DOE-NE Microreactor Program Review

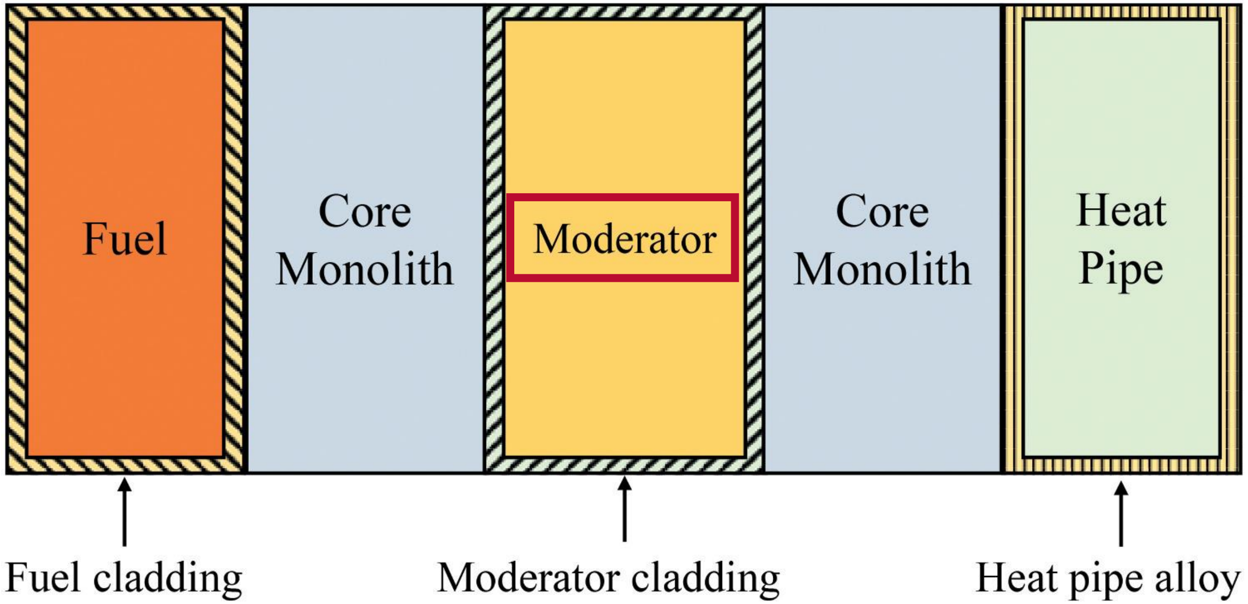
(Project 23-29784) Deciphering Irradiation Effects of YHx Through In-situ Evaluation and Micromechanics for Microreactor Applications

Eric Lang – UNM

NUCLEAR ENGINEERING

Microreactors

- Microreactor: <30 MW_{th} power and >600 °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

- TRL1: Basic principles of metal hydrides observed and reported
- TRL2: Hydride moderator concept or application formulated
- TRL3: Hydride moderator concept or application proven through analysis and experimentation
- TRL4:Basic prototype validated in laboratory environment

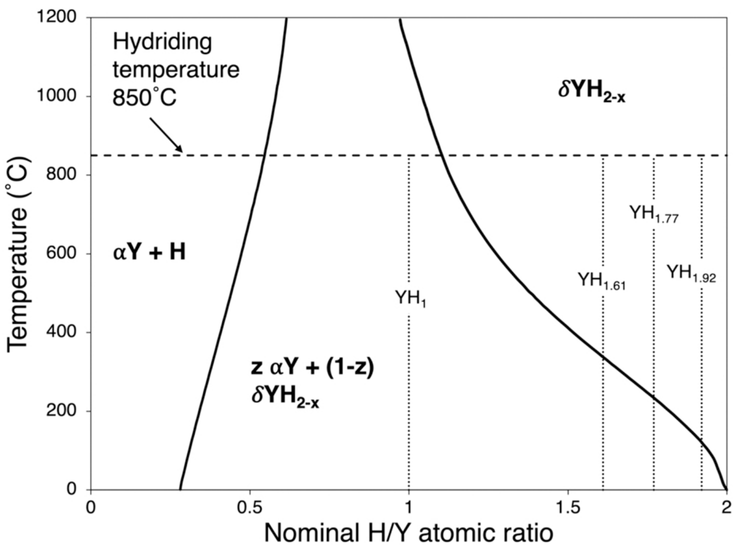
Experimental infrastructure is needed to assess hydrogen redistribution in condensed matter

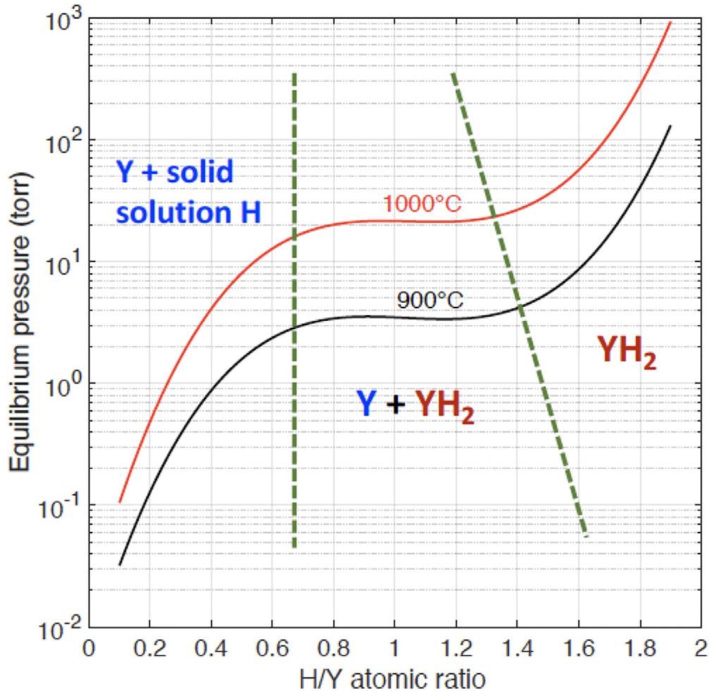
M. Nedim Cinbiz, Chase N. Taylor, Erik Luther, Holly Trellue & John Jackson (2023) Considerations for Hydride Moderator Readiness in Microreactors, Nuclear Technology, 209:sup1, S136-S145, DOI: 10.1080/00295450.2022.2121583

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?





J. Peng, M. Wu, F. Du, F. Yang, J. Shen, L. Wang, X. Ye, G. Yan, Thermodynamic modelling of Y–H and Y–Zr–H system aided by first-principles and its application in bulk hydride moderator fabrication, J. Nucl. Mater. 531 (2020), 152035, https://doi.org/10.1016/j.jnucmat.2020.152035.

G.M. Begun, J.F. Land, J.T. Bell, High temperature equilibrium measurements of the yttriumehydrogen isotope (H2, D2, T2) systems, J. Chem. Phys. 72 (5) (1980) 2959e\2966.

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

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)

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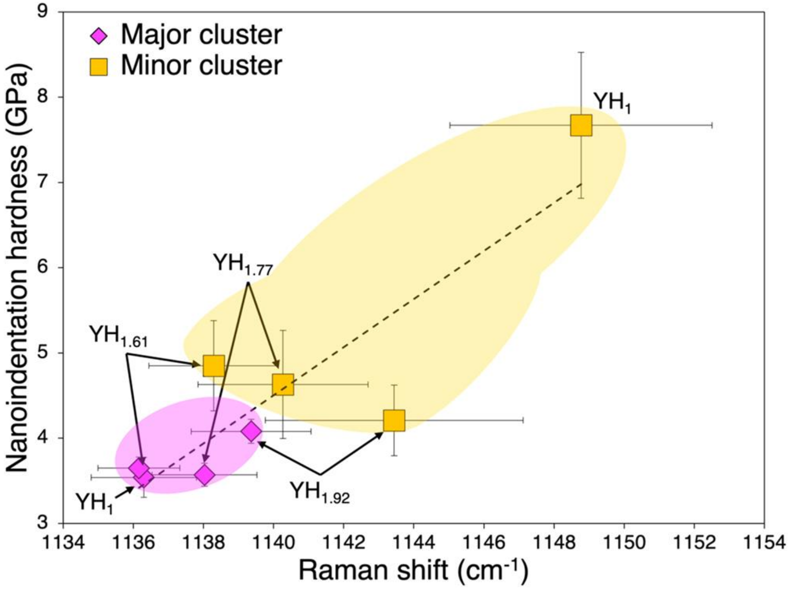
Prior work has established some structure-fabrication relationships

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

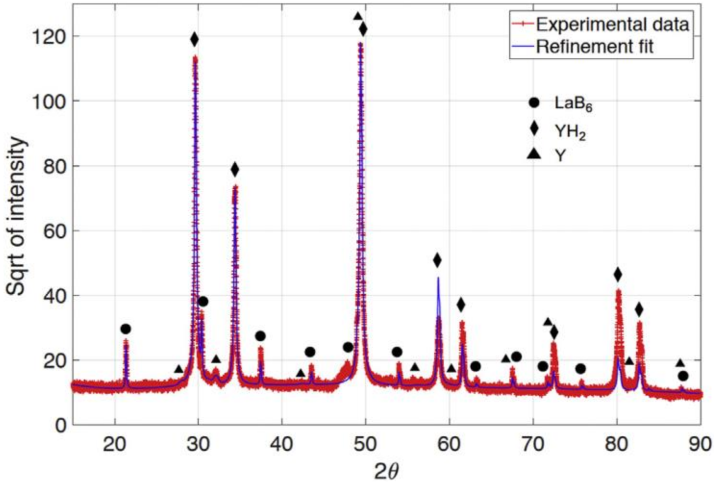
All samples investigated here fabricated at LANL



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

Fabrication of YH Specimens: Thrust 1

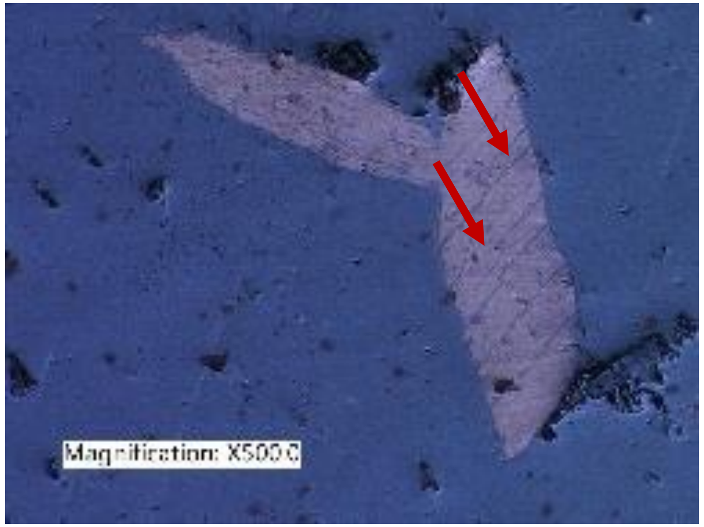
- Two sets of specimens fabricated at LANL

Sample	H:M
Sample 1 - Disc	1.8
Sample 2 – Disc	1.4
Sample 3 – Disc	1.7
Sample 4 – Disc	1.9
Sample 5 – Cube	1.6
Sample 6 – Cube	1.6
Sample 7 – Cube	1.7

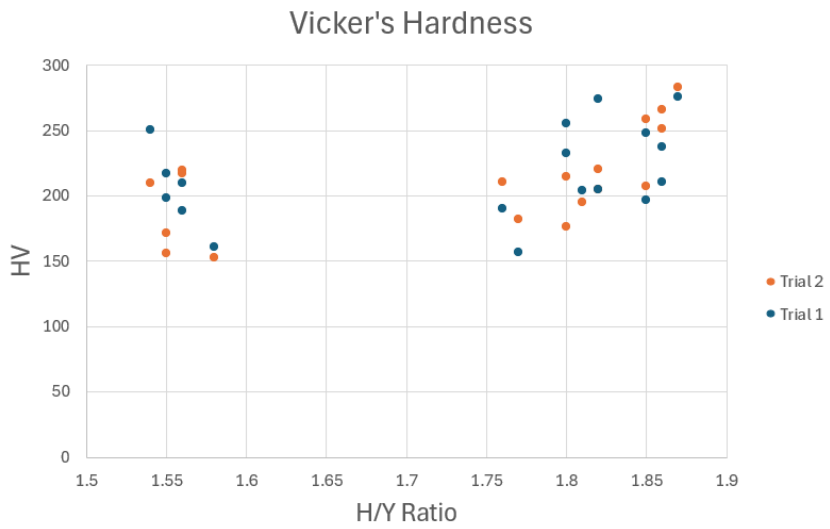
Sample	H:M	Number of Samples
Sample Set 1 – Disc	~1.6	10
Sample Set 2 – Disc	~1.7	10
Sample Set 3 – Disc	~1.8	8
Sample Set 4 – Disc	~2	9
Sample Set 5 – Cube	~1.55	6
Sample Set 6 – Cube	~1.8	12

Fabrication of YH Specimens: Thrust 1

YH1.5 sample: spinel-type structure



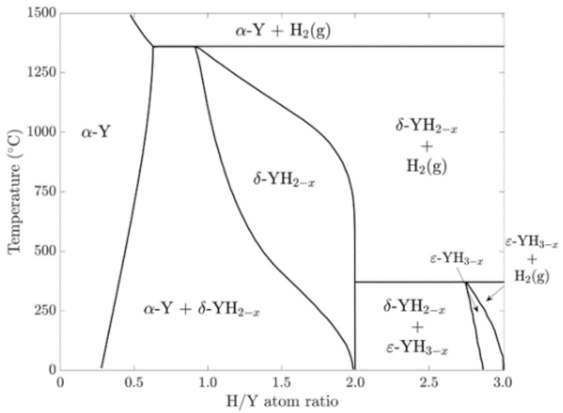
Multi-phase structure with interphase twins

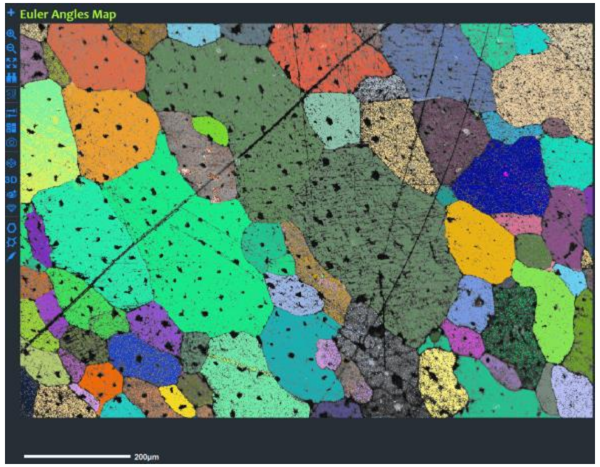


Hardness trend with H:M ratio agrees with other specimens

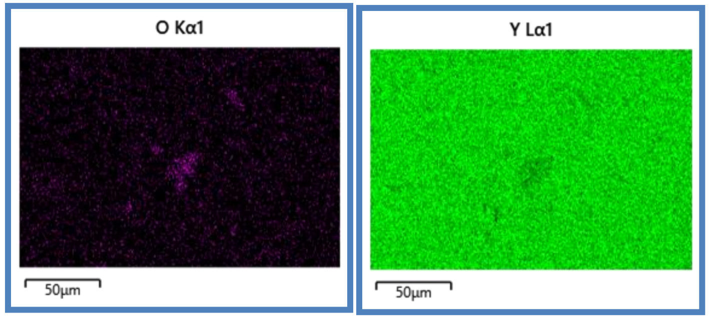
Fabrication of YH Specimens: Thrust 1

- 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.

NUCLEAR ENGINEERING

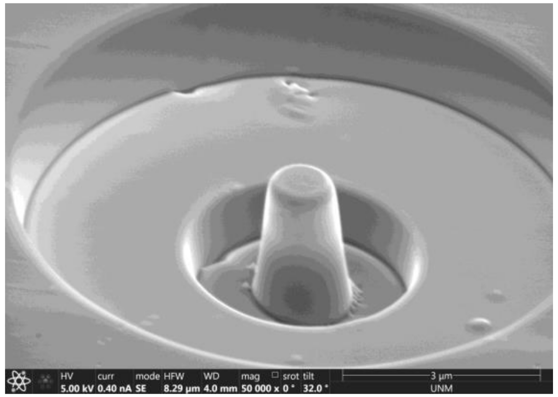
In-situ SEM Picoindenter

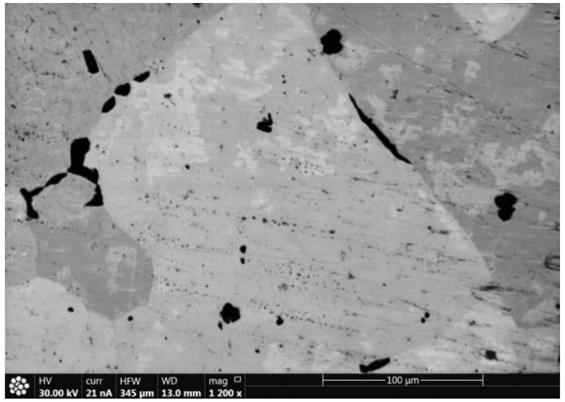
- Technically associated with NEUP Infrastructure DE-NE0009369
  - “Establishment of Hot Cell Irradiated Materials Micro and Nano-Mechanical Testing at the University of New Mexico”
  - Purchasing of SEM Picoindenter at UNM
  - Hysitron PI-85E Picoindenter delivered and installed April 2024 at UNM

PI-85E in our FIB at UNM

Nanomechanical properties

- Samples well-polished enough for single-grain identification -> crucial for single-crystal micropillars
- Sets of micropillars FIB machined on YH1.8 samples
- Preliminary compression tests indicate pillars need to have larger OD





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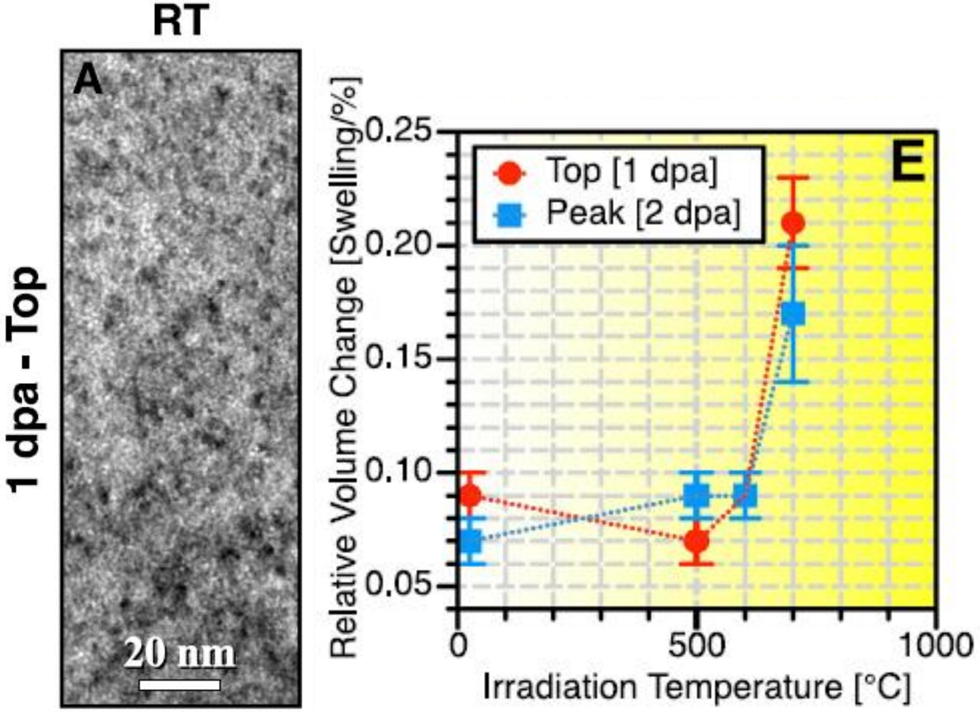
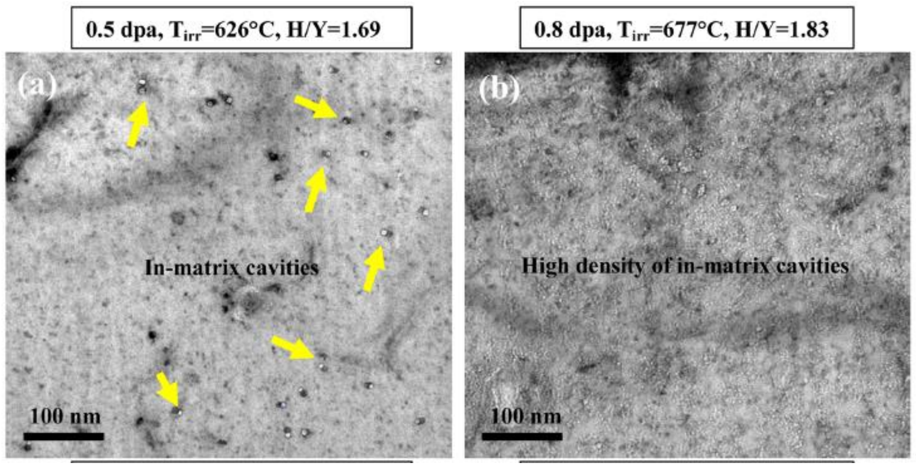
Prior work has established first effects of ion/neutron irradiation on YH

Ion irradiation of YH_2 specimens causes void formation



M.A. Tunes, et al. Acta Materialia 280 (2024) 120333

Neutron irradiation causes grain restructuring, voids, varying H content, lowered thermal conductivity, altered H release



A. Le Coq, et al. JNM 603 (2025) 155374

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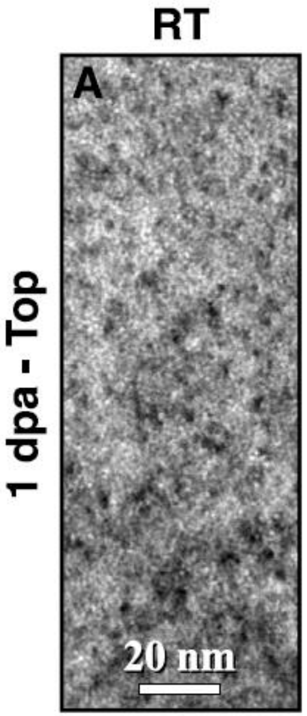

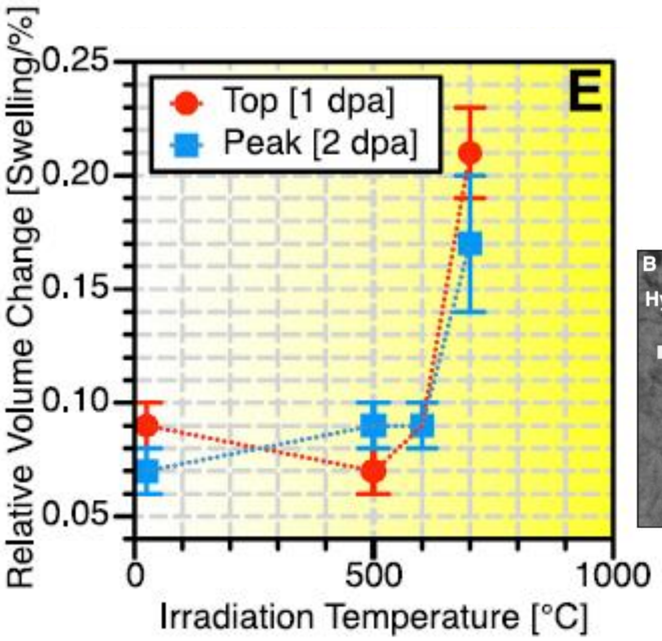

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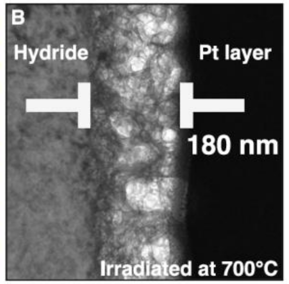


Only one ion species: 9 MeV Au

What ion species best emulates neutron irradiation damage in YH? We have this data for steels, why not for YH?

Oxide formed under irradiation

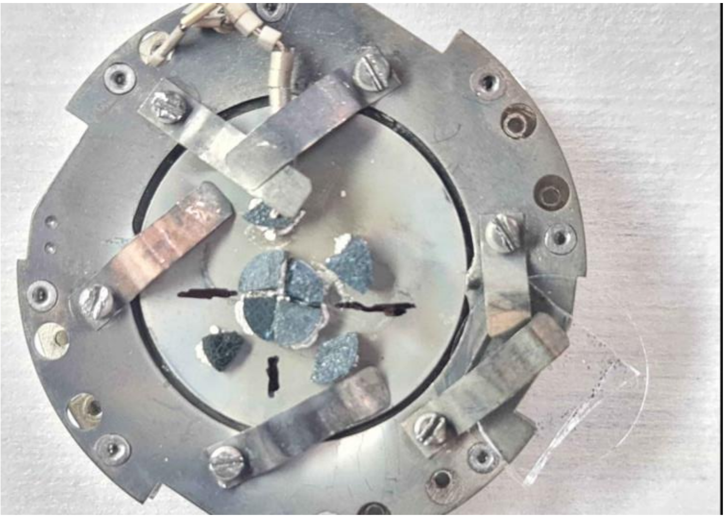
Effect of vacuum irradiation environment and no cladding material

H content studied in near-surface with ERD

More ion irradiation studies needed

Thrust 2 progress

- Thrust 2: Probe radiation damage effects with reactor-relevant doses of irradiation
  - UTK Tennessee Ion Beam Materials Lab
  - H ion irradiations carried out



Yttria powder cathode and new high temperature stage commissioned at UTK

Cyclotron Irradiation of Specimens

- Due to mechanical issues at TIBML in summer 2024, ion irradiations performed at UTK Cyclotron (100 MeV protons)
  - Enables through-sample irradiation (i.e. no unirradiated zone)
  - No implantation effects
  - Important for studying effects of irradiation on H release

#	Sample	Fluence [cm^{-2}]	Time (sec)	Average Flux
6	Gold plated Yttrium Disc	1.00E+11	438.6	2.28E+08
3	Gold plated Yttrium Disc	1.83E+09	5.1	3.59E+08
5	YH Disc	1.01E+11	324	3.12E+08
1	YH Disc	1.47E+09	8.4	1.75E+08

Gold plated to prevent H release during irradiation

Cyclotron Irradiation of Specimens

- Due to mechanical issues at TIBML in summer 2024, ion irradiations performed at UTK Cyclotron (100 MeV protons)
  - No mass change before and after irradiation a quick indication of no H loss during the irradiation experiment

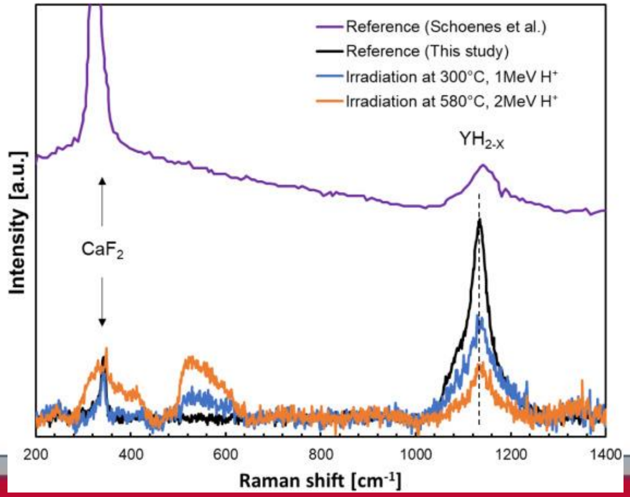
#	Sample	Fluence Fluence [cm^{-2}]	Mass Before (g)	Mass After (g)	Mass Change (g)
6	Gold plated Yttrium Disc	1.00E+11	0.1096	0.1098	+0.0002
3	Gold plated Yttrium Disc	1.83E+09	0.1024	0.1026	+0.0002
5	YH Disc	1.01E+11	0.0875	0.0875	0.0000
1	YH Disc	1.47E+09	0.0705	0.0703	-0.0002

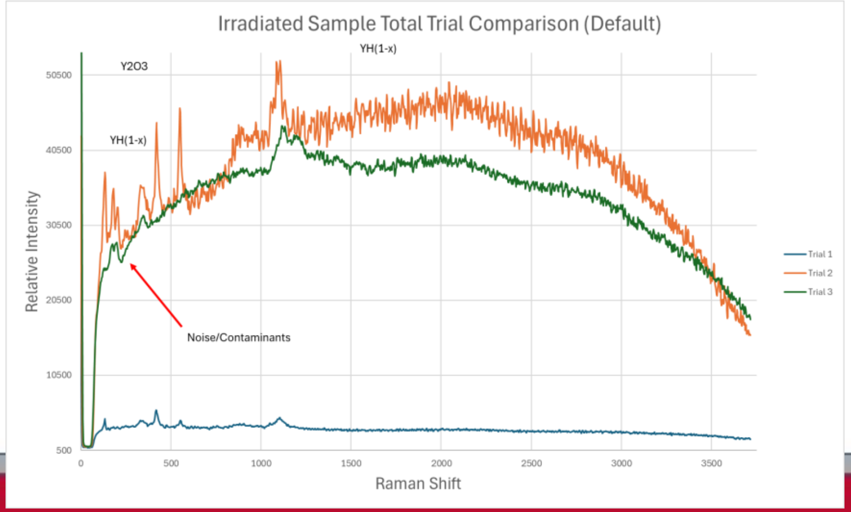
Gold plated to prevent H release during irradiation

Ion irradiation experiments

- Analysis focusing on non-destructive evaluation
- Voids not expected due to low fluence, high energy, low temperature
- Raman Spectroscopy
  - Used previously for NDE of YH -> identification of phases and correlation with H content

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

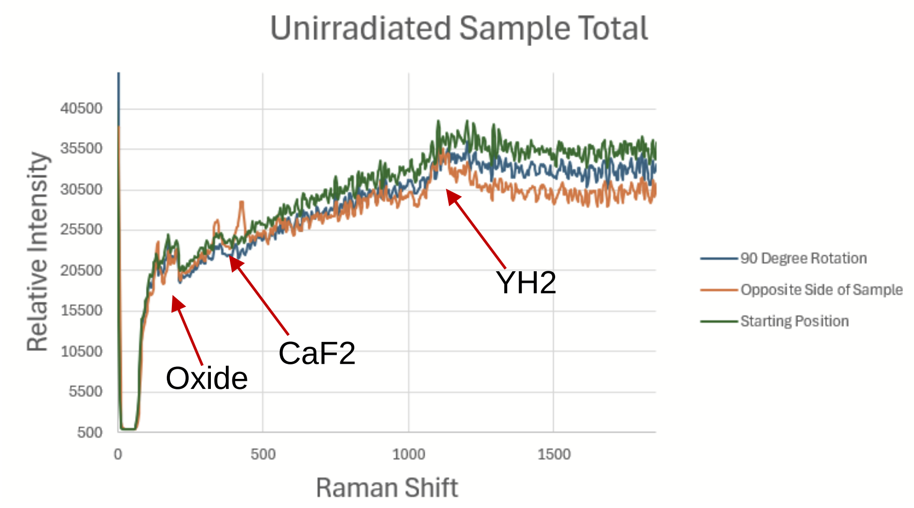


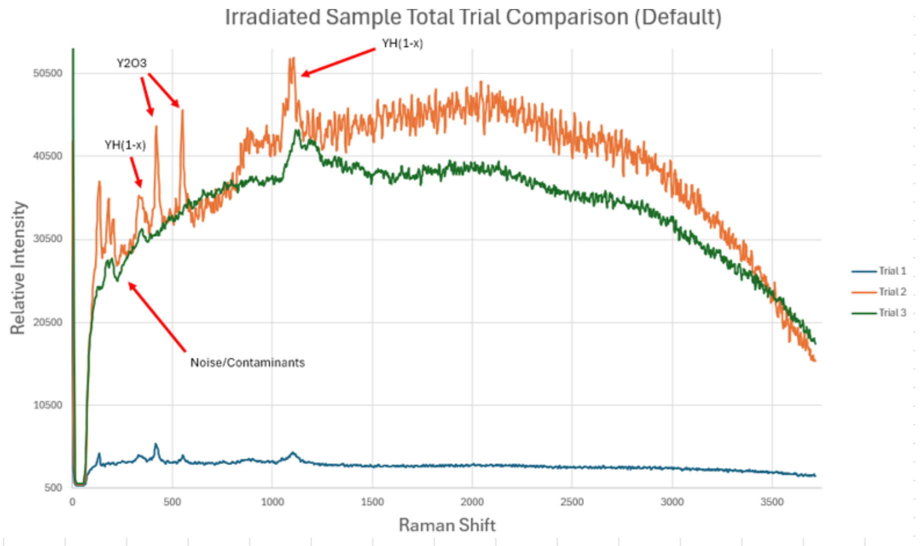


Our data

Ion irradiation experiments

- Raman Spectroscopy
  - Differences in sample position vs. irradiation



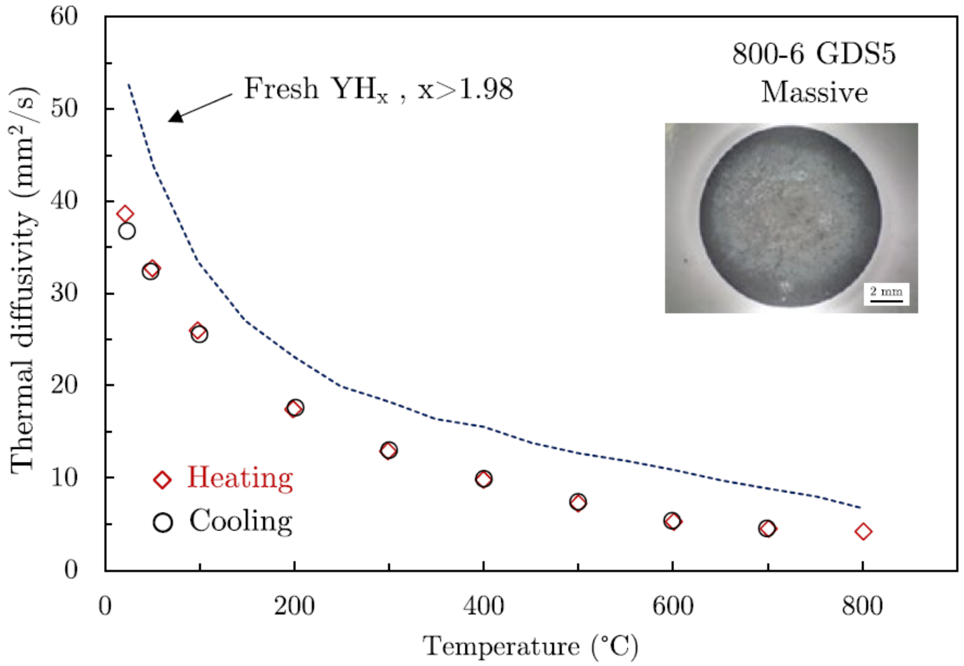


Ion irradiation experiments

- Analysis focusing on non-destructive evaluation
- Voids not expected due to low fluence, high energy, low temperature
- Positron Annihilation Spectroscopy to be performed at INL
  - View if nanoscale defects formed
  - Samples shipped to INL yesterday
- Thermal Desorption Spectroscopy planned
  - Impact of irradiation on thermal release of H
  - Neutron irradiation showed varied results, but irradiation temperature not well-controlled
  - This irradiation study will offer direct comparison results across samples

Thermal Properties Evaluation

- Changes in thermal conductivity due to irradiation may change H diffusion during irradiation, thus affecting moderating power
- Neutron irradiation has shown decrease in thermal conductivity following irradiation, but results vary across manufacturing
  - Results only taken post-mortem, no time resolution



M.N. Cinbiz, et al. INL/RPT-23-71531 Revision 0 Sept 2023

Thermal Properties Evaluation

- Currently, a Transient Grating Spectroscopy system is being constructed at TIBML for *ex-situ* and *in-situ* characterization on YH samples.
- Properties such as void growth and thermal diffusivity can directly be measured post-irradiation using this system.
- Photos of the optics (Fig. 3), the pump laser (Fig. 4), and the probing laser (Fig. 5) are shown.

Fig. 3

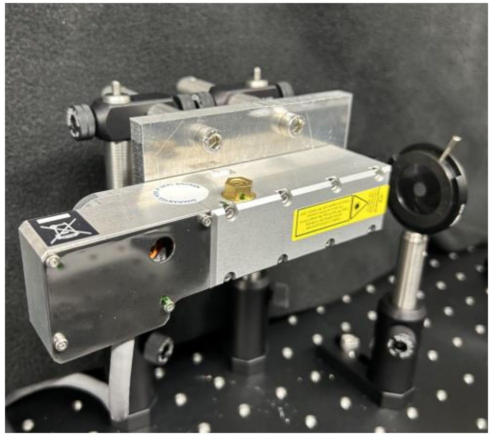


Fig. 4

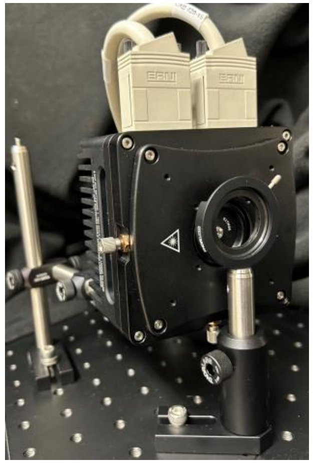


Fig. 5

Fabrication of YH Specimens: Thrust 1

- Second set of samples fabricated in October 2024

Sample	H:M	Number of Samples
Sample Set 1 – Disc	~1.6	10
Sample Set 2 – Disc	~1.7	10
Sample Set 3 – Disc	~1.8	8
Sample Set 4 – Disc	~2	9
Sample Set 5 – Cube	~1.55	6
Sample Set 6 – Cube	~1.8	12

Ion irradiation plan:

Systematic study of starting H content

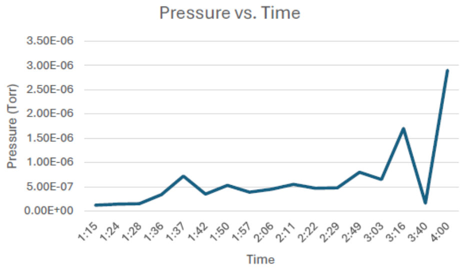
Au beam
>600 C
1-2 dpa

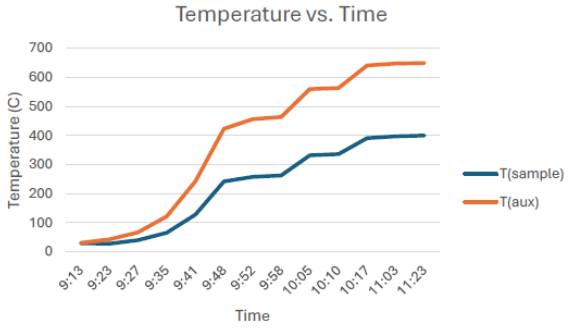
$$Au^{3+}: 9\ MeV, 400C$$

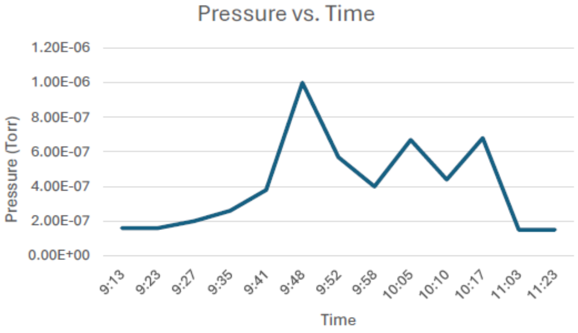
$$\emptyset = 2.33 * 10^{15} \frac{ions}{cm^2}$$

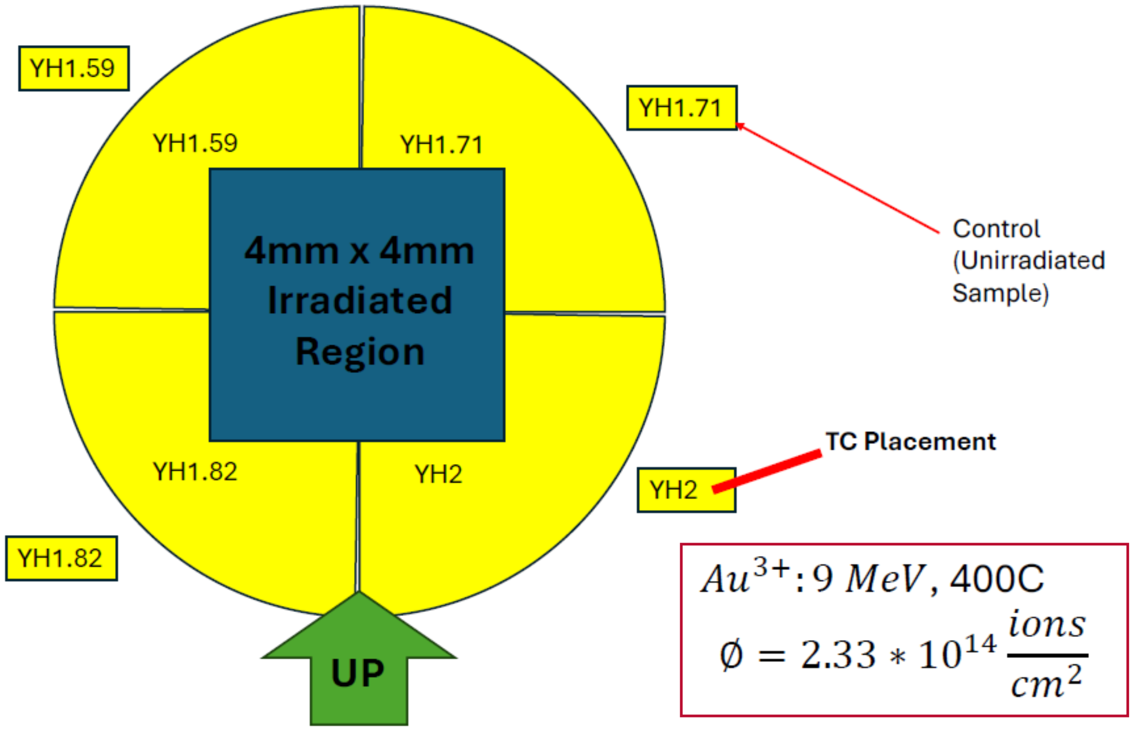
Ion Irradiated Specimens at UTK

- Need for gas-coverage experiments
  Outgassing above only 700 °C









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

$\emptyset = 2.33 * 10^{14} \frac{ions}{cm^2}$

Path and progress

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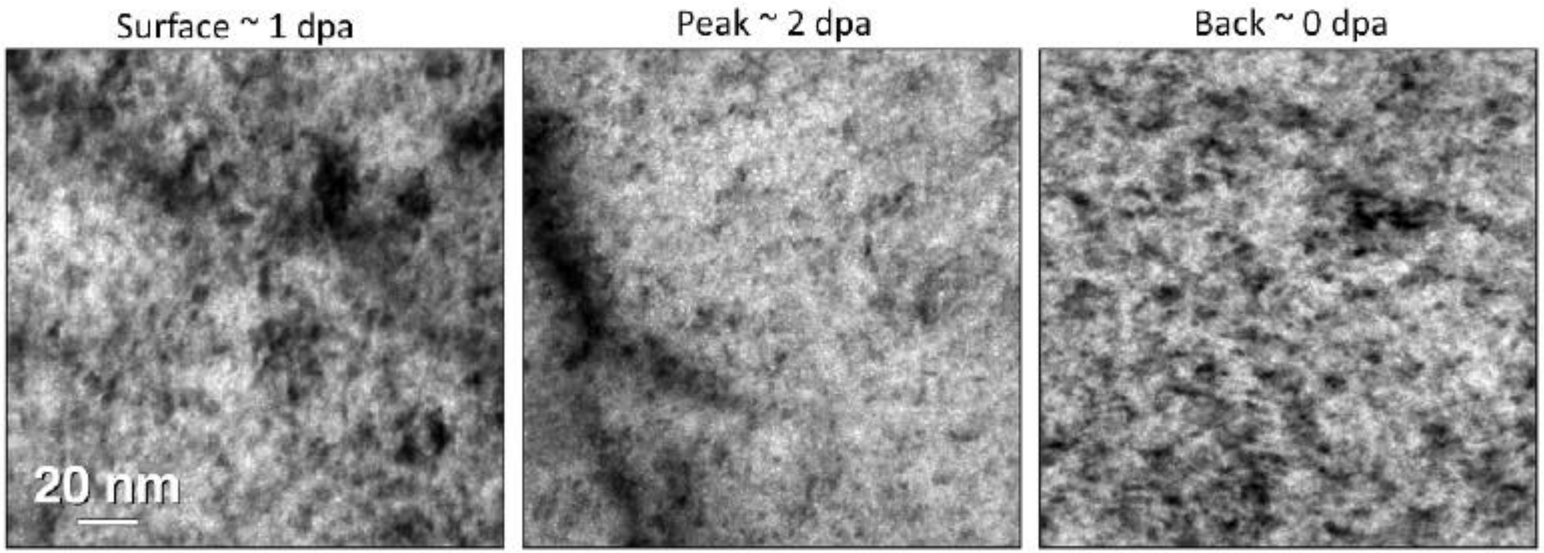
- **Thrust 3: Structure-property relationships of ion irradiated YH materials (Primary work: Q4-Q12)**
- Thrust 4: Decipher real-time YH dynamics and interplays with structural materials (Primary work: Q8-12)

Thrust 3 progress

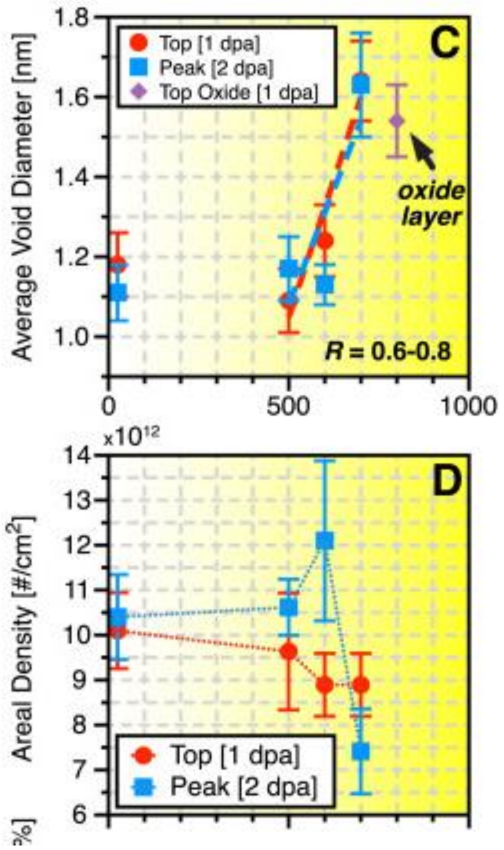
- Old ion irradiated specimens shipped from LANL to UNM for further characterization (APT specimen fabrication and micro/nano-cantilever fabrication)
- However, irradiated region only about 200 nm deep -> too thin for micropillar compression



Previously irradiated YH specimens with 9 MeV Au at 700 C

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
  - Collaboration with UNM Prof. Madura Pathirage, expert in Finite Element modelling of fracture of brittle solids with nanoscale voids



Uniform Distribution of 1.1-1.6 nm voids with above densities

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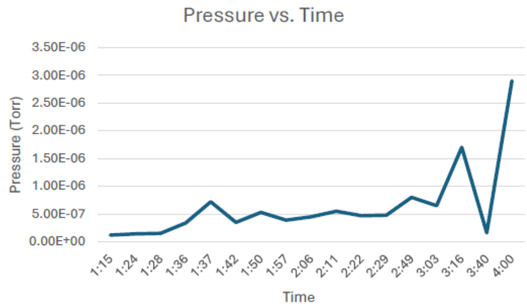
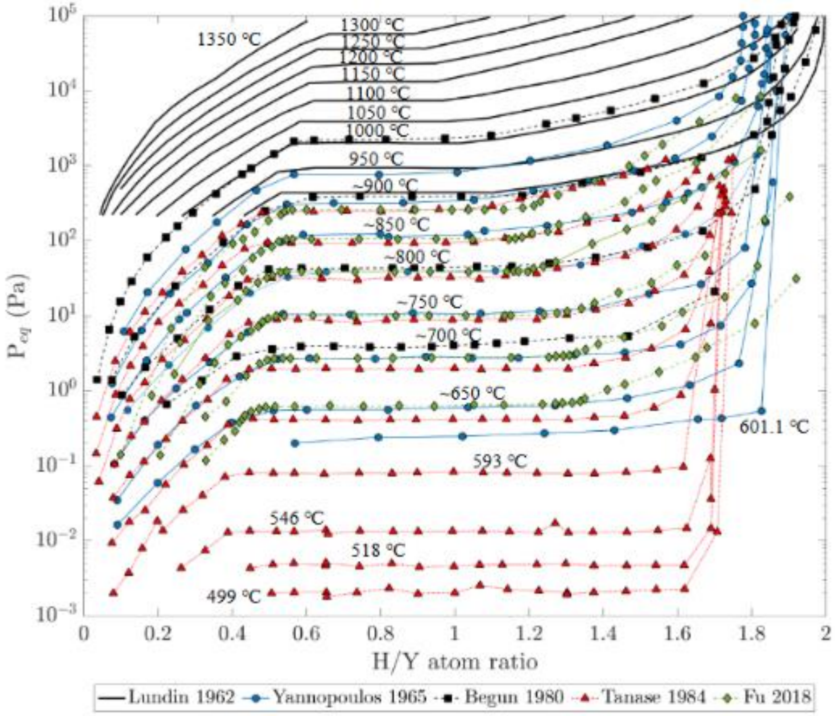
Thrust 4 progress: Decipher real-time YH dynamics and interplays with structural materials

- 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

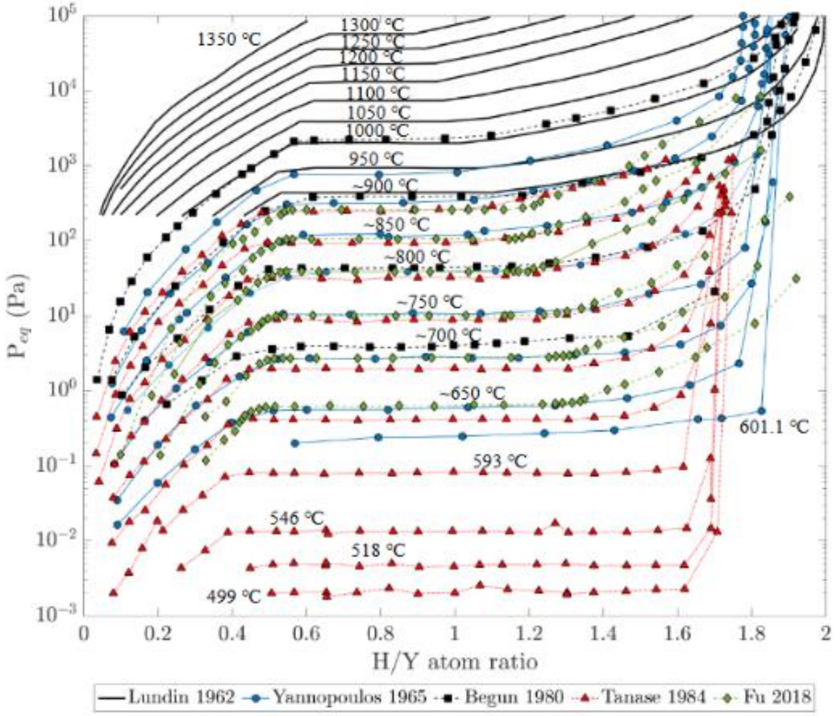




In-situ gas environment cell will need to be ~1 Torr

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

- Depositing YH thin films
  - Y sputter target acquired, reactive sputtering in $Ar+H_2$ gas
  - Deposition on SiN windows
    - SiN windows purchased to maintaining H environment in beamline
  - Deposition on a Mo:YH:Mo sandwich for through-layer irradiation to prevent H loss



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

Roadblocks & Mitigations

- Sample fabrication (Thrust 1)
  - LANL upgrades to furnace capabilities
  - No specimens fabricated between March and October 2024
  - Now have >20 samples fabricated in various H:M ratios and geometries
  - Polishing of specimens: grain pullout, pitting, etc.
- Ion irradiation (Thrust 2)
  - UTK TIBML down between July and October 2024
  - Now continuously operating since November 2024
    - Used cyclotron at UTK instead of TIBML
  - Au ion irradiation performed at high temperature now, though
- Mechanical testing (Thrust 1 and 3)
  - In-situ SEM Picoindenter fully installed and operating

Outcomes & Products

- LANL: Developing technique for fabricating YH with H/M < 1.9 in 6% flowing hydrogen
- UTK: Presentation planned at ANS Student 2025 Conference “Design and Construction of Transient Grating Spectroscopy (TGS) System”
- UNM: Presentation planned at TMS 2025 Conference
  - R. Pena, C. Kohnert, K. Hattar, E. Lang. “Effect of 100 MeV Irradiation on YH for Microreactor Operation”

Summary

- Work ongoing to investigate the structure property and ion irradiation properties of YH for microreactor applications
- Successful fabrication of a total of ~25 specimens in varying H:M ratios
- 4 specimens exposed to 100 MeV proton irradiation
  - Early results show no change in H concentration
  - To perform thermal desorption spectroscopy
- 8 specimens exposed to 9 MeV Au irradiatoin
- Micro/nano-mechanical properties being studied with newly installed PI-85E picoindenter at UNM
- YH films deposited on SiN to enable studies at elevated temperatures without oxidation/loss of H

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

Future goals and near-term outcomes

- Thrust 1:
  - Continued characterization of as-hydrided specimens recently fabricated at LANL
- Thrust 2:
  - Ion irradiations at elevated temperatures with H, Au, Y beams at UTK
  - Systematic study of different H:M ratios
- Thrust 3:
  - Pre-irradiation characterization of mechanical properties
  - In-situ SEM/TEM compression coupled with modelling
- Thrust 4:
  - Y films to be deposited on SiN windows for H gas cover experiments at UTK
  - Depositing films via CINT User Access proposal at LANL