

Plan

Yttrium Hydride Post-Irradiation Examination Plan



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Idaho National Laboratory

<p>YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN</p>	<p>Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22</p> <p>Page: ii of viii</p>
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YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: iii of viii</div>
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EXECUTIVE SUMMARY

The Department of Energy Office of Nuclear Energy's Microreactor program aims to provide infrastructure and a qualification bed for the development of micro nuclear reactors. One key need is the development and qualification of high-temperature moderators that reduce fuel loading and ensure their compact design for flexible operational purposes. Because microreactors must be compact to offer a flexible operational purpose, metal hydrides are expected to be used as neutron moderator materials due to their high hydrogen number density. Los Alamos National Laboratory (LANL) is developing qualified fabrication techniques for metals hydride, especially yttrium hydride (YH_x) which provides advantages over other metal hydride candidates. Qualification of YH_x involves the fabrication process, thermomechanical properties, and performance which must be met for the deployment of microreactors. In the support of the qualification process metal hydride, LANL has developed fabrication techniques based on direct hydriding and powder metallurgy. For both manufacturing techniques, fresh material properties of metal hydrides have been reported in the literature. To establish the performance limits and the qualification parameters, an irradiation campaign has been initiated at Idaho National Laboratory (INL). The irradiation campaign aims to investigate the critical irradiated YH_x properties as a function of irradiation temperature and fabrication method. The first phase of the irradiation campaign includes the effect of the operating temperature under neutron irradiation which provides initial data on the metal hydrides and informs the manufacturing processes. A drop-in irradiation experiment for use in the INL Advanced Test Reactor (ATR) has been designed. This document provides the post-irradiation examination (PIE) plan of the ATR irradiated YH_x . The objective of the PIE plan is to serve as a qualification basis for YH_x as a neutron moderator material by understanding the impacts irradiation has on the physical and thermophysical properties, to establish a link between fabrication and the irradiated YH_x properties, to institute a PIE strategy for other solid-state neutron moderator candidates, and to inform microreactor fuel performance and safety codes.

Idaho National Laboratory

<p>YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN</p>	<p>Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22</p> <p>Page: iv of viii</p>
---	--

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YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: v of viii</div>
--	---

CONTENTS

1.	INTRODUCTION	1
1.1	Background	1
1.2	Summary of LANL-MOD-1 Irradiations	1
1.3	Initial Condition of LANL Capsules Prior to Irradiation	5
1.4	Objectives of the Post-Irradiation Examinations	7
2.	REQUIREMENTS	7
3.	PRECAUTIONS AND LIMITATIONS	7
4.	PERSONNEL	8
5.	PRE-EXAMINATION SURVEY OF FRESH YTTRIUM HYDRIDES	8
6.	YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION	9
6.1	Scope	9
6.1.1	Non-Destructive Examinations (NDE)	9
6.1.2	Destructive Examinations (DE)	10
6.2	Non-Destructive Examinations	10
6.2.1	Cask Shipment and ATR Basket Retrieval	10
6.2.2	Neutron Radiography of the ATR Basket	10
6.2.3	Retrieval of ATR Fixtures from the ATR Basket	10
6.2.4	Retrieval of LANL Capsules and Fluence Wires from the ATR Fixtures	11
6.2.5	Packaging and Transport of Fluence Wires to Relevant Institution	11
6.2.6	Visual and Dimensional Inspections of LANL Capsules	11
6.2.7	Mass Measurement of LANL Capsules	11
6.2.8	Disposal of ATR Basket and Fixtures	11
6.2.9	Transfer of LANL Capsules to the Appropriate Facility for Disassembly	12
6.2.10	Disassembly of the LANL Capsules	12
6.2.11	Packaging of the Specimens and Melt Wires	12
6.2.12	Optical Inspection and Dimensional Measurements of Specimens	12
6.2.13	Mass and Volume Measurements of YH _x and TZM Specimens	13
6.2.14	X-ray Diffraction (XRD) of YH _x and TZM Specimens	13
6.2.15	Transfer of Selected Specimens to the External Institutions for Advanced PIE	13
6.3	DESTRUCTIVE EXAMINATIONS	13
6.3.1	Hydrogen Content Measurements	13
6.3.2	Metallography and Electron Microscopy/Spectroscopy of YH _x and TZM Samples	13
6.3.3	Mechanical Properties of YH _x Specimens	14
6.3.4	Thermophysical Properties of YH _x	15
6.3.5	Hydrogen Transport Analysis	15

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier:	PLN-6268
	Revision:	1
	Effective Date:	03/04/22
		Page: vi of viii

7. REFERENCES 16

Appendix A Sample Identifications..... 17

Appendix B PIE Flow (HFEF) 18

Appendix C PIE Flow (AL, IMCL, STAR)..... 19

FIGURES

Figure 1. Arrangement of ATR basket, ATR fixtures, LANL capsules, and sample placement in the LANL capsule..... 3

Figure 2. Schematics for an ATR fixture assembly steps. 4

Figure 3. Predicted irradiation temperatures and internal gas compositions of LANL capsules. 4

Figure 4. Picture of assembled capsules, except the capsule 2B. 5

Figure 5. A crack detected after welding of the LANL Capsule 2B during optical inspections, the crack initiated via welding process. 6

Figure 6. Arrangement of LANL capsules in the ATR basket and location of the defected capsule. 6

TABLES

Table 1. Dimensions of components in a single LANL capsule loaded into an ATR fixture (INL-DWG-822579)..... 2

Table 2. Layout of LANL capsules in each ATR fixture..... 6

Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier:	PLN-6268	
	Revision:	1	
	Effective Date:	03/04/22	Page: vii of viii

ACRONYMS

AL	Analytical Laboratory
APT	Atom probe tomography
ATR	Advanced Test Reactor
BSE	Backscattered Secondary Electron
DE	destructive
DOE	Department of Energy
DOE-NE	Department of Energy Nuclear Energy
DSC	differential scanning calorimetry
DWG	Drawing
EBSD	Electron backscatter diffraction
EDS	Energy dispersive spectroscopy
GDOES	Glow-Discharge Optical Emission Spectroscopy
HALEU	High Assay Low Enrichment Uranium
HFEF	Hot Fuel Examination Facility
IMCL	Irradiated Materials Characterization Laboratory
INL	Idaho National Laboratory
LANL	Los Alamos National Laboratory
LECO	Laboratory Equipment Corporation
LF	Laser-flash
LFA	Laser-flash analysis
LI	Laboratory Instruction
MFC	Materials and Fuels Complex
NDE	non-destructive
NRAD	Neutron Radiography Reactor
PAS	Positron Annihilation Spectroscopy
PCA	Performer Controlled Activity
PED	Precession electron diffraction
PI	Principal Investigator
PIE	Post-irradiation examination
QA	Quality Assurance

Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="float: right;">Page: viii of viii</div>
--	---

RUS	Resonant Ultra-Sound
SE	Secondary Electron
SEM	Scanning electron microscopy
SME	Subject Matter Expert
STAR	Safety and Tritium Applied Research
TEM	Transmission electron microscopy
TZM	Titanium-zirconium-molybdenum
VEM	Visual Examination Machine
WGS	Waste Generation Service
XRD	X-ray diffraction
YH _x	Yttrium hydride

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: 1 of 19</div>
--	---

1. INTRODUCTION

1.1 Background

The overarching goal of the Department of Energy Office of Nuclear Energy (DOE-NE) microreactor program is to develop technologies for the deployment of civilian microreactors by stakeholders¹. Microreactors are expressed as advanced transportable nuclear reactors operating at low power (<20MWth) but high temperatures (>600°C), as well as plug-and-play and inherently safe designs. One prerequisite of a microreactor is the compactness, so that a truck can transport the reactor under safe conditions with the current road infrastructure^{1,2}. The compactness of these reactors likely can be attainable by use of solid components for the essentials of the nuclear core, such as fuel, core heat removal components, reflectors, and moderators. Among these essentials, where fuel enrichment must remain < 20% to meet High Assay Low Enrichment Uranium criteria (HALEU), the largest contribution to the compactness is offered by use of solid moderators which benefit from light atomic weight elements, such as hydrogen, carbon, and beryllium². Among these, hydrogen-bearing materials, such as metal hydrides, are superior to other options from the lowest critical mass standpoint. Noting that, factors other than critical mass should be considered for a specific reactor design.

Yttrium- or zirconium-based metal hydrides have been down-selected due to their neutronic performance. In addition to the neutronic perspective, maintaining hydrogen within the metal hydride is important at the high operating temperatures proposed by advanced reactors. Yttrium hydride (YH_x) is, therefore, a proposed moderator material that offers better hydrogen retention at higher operating temperatures than zirconium hydrides due to higher retention and thermal stability of hydrogen in the metal³.

The irradiated materials properties of metal hydrides, in this case YH_x, must be assessed for the qualification of these moderators. Material testing and inspection processes must illustrate that the effects of dimensional and property changes on thermophysical and mechanical properties do not cause any significant changes on the microreactor safety, and the moderating power is maintained within design limits. Thus, the effect of irradiation on the thermophysical and mechanical properties must be determined. This post-irradiation examination (PIE) plan specifically aims to determine these properties for YH_x following Advanced Test Reactor (ATR) irradiation.

1.2 Summary of LANL-MOD-1 Irradiations

The Los Alamos National Laboratory (LANL)-MOD-1 irradiation is a drop-in experiment that aims to examine the irradiation performance of YH_x and investigate any dimensional changes and determine the thermophysical properties of irradiated YH_x. Details of the LANL-MOD-1 irradiations are described in the Idaho National Laboratory (INL) reports of “TFR-1049 LANL-MOD-1 Irradiation in the ATR NEFT Small B Positions”⁴ and “DP-157 LANL-MOD-1 Experiment Data Package.”⁵ Irradiation campaign uses one Advanced Test Reactor (ATR) basket that contains three stacked ATR fixtures as shown in Figure 1. Dimensions of the important components are listed in Table 1 as well.

Disk and cylindrical YH_x specimens were stacked in these LANL capsules (see Figure 1). Additional titanium-zirconium-molybdenum (TZM) disk-shaped sheets were placed in between Resonant Ultra-Sound (RUS), Glow-Discharge Optical Emission Spectroscopy (GDOES), and laser-flash analysis (LFA) samples. These thin TZM sheets will also be used as samples. At the bottom of the LANL capsule, differential scanning calorimetry (DSC) specimens were surrounded by a TZM ring.

Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier:	PLN-6268
	Revision:	1
	Effective Date:	03/04/22

Page: 2 of 19

LANL capsules were made of TZM alloy, and they were sealed via electron beam welding under a gas mixture of ~8 torr hydrogen and 592 torr helium pressures (total 600 torr).

Each of the three ATR fixtures consists of two LANL capsules and melt wires between two capsules (see Figure 1). Assembly steps of an ATR fixture are shown in Figure 2. LANL capsules and melt wires were inserted into a half piece of a clam shell ATR fixture, and the opposite piece of the clam shell ATR fixture was placed onto the other half. After this step, the ATR fixture was clamped from each side. The three clamped clam shells were slid inside a tube. This ATR basket was welded and, finally, was inserted into the B2 position of ATR. In total, six LANL capsules are subjected to one ATR cycle, 169A.

Table 1. Dimensions of components in a single LANL capsule loaded into an ATR fixture (INL-DWG-822579). Each ATR fixtures consist of two LANL capsules.

Component	Material	Quantity	Outer diameter (mm)	Inner diameter (mm)	Height (mm)
Sample Column	YH _x (RUS)	3	12.5	N/A	10.0
	YH _x (GDOES)	5	12.5	N/A	2.0
	YH _x (TEM)	1	12.5	N/A	2.0
	YH _x (LFA)	2	12.5	N/A	2.0
	YH _x (DSC)	6	5.0	N/A	1.5
	TZM sheet	6	12.5	N/A	0.5
	TZM ring	1	12.5	5.0	9.0
Capsule	TZM	1	14.0	13.0	63.6
Capsule lid	TZM	4	13.0	12.0	2.0
ATR Fixture	SS304L	1	Variable	14.43	174.9
Core holder	SS304L	1	21.01	18.47	543.3

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier:	PLN-6268
	Revision:	1
	Effective Date:	03/04/22
		Page: 3 of 19

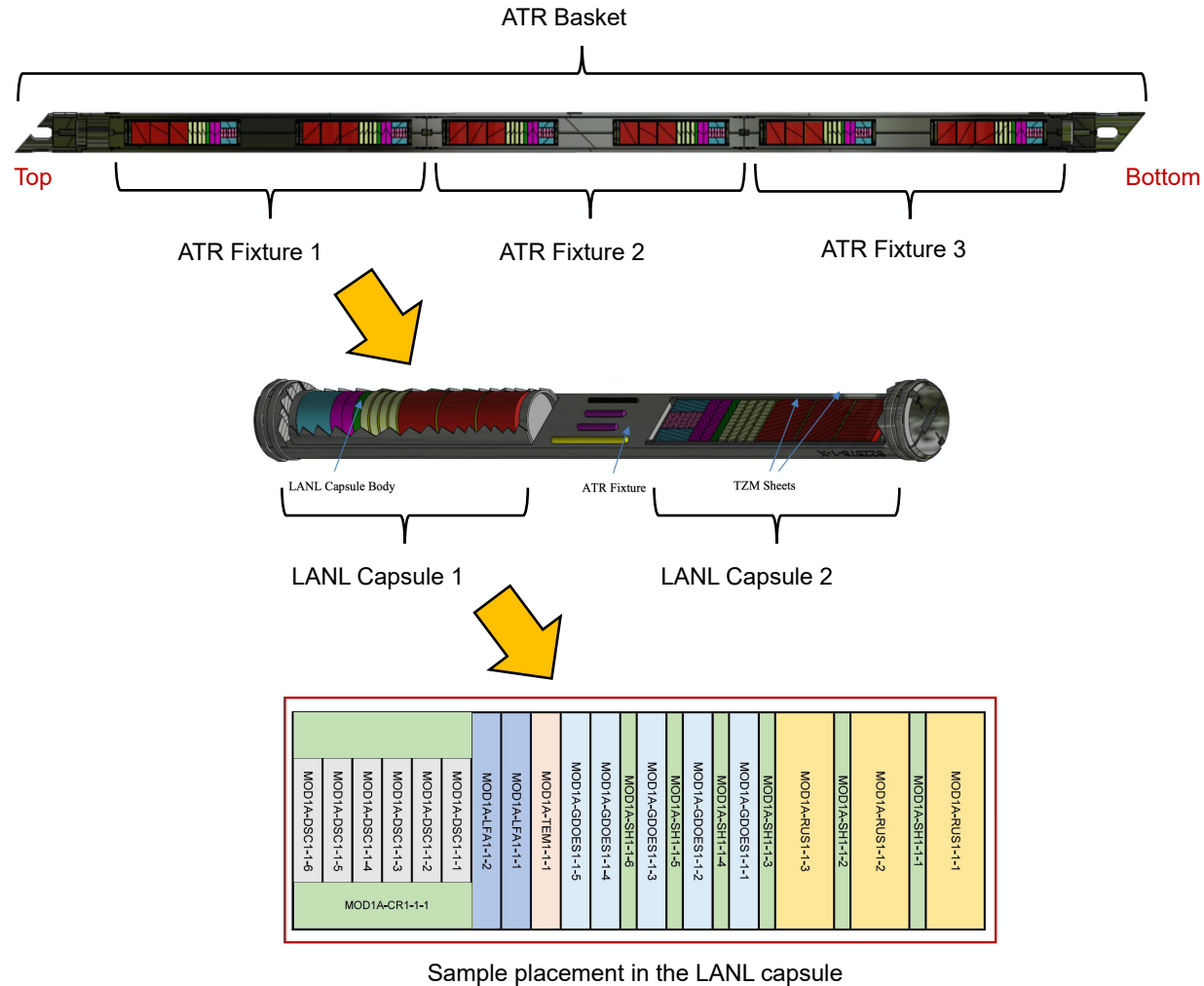


Figure 1. Arrangement of ATR basket, ATR fixtures, LANL capsules, and sample placement in the LANL capsule. In total, six LANL capsules are subjected to irradiations in one ATR basket. Melt wires are not shown in this schematic.

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier:	PLN-6268
	Revision:	1
	Effective Date:	03/04/22
		Page: 4 of 19

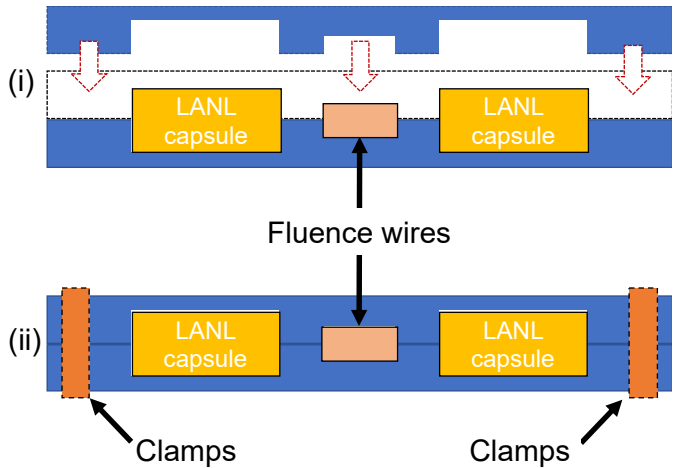


Figure 2. Schematics for an ATR fixture assembly steps.

Figure 3 shows the nominal temperatures of each ATR fixture during irradiation. Temperature predictions and other engineering data are reported in “INL-ECAR-4820 Thermal and Structural Safety Compliance Analysis for LANL-MOD-1 Experiment in the B-2 Position in ATR.”⁶ From the top to the bottom of the basket, the nominal temperature of YH_x samples is 600, 800, and 700°C, respectively. The maximum temperature gradient within each ATR fixture is calculated as 26°C. ATR fixture is filled with an Ar-He gas mixture up to 760 Torr (1 atm). In addition, the LANL cans were fabricated with a varying Ar-He gas composition plus 10 Torr hydrogen. The hydrogen was included to help maintain the given YH_x stoichiometry in the samples.

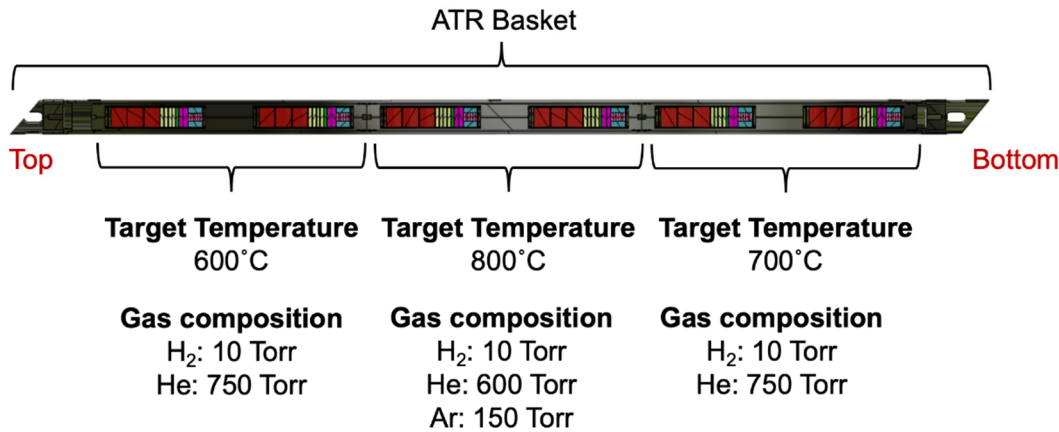


Figure 3. Predicted irradiation temperatures and internal gas compositions of LANL capsules.

Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier:	PLN-6268
	Revision:	1
	Effective Date:	03/04/22

Page: 5 of 19

1.3 Initial Condition of LANL Capsules Prior to Irradiation

LANL capsules were assembled at LANL facility. After installing specimens into TZM capsules, capsules were sealed with a lid made of TZM using an orbital arc-welder. Assembled capsules are shown in Figure 4, except capsule 2B. A surface oxidation on the capsule lids was observed after the welding process. Capsules (1, 3A, 4, 5, and 6) passed the helium gas leak check. The welding of the capsule 2B successively failed, and the helium gas leak check implied the presence of a crack. The crack at the weld section was also detected via optical microscopy as shown in Figure 5. The crack extended through the wall thickness of the TZM capsule. The samples in this can were fabricated via powder metallurgy technique.

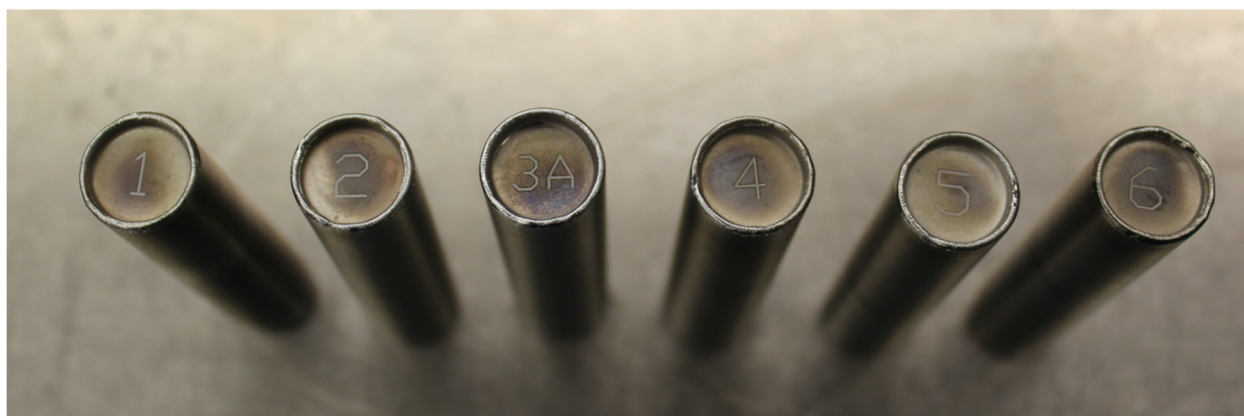


Figure 4. Picture of assembled capsules, except the capsule 2B.

During ATR irradiation, this location is expected to act as an open path for the gas communication between the samples in this can and the ATR fixture's free volume. The final step in the weld assembly of the ATR basket involves weld joining, which seals the basket under the Ar He gas mixture. During this process, the ATR basket gas will infiltrate the cracked 2B capsule during the final weld, and the 2B capsule will not contain the desired H_2 gas. A new thermal analysis has been performed for this "as-built" configuration, and the new projected temperatures are shown in Table 2. The WUPS process evacuated the basket assembly to -20 in Hg for 30 minutes or until the pressure stabilized, after which the basket was pressurized up to 20 psi with the Ar/He gas mixture. This pump-purge cycle was repeated five times to ensure that the air was removed from the cracked canister and basket assembly. Nevertheless, the cracked and air-filled capsule scenario is considered as the worst-case scenario. Under that condition, the average temperature of the capsule increases 10-15°C for all capsules other than 2B. The average temperature of the capsule 2B increases from 699°C (intact condition) to 769°C. Since installation will be performed under inert atmosphere, the cracked and evacuated condition of the capsule 2B is considered as the most likely situation during the irradiation period. The arrangement of individual LANL capsules and the defected capsule (2B) is also illustrated in Figure 6.

**YTTRIUM HYDRIDE
POST-IRRADIATION EXAMINATION
PLAN**

Identifier: PLN-6268

Revision: 1

Effective Date: 03/04/22

Page: 6 of 19

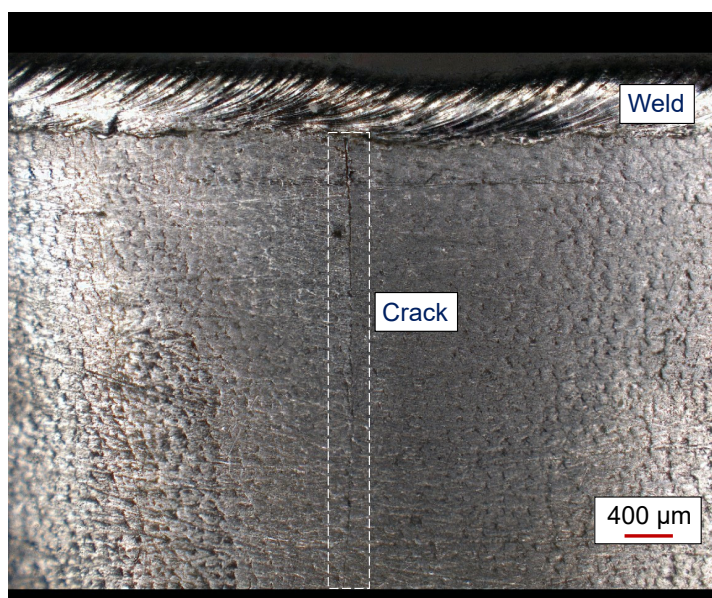


Figure 5. A crack detected after welding of the LANL Capsule 2B during optical inspections, the crack initiated via welding process.

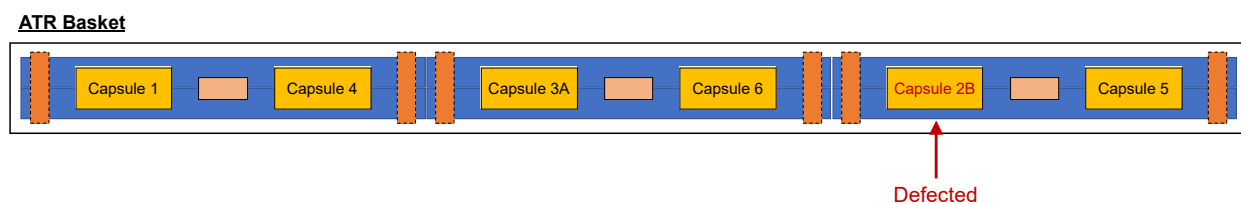


Figure 6. Arrangement of LANL capsules in the ATR basket and location of the defected capsule.

Table 2. Layout of LANL capsules in each ATR fixture. The original temperatures are shown as intact condition, as well as the projected temperatures for the as-built assembly under two potential scenarios. The "Evacuated cracked capsule" scenario is considered the most likely scenario.

Average Capsule Temperature (°C)	Condition of Capsule 2B			
	Capsule	Intact	Cracked and Evacuated (best scenario)	Cracked and Air Filled (worst scenario)
	1	600	601	609
	4	601	602	610
	3A	801	801	811
	6	800	803	813
	2B	699	756	769
	5	700	706	715

Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: 7 of 19</div>
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1.4 Objectives of the Post-Irradiation Examinations

The objective of this plan is to document data collection and handling requirements for the non-destructive (NDE) and destructive (DE) PIE of YH_x. Data collected from NDE and DE examinations will be utilized for one or more of the activities shown below.

1. Serve as a qualification basis for YH_x as a neutron moderator material, by understanding the impacts irradiation has on the physical and thermophysical properties;
2. Establish a link between manufacturing path and the irradiated YH_x properties;
3. Establish a PIE strategy for other solid-state neutron moderator candidates, and
4. Inform microreactor material performance and safety codes.

2. REQUIREMENTS

This PIE activity is supposed to use more than one facility. All work control documents will be completed as necessary and approved prior to commencing any testing activities. Experimental work will be conducted per an approved Laboratory Instruction (LI) or completed as a Performer Controlled Activity (PCA) depending on the hazards involved in performing the activity. Both LIs and PCAs will be utilized for conducting work associated with this test plan.

Equipment operators shall have backgrounds in engineering and/or appropriate knowledge, training, and experience to safely conduct the tests. Equipment operators shall meet the training requirements of the specified facilities. This information shall be included in the PIE documentation (official electronic laboratory notebook and PIE report).

Due to radiological contamination concerns, some equipment in radiological hoods and gloveboxes may only be calibrated prior to installation. In these instances, redundant measurement equipment should be used whenever possible. Potential sources of uncertainty and error in the measurement shall be identified, minimized as appropriate, and included in the PIE documentation.

3. PRECAUTIONS AND LIMITATIONS

All activities, at a minimum, shall be performed in accordance with LWP-21220, "Work Management."⁷[7] Further instruction on work scope execution is provided by this test plan.

Samples to be examined in this PIE plan must be handled with care due to the brittle nature of the samples. Because hydrogen is bound to the samples, hydrogen release and oxidation can occur. Precautions to avoid any unexpected or unplanned hydrogen release must be taken. This will require careful consideration before polishing samples and any heat treatments.

Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: 8 of 19</div>
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4. PERSONNEL

This PIE plan may include one or more principal investigators (PI), subject matter expert (SME), and performers/operators.

- Principal Investigator (PI) must be knowledgeable in the NDE and DE activities. The PI must have technical understanding of the Microreactor program goals, interpretation and use of the generated data, knowledge of Quality Assurance (QA) requirements. Responsibilities of the PI are (i) development of test plans, (ii) specification of test instructions and requirements, (iii) data analysis and reporting.
- Subject Matter Expert (SME) must be the expert on the relevant NDE or DE technique. SME must be familiar with the physical operation and limits of the relevant technique's equipment. SME must be familiar with the sample requirements and data-acceptance requirements of the relevant technique. Responsibilities of SME are (i) implementation of test plans, (ii) overseeing measurement equipment's calibration for testing conducted in accordance with the test plan, (iii) verifying performers/operators meet the training requirements of the relevant technique, (iv) supervision of the equipment's periodic operation and calibration verifications, completion of data review, data analysis, and reporting.
- Process Engineer/Performer/Operator must have background in science and engineering. Performer/Operator must be trained on safe and accurate execution of relevant tests and activities under SME's supervision, in accordance with training requirements indicated in applicable work control document, test standards, and methods. Responsibilities of the performer/operator are (i) configuration control of relevant equipment, (ii) ensuring the operational checks of the relevant equipment, (iii) completion of experimental instructions, records registered laboratory notebooks, and (iv) printed or electronic data management and archival.

5. PRE-EXAMINATION SURVEY OF FRESH YTTRIUM HYDRIDES

Fresh YH_x will be obtained from LANL. These samples will be used for performing baseline experiments to gain experience with the metal hydrides which are brittle in nature and oxidation-resistant. If necessary, the PI can ask for additional supporting examinations other than those listed below.

1. Testing is required to identify if hydrogen is emitted from the unirradiated YH_x while stored in a low hydrogen partial pressure atmosphere (i.e., argon atmosphere) to better understand the impact on irradiated materials and apply controls to minimize these impacts if needed.
2. Base-line examinations of YH_x comprise all the destructive examinations to be performed on fresh YH_x samples. These experiments are expected to be performed at the same facilities as destructive examinations. This effort will allow for the development of procedures and reduce the risk of experimental error.
3. Mockup LANL capsules will be obtained from LANL to practice at INL's mockup shop to develop a capsule cutting procedure and associated equipment.

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YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: 9 of 19</div>
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4. Handling of irradiated YH_x samples is challenging due to the brittle nature of the metal hydrides. Therefore, possible jigs, storage, and transfer methods will be investigated with the Materials and Fuels Complex (MFC) remote engineering support group.

6. YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION

6.1 Scope

The scope of PIE work is to determine the physical, thermal, and mechanical properties of YH_x moderator and to provide basic characterization on the moderator cladding candidate of TZM alloy specimens. PIE activities are categorized as non-destructive and destructive examinations and include:

6.1.1 Non-Destructive Examinations (NDE)

1. Cask shipment and ATR basket retrieval
2. Neutron radiography of the ATR basket
3. Retrieval of ATR fixtures from the ATR basket
4. Retrieval of LANL capsules and fluence wires from ATR fixtures
5. Packaging and transport of fluence wires to relevant institution
6. Visual and dimensional inspections of LANL capsules
7. Mass measurements of LANL capsules
8. Disposal of ATR basket and fixtures
9. Transfer of LANL capsules to the appropriate facility for disassembly
10. Disassembly of LANL capsules
11. Packaging of the specimens and melt wires
12. Optical inspection and dimensional measurements
13. Mass and volume measurements of YH_x and TZM samples
14. X-ray diffraction (XRD) measurements of YH_x and TZM samples
15. Transfer of selected YH_x and TZM samples to external institutions for advanced PIE

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YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: 10 of 19</div>
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6.1.2 Destructive Examinations (DE)

1. Hydrogen content measurements in YH_x and TZM samples
2. Metallography and electron microscopy/spectroscopy of YH_x and TZM samples
3. Mechanical properties of YH_x samples
4. Thermophysical properties of YH_x
5. Hydrogen Transport analysis in YH_x

All PIE activities shall be performed under the guidance of existing work controls, forms, available standards, and documentation of the relevant facilities. All the PIE data, including essential forms, and procedures shall be included in the PIE documentation (official lab notebook, the final report, or other). The digital data will be stored in the INL's digital data management system. All NDE, DE, and base-line examinations must be compliant with INL's quality assurance program PDD-1300 from the shipping cask acceptance to the final PIE data dissemination.

6.2 Non-Destructive Examinations**6.2.1 Cask Shipment and ATR Basket Retrieval**

The shipping cask will be transferred from ATR to HFEF using BR-3 cask. Once the shipping cask arrives at HFEF, the cask is mated with the HFEF hot cell, and the irradiation basket is unloaded from the shipping cask. Handling of the cask and irradiation basket are performed using cranes and manipulators using existing standard procedures. Documentation of the unloading process is kept by the HFEF facility and is excluded from the PIE documentation. The ATR basket retrieval will be documented via visual examination throughout the hot cell window using a digital camera.

6.2.2 Neutron Radiography of the ATR Basket

Neutron radiography will be performed using the Neutron Radiography (NRAD) Reactor facility in HFEF. Neutron radiography of the basket will be performed and documented in consultation with the PI. The transmitted data will include neutron radiographs and loading order photographs. Neutron radiography will be provided for the basket at a minimum of one azimuthal orientation. The axial position is determined from a gadolinium treated scale (marking spacing 0.025 in. and 1 mm) incorporated in the utilized NRAD rodlet fixture. Test shots will be performed before neutron radiography to verify system operation and to ensure that test parameters result in acceptable image quality. The quality of the final images will be validated by certified personnel. Neutron radiography data will be digitized at high-resolution. Certification records for the scale bars used during radiography will be included in the data package.

6.2.3 Retrieval of ATR Fixtures from the ATR Basket

The ATR basket (see INL-DWG-822576) containing three ATR fixtures will be disassembled by a process engineer by following standard sectioning procedures. To ensure no visible surface defect, each ATR fixture will be visually inspected throughout the hot cell window using digital camera, and digital photos will be included in the PIE documentation.

Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: 11 of 19</div>
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6.2.4 Retrieval of LANL Capsules and Fluence Wires from the ATR Fixtures

Each ATR fixture will be cut open to retrieve LANL capsules and fluence wires by a process engineer by keeping the identifications of the ATR fixtures, LANL capsules, and fluence wires. The cutting methodology will be decided after the neutron radiography analysis of the ATR basket. Each LANL capsule will be visually inspected throughout the hot cell window using digital camera, and digital photos will be included in the PIE documentation. Each LANL capsule will be labeled and stored in an appropriate container for tracking. Fluence wires will be labeled and stored in a different container for transportation to the relevant facility.

Warning

Capsule 2B has a crack. Take extra care and precaution while retrieving capsule 2B.

6.2.5 Packaging and Transport of Fluence Wires to Relevant Institution

Fluence wires will be secured and packaged under the guidance of the process engineer for transportation to the relevant institution (Pacific Northwest National Laboratory). Transportation will be performed by following existing work controls of the HFEF and the relevant institution. Release and receipt of the fluence wires will be included in the PIE documentation.

6.2.6 Visual and Dimensional Inspections of LANL Capsules

Digital photographs of LANL capsules will be collected through visual examination machine (VEM). As needed, higher magnification pictures will also be captured at a minimum of three viewing angles (e.g., 0, 120, and 240 degrees) under the PI's supervision. Each capsule will be optically inspected using VEM at window 8M. Photographs will be collected from a minimum of three viewing angles (e.g., 0, 120, and 240 degrees), and the whole LANL capsule (2.5 in.) length will be captured. Each LANL capsule is to be subjected to the length and diameter measurement via caliper or micrometer.

6.2.7 Mass Measurement of LANL Capsules

LANL capsule mass will be measured using the balance at HFEF's hot cell. Measurements will be recorded and verified according to the procedure to be developed by the HFEF Process Engineer and PI. Calibration mass measurements will be performed before and after the specimen measurements.

6.2.8 Disposal of ATR Basket and Fixtures

The shipping basket may be deconned and returned to ATR for re-use. If the contamination levels on the shipping basket are excessive, the basket will be disposed of per Waste Generation Service (WGS) direction (likely low-level waste). The ATR basket will also be disposed of per WGS direction. The TZM capsules disposition is under review as these components may be retained for further PIE.

Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: 12 of 19</div>
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6.2.9 Transfer of LANL Capsules to the Appropriate Facility for Disassembly

LANL capsules will be packaged by following the HFEF work controls and procedures. Documentation for the transfer will be kept in the HFEF, and it is excluded from the PIE documentation. The package will be transferred to the appropriate facility for disassembling LANL capsules. The release and receipt notices in between facilities will be recorded in the PIE documentation.

6.2.10 Disassembly of the LANL Capsules

LANL capsules will be disassembled at the appropriate facility. For disassembling, a capsule holder jig is to be built. Capsule sectioning process is to be performed via wire-saw or low-speed saw depending on the availability. The sectioning process will be documented via digital photography using digital camera, and sectioning parameters will be recorded in the PIE documentation.

WARNING

After this point YH_x samples must be handled very carefully due to their brittle nature. Any jigs for handling may be designed and used.

6.2.11 Packaging of the Specimens and Melt Wires

Each YH_x, TZM sheet/ring, and melt wires will be removed from the sectioned LANL capsule. Each specimen will be secured in an appropriate container with a unique identification as shown in Appendix A. If extensive cracking or disintegration is observed, the NDE and DE activities will be re-arranged, noted, and included in the PIE documentation.

6.2.12 Optical Inspection and Dimensional Measurements of Specimens

Optical inspection of each sample (YH_x, TZM sheet/ring, and melt wires) will be performed to determine any observable surface defects using a calibrated optical microscope or digital camera. Optical inspections can also be used for the diameter measurements. Caliper or other dimensional inspection tools can be utilized to perform dimensional measurements. Calibration records will be included in the PIE documentation.

Melt wires will be inspected using an optical microscope under the guidance of an SME who will make a decision based on the optical micrographs of the melt wires. Relevant optical micrographs, rationale of the decision, and the decision will be included in the PIE documentation

Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: 13 of 19</div>
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6.2.13 Mass and Volume Measurements of YH_x and TZM Specimens

Mass of each YH_x and TZM sheet/ring specimens will be measured using a calibrated balance. Minimum five measurements for each sample will be performed. Calibration mass measurements will be performed before and after the specimen measurements. Volume of each YH_x and TZM sheet/ring specimen will be measured using a gas displacement pycnometer system. The density of the specimens will be calculated via: density = mass / volume by the performer or PI.

6.2.14 X-ray Diffraction (XRD) of YH_x and TZM Specimens

XRD patterns of selected YH_x and TZM samples will be collected at ambient temperature in an appropriate facility. XRD samples will be selected by the PI. At minimum, x-ray diffraction profiles will be indexed, and existing phases will be identified. Additional x-ray data analysis, such as Rietveld refinement and others, may be requested by the PI. X-ray samples may be prepared via polishing to fit into specimen holders.

6.2.15 Transfer of Selected Specimens to the External Institutions for Advanced PIE

Selected YH_x specimens are to be encapsulated, packaged, and transferred to the Los Alamos National Laboratory and Brookhaven National Laboratory for neutron and x-ray scattering studies to elucidate the effect of the radiation damage on the YH_x. Encapsulation and packaging of specimens will be performed under the guidance of the external institutions' work controls, facility requirements, and laboratory instructions. These activities are to be documented in the relevant facilities, and the release and receipt of the specimens will be included in the PIE documentation.

6.3 DESTRUCTIVE EXAMINATIONS**6.3.1 Hydrogen Content Measurements**

Hydrogen content of pre-specified YH_x and TZM samples will be measured using the Laboratory Equipment Corporation (LECO) system available. Due to the high hydrogen levels in YH_x in the samples and potential saturation of the instrument detector, the calibration and measurement processes will be studied to establish the testing procedure for YH_x. All procedures, surrogate specimen data, test parameters, and equipment will be reported in the PIE documentation for the developed technique. For TZM specimens, standard LECO measurements will be performed by following the facility procedures. The generated data and uncertainties will be reported in the PIE documentation.

6.3.2 Metallography and Electron Microscopy/Spectroscopy of YH_x and TZM Samples

Metallography and electron microscopy/spectroscopy of pre-determined YH_x and TZM specimens will be performed at appropriate facilities. Metallography samples will be prepared for optical microscopy, Scanning Electron Microscopy (SEM), and Energy Dispersive Microscopy (EDS) via metallographic mount preparation techniques. If necessary, specific mounts can be designed. Details of mount preparation, grinding, and polishing procedures will be included in the PIE documentation. Mounts will be compliant with the SEM holders for electron microscopy characterizations.

Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: 14 of 19</div>
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Optical microscopy of selected YH_x specimens will be taken at low and high magnifications ranging from 5X to 100X. Expected magnifications are 25X and 50X. If necessary, the PI can suggest additional magnifications. Full cross section mosaics will be compiled from macrographs. The montage will be performed from partially overlapping images. The amount of overlapping should be at least 15%. Average grain size, any imperfection, defect, second phase will be investigated and reported. If optical microscopy resources are not available at the time of the PIE, the PI can decide to exclude optical microscopy from the PIE.

Electron microscopy characterization of selected YH_x and TZM specimens will be performed using SEM and EDS. Secondary Electron (SE) and Backscattered Secondary Electron (BSE) images will be acquired as directed by the PI. At a specific low magnification (as decided by the PI), the sample cross-section pictures will be collected and montaged to show whole mount-sample cross-section. EDS data will be collected from the edge of the mount to the center of the mount. Magnifications for EDS data acquisition will be decided by the PI. In addition, electron backscatter diffraction (EBSD) pattern may be collected to determine the grain orientations.

Further microscopy characterization of YH_x specimens will be performed using transmission electron microscopy techniques, including analytical (S/TEM) or high-resolution (HR-TEM), precession electron diffraction (PED), to investigate the effect of the irradiation on the fine-microstructure. Relevant TEM technique(s) will be decided by the PI based on the SEM, EDS, and EBSD data. If necessary, atom probe tomography (APT) may be performed to capture specific features.

6.3.3 Mechanical Properties of YH_x Specimens

Mechanical properties of selected YH_x specimens will be examined using RUS and nano-indentation techniques. RUS will be performed to determine the isotropic elastic constants and orthotropic elastic constants of YH_x at ambient temperature. As guided by the PI, high-temperature RUS examinations can be performed at elevated temperatures (maximum temperature is 800°C).

Further mechanical property examination will be performed using nanoindentation techniques where hardness and elastic modulus of YH_x can be determined. For nanoindentation tests, cube corner indent tip is planned to be use for the determination of basic parameters and the fracture toughness. If cube corner indent is not available at the time of the PIE, other indent tip geometries are to be used such as Berkovich, conical, cylindrical, or spherical as decided by the PI. Nanoindentation tests will be performed at ambient temperature. If needed, the PI can request high temperature nanoindentation tests. Minimum six (6) indents will be applied to the surface of each specimen for statistics. After the nanoindentation tests, the SEM picture of the deformed surface of the specimen will also be recorded. Calibration specimens will be used for both RUS and nanoindentation examinations before and after actual specimen tests. The procedures, test parameters, and the generated data will be included in the PIE documentation.

Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: 15 of 19</div>
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6.3.4 Thermophysical Properties of YH_x

NOTE: *Because yttrium hydride (YH_x) is prone to oxidation and hydrogen release, crucibles must be painted or coated with yttria (YO₂) to avoid hydrogen exchange between crucible and the specimen.*

Thermophysical properties of selected irradiated YH_x specimens will be examined via push-rod dilatometry, DSC, and laser-flash analysis (LF) techniques to determine linear thermal expansion, thermal diffusivity, heat capacity, and any thermal recovery effects. The experimental procedures, parameters, and generated data shall be included in the PIE documentation.

6.3.4.1 Push-Rod Dilatometry

Push-rod dilatometry will be performed to determine the linear thermal expansion of YH_x as a function of temperature up to 800°C. If the PI requests, the maximum temperature can be increased up to 1200°C where hydrogen starts to leave YH_x. Push-rod dilatometry experiments can perform as heating-cooling cycles, high-temperature soak, or a combination of two treatments under the guidance of the PI. Heating rates, cooling rates, maximum temperature, soak time (if experiment has a high-temperature soak), and the number of heating-cooling cycles will be determined by the PI with the performer. The PI may also request microstructural characterizations if a significant effect is observed with the dilatometry experiments.

6.3.4.2 DSC Measurements

Differential-scanning calorimetry measurements of selected YH_x specimens will be performed to determine the heat capacity and any phase transformation. The maximum temperature for DSC measurements is 1200°C. Heating rates, cooling rates, maximum temperature, soak time (if experiment has a high-temperature soak), number of heating-cooling cycles will be determined by the PI with the performer. The PI may also request microstructural characterizations if a significant effect is observed with the dilatometry experiments.

6.3.4.3 LFA Measurements

Laser-flash analysis will be performed on selected YH_x specimens to determine the thermal diffusivity of YH_x as affected by neutron-irradiation. A minimum of two heating-cooling runs will be performed. The experiment parameters will be determined by the performer and the PI.

6.3.5 Hydrogen Transport Analysis

Selected YH_x specimens will be subjected to positron annihilation spectroscopy (PAS) and GDOES to investigate the hydrogen effect in the YH_x crystal and any potential re-distribution of hydrogen in the material.

Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-6268 Revision: 1 Effective Date: 03/04/22 <div style="text-align: right;">Page: 16 of 19</div>
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7. REFERENCES

1. “A Microreactor Program Plan for The Department of Energy,” INL/EXT-20-58191, Idaho National Laboratory, Idaho Falls, 2020.
2. J. B. Vetrano, “Hydrides as neutron moderator and reflector materials,” *Nuclear Engineering and Design*, 14(3) (1971): 390-412.
3. W. M. Mueller CHAPTER 2 Hydrides in Nuclear Reactor Applications, in: W.M. Mueller, J.P. Blackledge, G.G. Libowitz (Eds.), *Metal Hydrides*, Academic Press, 1968.
4. Technical and Functional Requirements. “LANL-MOD-1 Irradiation in the ATR NEFT Small B Positions, TFR-1049, Revision 1, Idaho National Laboratory, Idaho Falls.
5. “LANL-MOD-1 Experiment Data Package,” DP-157 Revision 2, Idaho National Laboratory, Idaho Falls, 2020.
6. ECAR-4820, “Thermal and Structural Safety Compliance Analysis for LANL-MOD-1 Experiment in the B-2 Position in ATR,” Idaho National Laboratory, Idaho Falls, 2020.
7. LWP-21220, “Work Management,” Idaho National Laboratory, Idaho Falls, 2020.

Idaho National Laboratory

**YTTRIUM HYDRIDE
POST-IRRADIATION EXAMINATION
PLAN**

Identifier: PLN-6268

Revision: 1

Effective Date: 03/04/22

Page: 17 of 19

Appendix A

Sample Identifications

MOD1A-ASSY						
MOD1A-BSK-T						
MOD1A-BSK-PL1						
MOD1A-BSK-PL2						
MOD1A-FIXT1						
MOD1A-FIXH1-1						
MOD1A-FIXH1-2						
MOD1A-FIXT2						
MOD1A-FIXH2-1						
MOD1A-FIXH2-2						
MOD1A-FIXT3						
MOD1A-FIXH3-1						
MOD1A-FIXH3-2						
MOD1A-CAN1-1	MOD1A-CAN1-2	MOD1A-CAN2-1	MOD1A-CAN2-2	MOD1A-CAN3-1	MOD1A-CAN3-2	
MOD1A-LID1-1-1	MOD1A-LID1-2-1	MOD1A-LID2-1-1	MOD1A-LID2-2-1	MOD1A-LID3-1-1	MOD1A-LID32-2-1	lid
MOD1A-LID1-1-2	MOD1A-LID1-2-2	MOD1A-LID2-1-2	MOD1A-LID2-2-2	MOD1A-LID32-1-2	MOD1A-LID3-2-2	lid
MOD1A-RUS1-1-1	MOD1A-RUS1-2-1	MOD1A-RUS2-1-1	MOD1A-RUS2-2-1	MOD1A-RUS3-1-1	MOD1A-RUS3-2-1	RUS sample
MOD1A-RUS1-1-2	MOD1A-RUS1-2-2	MOD1A-RUS2-1-2	MOD1A-RUS2-2-2	MOD1A-RUS3-1-2	MOD1A-RUS3-2-2	RUS sample
MOD1A-RUS1-1-3	MOD1A-RUS1-2-3	MOD1A-RUS2-1-3	MOD1A-RUS2-2-3	MOD1A-RUS3-1-3	MOD1A-RUS3-2-3	RUS sample
MOD1A-GDOES1-1-1	MOD1A-GDOES1-2-1	MOD1A-GDOES2-1-1	MOD1A-GDOES2-2-1	MOD1A-GDOES3-1-1	MOD1A-GDOES3-2-1	GDOES sample
MOD1A-GDOES1-1-2	MOD1A-GDOES1-2-2	MOD1A-GDOES2-1-2	MOD1A-GDOES2-2-2	MOD1A-GDOES3-1-2	MOD1A-GDOES3-2-2	GDOES sample
MOD1A-GDOES1-1-3	MOD1A-GDOES1-2-3	MOD1A-GDOES2-1-3	MOD1A-GDOES2-2-3	MOD1A-GDOES3-1-3	MOD1A-GDOES3-2-3	GDOES sample
MOD1A-GDOES1-1-4	MOD1A-GDOES1-2-4	MOD1A-GDOES2-1-4	MOD1A-GDOES2-2-4	MOD1A-GDOES3-1-4	MOD1A-GDOES3-2-4	GDOES sample
MOD1A-GDOES1-1-5	MOD1A-GDOES1-2-5	MOD1A-GDOES2-1-5	MOD1A-GDOES2-2-5	MOD1A-GDOES3-1-5	MOD1A-GDOES3-2-5	GDOES sample
MOD1A-TEM1-1-1	MOD1A-TEM1-2-1	MOD1A-TEM2-1-1	MOD1A-TEM2-2-1	MOD1A-TEM3-1-1	MOD1A-TEM3-2-1	TEM Sample
MOD1A-LFA1-1-1	MOD1A-LFA1-2-1	MOD1A-LFA2-1-1	MOD1A-LFA2-2-1	MOD1A-LFA3-1-1	MOD1A-LFA3-2-1	LFA sample
MOD1A-LFA1-1-2	MOD1A-LFA1-2-2	MOD1A-LFA2-1-2	MOD1A-LFA2-2-2	MOD1A-LFA3-1-2	MOD1A-LFA3-2-2	LFA sample
MOD1A-DSC1-1-1	MOD1A-DSC1-2-1	MOD1A-DSC2-1-1	MOD1A-DSC2-2-1	MOD1A-DSC3-1-1	MOD1A-DSC3-2-1	DSC sample
MOD1A-DSC1-1-2	MOD1A-DSC1-2-2	MOD1A-DSC2-1-2	MOD1A-DSC2-2-2	MOD1A-DSC3-1-2	MOD1A-DSC3-2-2	DSC sample
MOD1A-DSC1-1-3	MOD1A-DSC1-2-3	MOD1A-DSC2-1-3	MOD1A-DSC2-2-3	MOD1A-DSC3-1-3	MOD1A-DSC3-2-3	DSC sample
MOD1A-DSC1-1-4	MOD1A-DSC1-2-4	MOD1A-DSC2-1-4	MOD1A-DSC2-2-4	MOD1A-DSC3-1-4	MOD1A-DSC3-2-4	DSC sample
MOD1A-DSC1-1-5	MOD1A-DSC1-2-5	MOD1A-DSC2-1-5	MOD1A-DSC2-2-5	MOD1A-DSC3-1-5	MOD1A-DSC3-2-5	DSC sample
MOD1A-DSC1-1-6	MOD1A-DSC1-2-6	MOD1A-DSC2-1-6	MOD1A-DSC2-2-6	MOD1A-DSC3-1-6	MOD1A-DSC3-2-6	DSC sample
MOD-1A-SH-1-1-1	MOD-1A-SH-1-2-1	MOD-1A-SH-2-1-1	MOD-1A-SH-2-2-1	MOD-1A-SH-3-1-1	MOD-1A-SH-3-2-1	TZM sheet
MOD-1A-SH-1-1-2	MOD-1A-SH-1-2-2	MOD-1A-SH-2-1-2	MOD-1A-SH-2-2-2	MOD-1A-SH-3-1-2	MOD-1A-SH-3-2-2	TZM sheet
MOD-1A-SH-1-1-3	MOD-1A-SH-1-2-3	MOD-1A-SH-2-1-3	MOD-1A-SH-2-2-3	MOD-1A-SH-3-1-3	MOD-1A-SH-3-2-3	TZM sheet
MOD-1A-SH-1-1-4	MOD-1A-SH-1-2-4	MOD-1A-SH-2-1-4	MOD-1A-SH-2-2-4	MOD-1A-SH-3-1-4	MOD-1A-SH-3-2-4	TZM sheet
MOD-1A-SH-1-1-5	MOD-1A-SH-1-2-5	MOD-1A-SH-2-1-5	MOD-1A-SH-2-2-5	MOD-1A-SH-3-1-5	MOD-1A-SH-3-2-5	TZM sheet
MOD-1A-SH-1-1-6	MOD-1A-SH-1-2-6	MOD-1A-SH-2-1-6	MOD-1A-SH-2-2-6	MOD-1A-SH-3-1-6	MOD-1A-SH-3-2-6	TZM sheet
MOD-1A-CR-1-1-1	MOD-1A-CR-1-2-1	MOD-1A-CR-2-1-1	MOD-1A-CR-2-2-1	MOD-1A-CR-3-1-1	MOD-1A-CR-3-2-1	centering ring

**YTTRIUM HYDRIDE
POST-IRRADIATION EXAMINATION
PLAN**

Identifier: PLN-6268

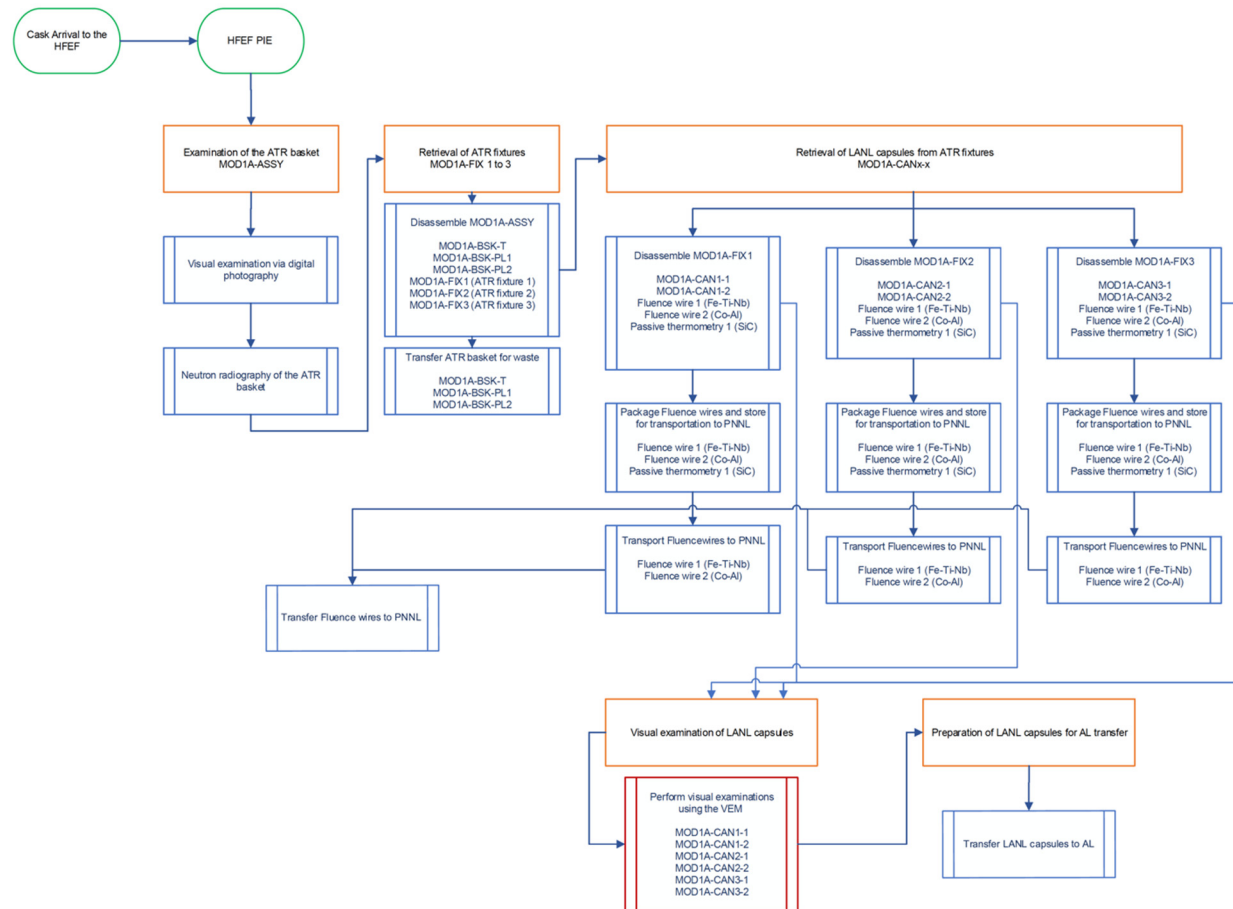
Revision: 1

Effective Date: 03/04/22

Page: 18 of 19

Appendix B

PIE Flow (HFEF)



Idaho National Laboratory

YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION PLAN

Identifier: PLN-6268

Revision: 1

Effective Date: 03/04/22

Page: 19 of 19

Appendix C

PIE Flow (AL, IMCL, STAR)

