. The overall goal of the project is to provide insights to the performance of these seals when exposed to beyond-design-basis temperature conditions that could result due to a severe fire.

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