Post-Irradiation Examination of Yttrium Hydride at Oak Ridge National Laboratory

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Annual Program Review Meeting – March 5, 2025

Outline

- Evolution of hydride irradiations
- Motivation: Why are we doing PIE on yttrium hydrides?
- Specimens and HFIR irradiations
- PIE plan
- PIE results
  - Optical examinations, dimensional change
  - Thermal property measurements and data analysis
  - XRD approach and preliminary results
  - Microstructure characterizations
- Conclusions

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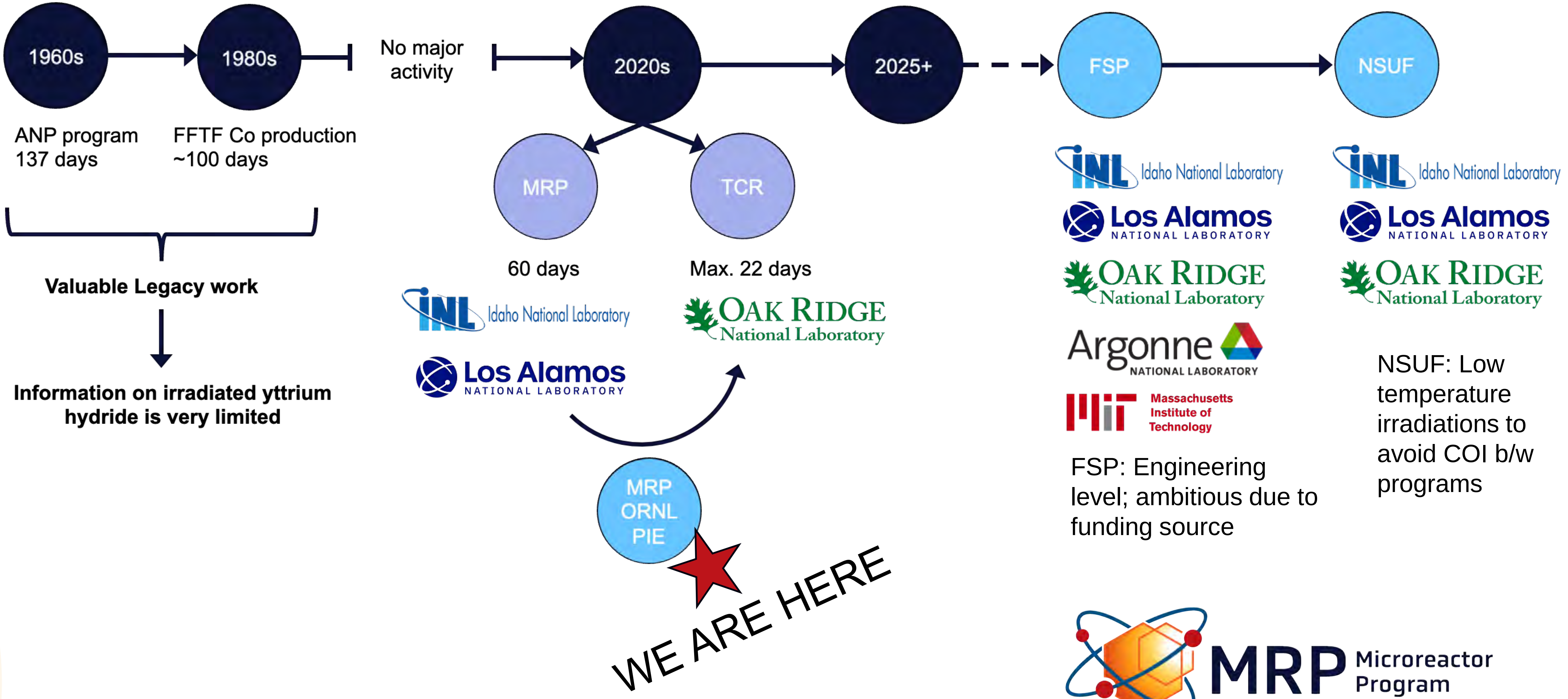
Major steps in yttrium hydrides irradiations and PIE



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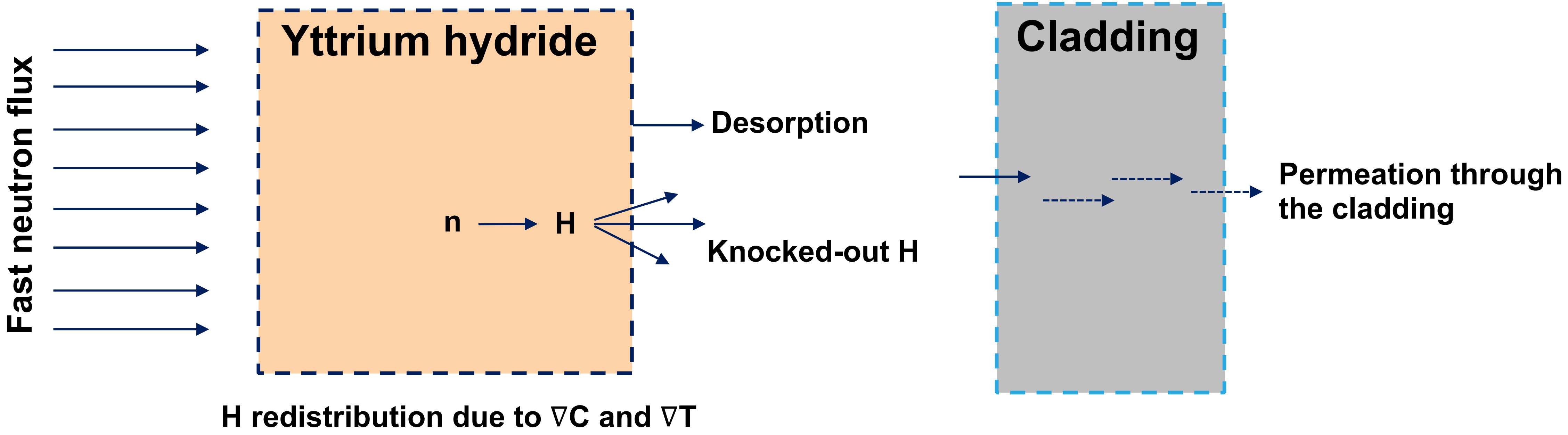
The Neutron Displacement Damage Can Assist Hydrogen Loss From Yttrium Hydride During Reactor Operation



The Phase Stability Of YH_x Needs To Be Addressed From The H Retention Aspect Of Hydride-Moderated Microreactors

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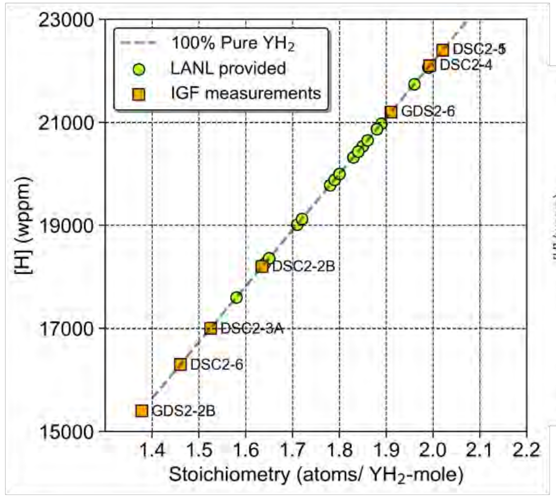
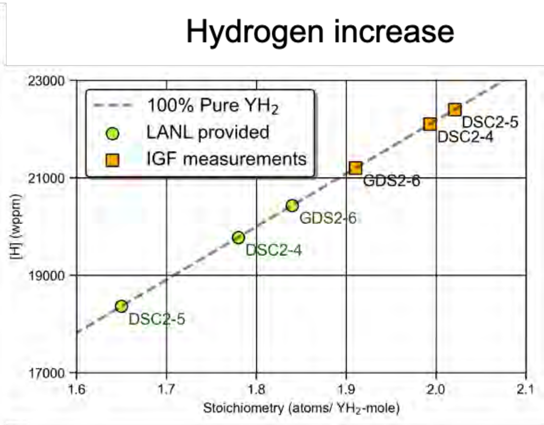
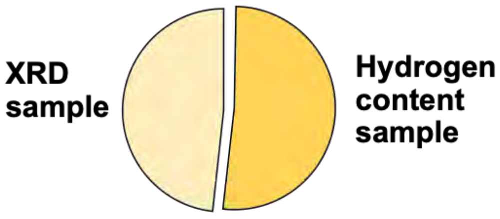
Major Takeaways From Microreactor PIE ?

Thermal property measurements indicated H redistribution in same capsule specimens





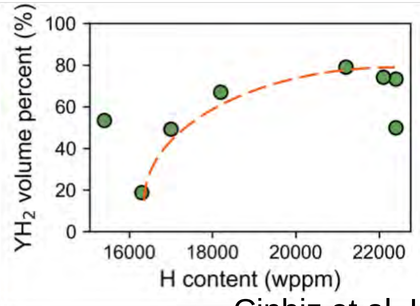
Laboratory XRD and IGF also confirmed H loss/gain



Rietveld refinement results

Post-Irradiation [H] (ppm)	XRD Volume fractions (%) YOF	Y_2O_3	Y	YH_2
18200	2.40	6.19	23.58	67.04
15400	0.86	21.52	23.97	53.41
22400	11.09	11.98	2.42	73.38
22100	0.13	16.41	9.28	74.18
17000	4.84	32.87	12.08	49.22
22400	0.89	3.40	45.81	49.89
16300	1.03	0.98	79.15	18.83
21200	3.40	2.09	14.65	79.08

XRD and IGF results show a systematic trend, except one specimen



XRD can be correlated to H content

Challenges: local hydrogen content variations, interaction volume of x-rays for the laboratory XRD

Cinbiz et al. INL report 2023

A Robust H Retention Metric For Irradiated Specimens Is Needed

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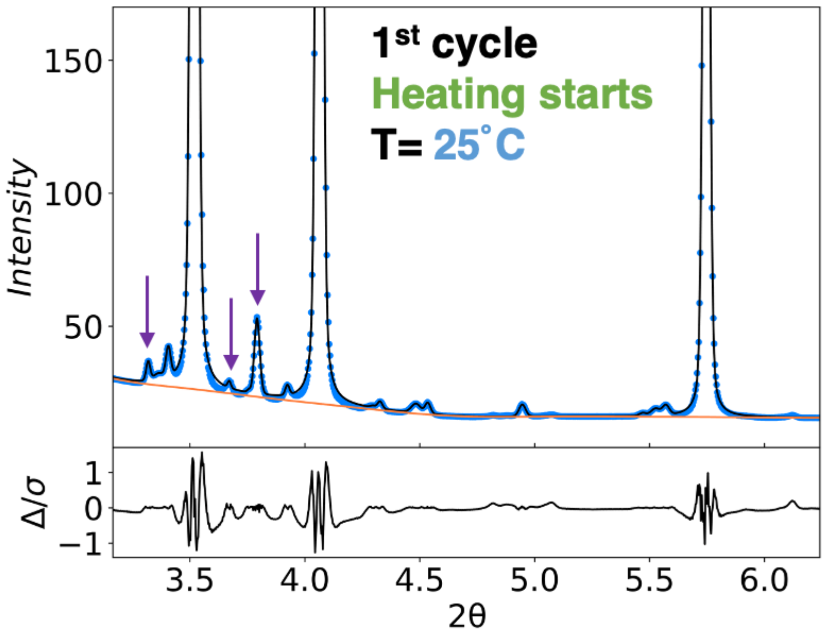
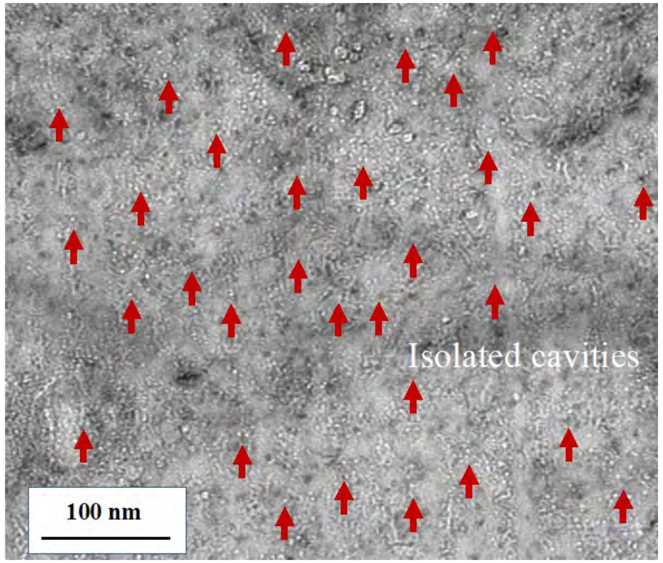
Major Takeaways From ORNL/HFIR Irradiations in 2023

- High energy X-rays indicated unexpected H recovery during heating of irradiated specimens
- Microstructure characterization indicated cavities in the matrix due to irradiation damage





Cinbiz et al., Materialia, 2023

Cavities or closed-pores are likely beneficial for H retention

Impact of Cavities on H Retention Needs To Be Determined

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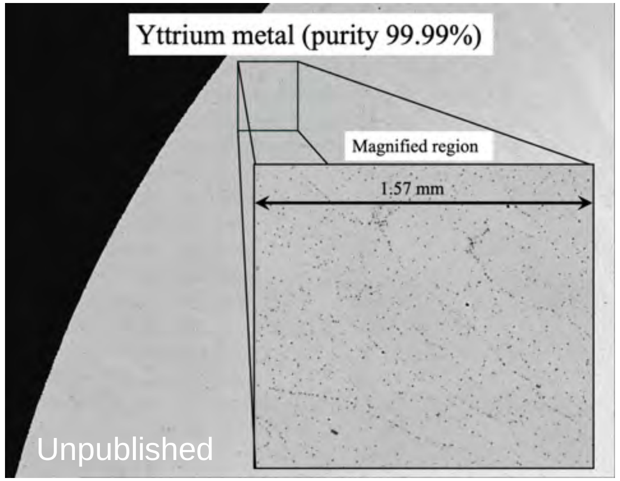
Based On What We Learned From Microreactor and ORNL Specimens PIE, Goal Is Redefined As

Can We determine a H retention/redistribution metric for irradiated moderator that supports material qualification and **reactor designers?**

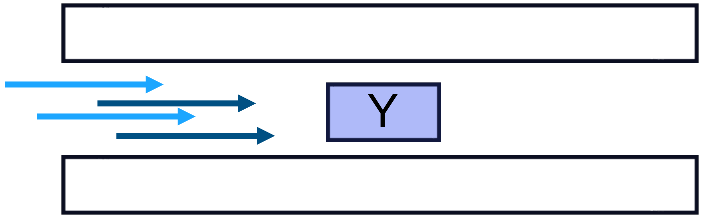
Materials: Yttrium Hydride Fabrication Was Performed Using Gaseous Diffusion Under Legacy TCR Program

Optical microstructure of a typical starting Y



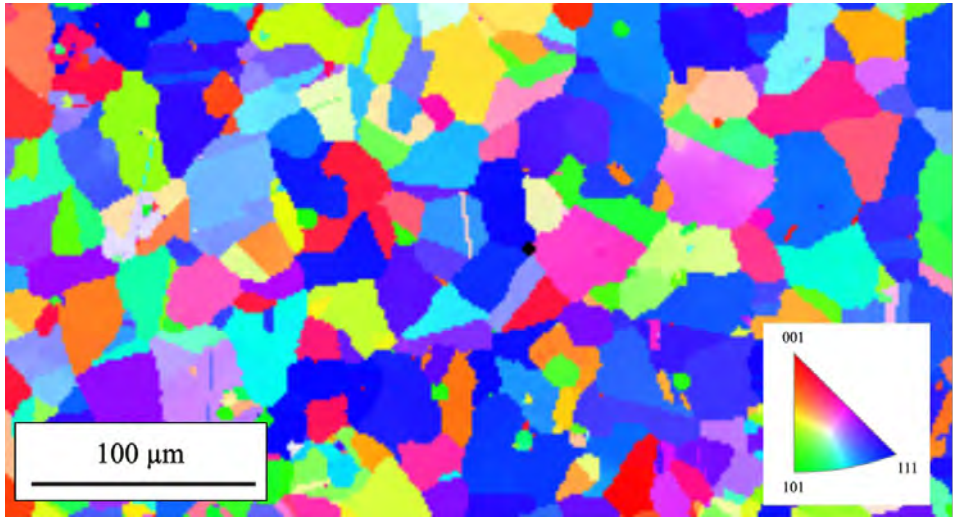
Starting Y (99.99% TREM) metal Main impurities were CaF_2 ,YOF, Y_2O_3

Flowing hydrogen gaseous diffusion (massive hydriding)



Pure H_2 flow

EBSD of $YH_{1.83}$



Cinbiz et al, Materialia, 2023

Starting material is like LANL specimens used in Microreactor irradiations

Hu et al, JNM, 2020

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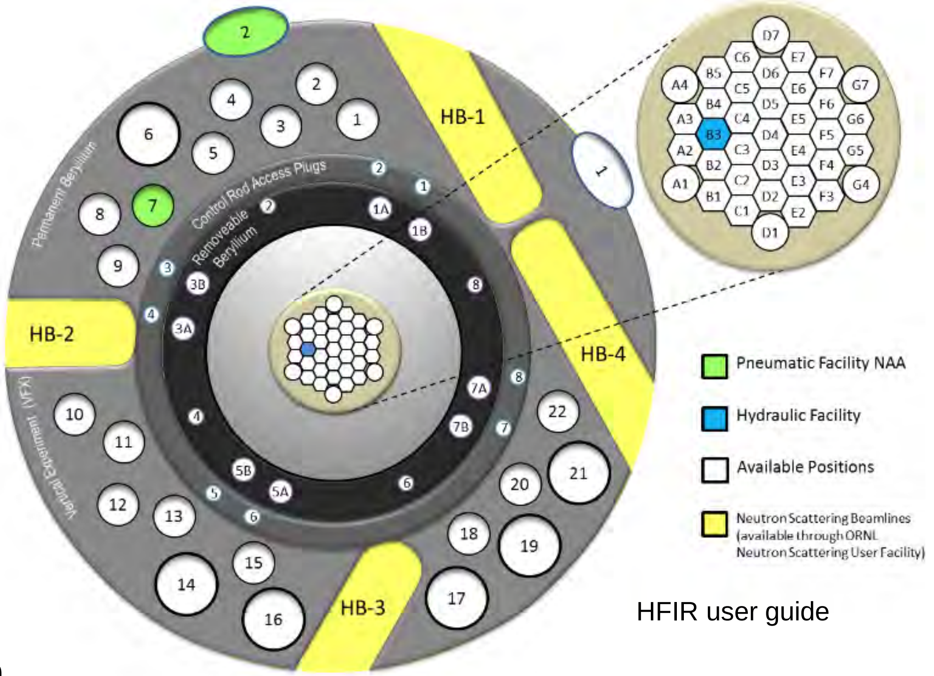
High Flux Isotope Reactor (HFIR) Is A Multi Role Reactor With Isotope Production, Materials Irradiation, And Neutron Science

- HFIR positions enable to accumulate significant dose in samples over a short duration
- 1 HFIR cycle ~ 24 days
- 5-7 cycles per year
- Constant power (85MW)
- Instrumented and drop-in (passive thermometry) irradiation capabilities



HFIR user guide

The ultimate advantage of HFIR is THE STEADY-STATE operation for successive reactor cycles

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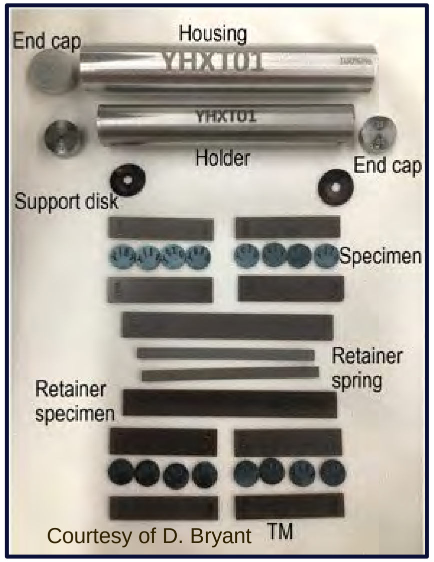
Yttrium Hydride Specimens Were Irradiated With A Drop-In Capsule With Passive SiC Thermometry

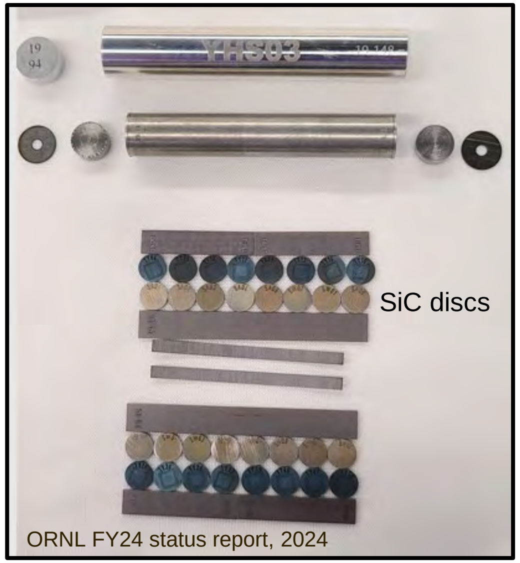
Capsule average parameters

Capsule	H/Y	dpa	T_{irr} (°C)	
YHXT			Calc.	Measured
1	1.69	0.1	596	595
2	1.69	0.5	600	626
13	1.69	2.0	602	663
4	1.69	0.1	895	847
5	1.69	0.9	897	794
14	1.69	1.7	901	655
7	1.83	0.1	596	616
8	1.83	0.9	600	640
9	1.83	2.0	602	536
10	1.83	0.1	894	878
11	1.83	0.8	896	677
12	1.83	2.0	903	672
YHS				
1	1.87	2.0	600	532
2	1.87	2.0	599	597

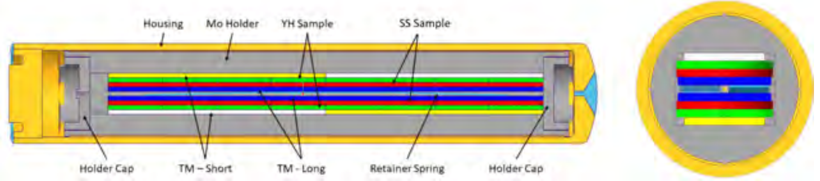
YHXT 1–12: 1st campaign

YHS 1–2: 2nd campaign





Each capsule holds 16 disc-shaped samples and 8 SiC thermometry (TM)



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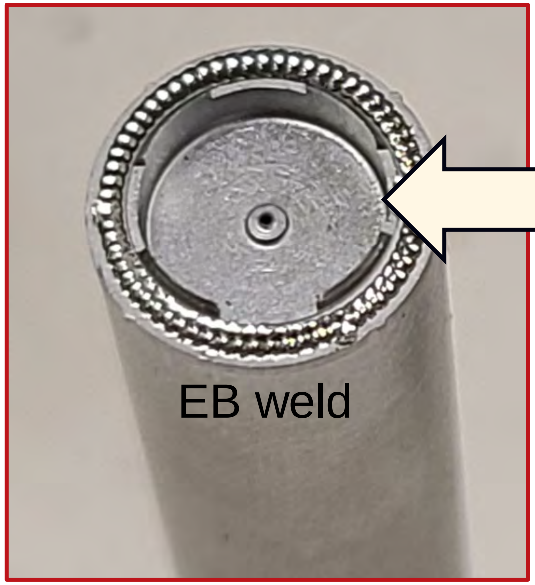
See P. A. Champlin et al., ORNL/TM-2019/1310, 2019 for details of HFIR capsules

Pure Molybdenum Tubing Was Used As Hydrogen Barrier During Irradiations



- Mo end caps welded on both sides of the Mo holder using EB welding
- Weld testing included bubble test, and He leak test

To mature hydride technology, cladding joining techniques need a dedicated R&D

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PIE Activities and Status

PIE Activity	Goal	Comments	Status
Inventory and basic visual examination	Observe structural integrity	Completed	
Dimensional inspection	Determine dimensional stability	Completed	37 specimens were examined
SiC TM analysis	Determine irradiation temperature	50% completed; decision making	4 TMs were tested
Specific heat capacity	Determine transition temperature for H retention metric		On-going; 6 specimens were tested; 5 remaining*
Thermal diffusivity	Same as specific heat	Merged with DSC due to shared goal	
Hardness	Structural integrity		
X-ray diffraction	Determine the local phase fractions in a sample	BNL tests on March 10th	6 specimens were tested; 10 remaining
Microstructure characterizations	Determine cavity microstructure and their density for H gain/loss behavior		3 samples are completed; 2 remaining

In total, 40 irradiated specimens will undergo PIE

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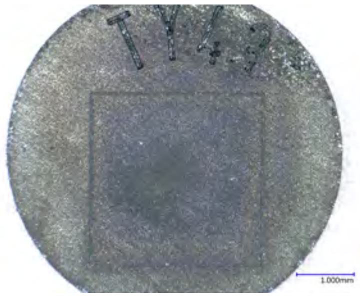
Visual Examination Of specimens

YHS01	Intact	Broken	Intact	Intact	Intact	Intact	Intact	Broken
	Chipped	Chipped	Intact	Intact	Intact	Intact	Intact	Intact
YHS03	Intact	Intact	Intact	Intact	Intact	Intact	Intact	Intact
	Intact	Intact	Intact	Intact	Intact	Intact	Intact	Intact

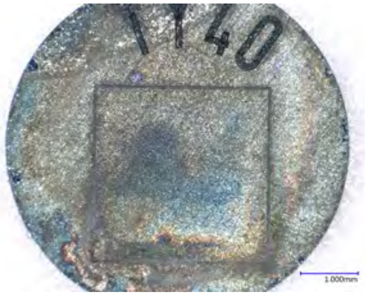
Specimens were laser marked to determine dimensional change

TY43

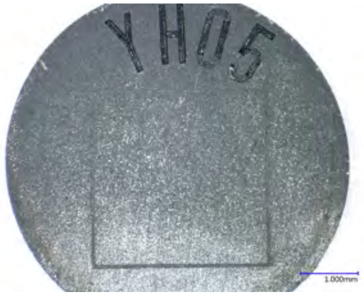


TY62

TY40



YH05



Similar Surface Features Were Present As Observed In MRP PIE

Samples characterized from the 1st campaign

IDs	H/Y	Condition
M004	1.83	Intact
M007	1.83	Intact
M100	1.83	Intact
M011	1.83	Intact
M013	1.83	Intact
M081	1.83	Intact
M088	1.83	Intact
M052	1.83	Intact

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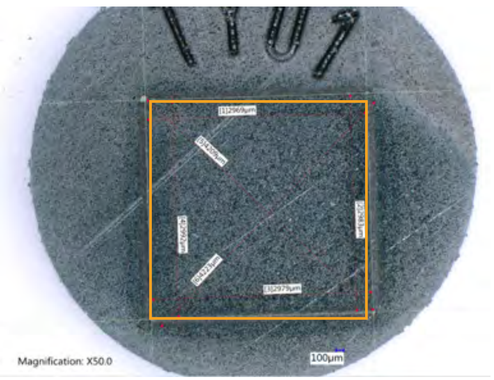
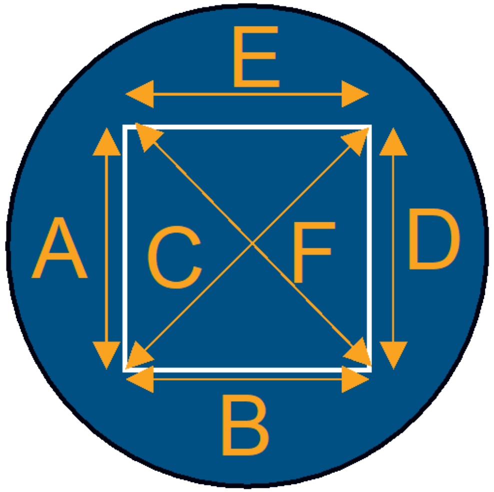
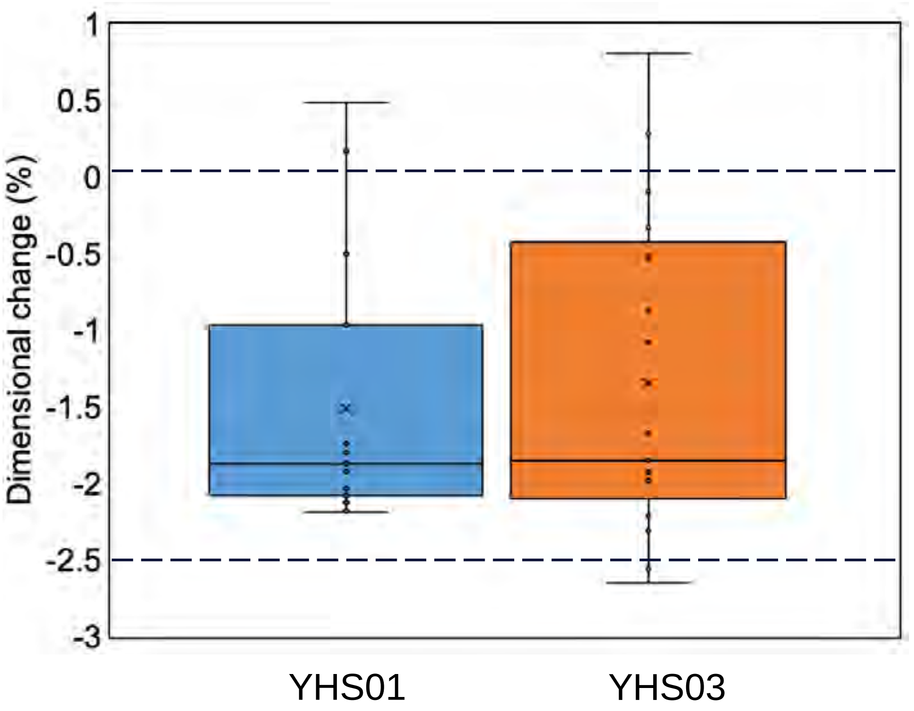
Dimensional Analysis of Specimens





Average dimensional change (A+B+C+D+E+F)



Overall Specimens Tend To Shrink → Density Increase → It Is Not Directly Related To H Retention → MRP PIE Results

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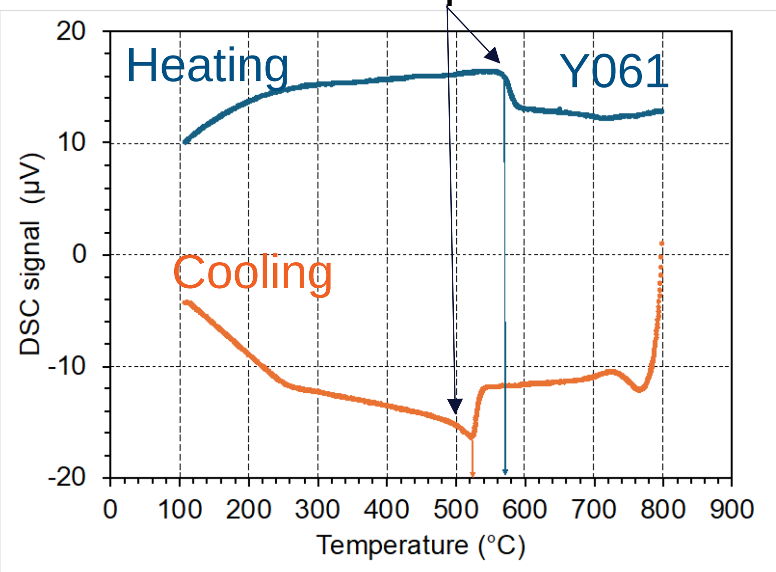
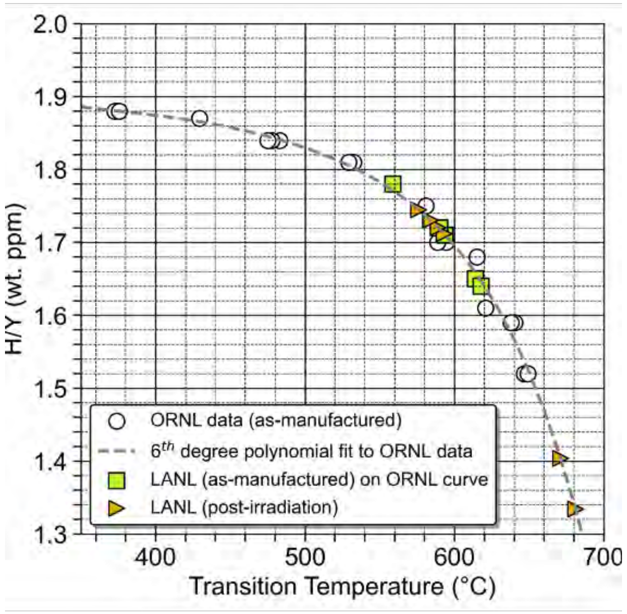
Differential Scanning Calorimetry Results

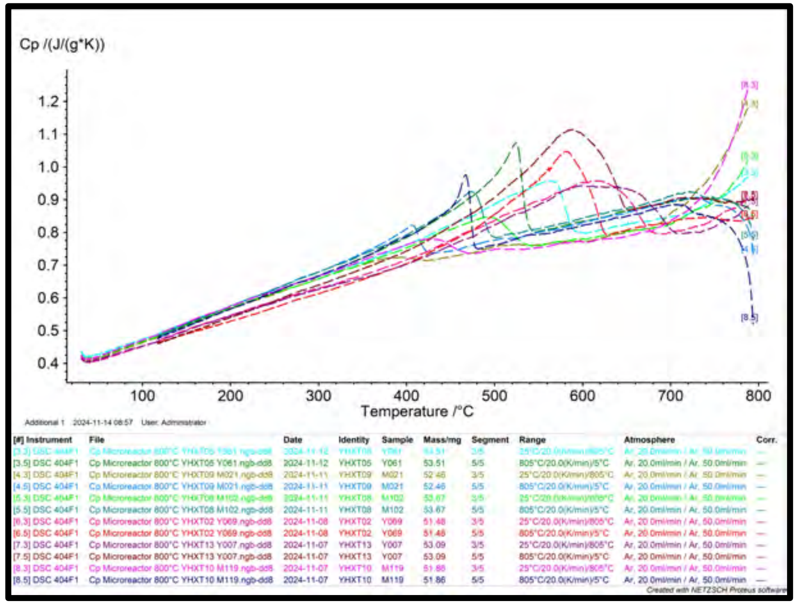
We will determine the H/Y ratio after irradiation and heat treatment to determine the amount of recovery

Transition temperature





Raw data example on irradiated specimens



There is ΔT between heating and cooling transition temperatures which is directly related to H/Y ratio, thus H retention

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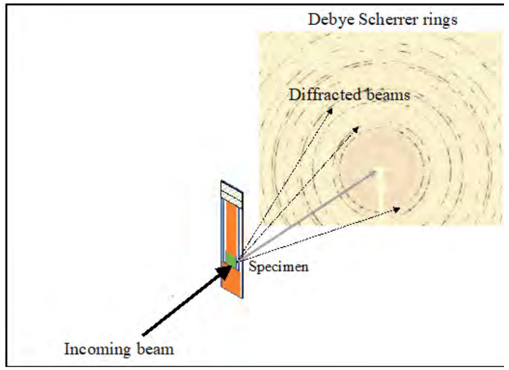
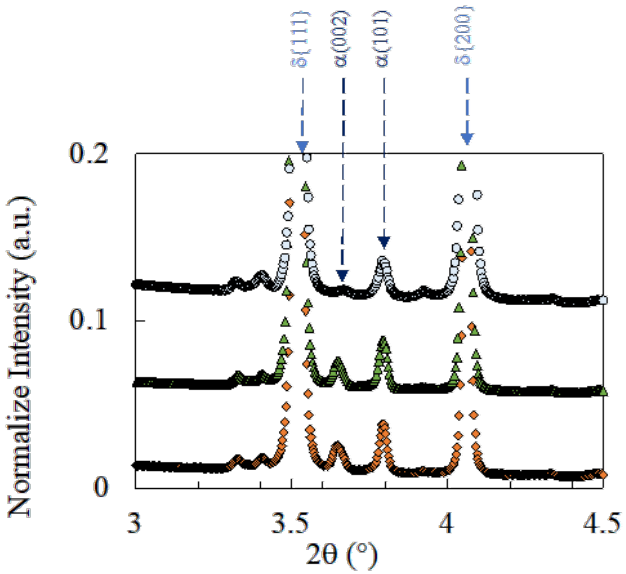
High Energy X-ray Diffraction in Transmission Mode

High-energy XRD

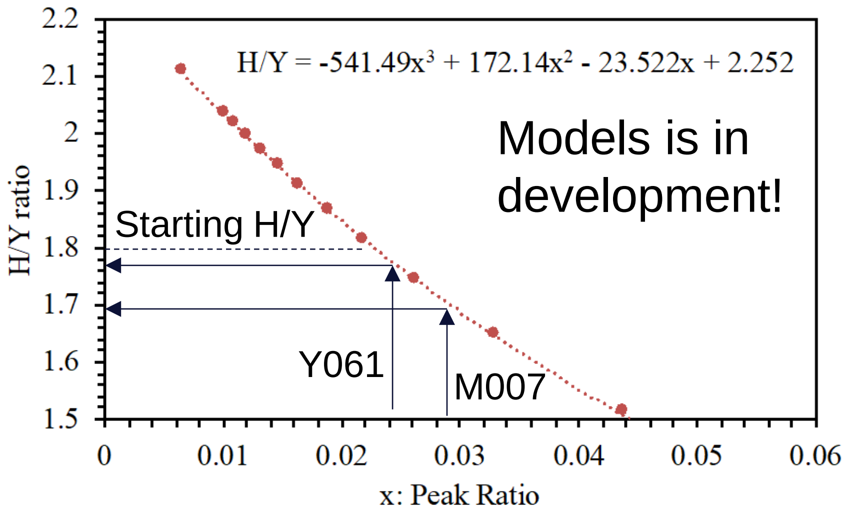


February 7: BNL also performed base XRD collection on samples with different H/Y ratios

Increase in αY peaks is related to H loss or trapping in cavities



We are working on developing a correlation b/w hydride and yttrium peak intensities



Data Analysis Of Tested Specimens Is Ongoing Cavity Contribution Needs To Be Understood

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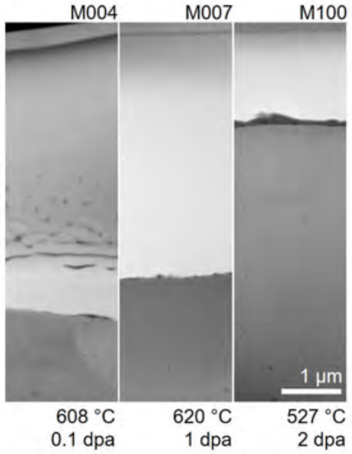
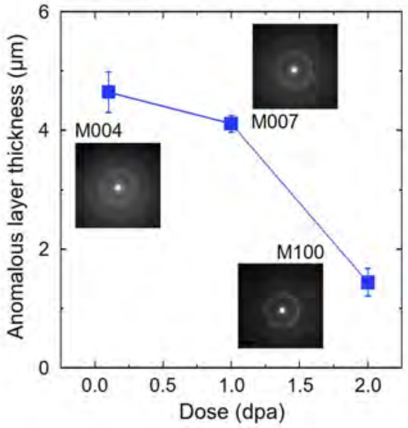
Microstructure Have More Impact On H Retention Behavior

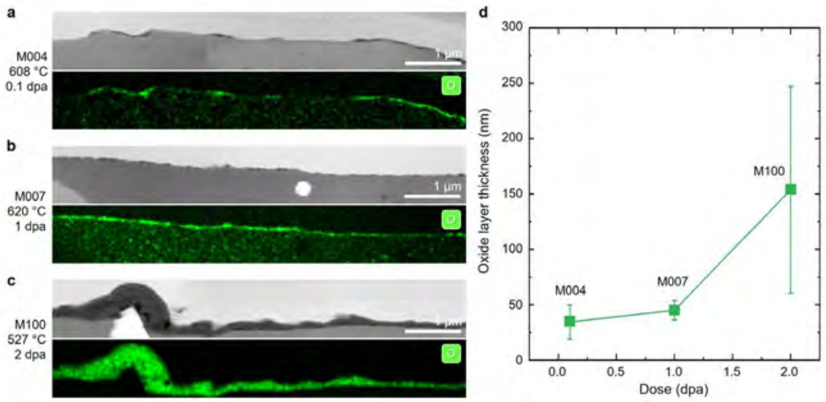
Amorphous region with Ca-rich



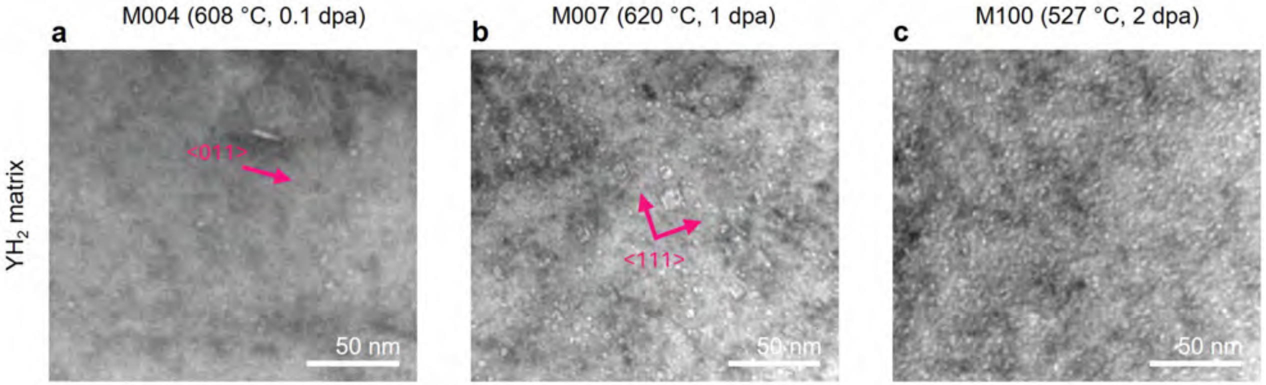
As dose increase amorphous region gets thinner



Continuous oxide formation with increasing dose



As dose increase cavity density increases while cavity size is intact



Hydride's H retention may be improved by impurity traps

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Milestone

Title	Description	Progress/Due Date	Challenges
M2AT-25OR0804091 - Complete full Post Irradiation Examination of HFIR irradiated YHx specimens to fill irradiation performance knowledge gaps	**Level 2 Milestone** (ORNL Technical Manuscript) - YHx material properties under combined effects of high flux irradiation targeting ~2dpa at target test temperatures of 600C.	On Track - September 26, 2025	Nothing to report

Everything is on-going as planned!

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Conclusions

- Confirmed that dimensional measurements (MRP ORNL PIE), volume, and mass (MRP PIE + TCR PIE) are misleading to assess H/Y ratio of irradiated specimens due to microstructure evolution
- Microstructure characterization indicated an impact of radiation damage on amorphous region, oxide development, and cavity density; which can act as H retention relevant features
- DSC method is also a strong technique to determine the transition temperature and H/Y ratio
- Transmission mode XRD is a very strong tool to determine the phase fractions of hydride and yttrium metal, but XRD does not see H in cavities

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Acknowledgements

- **Diane Li, John Jackson, and Holly Trellue** for their kind funding support
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- **Shaileyee Bhattacharya and Yan-Ru Lin** (ORNL) for XRD data analysis and TEM characterizations
- **David Sprouster** (BNL) for XRD

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