Technology Enabling Zero-EPZ Micro Modular Reactors

Milestone M2.2.3
Produce ZrH Moderator Material

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Award Number: DE-AR0000977
Prime Recipient: Stony Brook University
Sub-recipients: University of Tennessee Knoxville Oak Ridge National Laboratory
Project Period: 03/13/2019 – 09/12/2021
Milestone report #: 2.2.3
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Due date: 03/12/2021
Report submission date: 03/12/2021

SUMMARY

This report complete Milestone M2.2.3 — *"Produce zirconium hydride moderator material"*. In this milestone, we report the successful fabrication of FeCrAl cladded zirconium hydride moderator as well as its thermal stability. Following the demonstration of successfully fabricating delta-phase bulk zirconium hydride in M2.2.2, the remaining challenge to produce zirconium hydride moderator compact is the development of cladding technique. Different types of cladding designs were presented in this report, including the basic design with bar cladding material, basic design with tube cladding material, and crucible design with position holding function. Mo and FeCrAl were used as the trial cladding materials. The welding techniques and procedures were reported and discussed. Three welding techniques were adopted in the development of moderator cladding, electron beam welding (EBW), laser beam welding (LBW), and gas tungsten arc welding (GTAW). After welding, all cladded crucibles were evaluated with two kinds of leak testing, the helium leak test for minor leakage and the bubble test for major leakage. Due to the concern of poor neutronics performance, Mo was not ideal for future moderator cladding application. Therefore, detailed characterization of the Mo cladded zirconium hydride was not pursued. Instead, the characterization of FeCrAl cladded zirconium hydride was performed. Two material conditions (i.e., as machined and pre-oxidized conditions) were discussed prior to the cladding process. Thermal stability of the cladded zirconium hydride moderator through directly measuring hydrogen release and characterization of the zirconium hydride following the thermal desorption measurement were evaluated. The results showed pre-oxidized FeCrAl is capable of efficiently preventing hydrogen release from the moderator assembly. X-ray computed tomography analysis showed that there is no visible deformation of the moderator assembly following the heat treatment. High energy X-ray diffraction measurement of the zirconium hydride pieces extracted from the moderator assemblies following hydrogen release testing showed that delta-phase zirconium hydride is still dominant (> 97.9%) and the formation of new metallic phases (< 0.3%), $(Fe, Cr)_2Zr$, was observed.

CONTENTS

FIGURES

TABLES

ACRONYMS

°C	Degree Celsius
"	Inch
Al	Aluminum
ARPA-E	Advanced Research Projects Agency-Energy
ATM	Atmosphere Pressure
C	Carbon
cc	Cubic Centimeter
Cr	Chromium
EBW	Electron Beam Welding
Fe	Iron
g	Gram
GTAW	Gas Tungsten Arc Welding
H	Hydrogen
K	Kelvin
LCAC	Low Carbon Arc Cast
LBW	Laser beam welding
MEITNER	Modeling-Enhanced Innovations Trailblazing Nuclear Energy Reinvigoration
m	Meter
ml	Milliliter
mm	Millimeter
Mo	Mo
N	Nitrogen
Ni	Nickel
O	Oxygen
ORNL	Oak Ridge National Laboratory
sec	Second
SEM	Scanning Electron Microscopy
Si	Silicon
std	Standard
W	Watt
XCT	X-ray Computed Tomography

XRD	X-ray Diffraction
Y	Yttrium
Zr	Zirconium
ZrHx	Zirconium Hydride

ACKNOWLEDGMENTS

The authors gratefully acknowledge Yukinori Yamamoto and Andrew Nelson for providing FeCrAl material and information. Alan Frederick, Eric Vidal, Doug Kyle, and Randy Parten for technical support of welding, leak testing and crucible fabrication. Tom Geer and Victoria Cox for metallographic work. Douglas Stringfield for parts fabrication coordination, and Kevin Faraone and Ben Garrison for reviewing the report. The authors appreciate the support of the Advanced Research Projects Agency-Energy (ARPA-E) program: Modeling-Enhanced Innovations Trailblazing Nuclear Energy Reinvigoration (MEITNER) under contract DE-AR0000977.

1 Introduction

Metal hydrides are particularly well suited to thermal reactor systems in which core weight and volume need to be minimized, where they serve as a constituent in fuels and in moderator and shield materials [1]. Due to its very low neutron absorption cross section, zirconium hydride (ZrHx) is particularly attractive for the high-performance moderator application. We have demonstrated the successful fabrication of bulk delta-phase crack free ZrHx through carefully controlling the hydrogen flow rate and process temperature by using the ORNL bulk metal hydriding system, as reported in [2].The deployment of ZrHx moderator at high temperature is challenging due to the thermally-driven hydrogen desorption[3]. Cladding ZrHx with materials having low hydrogen permeability is proposed here to mitigate the hydrogen loss from hydrides. In addition to the low hydrogen permeability, other factors also need to be considered including neutronics, chemical compatibility, radiation stability, and machinability[4].

ZrHx moderator cladded with two metallic cladding materials were demonstrated in this report. One was low carbon arc cast (LCAC) unalloyed molybdenum (Mo) due to its good machinability, fare weldability, and low hydrogen permeation. However, due to the poor neutronics performance of Mo, the detailed characterization of ZrHx cladded with Mo was not pursued, suggested by the Resource Team[4]. The other one was nuclear-grade FeCrAl alloys developed by ORNL for the application as accident tolerant fuel cladding [5]. The FeCrAl was C26M2 with a nominal composition of Fe-12Cr-6Al-2Mo-0.2Si-0.03Y, in weight percent [6]. Crucibles were designed and fabricated from Mo and FeCrAl bars and tubes. Electron beam welding (EBW) and laser beam welding (LBW) were selected as welding techniques because of their high energy density, high quality and high integrate processes. Gas tungsten arc welding (GTAW) was also used as an alternative welding technique. Metallographic works were carried out to check welded cross section and microstructures. Helium leak and bubble tests were performed on welded crucibles for minor leakage and major leakage, respectively. Thermal stability of ZrHx moderator cladded with bare FeCrAl exhibits unacceptable hydrogen release, motivating the development of a hydrogen permeation barrier on the FeCrAl surface. Learning from the M3.1.1 milestone report, "Downselection of cladding materials for zirconium hydride moderator" [4], Al_2O_3 film on the surface of FeCrAl alloys is an effective hydrogen permeation barrier. Air oxidation of FeCrAl tubes at high temperature is sufficient to generate such a protective oxide layer. Material weldability with and without oxide layers, welding heat input effects on cladded zirconium hydride, zirconium hydride position holding inside the crucible, and hydrogen release at elevated temperature are summarized in this report.

2 Crucible Materials, Designs and Conditions

2.1 Crucible materials

Two metals were chosen initially as the cladding material candidates: commercially available LCAC Mo and nuclear-grade FeCrAl alloys developed at ORNL. The purchased 0.5" (12.7 mm) diameter LCAC Mo bar was certified to ASTM B387 Type 365 specification [7], and the chemical composition from the quality certificate is shown in Table 1. The analyzed FeCrAl (Heat #17025001) compositions are shown in Table 2. There are two material forms of the FeCrAl: tube and bar. The FeCrAl tube dimensions were 10.25 mm outer diameter and 0.39 mm wall thickness, and the FeCrAl bar diameter was 0.6” diameter. Both FeCrAl tube and bar were produced in house at ORNL.

Table 1. LCAC Mo chemical composition, wt.% [3]

Mo, %	O, %	N, %	Ni, %	C, %	Fe, %	Si, %
> 99.97	0.0015	0.0002	< 0.0010	0.0050	< 0.0010	< 0.0010

Table 2. Analyzed composition of C26M2 (heat #17025001), balanced Fe, wt.%

Element	Cr	Al	Mo	Si	Y	C	S
Nominal	12	6	2	0.2	0.03	-	-
Analyzed*	11.87	6.22	1.98	0.20	0.03	<0.01	<0.005
* By an induction coupled plasma optical emission spectroscopy (ASTM E1097-12, for major elements) and a combustion analysis (ASTM E1019-11, for C and S)							

2.2 Crucible design and fabrication

Depending on the material type and the required function, there were several crucible designs generated. The basic requirement of the crucible is to enclose the zirconium hydride cylinder with 1 atmosphere internal helium pressure (1 ATM). Therefore, the basic crucible was designed so that a zirconium hydride cylinder can be placed into the crucible, then a cover can be welded on the crucible containing the zirconium hydride cylinder, thus seal and maintain and seal the required atmosphere (1 ATM helium). Following the functional requirement, the basic crucible design is shown in Figure 1. After the zirconium hydride was placed in the metal tube, the cover was placed on the top of the tube. The EBW was planned to be performed first to seal the crucible circumference interface under the vacuum condition, then the LBW was planned to be performed to seal the center hole under the 1 ATM helium condition, as shown in Figure 1. All LCAC Mo crucibles were fabricated following the design shown in Figure 1.

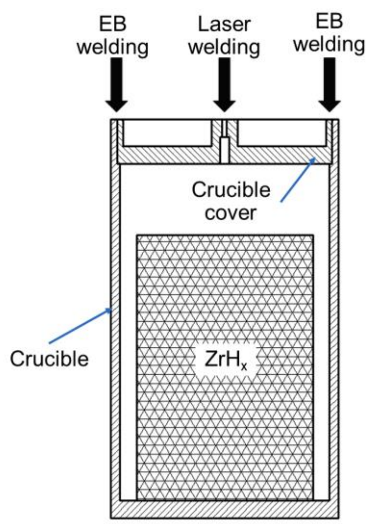


Figure 1. Basic design concept of the crucible containing a zirconium hydride cylinder

Since both FeCrAl tube and bar were available, the FeCrAl crucible basic design was similar but slightly different from the Mo crucible design shown in Figure 1. Beside the top cover, the FeCrAl crucible design also had a bottom cover so that the FeCrAl tube could be sealed by welding from both sides. A schematic of the FeCrAl crucible basic design is shown in Figure 2. The bottom cover was welded along the tube circumference interface by EBW in vacuum condition first. A zirconium hydride rod was placed in after that, followed by EBW of the top cover along the circumference interface in vacuum condition. A final LBW was completed to seal the center hole on the top cover under 1 ATM helium environment.

Beside the FeCrAl crucible basic design, a FeCrAl crucible design with zirconium hydride cylinder position holding function was also carried out. To achieve the cylinder position holding, a cavity with the diameter slightly larger than the zirconium hydride diameter was designed on the bottom cover to hold the cylinder in position, and a stainless steel spring was placed in between the top cover and the cylinder top surface to apply compressive force to the cylinder. The cylinder position holding design schematic is shown in Figure 3. The welding procedures of this crucible design were about the same as the basic FeCrAl crucible design, except the involvement of springs and an extra fixture to hold the crucible top cover down before EBW.

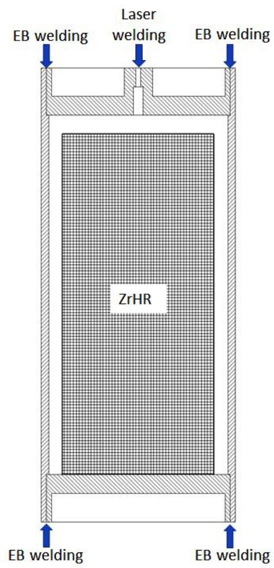


Figure 2. FeCrAl crucible basic design

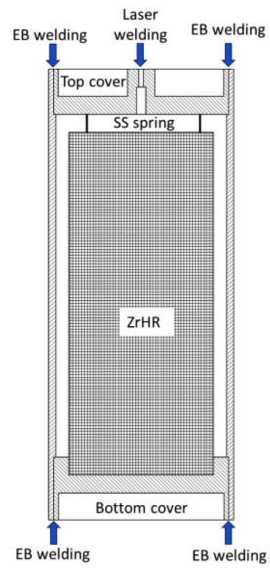
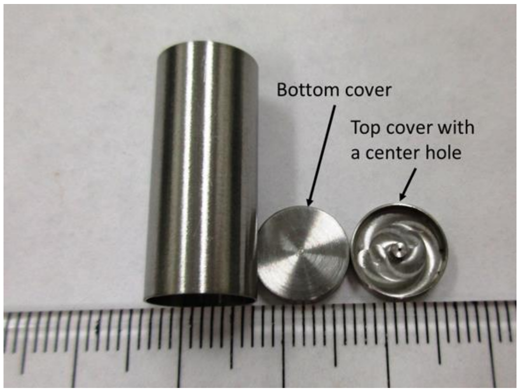


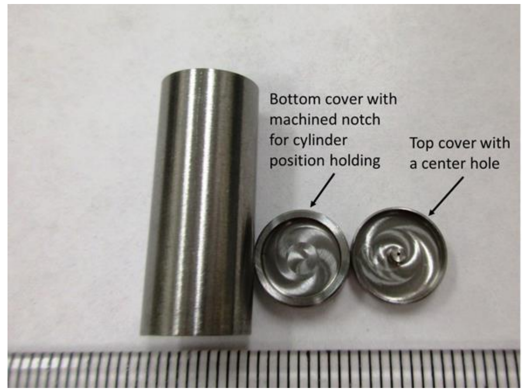
Figure 3. FeCrAl crucible design with position holding

Figure 4. Fabricated LCAC Mo crucible

Mo crucibles were fabricated from 0.5” diameter LCAC Mo bars following the basic design shown in Figure 1, the tube wall thickness was 0.5 mm and the top cover thickness was 1 mm. Two fabricated Mo tubes and covers are shown in Figure 4. FeCrAl crucibles were fabricated following the basic design (Figure 2) and position holiding design (Figure 3). Examples of fabricated FeCrAl crucibles with both designs are shown in Figure 5.



(a) Basic design



(b) Position holder design

Figure 5. Fabricated FeCrAl crucibles with the basic and position holding designs

2.3 Crucible conditions prior to welding

The LCAC Mo crucibles were all in as machined condition before welding. The FeCrAl crucibles had two conditions prior to welding: As machined and pre-oxidized. The main purpose to add a layer of oxide at the FeCrAl tube and cover surfaces is to decrease the hydrogen permeability at elevated temperature as Al_2O_3 is considered an effective hydrogen permeation barrier. The oxidation on the FeCrAl tubes and covers was achieved by heat treatment in air (1178 °C × 2.6 hours). Before the heat treatment, all crucibles were cleaned with alcohol or acetone. An as machined FeCrAl crucible and a heat treated FeCrAl crucible are shown in Figure 6.

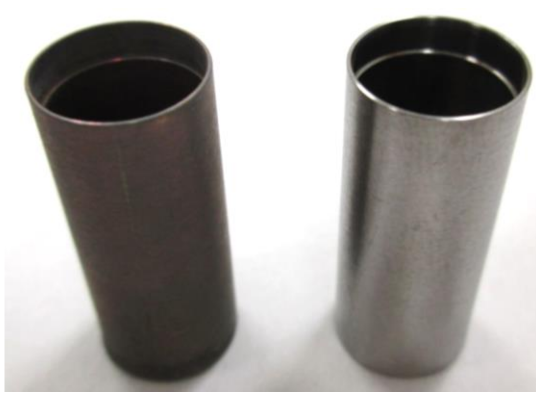


Figure 6. Heat treated (left) and as machined (right) FeCrAl crucibles

Scanning electron microscopy (SEM) was used to characterize the oxide layer thickness after the heat treatment. Results showed that ~1 μm oxide scale was measured on the crucible surface after 1178 °C ×2.6 hours heat treatment in air. The cross section and element mapping of a FeCrAl specimen after 1178 °C × 2.6 hours heat treatment in air are shown in Figure 7. From Figure 7, it is apparent that an oxide layer enriched in Al and O was formed at the FeCrAl surface and the oxide layer thickness was about 1 µm.

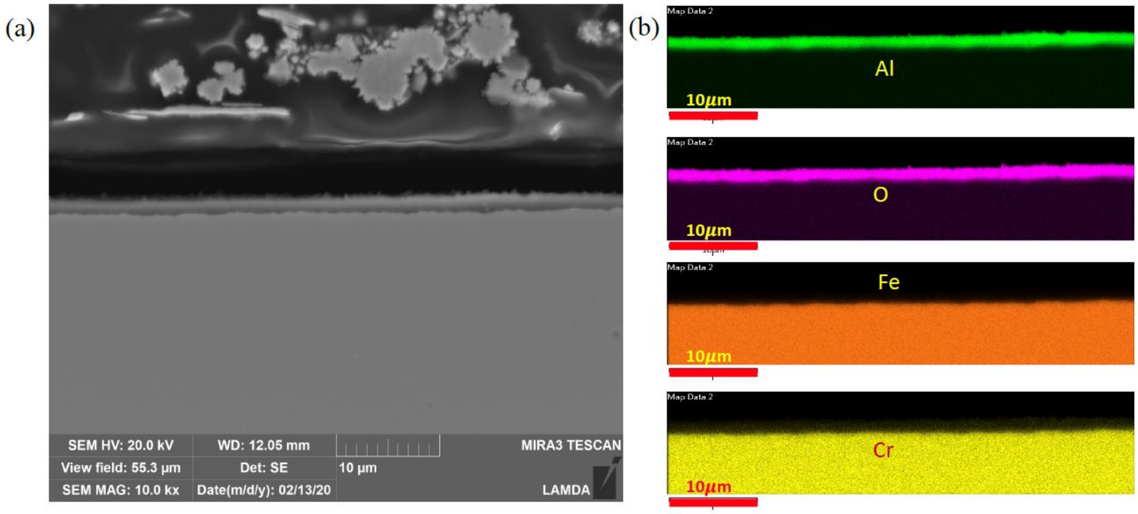


Figure 7. (a) SEM image and (b) element mapping of the cross section of heat treated (1178 °C, 2.6 hours) FeCrAl.

3 Crucible welding and evaluation

3.1 Electron beam welding

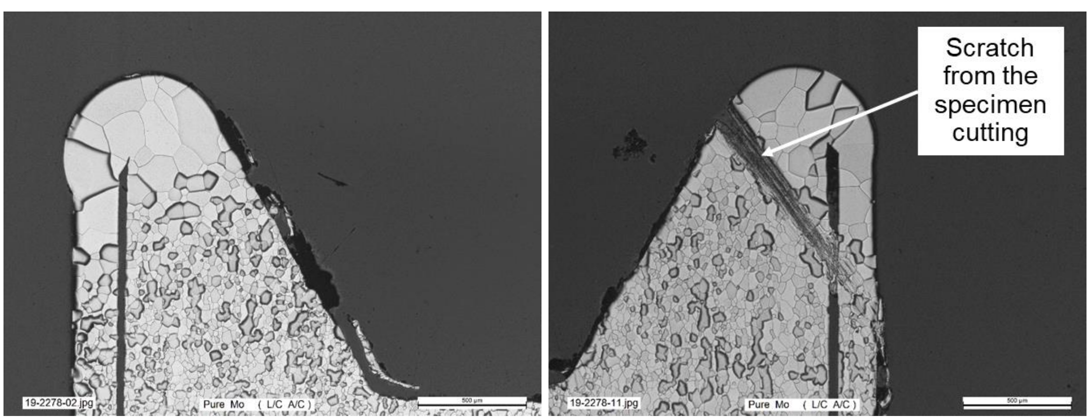
The EBW uses high-velocity electrons to join materials. The high energy density of EBW ensures high speed welding at high efficiency resulting in high penetration with narrow HAZ width and high welding quality. The EBW needs to be carried out under a vacuum condition.

Figure 8. The EBW equipment

EBW was used for circumference welding of all crucibles and covers for both LCAC Mo and FeCrAl materials, as their designs are shown in Figure 1 – Figure 3. The EBW equipment is shown in Figure 8.

Figure 9. Circumference EB welded LCAC Mo crucible

An EB welded LCAC Mo crucible is shown in Figure 9. After the EBW, the welded crucible was sliced open, and the joint portion was mounted, ground, polished and etched with Murakami's etchant (100 ml H_2O, 10 g KOH, and 10 g Potassium ferricyanide ($K_3Fe(CN)_6$). Welded joints at the left and right side of the cross section are shown in Figure 10. From Figure 10, it is clear that the interface of the cover and the tube was well sealed by EBW, and the grains in the weld zone were much larger than those in the LCAC Mo base metal. Using line intersection method, the average EB weld zone grain size was about 121 µm, and the LCAC BM average grain size was about 26 µm.



(a) Left side of the cross section *(b) Right side of the cross section*

Figure 10. Microstructure of the EB welded joint of the LCAC Mo crucible

By using the same machine, circumference joints of FeCrAl tubes and covers were welded by EBW. The circumference welded joints at the top and bottom of a FeCrAl crucibles are shown in Figure 11.

(a) *EB weld on the top*

(b) *EB weld at the bottom*

Figure 11. EB circumference welds at the top and bottom covers of a FeCrAl crucible

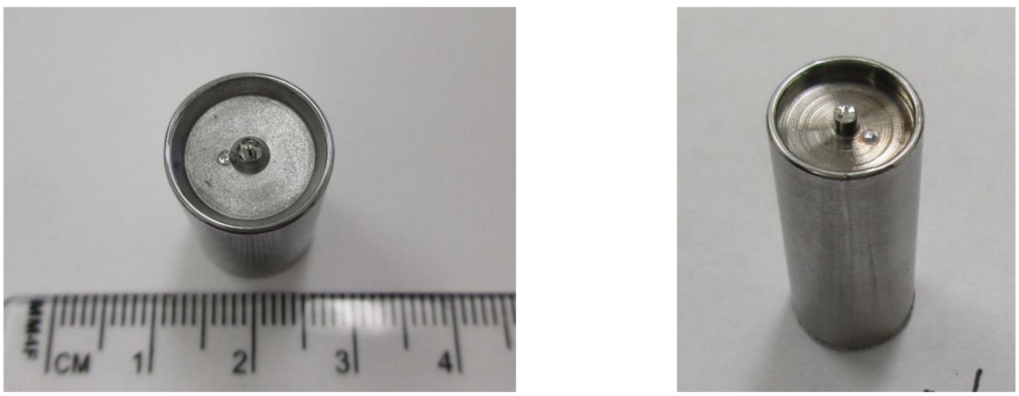
For oxidized FeCrAl crucibles, the oxide layer at the to-be-joined interfaces was removed and cleaned before EBW. The removal of the oxide layer followed by alcohol wipe cleaning ensured high welding quality. A EB welded oxidized FeCrAl crucible is shown in Figure 12.

Figure 12. EB welded FeCrAl crucible with surface oxidation

3.2 Laser beam welding

After EBW, LBW was used to seal the center hole on the top cover at 1 ATM helium atmosphere. The LBW head and chamber used in this study are shown in Figure 13. To ensure 1ATM helium pressure inside crucibles, the working chamber was evacuated to about 50 millitorrs using a small vacuum pump after EB welded crucibles were loaded in. The chamber was then back filled with 1 ATM helium. A pulsed laser was used to seal the top cover center hole after the chamber stabilized with 1 ATM helium. The laser seal welded Mo crucible and FeCrAl crucible are shown in Figure 14.

Figure 13. The LBW head and chamber



(a) LCAC Mo crucible *(b) FeCrAl crucible*

Figure 14. Centers holes laser seal welded crucibles

3.3 Leak test

After welding, leak tests were carried out on all crucibles to evaluate the EBW and LBW quality. The leak test contained two experiments, helium leak test and bubble test. The helium leak test was performed for minor leakage, and the bubble test was performed for major leakage. The passing criteria of the helium leak test was set to 1×10^{-8} std-atm-cc/sec leak rate. If a leakage was detected, further checks were performed to identify the location.

All welded as machined FeCrAl crucibles passed the helium leak test. However, the LCAC Mo crucibles couldn't pass the leak test. Further evaluation showed that the EB weld didn't have any leakage and the leaking location was at the top cover laser welded center hole. In addition, a couple of heat treated crucibles didn't pass the leak test either, and the leaking location was identified at the EB welds. The LCAC Mo crucible leakage was resolved by applying GTAW to seal the top cover center hole. The oxidized FeCrAl crucible leakage was resolved by more careful oxide removal at the to-be-joined interfaces prior to welding. Both types of crucibles passed the leak test after technique was changed/improved.

Leak test report samples are listed in the Appendix.

3.4 Issues and solutions in welding

3.4.1 LCAC Mo crucible center hole seal welding

Comparing with FeCrAl, pure Mo has higher melting temperature (2623 °C vs. 1500 °C) and much higher thermal conductivity (138 W/mK vs. 11W/mK at 20 °C). Therefore, it is more difficult to weld the LCAC Mo than the FeCrAl. The energy density of the EBW were high and it joined the LCAC Mo without any problem. On the other hand, the maximum energy of the LBW equipment was relatively low and insufficient to produce a high quality weld to seal the center hole. A metallographic picture of the LCAC Mo crucible at the center hole area after LBW is shown in Figure 15. Clearly the laser weld shown in this picture does not look as promising as the EB weld shown in Figure 10 on LCAC Mo.

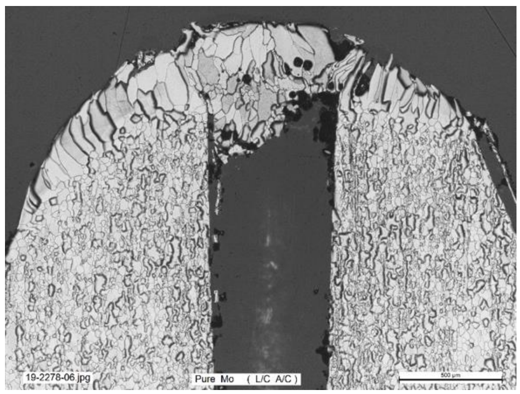
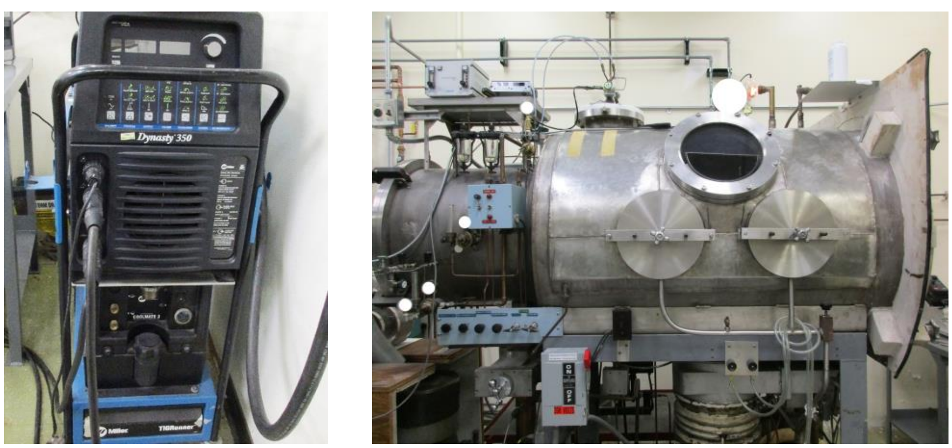


Figure 15. Laser welded LCAC top cover center hole

GTAW can easily generate higher heat input than that of the LBW equipment used in this report. Therefore, applying GTAW inside an environment-controlled chamber may produce sealed crucibles with 1 ATM helium pressure. The GTAW power source and the welding chamber used in this report are shown in Figure 16.



(a) GTAW power source *(b) Environment-controlled welding chamber*

Figure 16. GTAW power source and welding chamber as the alternative way to weld LCAC Mo crucibles

The laser welded LCAC Mo crucibles were drilled open at the center hole location for reliable environment and pressure control. Afterwards, they were placed in the welding chamber shown in Figure 16(b). The welding chamber was evacuated first to about 0.1 millitorrs then backfilled with 1 ATM helium. Finally, GTAW was used to melt Mo around the center hole and seal the crucible. Two crucibles welded inside the welding chamber using GTAW are shown in Figure 17. The center weld made with GTAW (Figure 17) was larger than that made with LBW (Figure 14(a)), because the high heat input GTAW caused more melting of the LCAC Mo.

Figure 17. Center holes sealed by GTAW pure Mo crucibles

With center holes welded by GTAW, both crucibles passed the leak test with the criteria of 1×10^{-8} std-atm-cc/sec leak rate. The more material consumed by GTAW secured the center hole seal of LCAC Mo crucibles. Moreover, a higher energy LBW than the one used in this report may seal weld the LCAC Mo crucible center holes as well.

3.4.2 Oxidized FeCrAl crucibles welding

When EBW was applied on oxidized FeCrAl crucibles, some of them had blowout and/or lack of fusion issues. Repeated welding passes had to be applied on the crucible circumference interface, to form a good weld. However, those crucibles couldn't pass the leak tests described in section 3.3, and the leaking locations were identified at the circumference EB welds. Optical microscope observation of failed welds indicated that the poor weld quality might be caused by too much oxide remaining at the welding interface. Therefore, careful oxide removal and cleaning at the to be joined interfaces were performed prior to welding. After thorough oxide removal and cleaning at the interface, both oxidized top and bottom covers were EB welded onto the oxidized crucible with good surface finish. After LBW on the center hole, the FeCrAl crucible passed the leak test. An oxidized crucible with poor weld quality and a bad surface finish and an oxidized crucible with a high weld quality and a good surface finish are shown in Figure 18.

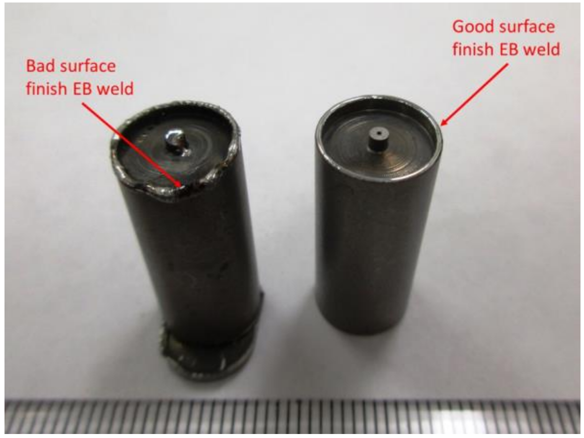


Figure 18. A poor welding quality with bad surface finish oxidized FeCrAl crucible (Left) and a high welding quality with good surface finish oxidized FeCrAl crucible (Right)

4 EXAMINATIONS OF WELDED CRUCIBLES

Following the recommendation from the Resource Team, Mo cladding for zirconium hydride moderator was not further investigated due to its unacceptably poor neutronics performance. The machining and welding experience of Mo developed in this project could be useful for other purposes when Mo tubes are needed. We will focus on the characterization of FeCrAl cladded zirconium hydride moderator in this section.

4.1 Welding heat input effects on Zirconium hydride cylinder

During EBW and LBW of the FeCrAl crucible, welding heat inputs could be a concern that impacts the hydrogen content of the zirconium hydride cylinder in the crucible, considering the sensitivity of hydrogen desorption to heat. Both EBW and LBW are high energy density welding technologies, and welding processes were completed in a short time period of less than a couple of minutes. For that reason, heat affected areas should be quite limited. Therefore, the heat input impact is expected to be very limited to the zirconium hydride which was enclosed in the crucible. To verify that, a zirconium hydride, ZrH-6, was weighed and its chemical composition was examined by X-ray diffraction (XRD). The ZrH-6 was placed in a FeCrAl crucible and sealed with EBW and LBW using the same procedures as described in Section 3. After the welded crucible tested and passed the leak test, the top cover of that FeCrAl crucible was cut open, and the ZrH-6 was extracted for weight measurement and XRD analysis again. The cut open FeCrAl crucible and the extracted ZrH-6 are shown in Figure 19. The weight of the contained ZrH-6 changed from 5.06946 g before welding to 5.06679 g after welding. A ~0.05% weight change, corresponding to 0.7% hydrogen loss was measured. XRD analysis showed that the as-fabricated ZrH-6 has both δ phase zirconium hydride and α-phase zirconium with the phase fractions of 86.7% and 13.3%, respectively. After welding, the phase fractions of the two phases changed to 86.1% and 13.9%, respectively. The XRD patterns of the ZrH-6 before and after welding are shown in Figure 20. Results from both tests are listed in Table 3. Overall, both the weight measurement and the XRD analysis indicated that the EBW and LBW had negligible impact on the hydrogen content of the encapsulated zirconium hydride pellet. These results were reported in Milestone 2.2.1, “Stability of zirconium hydride” [8].

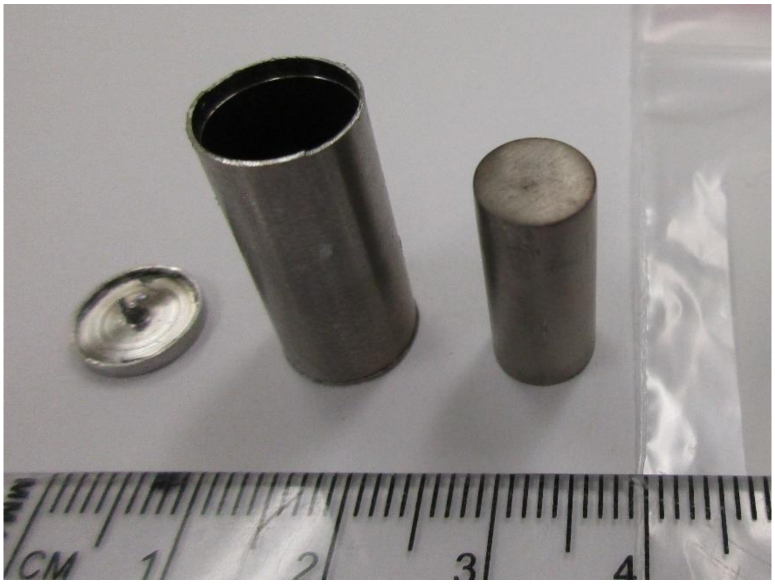


Figure 19. Opened FeCrAl crucible and extracted ZrH-6 cylinder after welding [8].

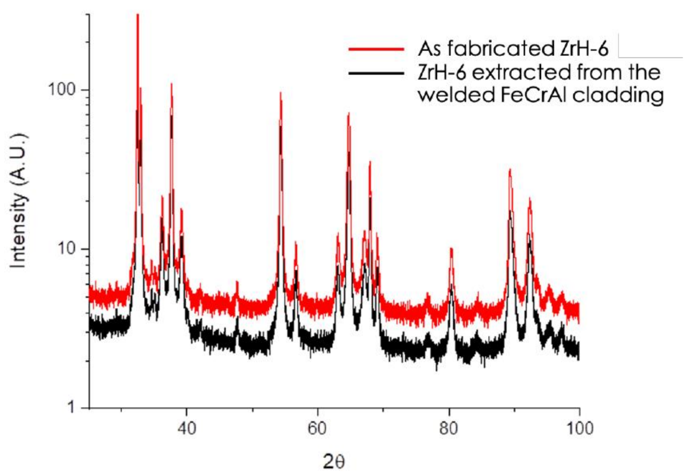


Figure 20. XRD pattern of ZrH-6 (H/Zr=1.398) as fabricated and after welding [8].

Table 3. Measurements and changes in ZrH-6 before and after welding [8].

Test	Before welding	After welding	Changes
Weight, g	5.06946 g	5.06679 g	-0.05%
XRD	86.7% ZrH 13.3% Zr	86.1% ZrH 13.9% Zr	-0.6% +0.6%

4.2 Hydrogen release measurement and crucible oxidation effects on thermal stability at elevated temperatures

Two delta-phase zirconium hydride cylinders ($ZrH_{1.6}$) were placed and seal welded in two FeCrAl crucibles respectively. The two FeCrAl crucibles were machined to the same dimensions but one was used with as machined condition and the other one was used with oxidized layer condition

(1178 °C × 2.6 hours heat treated in air). After they passed the leak test with 1×10^{-8} std-atm-cc/sec criteria, hydrogen releases at elevated temperatures of the two crucibles were directly measured by using the thermal desorption spectroscopy technique. Measurement results are shown in Figure 21. The hydrogen signal increase during the ramping process stems from the degas from the sample surface. When stabilizing at 650°C, hydrogen release from the bare FeCrAl continuously decreases as the hydrogen pressure inside the FeCrAl crucible decreases due to the hydrogen concentration change of the $ZrH_{1.6}$ rod, of which the lower hydrogen concentration corresponds to the lower equilibrium hydrogen pressure. The pre-oxidized FeCrAl efficiently prevented the hydrogen release, manifested by the fact that the stable hydrogen release from the pre-oxidized FeCrAl is overlapping with the background signal, 1.0×10^{-10}A.

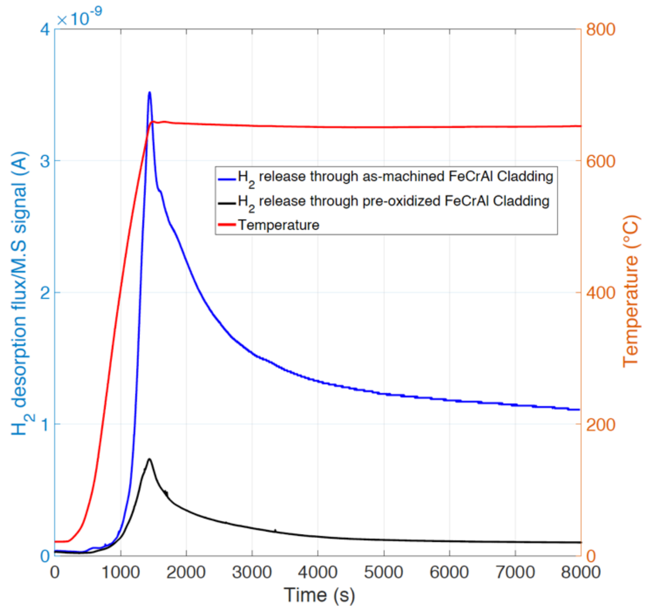


Figure 21. Hydrogen release through as-machined and pre-oxidized FeCrAl cladding containing ZrH1.6 pellet

4.3 X-ray Computed Tomography of ZrHx after thermal desorption

Following the thermal desorption testing, X-ray computed tomography (XCT) was performed to characterize the zirconium hydride cladded with bare and pre-oxidized FeCrAl. Figure 22 shows the reconstructed XCT slices through the two types of FeCrAl cladded zirconium hydride moderators. It is evident that no obvious deformation of the cladding was identified following the 2 hour heat treatment at 650°C. High resolution XCT analysis of the included zirconium hydride rods revealed minor surface cracking, which is believed to be formed during the hydriding process, as shown in the Milestone Report M2.2.2 [2].

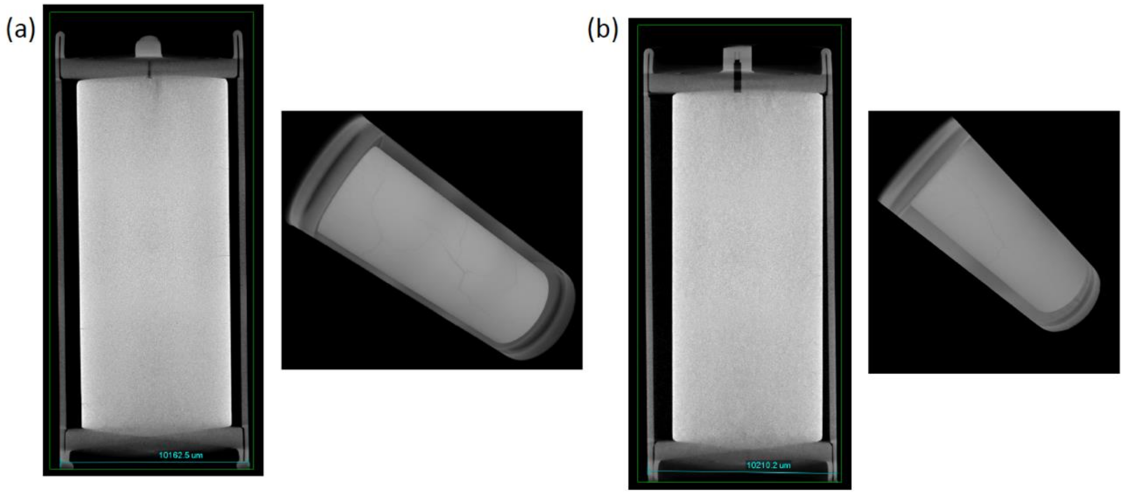


Figure 22 XCT images of (a) bare FeCrAl-ZrHx assembly and (b) pre-oxidized FeCrAl-ZrHx assembly

Following the XCT measurement, the moderator assemblies were cut open to extract zirconium hydride for further analysis, as shown in Figure 23. Two discs removed from the zirconium hydride pellets were then measured with XRD to determine if the heating to 650 C leads to any decomposition in the hydride phase.

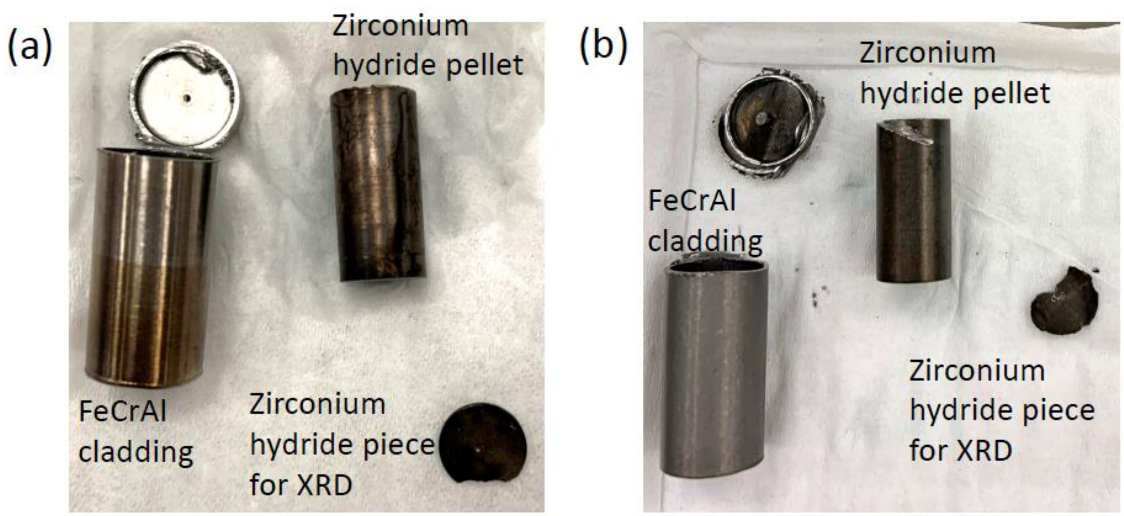


Figure 23 Pictures of the moderator assemblies following cut: (a) bare FeCrAl and (b) pre-oxidized FeCrAl cladded zirconium hydride.

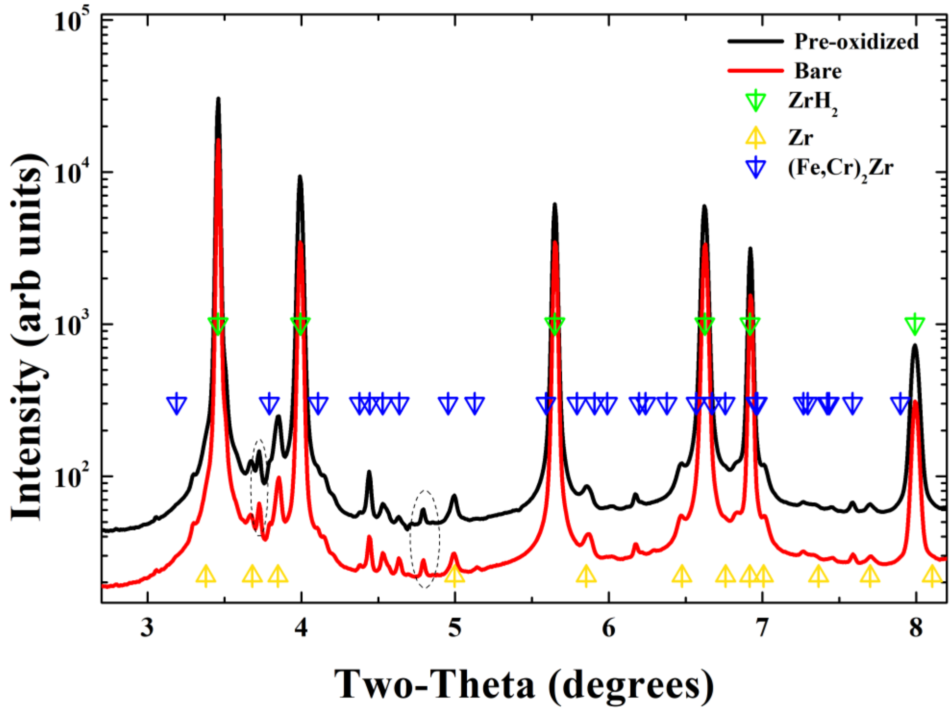


Figure 24. XRD patterns for zirconium hydride discs cut from zirconium hydride cladded with the pre-oxidized and bare FeCrAl, post heat treatment. The pre-oxidized XRD pattern is shifted in the vertical direction for clarification. Two open dashed circles on the patterns show peaks from an unidentified minor phase.

Table 4. Results from the initial XRD phase, and quantitative microstructural analysis.

	Phase	wt.%	a (Å)	c (Å)
Bare	ZrH	98.4 ± (0.7)	4.777 ± (0.001)	
	Zr	1.4 ± (0.2)	3.259 ± (0.003)	5.185 ± (0.011)
	(CrFe)2Zr	0.2 ± (0.1)	5.033 ± (0.012)	8.209 ± (0.033)
Pre-oxidized	ZrH	97.9 ± (0.6)	4.7788 ± (0.001)	
	Zr	1.8 ± (0.2)	3.261 ± (0.003)	5.184 ± (0.008)
	(CrFe)2Zr	0.3 ±(0.1)	5.033 ± (0.008)	8.232 ± (0.021)

High energy XRD patterns for the zirconium hydride discs shown in Figure 23 were collected at the National Synchrotron Light Source-II at the Pair Distribution Function Beamline [9, 10]. The reduced, and background corrected XRD patterns for extracted zirconium hydride specimens are shown in Figure 24 with phases identified overlaid for reference. It is apparent that the delta-phase zirconium hydride is the dominant phase in both specimens and only minor peaks associated with metallic Zr, $(Fe, Cr)_2Zr$ and an (as yet) unidentified phase are present in both specimens. The initial phase quantification results from analyzing the XRD patterns are listed in Table 4. The quantification analysis confirms that the bare and pre-oxidized specimens have similar crystal phases and lattice parameters (within error). The results indicate that the hydrogen release during the 2-hour heat treatment has little impact on the phase change of zirconium hydride. The formation of metallic phases confirms the thermodynamic calculations shown in Milestone M3.1.1, "Downselection of cladding materials for zirconium hydride moderator" [4].

5 Summary

In this report, we summarized our efforts to develop zirconium hydride cladding technique using EBW and LBW with 1×10^{-8} std-atm-cc/sec leak rate criteria, and downselect a metallic material as the cladding material. Different crucibles were designed for the two candidate cladding materials, LCAC Mo and FeCrAl, and machinability and weldability of the two materials were investigated. All crucibles with basic and position holding designs reached the design goals. Between the two metal candidates, FeCrAl had better machinability, weldability, and neutronics performance than the LCAC Mo. The FeCrAl crucible conditions prior to welding included as machined and pre-oxidized by heat treatment. Welded FeCrAl crucibles with both material conditions exhibited good thermal stability at elevated temperatures. Hydrogen release measurement by using thermal desorption spectroscopy technique showed that the oxide film formed on FeCrAl cladding effectively prevented hydrogen release.

6 References

1. Van Houten, R., *Selected Engineering And Fabrication Aspects Of Nuclear Metal Hydrides (Li, Ti, Zr, And Y).* Nuclear Engineering and Design, 1974. **31**: p. 434-448.
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7 APPENDIX

ACCREDITED

ORNL Surveillance & Inspection Organization - Certificate #4121.01/Scope of Accreditation to ISO/IEC 17020:2012

Report Number: 11/11/19-1

LEAK TEST REPORT

Test Requested by: W. TANG	Allowable Leak Rate: < 1.0 E-8 Std-Atm-cc/s
Date Requested: 10/16/19	Date Required: 11/7/19
Work Order Number: 3798305	Test Pressure Req. Across Boundary: -1 ATM
Item Tested: 2 EA. Mo CRUCIBLES	Customer: MSTD
Specification: — NDE 70, Rev: 7	Technique Used: INSIDE-OUT Rev: 0 ☒ Inside - Out ☐ Outside - In

EQUIPMENT

LEAK DETECTOR		STANDARD LEAK	
Make and Model: ADIXEN ASM 340		Manufacturer: VTI	Tracer Gas: He
Serial Number: HLD 1601393		Model: CPP9-He-118T	Serial Number: 4450
		Leak Rate: 5.09E-9 Atm-cc/s @ -1 atm @ 22.6 °C	
TEST GAUGES		Correlation Formula: $[1 - (T_{cal} - T_{surf}) C_T]$ LR	Temp Coefficient: 4.0 %/°C
Temp Gauges: A0019S2	Due: 9/12/20	Correlated LR: 5.0E-9 Atm-cc/s @ -1 atm @ 22.2 °C	
Pressure Gauges: —	Due: —	Calibration Due Date: 01/21/20	

RESULTS ☒ Quantitative ☐ Semi - Quantitative

MACHINE CALIBRATION	SYSTEM TEST CONDITIONS
System Pressure: 1.7E-2 Mb	System Temperature: 22.2 °C ☒ Surface ☐ Internal Gas
Background: < 1.0 E-10 Atm-cc/s	delta P Test Boundary: -1 ATM
Leak Response: 5.0 E-9 Atm-cc/s	Tracer Gas: He % Concentration: 100
Minimum Detectable Leak: 1.0E-9 Atm-cc/s	System Response Time: ~ 5s
System Sensitivity: 2.0E-9 Atm-cc/s	System Response: ≤ 5.0 E-9 Atm-cc/s
Response Time: ~ 3s	Duration of Test: ~ 30s

Aux. Equipment:

☒ ACCEPT ☐ REJECT ☐ SKETCH / DATA ATTACHED | System Leak Rate (w/ stated tracer gas): < 1.0E-8 Atm-cc/s @ -1 atm @ 22.2 °C

COMMENTS:

Mo CRUCIBLES # 1, 3 FINE LT

Test Conducted By: E. VIDAL [signature] Level: III Date: 11/11/19 Time: 9:30

Form NDE 70-MS, Rev. 1 CN02 IDMS: 21077

ORNL Surveillance and Inspection Organization / Certificate #4121.01 / Scope of Accreditation to ISO/IEC 17020:2012

ACCREDITED

Report Number: 11/11/19-2

LEAK TEST REPORT - BUBBLE TEST

Test Requested by: W. TANG	Customer: MSTD
Date Requested: 10/16/19	Date Required: 11/7/19
Work Order Number: 3798305	NDE 70, Rev: 7 — Tech, NDE 70 - BT Rev: 0
Item Tested: 2 EA Mo CRUCIBLES	Test Pressure Required: 15" Hg
Specification: –	Inspection Criteria: NO BUBBLES @ 2 MIN
Technique Used: VAC BOX	Liquid Media Used: ALCOHOL
Test Gas Used: VAC	Liquid Applicator Type: IMMERSION
Inspection Light Intensity: >100 FC	Post Cleaning Method: WIPE DRY
Other Apparatus Used: FLASHLIGHT	

Direct Pressure Technique ☐ **Vacuum Pressure Technique** ☒

Component Limits of Test:

GROSS LT

Component Test Site 5500	Component Installation Site

Gauges				Test Pressure		Temperature	
Mfg	ID No	Calibration Date	Range	Beginning	End	Beginning	End
	A002126	8/26/19	0-30" Hg	15" Hg	15" Hg		

Temperature Measuring Device

Mfg.	Model	Range	I.D. Number

RESULTS ☒ ACCEPT ☐ REJECT POST CLENING PERFORMED: ☒ Y ☐ N

Comments:

Mo CRUCIBLES #1, 3

Test Conducted By: E. VIDAL [signature]	Level: III	Insp. Date: 11/11/19

Form NDE-70-Bubble Rev. 1 CN02 IDMS: 10960

ORNL Surveillance & Inspection Organization - Certificate #4121.01/Scope of Accreditation to ISO/IEC 17020:2012

ACCREDITED

Report Number: 1/22/20 - 5

LEAK TEST REPORT

Test Requested by: W.TANG	*Allowable Leak Rate:* < 1.0 E-8 *Std-Atm-cc/s*
Date Requested: 10/16/19	*Date Required:* 1/22/19
Work Order Number: 3798305	*Test Pressure Req. Across Boundary:* -1 ATM
Item Tested: 1 EA. FeCrAl CRUCIBLE	*Customer:* MSTD
Specification: — *NDE 70, Rev:* 7	*Technique Used:* INSIDE-OUT *Rev:* 0 ☒ *Inside - Out* ☐ *Outside - In*

EQUIPMENT

LEAK DETECTOR		STANDARD LEAK	
Make and Model: ADIXEN ASM 340		*Manufacturer:* VEECO	*Tracer Gas:* He
Serial Number: HLD 1601393		*Model:* SC-4	*Serial Number:* 18091
		Leak Rate: 2.57E-8 *Atm-cc/s @* -1 *atm @* 23.5 *°C*	
TEST GAUGES		*Correlation Formula:* $[1 - (T_{cal} - T_{surf})\ C_T]\ LR$	*Temp Coefficient:* 3.0 *%/°C*
Temp Gauges: A001957	*Due:* 7/30/20	*Correlated LR:* 2.5E-8 *Atm-cc/s @* -1 *atm @* 22.6 *°C*	
Pressure Gauges: —	*Due:* —	*Calibration Due Date:* 10/1/20	

RESULTS ☒ Quantitative ☐ Semi - Quantitative

MACHINE CALIBRATION		SYSTEM TEST CONDITIONS	
System Pressure: 1.6 E-2 Mb		*System Temperature:* 22.7 *°C* ☒ *Surface* ☐ *Internal Gas*	
Background: < 4.0 E-10	*Atm-cc/s*	*delta P Test Boundary:* -1 ATM	
Leak Response: 2.5E-8	*Atm-cc/s*	*Tracer Gas:* He	*% Concentration:* 100
Minimum Detectable Leak: 1.0 E-9	*Atm-cc/s*	*System Response Time:* ~ 5s	
System Sensitivity: 2.0 E-9	*Atm-cc/s*	*System Response:* 1.5 E-9	*Atm-cc/s*
Response Time: ~ 3s		*Duration of Test:* ~ 30s	
Aux. Equipment:			
☒ **ACCEPT** ☐ **REJECT** ☐ SKETCH / DATA ATTACHED		***System Leak Rate:*** *w/ stated tracer gas* < 1.0E-8 *Atm-cc/s @* -1 *atm @* 22.7 *°C*	

COMMENTS:

Test Conducted By: EVIDAL [signature]	*Level:* III	*Date:* 1/22/20	*Time:* 12:35

Form NDE 70-MS, Rev. 1 CN02

IDMS: 21077

ORNL Surveillance and Inspection Organization / Certificate #4121.01 / Scope of Accreditation to ISO/IEC 17020:2012

Report Number: 1/22/20-6

LEAK TEST REPORT - BUBBLE TEST

Test Requested by: W. TANG	Customer: MSTD
Date Requested: 10/16/19	Date Required: 1/22/20
Work Order Number: 3798305	NDE 70, Rev: 7 — Tech, NDE 70 - BT Rev: 0
Item Tested: 1 EA. FeCrAl CRUCIBLE	Test Pressure Required: 15" Hg
Specification: –	Inspection Criteria: NO BUBBLES @ 2 MIN
Technique Used: VAC BOX	Liquid Media Used: GM 200 @ 20% SOLN
Test Gas Used: VAC	Liquid Applicator Type: IMMERSION
Inspection Light Intensity: > 100 FC	Post Cleaning Method: DI RINSE
Other Apparatus Used: FLASHLIGHT	

Direct Pressure Technique ☐ Vacuum Pressure Technique ☒

Component Limits of Test:

Component Test Site 5500	Component Installation Site

Gauges				Test Pressure		Temperature	
Mfg	ID No	Calibration Date	Range	Beginning	End	Beginning	End
	A002126	8/26/19	0-30" Hg	15" Hg	15" Hg		

Temperature Measuring Device

Mfg.	Model	Range	I.D. Number

RESULTS ☒ ACCEPT ☐ REJECT POST CLENING PERFORMED: ☒ Y ☐ N

Comments:

Test Conducted By: E. VIDAL [signature] Level: III Insp. Date: 1/22/20

Form NDE-70-Bubble Rev. 1 CN02 IDMS: 10960