

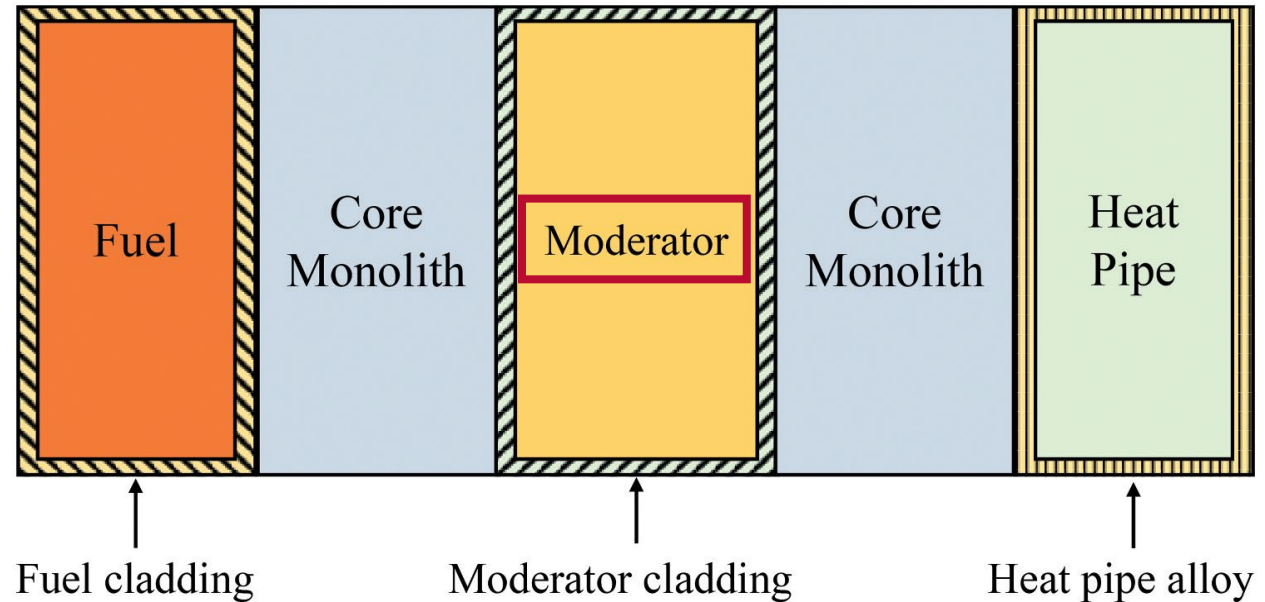
DOE-NE Microreactor Program Review

*(Project 23-29784) Deciphering Irradiation Effects of YHx Through In-situ
Evaluation and Micromechanics for Microreactor Applications*

Eric Lang – UNM

Microreactors

- Microreactor: $<30 \text{ MW}_{\text{th}}$ power and $>600 \text{ }^\circ\text{C}$ temperatures
- Hydrogen: attractive neutron moderator
- Metal hydrides: dense H concentration and high temperature stability



Schematic of microreactor core. This work focuses on the moderator

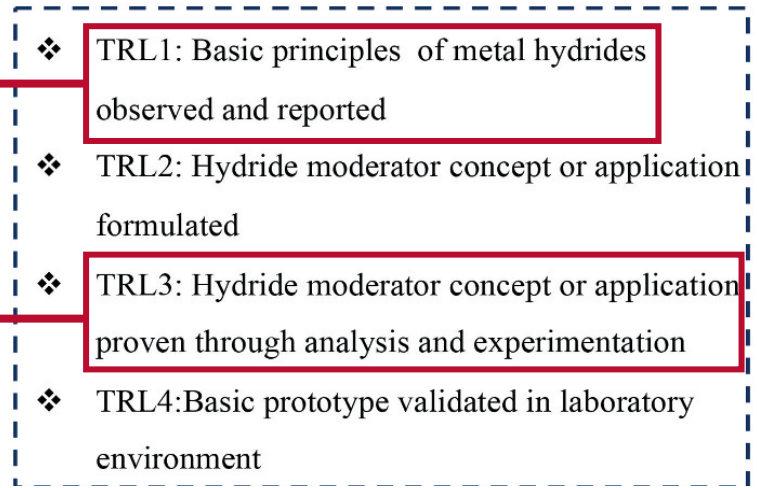
M. Nedim Cinbiz, Chase N. Taylor, Erik Luther, Holly Trellue & John Jackson
(2023) Considerations for Hydride Moderator Readiness in Microreactors, Nuclear Technology, 209:sup1, S136-S145, DOI: 10.1080/00295450.2022.2121583

Metal Hydrides for Moderator

- Base properties of hydrides starting to be re-investigated as fabrication re-starts

- Structure, phases, thermo-mechanical properties as a function of H content and fabrication route

- Performance of YH in thermal and irradiation environments



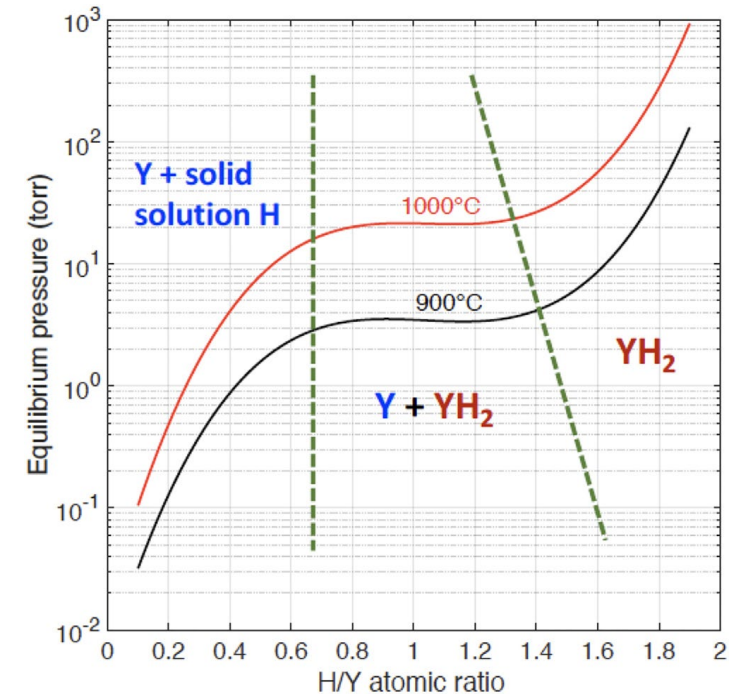
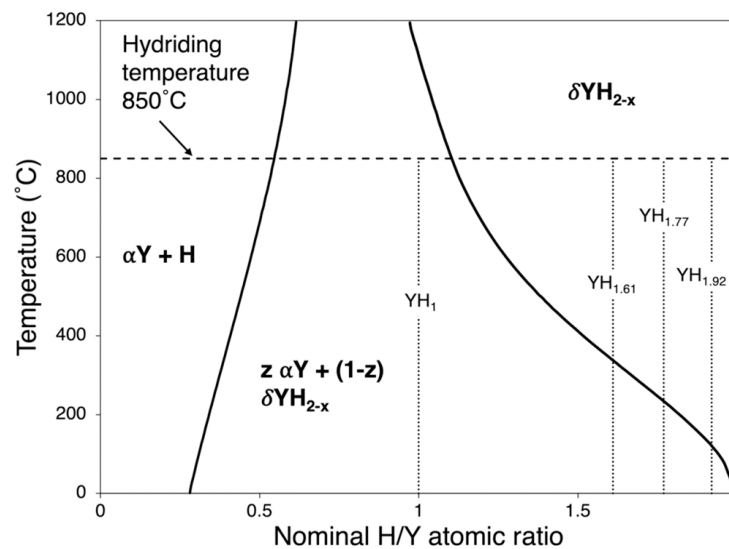
Experimental infrastructure is needed to assess hydrogen redistribution in condensed matter

M. Nedim Cinbiz, Chase N. Taylor, Erik Luther, Holly Trelue & John Jackson (2023) Considerations for Hydride Moderator Readiness in Microreactors, Nuclear Technology, 209:sup1, S136-S145, DOI: 10.1080/00295450.2022.2121583

Why Yttrium Hydride?

- H incorporated into the Y lattice, forms FCC delta phase
- Desirable to operate within this phase
- However, over irradiation or thermal environments, the phase may change

What is the effect of changing from single to mixed phase or vice-versa in a reactor environment?



Open questions

- What is the effect of ion irradiation on the YH microstructure and phase stability at elevated temperature?
- How do radiation-induced defects, such as voids and dislocation loops, affect mechanical properties?
- Does H gas exposure (in addition to incorporation in the lattice) change the material response?

Goals, Objectives, and Team

- **Goal:** Evaluate the high temperature irradiation effects on YH_x to decipher phase stability, H retention, and corresponding impact on mechanical properties through ion irradiation and micro-mechanical testing to help determine the efficacy of YH_x as a microreactor moderator material.
- **Team:**
 - **PI:** Eric Lang, University of New Mexico
 - **Co-PI:** Khalid Hattar, University of Tennessee-Knoxville
 - **Co-PI:** Adi Shivprasad, Los Alamos National Laboratory
 - **Co-PI:** Caitlin Kohnert, Los Alamos National Laboratory
 - **Two** UNM graduate students
 - **One** UTK graduate student

Goals, Objectives, and Team

The overall objectives of this project are to:

- Identify the effects of ion irradiation on the YH microstructure and phase stability at elevated temperature
 - i.e. H decoration surrounding voids
- Understand how radiation-induced defects, such as voids and dislocation loops, affect mechanical properties
- Establish a facility for further ion irradiation studies of YH and nuclear materials under gas exposure
 - i.e. limit loss of H under vacuum ion irradiation

Path and progress

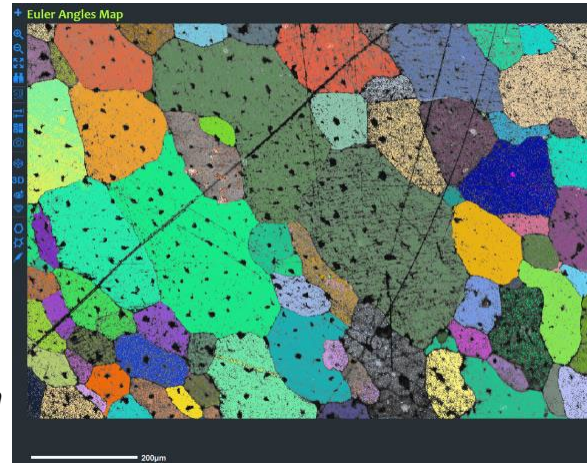
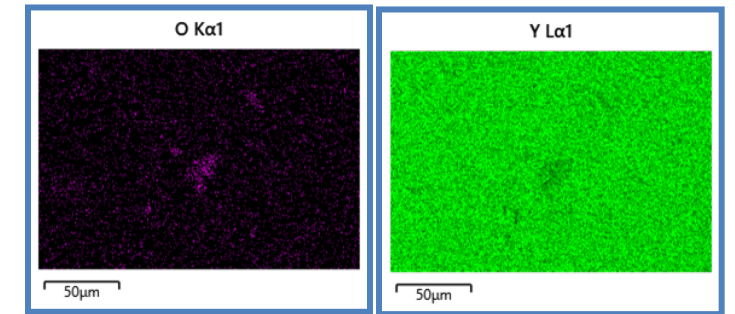
- Thrust 1: Establish a baseline understanding of YH fabricated properties vs. H content (Primary work: Q1-Q5)
- Thrust 2: Probe radiation damage effects with reactor-relevant doses of irradiation (Primary work: Q2-Q8)
- Thrust 3: Structure-property relationships of ion irradiated YH materials (Primary work: Q4-Q12)
- Thrust 4: Decipher real-time YH dynamics and interplays with structural materials (Primary work: Q8-12)

Path and progress

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Fabrication of YH Specimens: Thrust 1

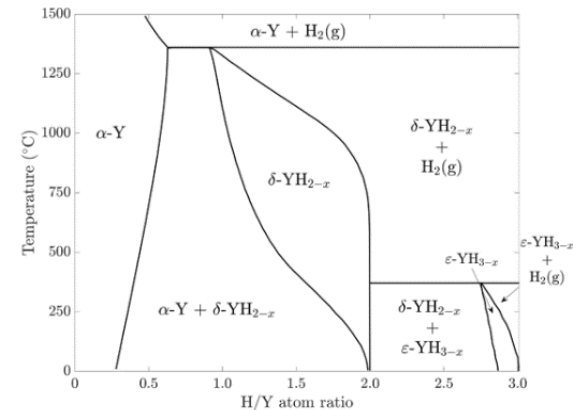
- Small oxide impurities observed in all samples
- No macroscopic CaF_2 impurities observed
- Polycrystallinity observed
 - FCC delta phase



SEM-EBSD map of grain orientation showing large micron-sized grains

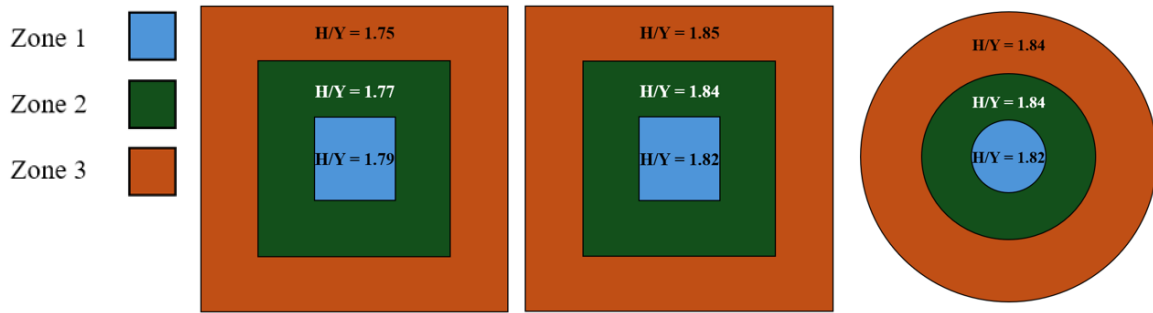
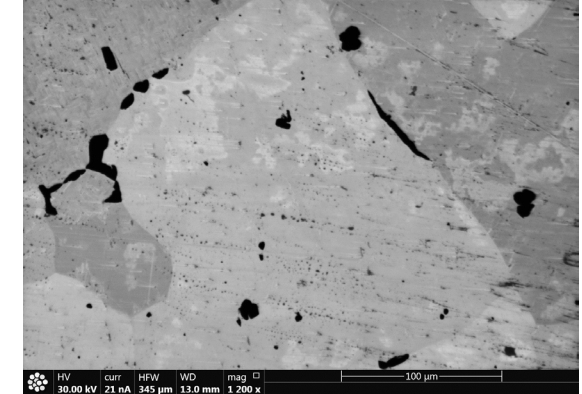
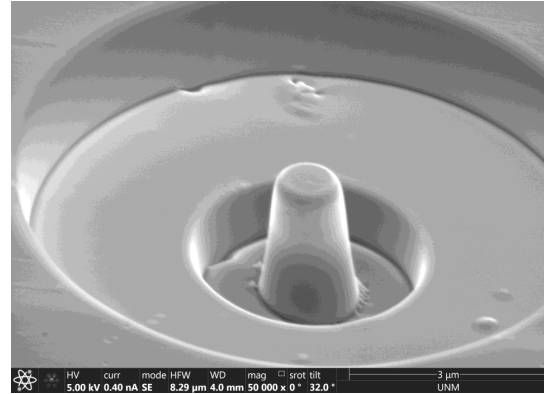


SEM-EDS map of YH1.9 specimen showing elemental map, showing small (~10 μm) yttrium oxide particles in the YH matrix.

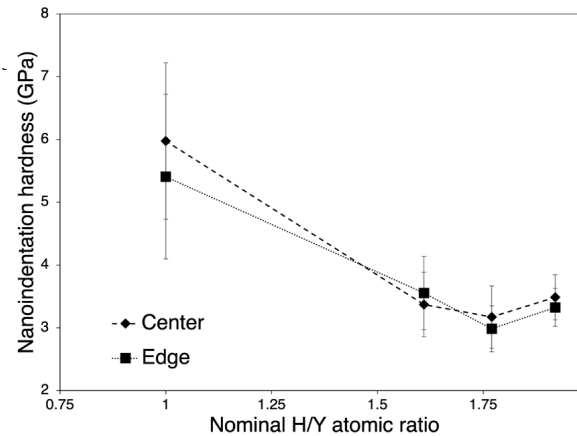


Nanomechanical properties

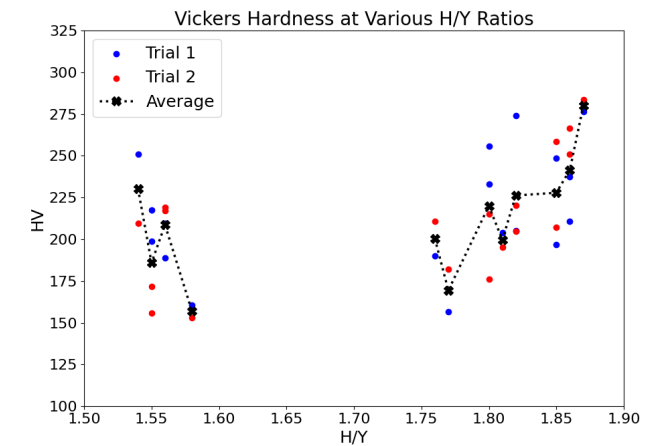
- Samples well-polished enough for single-grain identification -> crucial for single-crystal micropillars
- Hardness tracking vs. H/Y ratios



Hardness values correlating to estimated H/Y ratios across surfaces of fabricated samples - non-uniform stoichiometry throughout



H. Gietl, et al. Journal of the European Ceramic Society 43 (2023) 3216–3227



Path and progress

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Irradiation of YH Specimens

- In-vacuum ion irradiation at partner UTK

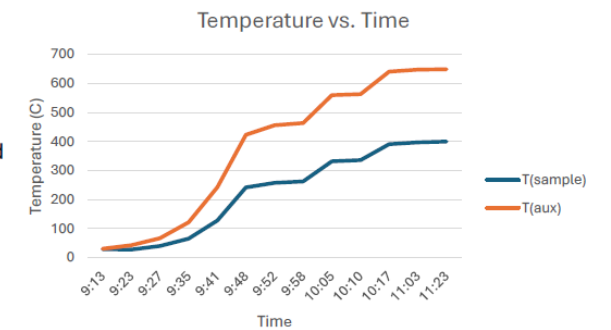
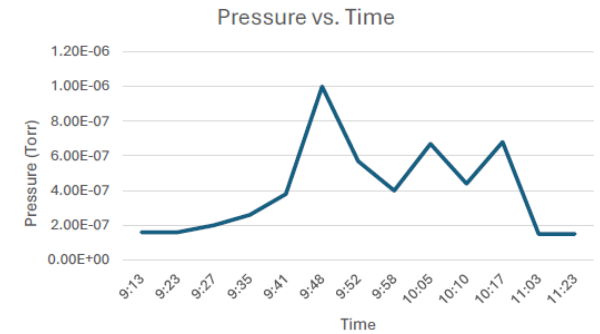
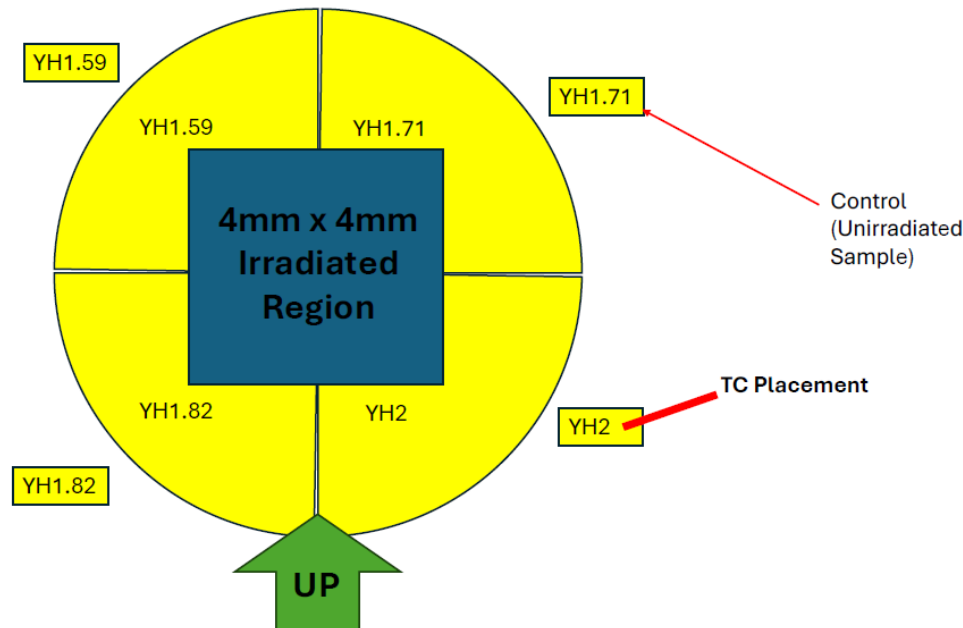
Systematic study of starting
H content

YHx: x=1.6, 1.8, 2.0

1-2 dpa

Au^{3+} : 9 MeV, 400C

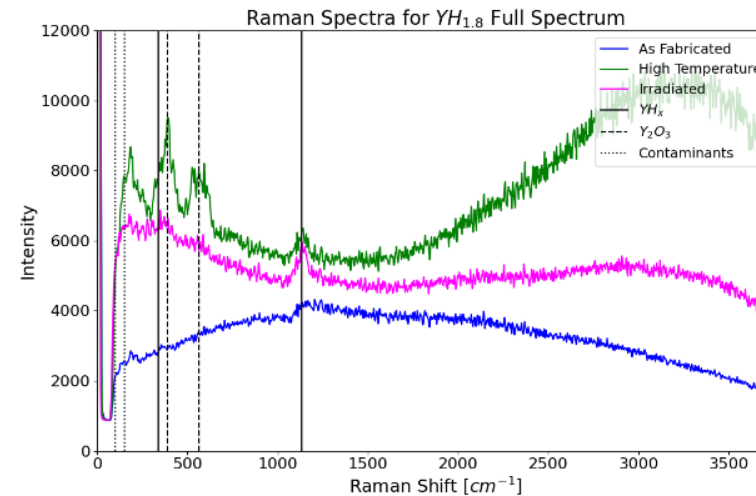
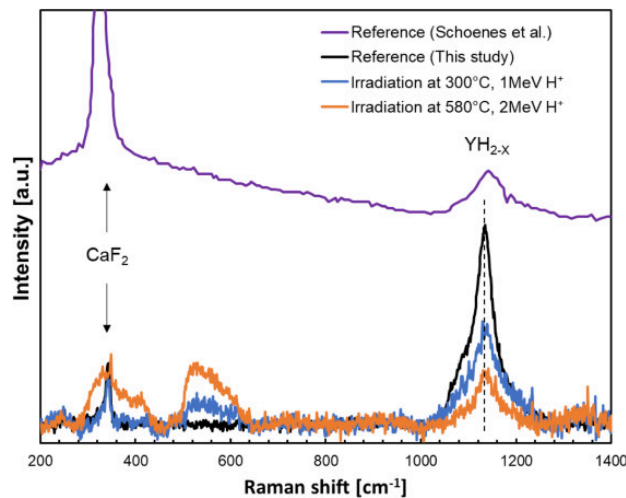
$$\phi = 2.33 * 10^{15} \frac{\text{ions}}{\text{cm}^2}$$



Some outgassing
observed during
temperature ramp, some
H loss expected

Ion irradiation experiments

- Analysis focusing on non-destructive evaluation
- Raman Spectroscopy
 - Used previously for NDE of YH -> identification of phases and correlation with H content

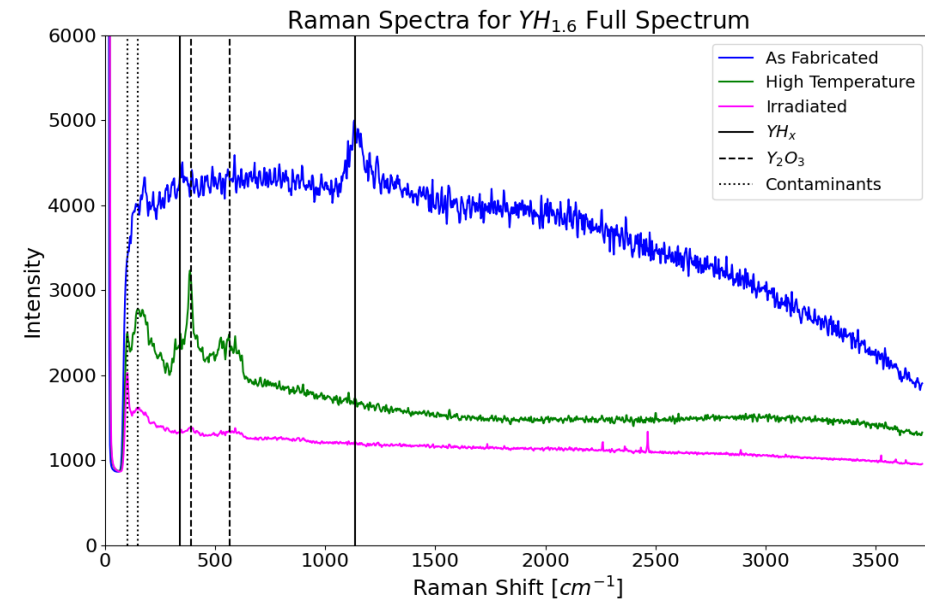
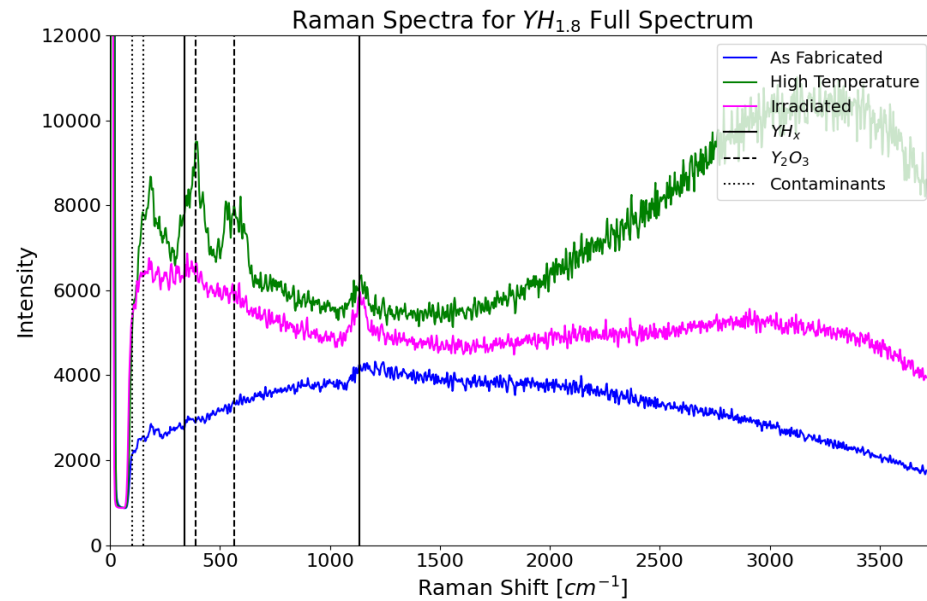


Our data on similar composition sample

Raman spectra of ion irradiated YH
[Taller, et al. 2025]

Ion irradiation experiments

- Raman Spectroscopy
 - Differences in sample composition vs. irradiation
 - YH1.6 exhibits increased H loss under exposure conditions vs. YH2 or YH1.8



Ion irradiation experiments

- GIXRD
 - Probing near-surface phase evolution
 - Loss of YH phase after high temperature
 - Simultaneous irradiation has convoluted impact on YH content

Stoichiometry	As-Fabricated	High Temperature	Irradiated
YH _{1.6}	3.59 ± 4.52 GPa (5 indents)	1.55 ± 1.18 GPa (22 indents)	2.51 ± 1.27 GPa (8 indents)
YH _{1.8}	7.27 ± 0.62 GPa (9 indents)	0.39 ± 0.65 GPa (8 indents)	2.04 ± 1.59 GPa (5 indents)
YH ₂	5.97 ± 2.77 GPa (24 indents)	2.89 ± 1.18 GPa (6 indents)	0.62 ± 0.34 GPa (7 indents)

Stoichiometry	As-Fabricated	High Temperature	Irradiated
YH _{1.6}	Y: 5.6% ± 0.7 YH ₂ : 94.4% ± 9.72	Y: 81.8% ± 5.6 YH ₂ : 18.2% ± 1.8	Y: 90.2% ± 6.4 YH ₂ : 9.8% ± 1.6
YH _{1.8}	Y: ~ 0% YH ₂ : > 99.9%	Y: 42.9% ± 5.7 YH ₂ : 57.1% ± 6.2	Y: 25.6% ± 1.7 YH ₂ : 74.4% ± 4.8
YH ₂	Y: ~ 0% YH ₂ : > 99.9%	Y: 41.4% ± 3.6 YH ₂ : 58.6% ± 6.3	Y: 94.5% ± 8.7 YH ₂ : 5.5% ± 0.5

Connection between H content in GIXRD and nanoindentation hardness in different irradiation states

Future Property Evaluation

- Currently, a Transient Grating Spectroscopy system is being constructed at TIBML for *ex-situ* and *in-situ* characterization on YH samples.
- Changes in thermal conductivity due to irradiation may change H diffusion during irradiation, thus affecting moderating power
- TEM analysis at UNM on-going to decipher void formation in irradiated specimens



Fig. 3

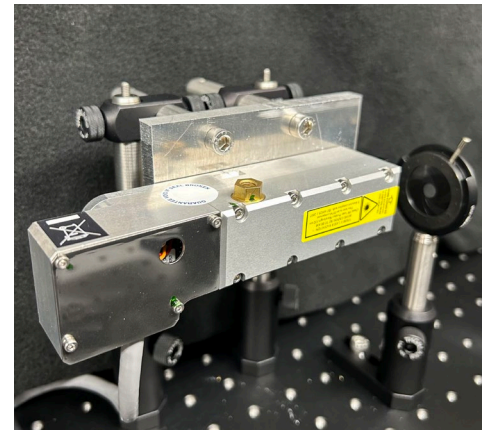


Fig. 4



Fig. 5

Path and progress

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Nanoindentation of irradiated YH

- Set of irradiated YH_x samples (x=1.6, 1.8, 2.0), 1-2 dpa, 400 C
- Higher H content correlates with higher indentation hardness
- High temperature exposure causes drop in hardness
 - H desorption?
- High temperature irradiation causes mixed effects

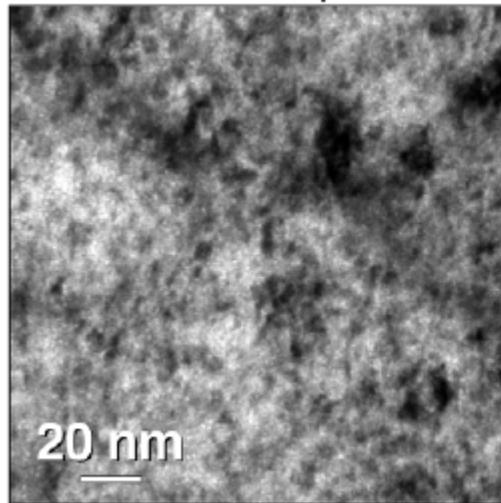
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Thrust 3 progress

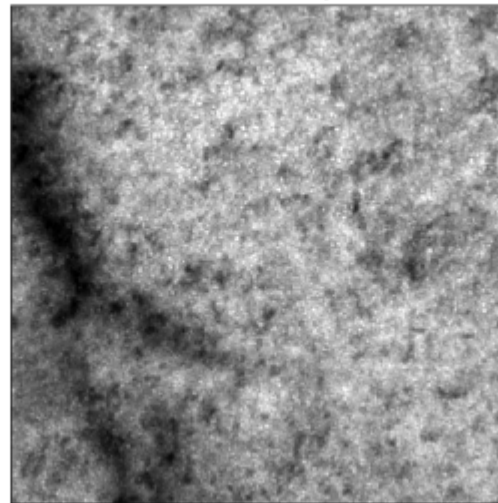
- Old ion irradiated specimens shipped from LANL to UNM for further characterization (APT specimen fabrication and micro/nano-cantilever fabrication)
- However, irradiated region only about 200 nm deep -> too thin for micropillar compression

Previously irradiated YH specimens with 9 MeV Au at 700 C

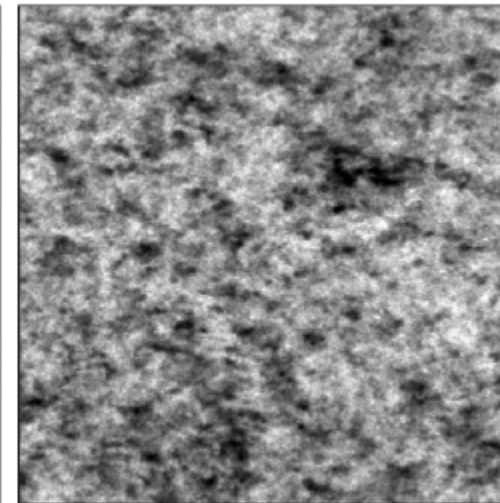
Surface ~ 1 dpa



Peak ~ 2 dpa

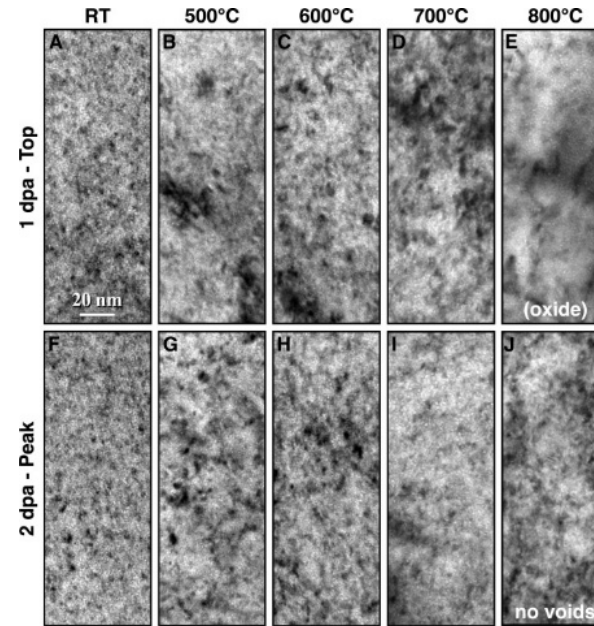


Back ~ 0 dpa

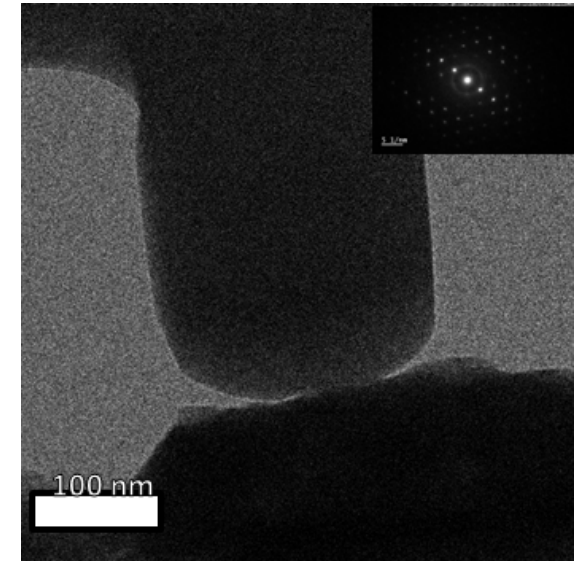
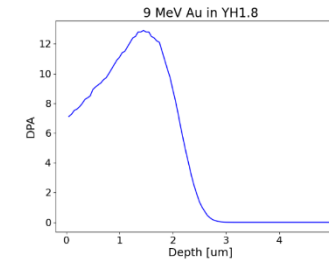
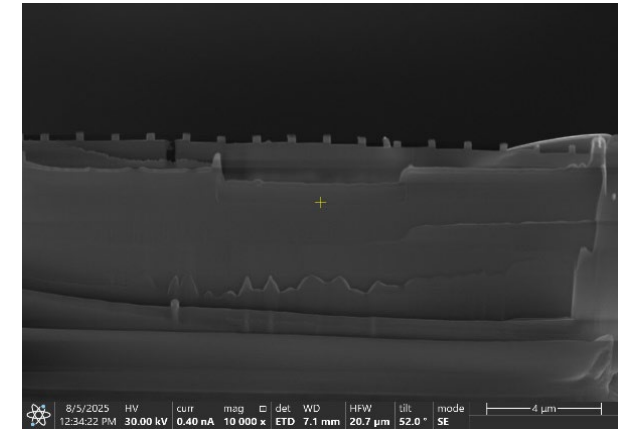


Thrust 3 progress

- Irradiated region only about 200 nm deep: too shallow for micropillar compression
- TEM nanopillar compression will directly probe this irradiated region
 - Nanopillars fabricated via FIB milling: Inform the results on the nanopillar compression experiments



Uniform distribution of 1.1-1.6 nm voids with above number densities

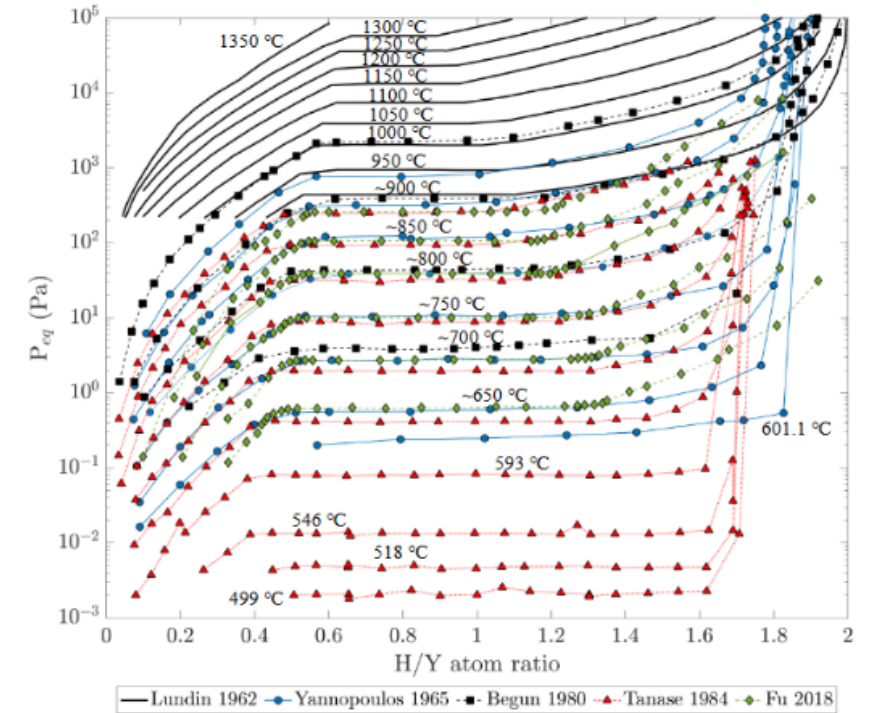
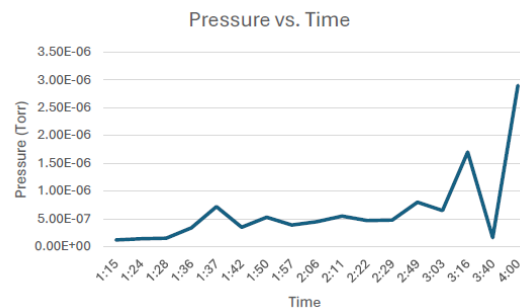


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Thrust 4 progress: Decipher real-time YH dynamics and interplays with structural materials

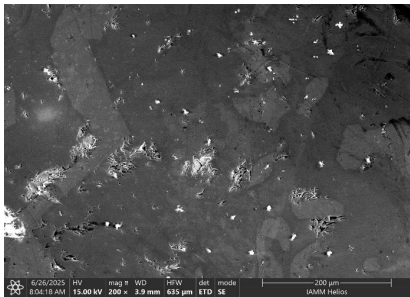
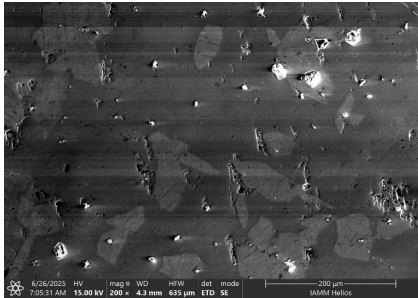
- Residual gas analyzer (RGA) attached to UTK beamline to decipher real-time desorption monitoring of H during irradiation
 - Some real-time measure of H release during irradiation+heating
 - Less quantitative, but certainly qualitative understanding of H being released



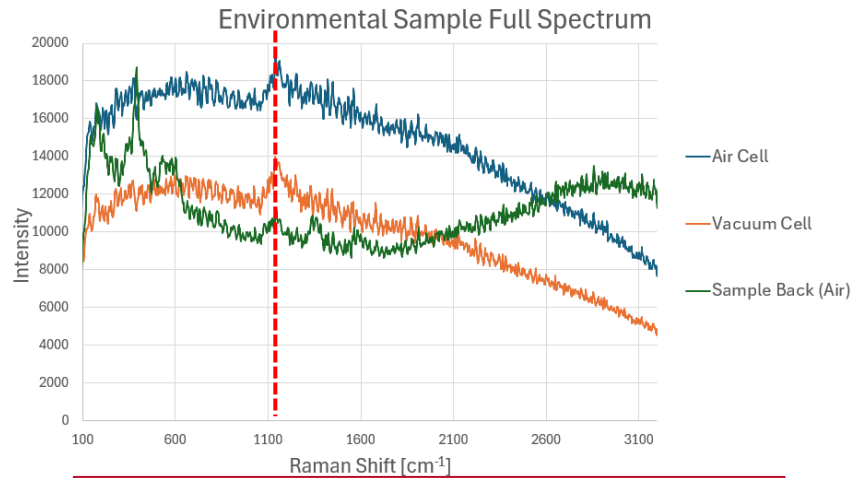
In-situ gas environment cell will need to be ~1 Torr

In-vacuo vs in air irradiation effects

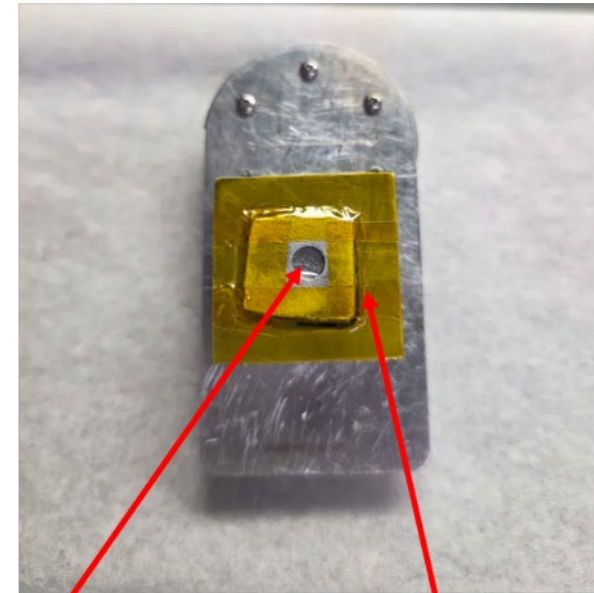
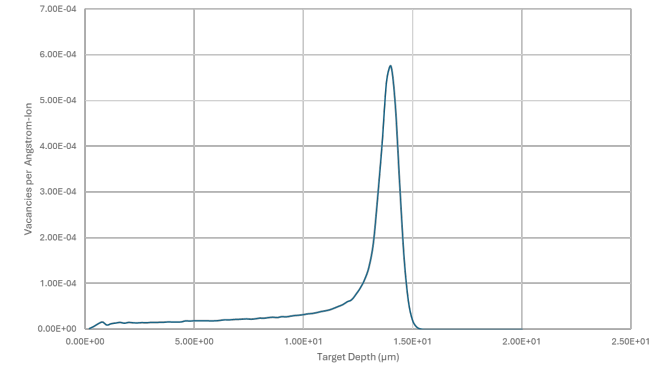
- UTK fabricated an environmental irradiation cell



Top: SEM of YH_{1.8} sample irradiated in vacuum. Bottom: SEM of YH_{1.8} sample irradiated in JMAW cell.



Raman Spectroscopy shows no clear oxidation or second phase development for YH sample irradiated in air



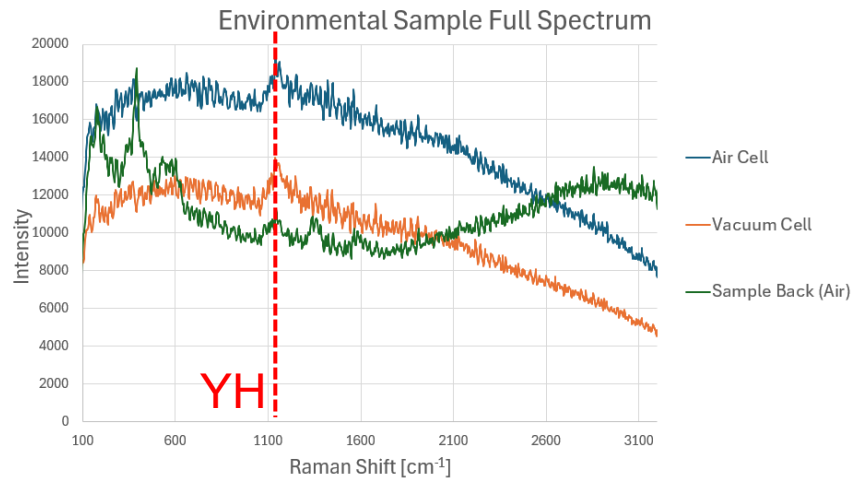
.8 μm thick Al foil

Kapton Tape

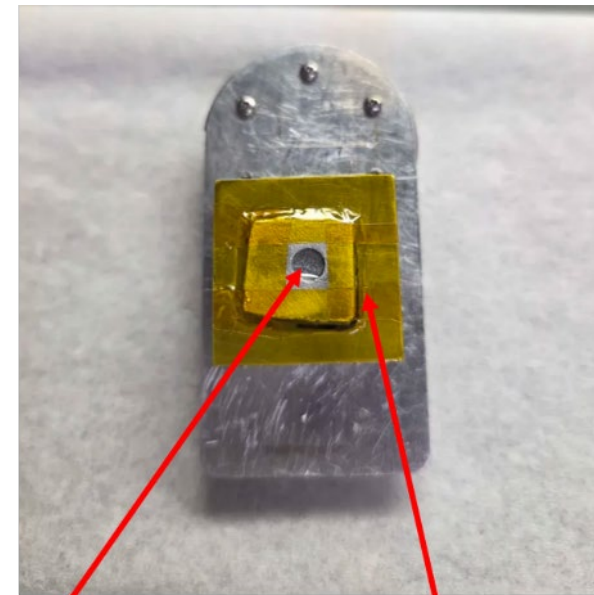
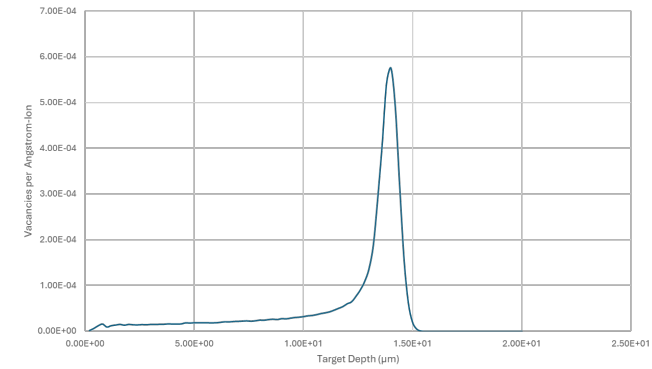
In-vacuo vs in air irradiation effects

- UTK fabricated an environmental irradiation cell

Sample	Hardness (GPa)
YH1.8 - Air Cell (3 MeV H)	4.6 ± 0.45
YH1.8 - Vacuum (3 MeV H)	4.2 ± 0.6
YH1.8 - Vacuum (9 MeV Au, 400 C)	4.9 ± 1.7
YH1.8 - Vacuum (400 C, no dose)	2.9 ± 1.8
YH1.8 Pristine	4.6 ± 0.29



Nanoindentation hardness shows air cell irradiation did not result in significant H loss



.8 μm thick Al foil

Kapton Tape

Roadblocks & Mitigations

- Mechanical testing (Thrust 1 and 3)
 - In-situ SEM Picoindenter fully installed and operating
 - FIB issues at UNM – CINT proposal has mitigated some concerns
 - PI-95 at CINT – electrical grounding issue, using UTK PI-95
- Thrust 4
 - In-situ TEM irradiation at UTK delayed in development
 - Will leverage my joint appointment at Sandia National Labs IBL for H gas environment coverage ion irradiation

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- **All work on all thrusts currently ongoing**

Summary

- Work ongoing to investigate the structure property and ion irradiation properties of YH for microreactor applications
- 8 specimens exposed to 9 MeV Au irradiation
- Effects of ion irradiation environment evaluated
- Micro/nano-mechanical properties being studied with newly installed PI-85E picoindenter at UNM
- Nanomechanical properties studied with TEM studies

Future goals and near-term outcomes

- Thrust 1:
 - Size-dependent mechanical testing on pillar specimens
- Thrust 3:
 - In-situ SEM/TEM compression coupled with modelling
 - More to be done next week
- Thrust 4:
 - In-situ TEM ion irradiations planned at SNL IBL next week

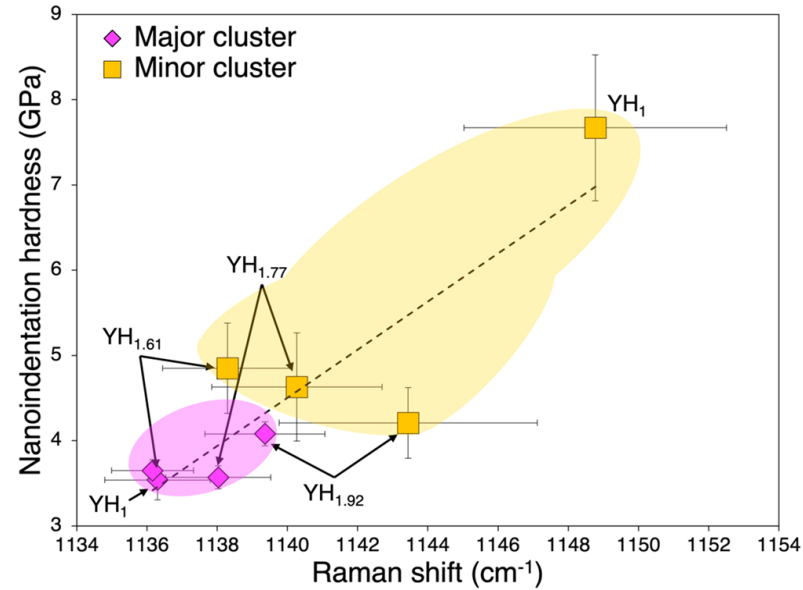
Outcomes & Products

- LANL: Developing technique for fabricating YH with $H/M < 1.9$ in 6% flowing hydrogen
- UTK: H. Osborne, “Development of transient grating spectroscopy (tgs) to analyze thermal and mechanical properties of yttrium hydride during in situ ion irradiation” M.S. Thesis, UTK, 2026
- UNM: Presentations planned at TMS 2026 Conference
 - R. Pena, “Non-destructive characterization of ion irradiated yttrium hydride for microreactor applications” M.S. Thesis, University of New Mexico, 2025.
 - Previous presentation at TMS 2025

Prior work has established some structure-fabrication relationships

- Fundamental relationships between H concentration and nano-hardness established
- Identification of common impurities and phases

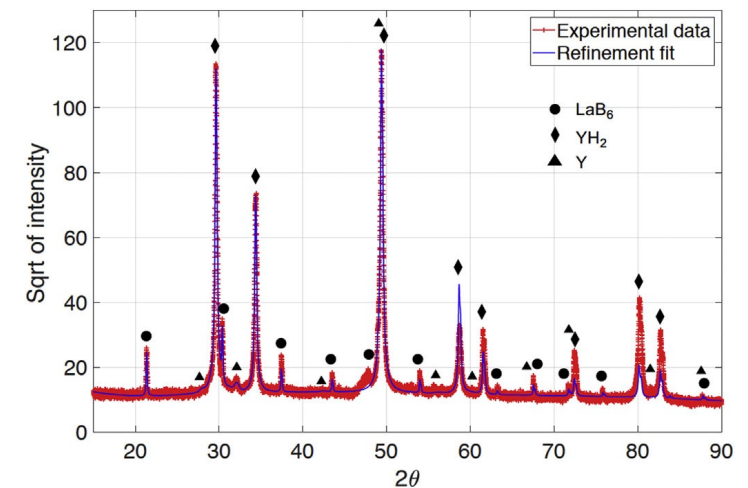
All samples investigated here fabricated at LANL



INL: multi-scale NDE of YH properties and composition

H. Gietl, et al. Journal of the European Ceramic Society 43 (2023) 3216–3227

ORNL fabricated relatively high purity YH samples



X. Hu, et al. Journal of Nuclear Materials 539 (2020) 152335