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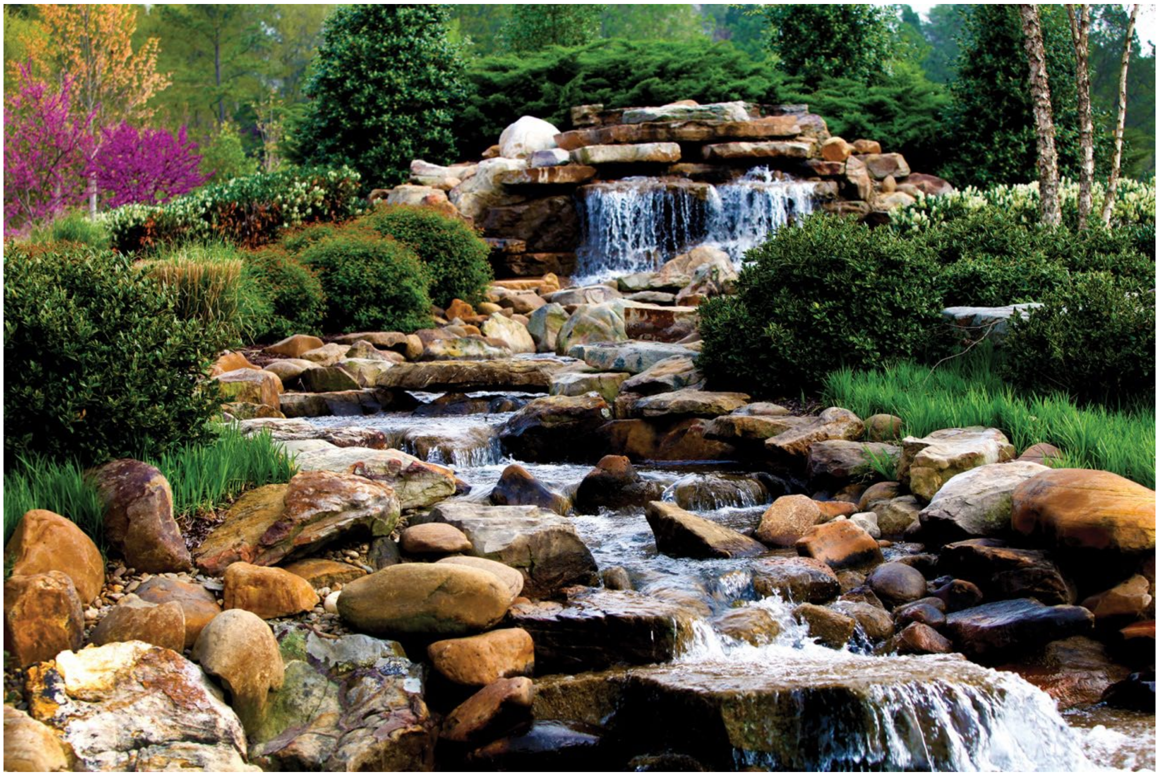
Testing and Qualification of Molybdenum Subcapsule Welds for MiniFuel Experiments



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December 2022

OAK RIDGE National Laboratory

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Nuclear Energy and Fuel Cycle Division

TESTING AND QUALIFICATION OF MOLYBDENUM SUBCAPSULE WELDS FOR MINIFUEL EXPERIMENTS

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ABSTRACT

This report documents the testing and qualification efforts for welding molybdenum components to support MiniFuel irradiation campaigns. Other details such as general weld parameters, weld joint geometries, and performance limits are also provided in this report. The American Welding Society's specifications were used to select the nondestructive and destructive tests, which include: gross and fine leak testing, internal pressure proof testing, and creating metallographic mounts of welds. Leak testing and metallographic mounts provide qualitative information about the weld integrity, while the proof testing indicated pressure/usage limits of the molybdenum subcapsule. Based on the testing results provided in the is report, the capsule can be exposed to an internal pressure of at least 20.7 MPa (3000 psi). A sealed capsules is also capable of withstanding the 3.2 MPa (468 psi) external hydrostatic operating pressure of the High Flux Isotope Reactor.

1. Purpose

Research to support characterization and qualification of advanced nuclear fuels is a major domestic nuclear energy focus. The MiniFuel irradiation platform provides flexible irradiation vehicles for testing nuclear fuels within various facilities in the High Flux Isotope Reactor (HFIR). A recent innovation within the MiniFuel design is the usage of sealed molybdenum subcapsules to provide a secondary barrier for the fuel samples. This secondary containment is necessary to meet various programmatic and safety basis requirements, such as ensuring subcapsule fission gas retention or isolating sodium from the reactor coolant through double containment. This work describes the preliminary development and testing used to qualify the molybdenum weld parameters used for the MiniFuel subcapsules.

2. Description of weld schedules and geometries

The MiniFuel molybdenum subcapsule consists of a holder and an end cap. The end cap fits into the open end of the holder to create a trepan weld joint, and possesses a backfill hole in the cap center. The weld and assembly configurations are shown in Figure 2-1. Both welds are intended to be accomplished through an autogenous process (i.e., no filler metal is added during the welding process).

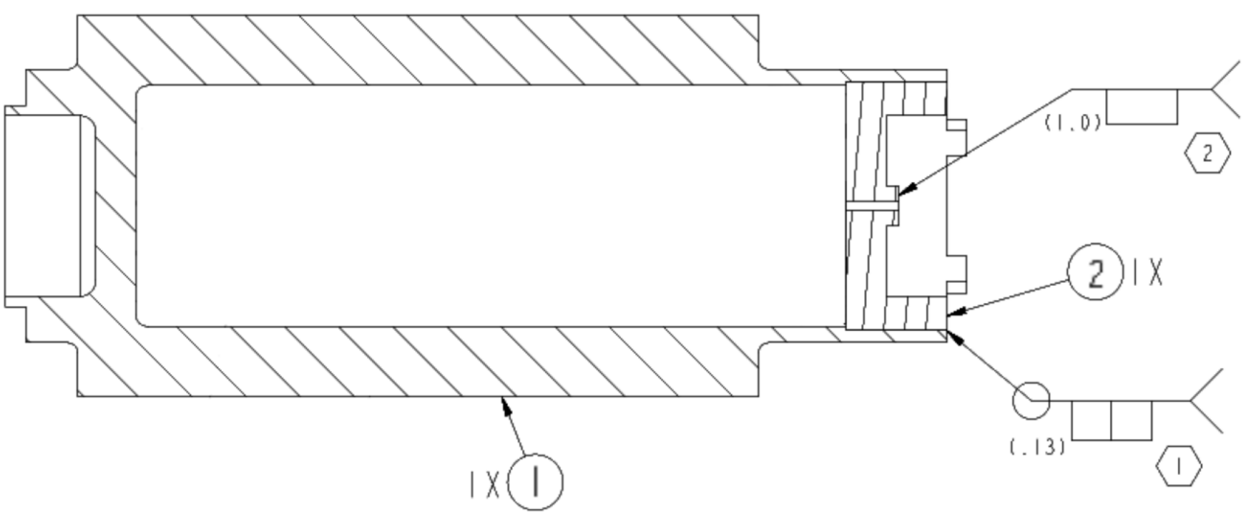


Figure 2-1. Engineering rendering of the MiniFuel subcapsule assembly, denoting welds.

The trepan joint (weld 1, Figure 2-1) is fused using an electron beam weld process. The subcapsule assembly is installed into the machine, and the weld is performed by focusing an electron beam onto the weld joint to autogenously fuse the end cap to the holder. Electron beam power is established by controlling beam current and voltage, which are recorded as essential variables for the weld schedule. The weld is performed under high vacuum condition and no fill gas is credited. Finally, the work platform is computer numerical controlled to establish weld travel speed, and to complete the round trepan weld pattern. See Table 2-1 for the selected variable settings for the electron beam weld schedule.

Table 2-1. Nominal variables for the electron beam trepan weld

Variable	**Nominal Setting**
Voltage	150 kV
Amperage	1.8 mA
Upslope/downslope	None
Pulsing	41.7 Hz, 33%
Sharp focus (welding machine/fixture dependent)	650
CNC welding deflection (welding machine/joint dependent)	Circular deflection x=28.5, y = 28.5, Hz = 0.4

The seal weld (weld 2, Figure 2-1) is performed using a manual direct current (DC) tungsten inert gas (TIG) welding process within a sealed pressure vessel. The system is designed to allow for a vessel containing the subcapsule to be purged and create a desired atmosphere that meets design requirements. The manual TIG process is performed with roughly 900 W of electrical power (80A/11V) with a helium cover gas. The weld technician attempts a very short pulse at full power in the fashion of a spot weld to seal the backfill hole and minimize the amount of heat imparted on the subcapsule.

3. Specification development and testing

3.1 Specification development for the MiniFuel subcapsule

The MiniFuel subcapsule is fabricated from molybdenum, which is a high strength refractory metal with low ductility. The American Society for Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC) does not consider molybdenum a code qualified material for pressure vessels, which prohibits qualifying such welds under the Section IX guidance. Alternative destructive and non-destructive tests were chosen to demonstrate the sealed subcapsule will perform properly under irradiation conditions.

Specific functions that must be performed by the sealed subcapsule include:

- Create a hermetic seal to maintain the capsule internal atmosphere
- Withstand subcapsule pressurization due to fission gas release, heating of internal atmosphere, etc.
- Prevent water ingress within the subcapsule if the primary containment fails and is flooded with reactor coolant

Hermeticity is generally verified by subjecting one side of a weld to a mass spectrometer and impart a tracer gas (typically helium) to the other side of the weld. The mass spectrometer will report an apparent leak rate across the welded membrane. The accepted maximum leak rate for a hermetically sealed weld intended for use is HFIR is 10^{-7} std. cc He/sec.

Capsule integrity is generally a function of wall thickness, but weld strength should also be verified for assemblies made because welds are integral parts of a vessel containment. The MiniFuel subcapsule was designed as a general use vehicle, so pressure limits for the subcapsule were established to be higher than any expected conditions that could be present during irradiation. For this case, the internal pressure limit is 20.7 MPa (3000 psi), which is verified through internal hydrostatic testing described in Section 3.2. The other limiting condition is to show the subcapsule (wall and welds) can withstand external 3.2 MPa (468 psi) hydrostatic compression from the HFIR coolant in the event the primary capsule is breached. This can be shown by relating internal working pressure to external buckling pressure through the following relationships [1]:

Internal pressure limit for cylindrical shell (longitudinal stress):

$$T = \frac{P_i \cdot r}{S \cdot E_j + 0.4 \cdot P_i}$$

Where:

- P_i is the internal pressure
- T is the shell thickness
- r is the outer radius
- S is allowable stress for the material (assumed as yield stress for this case)
- E_j is the weld joint efficiency

This relationship is valid for cylinders with length to diameter (L/D) ratio 50 < L/D <0.5. The MiniFuel subcapsule has an L/D ≈ 2.5.

Internal pressure limit for cylindrical shell (assume conservative D_o/T <10):

$$P_e = \frac{2A \cdot E}{3\left(\frac{2r}{T}\right)} = \frac{1.1}{\left(\frac{2r}{T}\right)^2}\frac{2E}{3\left(\frac{2r}{T}\right)} = \frac{2.2E}{3\left(\frac{2r}{T}\right)^3}$$

Where:

- P_e is the external pressure
- T is the shell thickness
- r is the outer radius ($D_o/2$)
- E is the material Young's Modulus

The following expression is formed by setting both equations in terms of T:

$$\frac{P_i \cdot r}{S \cdot E_j + 0.4 \cdot P_i} = 2r\sqrt[3]{\frac{3P_e}{2.2E}}$$

The equation can be rearranged to the following form:

$$P_e = \frac{2.2E}{3}\left(\frac{P_i}{S \cdot E_j + 0.4 \cdot P_i}\right)^3$$

Therefore, assuming the MiniFuel subcapsule has an internal pressure limit of 20.7 MPa, allowable stress of 300 MPa [2], Young's Modulus of 300 GPa [2], and a joint efficiency of 1 to minimize the allowable external pressure, then:

$$P_e = \frac{300GPa}{1.36}\left(\frac{20.7\ MPa}{2(300MPa + 0.4 \cdot 20.7MPa)}\right)^3 = 8.3\ MPa\ (1204\ psi)$$

The external pressure limit of 8.3 MPa is greater than the 3.2 MPa HFIR operating pressure and appropriate for the MiniFuel subcapsule specification, assuming testing supports at least a 20.7 MPa internal pressure limit.

3.2 Destructive and non-destructive testing

Destructive and nondestructive tests are intended to examine welds for defects and understand performance limitations. Codes and standards such as the American Weld Society (AWS) B2.1 are used to identify the appropriate test paradigms to develop useful engineering welding specification and to formally qualify weld schedules, if necessary. For this work, no formal weld qualifications are created, but these standards were consulted to ensure the welded MiniFuel subcapsules met the specifications outlined in Section 3.1. The primary tests included visual inspection, proof testing (internal hydrostatic compression), helium leak testing, and metallography. Table 3-1 correlates these tests to their respective applicable detectable defects. Other tests may be included in future work to identify mechanical properties, etc. but these tests were deemed appropriate for this series of MiniFuel subcapsules.

Table 3-1. Testing techniques with corresponding detectable defects (condenses from AWS C6.1-89 [3]).

Inspection technique	Defects or characteristics	Destructive or Nondestructive
Visual	Cracks, incomplete welds	Nondestructive
Proof testing	Cracks, incomplete welds, performance limits	Nondestructive and destructive
Leak testing	Cracks, incomplete welds	Nondestructive
Metallography	Cracks, incomplete welds, inclusions, material composition, Weld and heat affected zone position	Destructive

Two subcapsules, identified as 22-1527-1 and 22-1527-2, were used for this exercise. Images of these articles are found in Figure 3-1, with both sets of electron beam trepan welds and seal welds visible. The visual inspection of both articles was sufficient to proceed with the remaining tests. A quantitative helium leak test was performed on each of the articles prior to hydrostatic testing, and both had leak rates less than 10^{-7} std. cc He/sec.

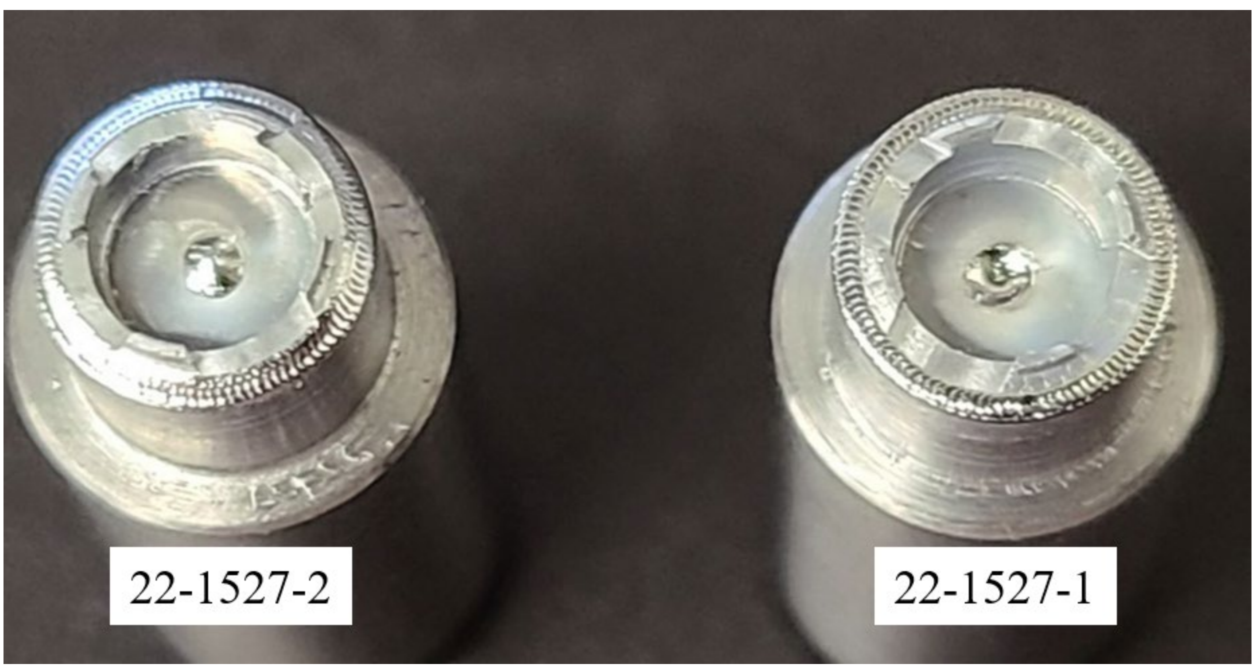


Figure 3-1. MiniFuel test articles used for nondestructive and destructive testing (12.

Both test articles were modified on the unwelded ends to be installed into a piping fixture used to perform the hydrostatic tests. The fixture consisted of a male and female end that compressed the test article against a water inlet nozzle, which sealed around an O-ring installed in the modified end of the article. See Figure 3-2 and Figure 3-3 for images of the piping fixture in an unassembled and assembled state, respectively.

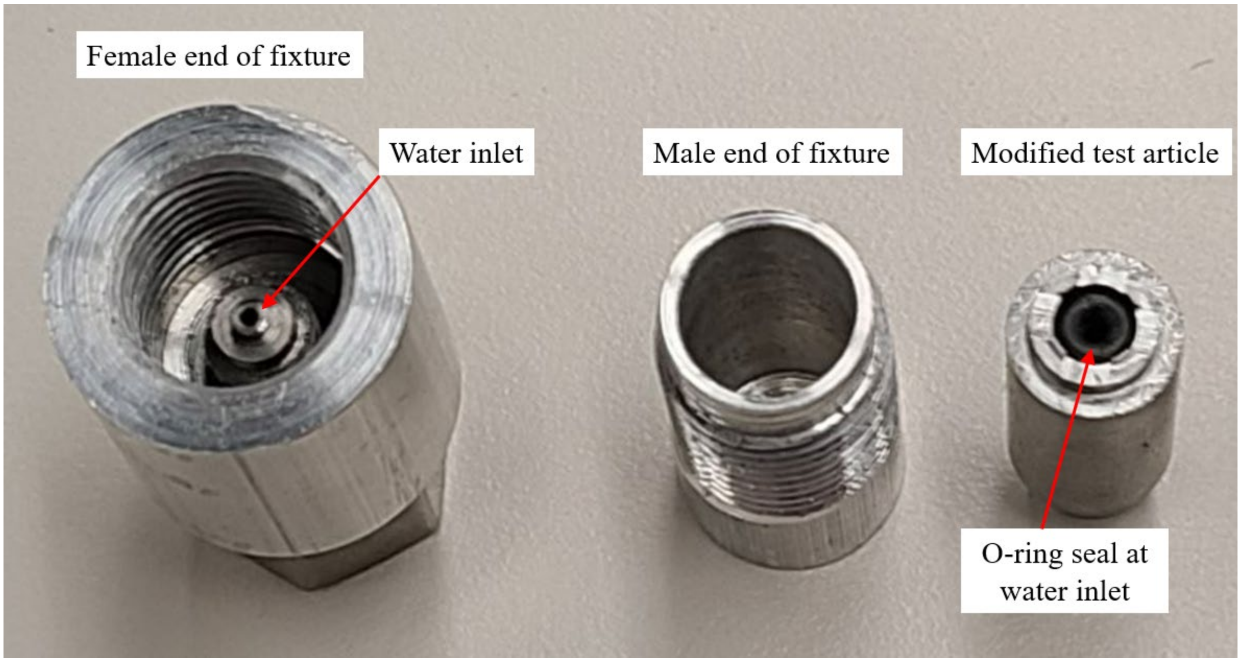


Figure 3-2. Constituent components of the piping fixture, showing modified test article with O-ring installed.

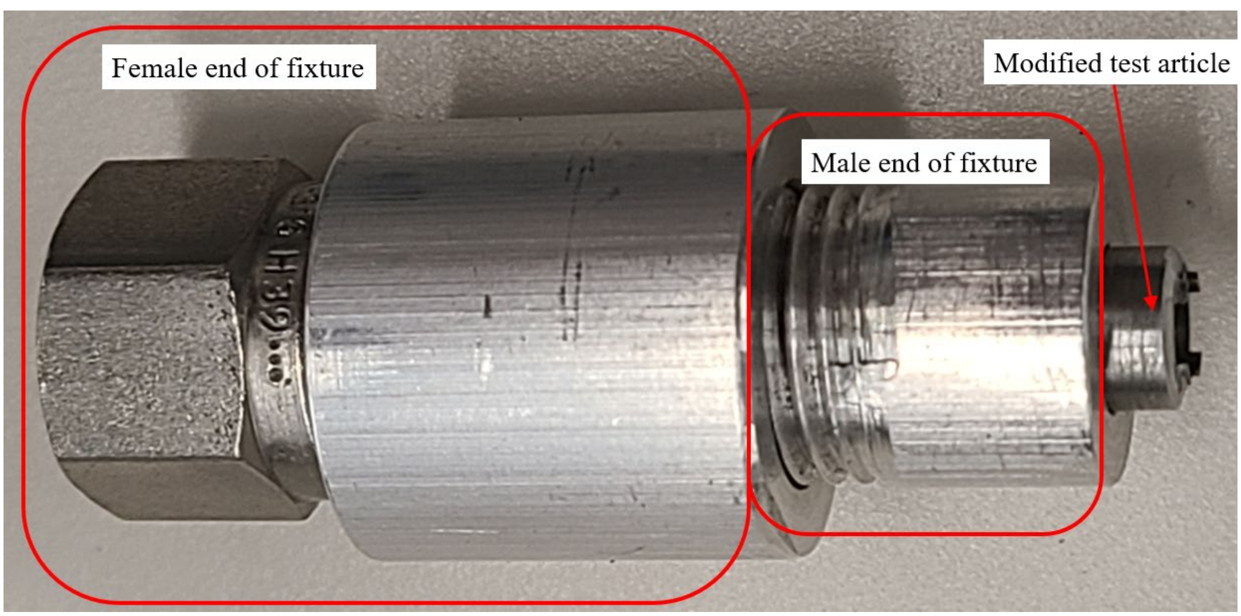


Figure 3-3. Assembled piping fixture ready for testing.

The assembled piping fixture was installed onto the hydrostatic compression test loop (HCTL), which was developed for proof testing to hydrostatically load test articles to explore failure and operating limits, etc. This loop includes a manually operated hand pump, pressure relief valve, calibrated gauge, bleeder valve, and required plumbing. A physical layout of the HTCL is shown in Figure 3-4. The HCTL requires an outside water source to operate. Once a test article is properly installed and the water source is established, the loop is purged of air. This is accomplished by opening the bleeder valve and running water through the loop until air is no longer ejected from the valve outlet. The hand pump is used to pressurize water within the test article until operating limits are verified. The two MiniFuel subcapsules were loaded to pressures in excess of 34.5 MPa (5000psi), as shown from the gauge readout images in Figure 3-5, and no plastic deformation or weld failures were observed during these tests. These results verified the internal operating pressure specification of 20.7 MPa is appropriate and conservative. Further, hydrostatic compression tests serve to demonstrate the absence of gross leaks in the welds. No such leaks were observed for either test articles.

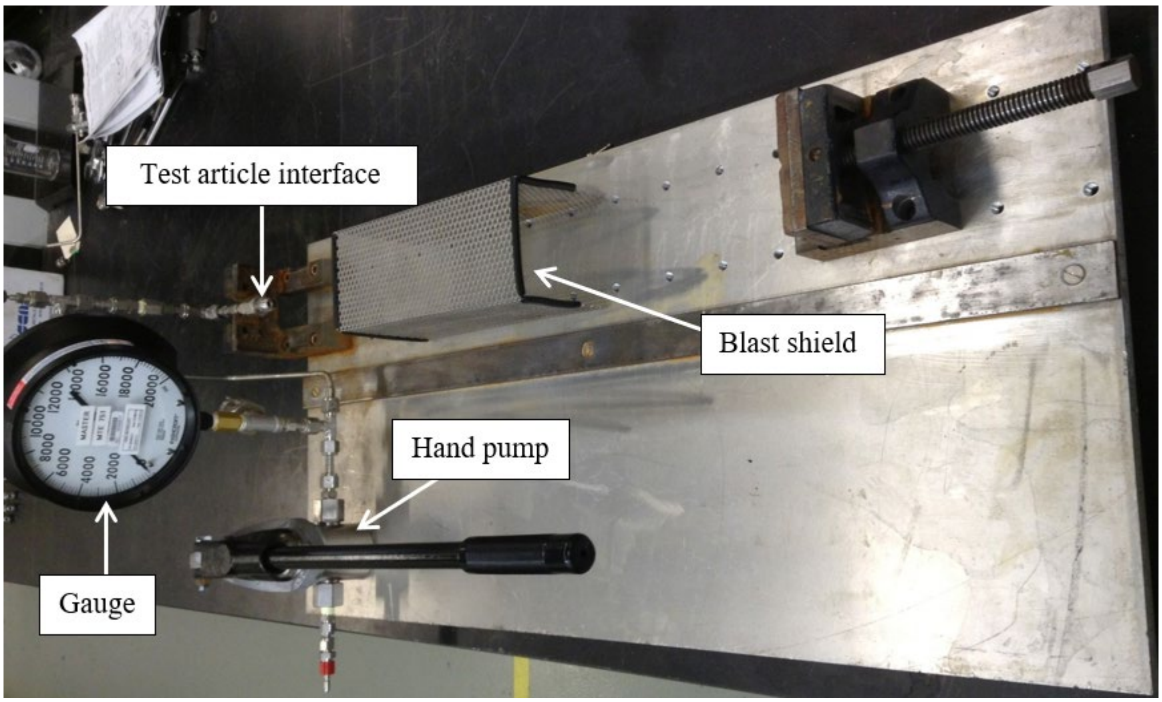


Figure 3-4. The HTCL with annotated subcomponents.

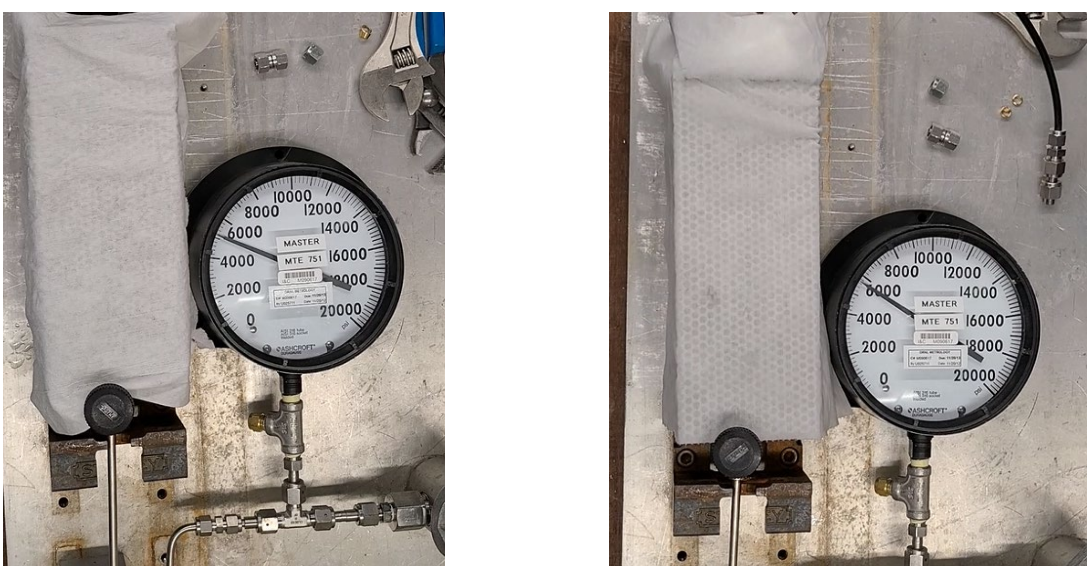


Figure 3-5. Peak pressure readouts for the MiniFuel subcapsule test articles (gauge units are psi).

Metallographic mounts were taken from the two test samples after the hydrostatic tests for weld and heat affected zone (HAZ) interrogations. Images of the 22-1527-1 and 22-1527-2 mounts are seen in Figure 3-6 and Figure 3-7, respectively. The weld fusion and HAZ sites are clearly indicated by the areas of large grain growth due to melting and solidification of the molybdenum base metal. Grain growth at areas in and around the welds is normal can further reduce the ductility of the molybdenum base metal. However, the proof testing performance showed that there is sufficient strength and support in the welds to withstand the design pressures intended for the MiniFuel subcapsule. The metallographic mounts revealed small pore defects in the weld zone, but these did not impact proof testing performance and are much smaller than the weld joint thickness (i.e., less than 1/3 wall thickness). Figure 3-6 shows a crack feature on the left side of weld 1, but there were observed deleterious effects caused by the defect. In summary, the metallographic mounts revealed that the welds have adequate penetration, and indicated that no unexpected crystallization/defect formation occurred in the weld sites.

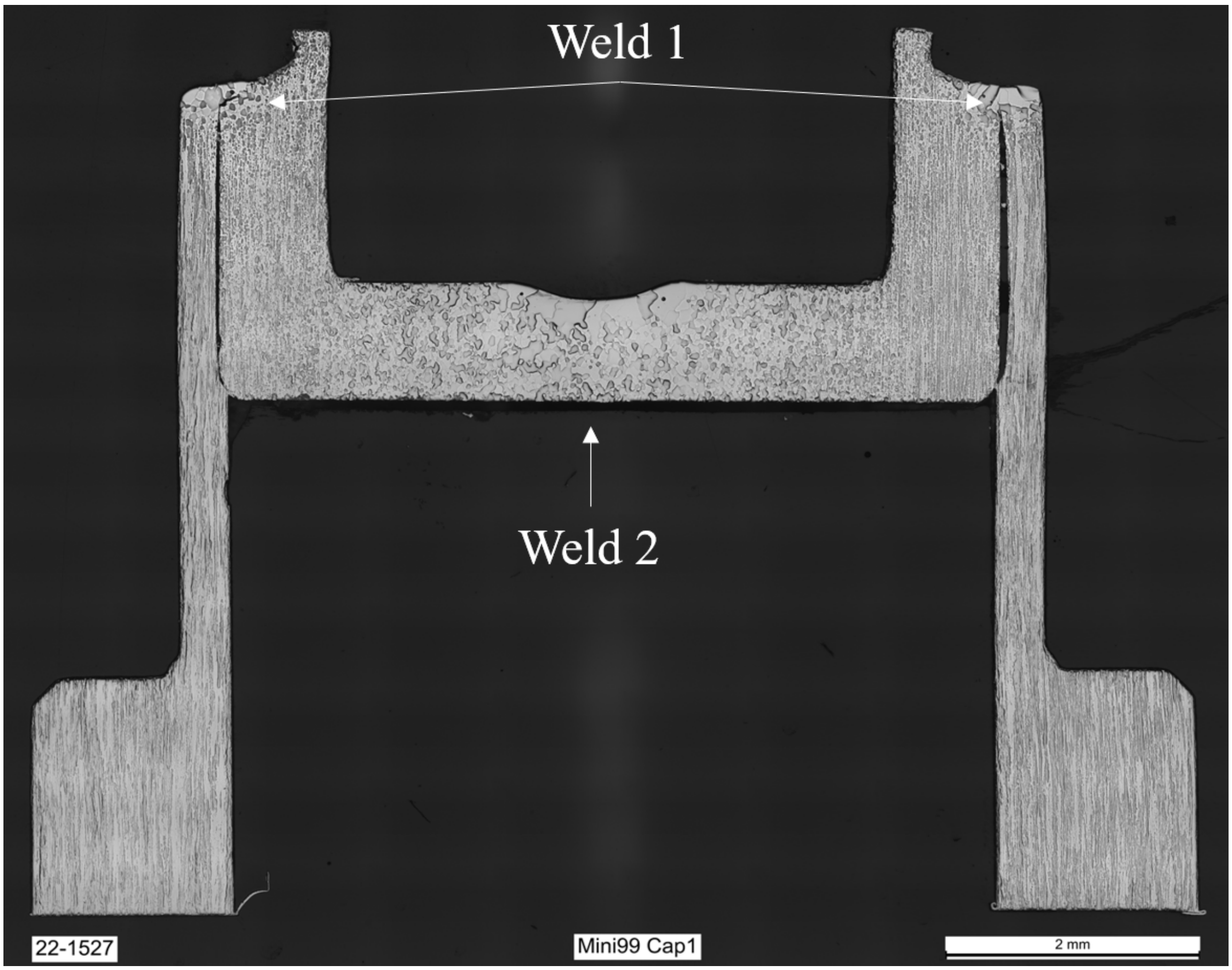


Figure 3-6. Metallographic mount of 22-1527-1

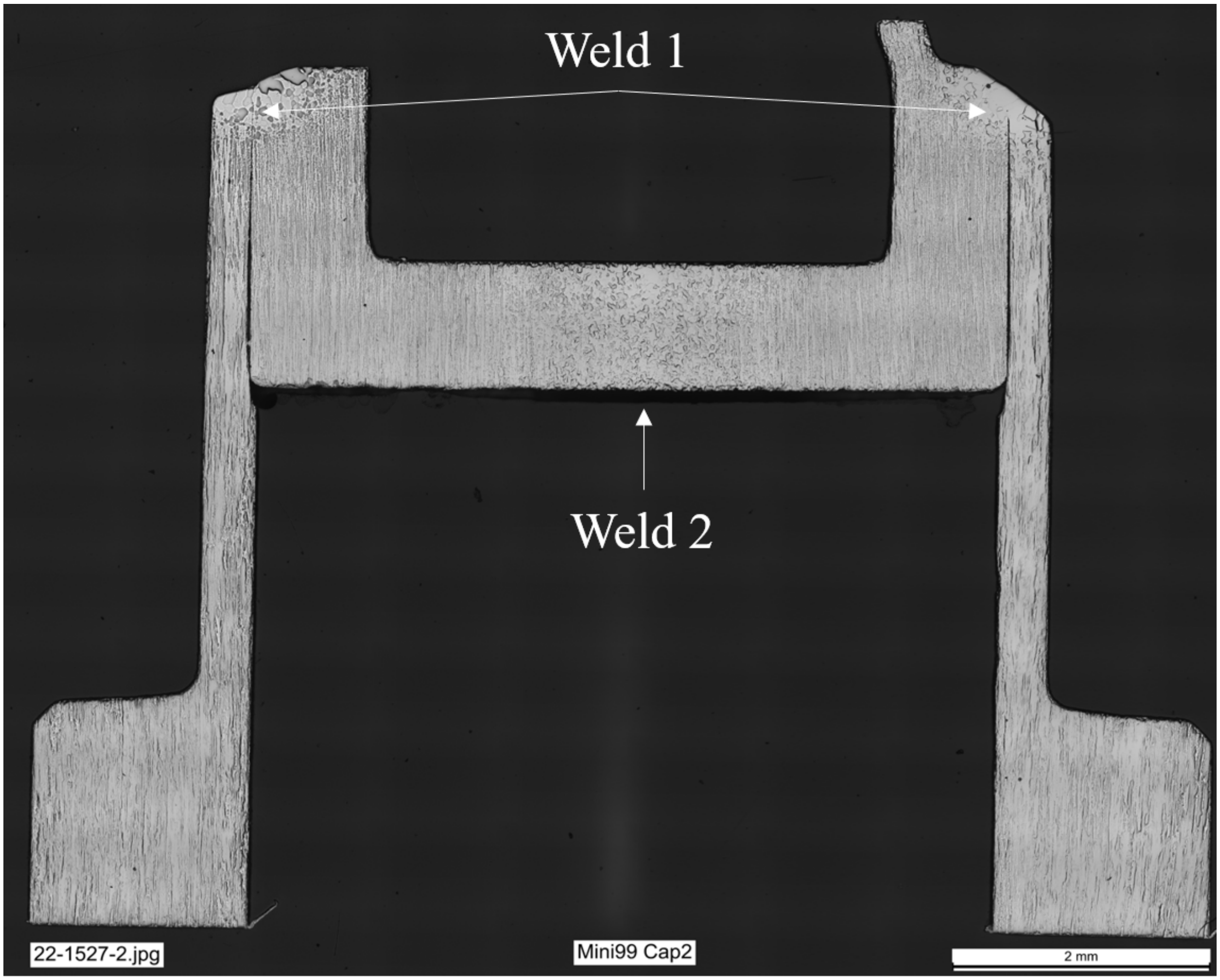


Figure 3-7. Metallographic mount of 22-1527-2.

4. Conclusions

This report details the weld specification and testing outcomes for the molybdenum MiniFuel subcapsule. Various destructive and nondestructive tests were performed to ensure the welds and subcapsule assembly performed at or beyond design specifications. These specifications dictate the subcapsule shall:

- Be hermetically sealed, with leak rates less than 10^{-7} std. cc He/sec
- Withstand a minimum of 20.7 MPa (3000 psi) internal pressure loading
- Withstand a minimum of 3.2 MPa (468 psi) external pressure loading
- Prevent water ingress into the subcapsule internal volume in the event the primary containment is breached

Test results reported in this document indicate that the weld schedule and subcapsule design meets these specifications. Therefore, the weld schedule presented here is appropriate for the MiniFuel irradiation platform. It is recommended that various nondestructive tests, such as visual inspection, external pressurization, and leak testing, be performed on all welds to ensure weld integrity of as-bult subcapsules prior to reactor insertion.

5. Acknowledgments

The authors would like to acknowledge Alan Frederick, Doug Kyle, Caitlin Duggan, and Annabelle Le Coq for their invaluable contributions to this work. The work was funded by the DOE/NNSA Office of Material Management and Minimization (NA-23).

6. References

[1] "Rules for Construction of Pressure Vessels, ASME Boiler and Pressure Vessel Code, Section VIII, Division I," The American Society for Mechanical Engineers, 2021.

[2] H.Shinno, M.Kitajima and M.Okada, "Thermal Stress Analysis of High Heat Flux Materials," *Journal of Nuclear Materials,* Vols. 155-157, pp. 290-294, 1988.

[3] "Recommended Practices for Friction Welding," The American Welding Society, ANSI/AWS C6.1-89R, 1989.

APPENDIX A. Select Attachments

Appendix A. ASME BPVC excerpts

R = inside radius of the shell course under consideration,[19]

S = maximum allowable stress value (see UG-23 and the stress limitations specified in UG-24)

t = minimum required thickness of shell

(c) Cylindrical Shells. The minimum thickness or maximum allowable working pressure of cylindrical shells shall be the greater thickness or lesser pressure as given by (1) or (2) below.

(1) Circumferential Stress (Longitudinal Joints). When the thickness does not exceed one-half of the inside radius, or P does not exceed $0.385SE$, the following formulas shall apply:

$$t = \frac{PR}{SE - 0.6P} \quad \text{or} \quad P = \frac{SEt}{R + 0.6t} \qquad (1)$$

(2) Longitudinal Stress (Circumferential Joints).[20] When the thickness does not exceed one-half of the inside radius, or P does not exceed $1.25SE$, the following formulas shall apply:

$$t = \frac{PR}{2SE + 0.4P} \quad \text{or} \quad P = \frac{2SEt}{R - 0.4t} \qquad (2)$$

(d) Spherical Shells. When the thickness of the shell of a wholly spherical vessel does not exceed $0.356R$, or P does not exceed $0.665SE$, the following formulas shall apply:

$$t = \frac{PR}{2SE - 0.2P} \quad \text{or} \quad P = \frac{2SEt}{R + 0.2t} \qquad (3)$$

(e) When necessary, vessels shall be provided with stiffeners or other additional means of support to prevent overstress or large distortions under the external loadings listed in UG-22 other than pressure and temperature.

(f) A stayed jacket shell that extends completely around a cylindrical or spherical vessel shall also meet the requirements of UG-47(c).

(g) Any reduction in thickness within a shell course or spherical shell shall be in accordance with UW-9.

(21) UG-28 THICKNESS OF SHELLS AND TUBES UNDER EXTERNAL PRESSURE

(a) Rules for the design of shells and tubes under external pressure given in this Division are limited to cylindrical shells, with or without stiffening rings, tubes, and spherical shells. Three typical forms of cylindrical shells are shown in Figure UG-28. Charts used in determining minimum required thicknesses of these components are given in Section II, Part D, Subpart 3.

(b) The symbols defined below are used in the procedures of this paragraph:

A = factor determined from Section II, Part D, Subpart 3, Figure G and used to enter the applicable material chart in Section II, Part D, Subpart 3. For the case of cylinders having D_o/t values less than 10, see (c)(2).

B = factor determined from the applicable material chart or table in Section II, Part D, Subpart 3 for maximum design metal temperature [see UG-20(c)]

D_o = outside diameter of cylindrical shell course or tube

E = modulus of elasticity of material at design temperature. For external pressure design in accordance with this Section, the modulus of elasticity to be used shall be taken from the applicable materials chart in Section II, Part D, Subpart 3. (Interpolation may be made between lines for intermediate temperatures.)

L = total length, in. (mm), of a tube between tubesheets, or design length of a vessel section between lines of support (see Figure UG-28.1). A line of support is:

(a) a circumferential line on a head (excluding conical heads) at one-third the depth of the head from the head tangent line as shown on Figure UG-28;

(b) a stiffening ring that meets the requirements of UG-29;

(c) a jacket closure of a jacketed vessel that meets the requirements of 9-5;

(d) a cone-to-cylinder junction or a knuckle-to-cylinder junction of a toriconical head or section that satisfies the moment of inertia requirement of 1-8.

P = external design pressure [see Note in (f)]

P_a = calculated value of maximum allowable external working pressure for the assumed value of t, [see Note in (f) below]

R_o = outside radius of spherical shell

t = minimum required thickness of cylindrical shell or tube, or spherical shell, in. (mm)

t_s = nominal thickness of cylindrical shell or tube, in. (mm)

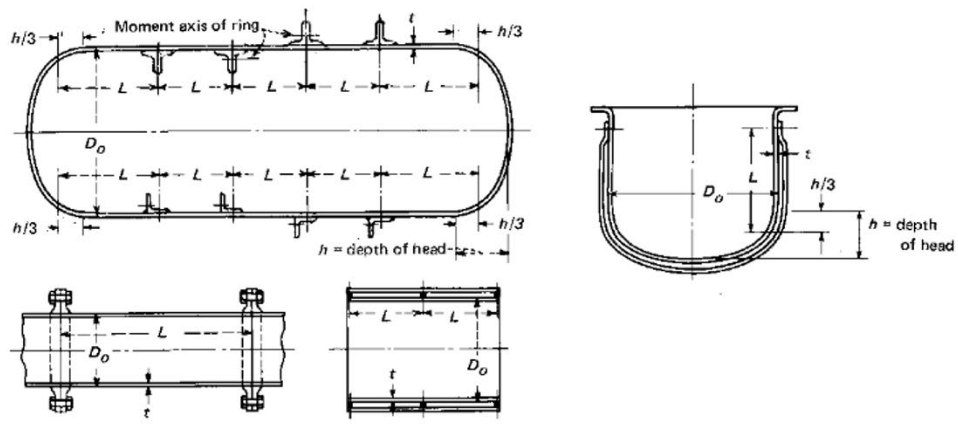
(c) Cylindrical Shells and Tubes. The required minimum thickness of a cylindrical shell or tube under external pressure, either seamless or with longitudinal butt joints, shall be determined by the following procedure:

(1) Cylinders having D_o/t values ≥ 10:

Step 1. Assume a value for t and determine the ratios L/D_o and D_o/t.

Step 2. Enter Section II, Part D, Subpart 3, Figure G at the value of L/D_o determined in Step 1. For values of L/D_o greater than 50, enter the chart at a value of L/D_o = 50. For values of L/D_o less than 0.05, enter the chart at a value of L/D_o = 0.05.

Figure UG-28
Diagrammatic Representation of Variables for Design of Cylindrical Vessels Subjected to External Pressure



Step 3. Move horizontally to the line for the value of D_o/t determined in Step 1. Interpolation may be made for intermediate values of D_o/t; extrapolation is not permitted. From this point of intersection move vertically downward to determine the value of factor A. For values of A greater than 0.10, use a value of 0.10.

Step 4. Using the value of A calculated in Step 3, enter the applicable material chart in Section II, Part D, Subpart 3 for the material under consideration. Move vertically to an intersection with the material/temperature line for the design temperature (see UG-20). Interpolation may be made between lines for intermediate temperatures. If tabular values in Section II, Part D, Subpart 3 are used, linear interpolation or any other rational interpolation method may be used to determine a B value that lies between two adjacent tabular values for a specific temperature. Such interpolation may also be used to determine a B value at an intermediate temperature that lies between two sets of tabular values, after first determining B values for each set of tabular values.

In cases where the value of A falls to the right of the end of the material/temperature line, assume an intersection with the horizontal projection of the upper end of the material/temperature line. If tabular values are used, the last (maximum) tabulated value shall be used. For values of A falling to the left of the material/temperature line, see Step 7.

Step 5. From the intersection obtained in Step 4, move horizontally to the right and read the value of factor B.

Step 6. Using this value of B, calculate the value of the maximum allowable external working pressure P_a using the following equation:

$$P_a = \frac{4B}{3(D_o/t)}$$

Step 7. For values of A falling to the left of the applicable material/temperature line, the value of P_a can be calculated using the following equation:

$$P_a = \frac{2AE}{3(D_o/t)}$$

If tabular values are used, determine B as in Step 4 and apply it to the equation in Step 6.

Step 8. Compare the calculated value of P_a obtained in Step 6 or Step 7 with P. If P_a is smaller than P, select a larger value for t and repeat the design procedure until a value of P_a is obtained that is equal to or greater than P.

(2) Cylinders having D_o/t values <10:

Step 1. Using the same procedure as given in (1), obtain the value of B. For values of D_o/t less than 4, the value of factor A can be calculated using the following equation:

$$A = \frac{1.1}{(D_o/t)^2}$$

For values of A greater than 0.10, use a value of 0.10.