



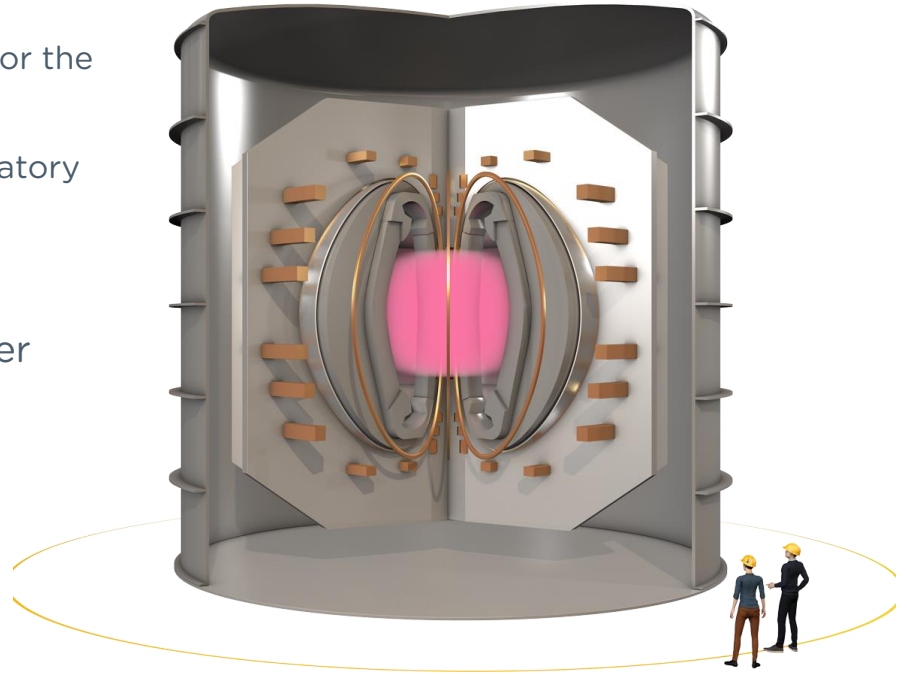
# Fabrication and characterization of transition metal hydrides for radiation shielding in tokamak devices

Gurdeep S. Kamal & Caitlin Kohnert



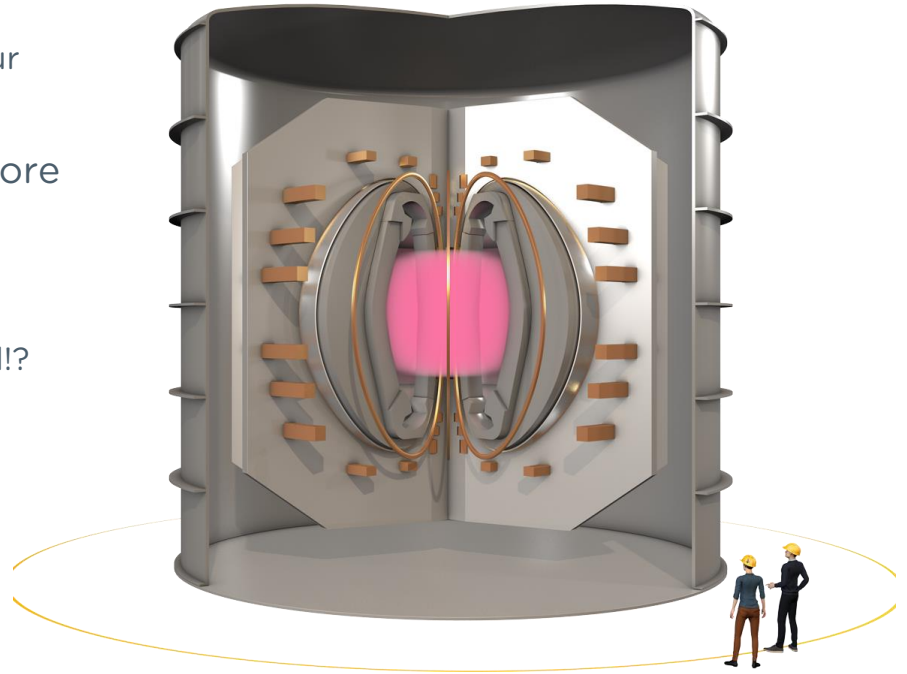
# Successful INFUSE applications are written considering the following

- Early engagement with national laboratory
  - Within the interest of the lab PI or the mission for the national laboratory
  - Clear understanding on what the national laboratory capabilities/experiences are
  - Keep a record of discussion!
- Underlying scientific basis on subject matter
- Be open and honest on your aspirations
- Have clear milestones to your end goal
- Working knowledge and theoretical understanding



# Successful INFUSE applications are written considering the following

- Relate the proposal to the application
  - Have clarity on how this proposal will assist your organization's end goal
- Peer review of your proposal internally before submission
  - is it valid to you and your organization!?
  - Does it meet your organization's quality control!?



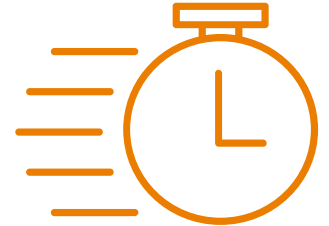


# Our INFUSE project was successful for the following reasons

- Good working relationship with national labs
  - Began talks in ample time to ensure all entities are in alignment
- Comprehensive and productive dialogue on-going during the project
  - In person kick off is key! [Our Lesson Learned]
  - Constant review on progression of milestones
  - Be as pro-active as possible for the project
  - Early knowledge transfer
- Build in adaptability into the project



Collaboration



Agility



Safety



Creativity



Respect

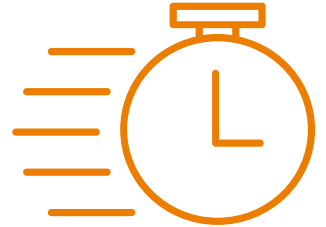


# Our INFUSE project was successful for the following reasons

- Have a team complimentary to one another
- Identifying risks early and attempt to burndown your risks when opportunities present themselves
- Be agile to pandemics



Collaboration



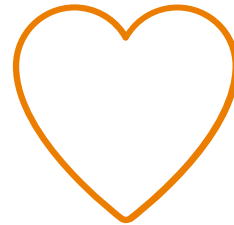
Agility



Safety



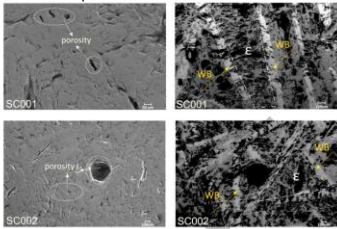
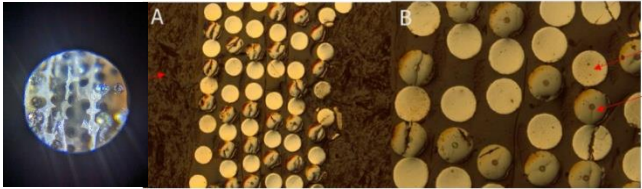
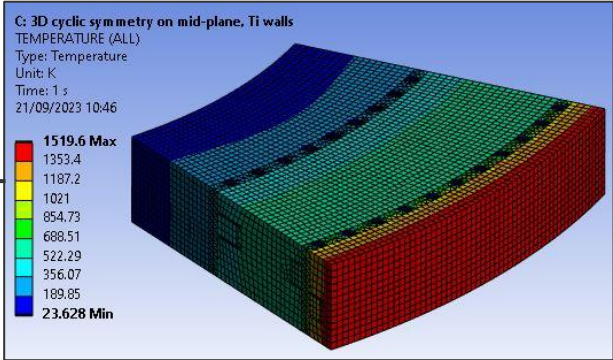
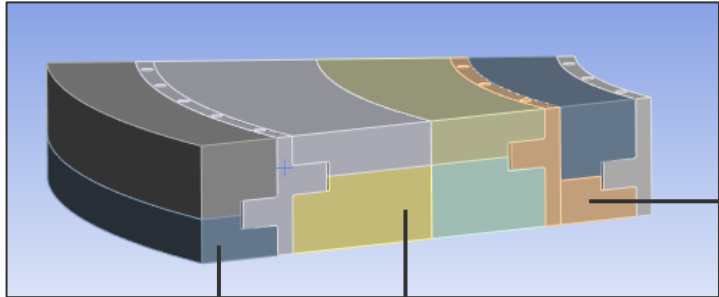
Creativity



Respect

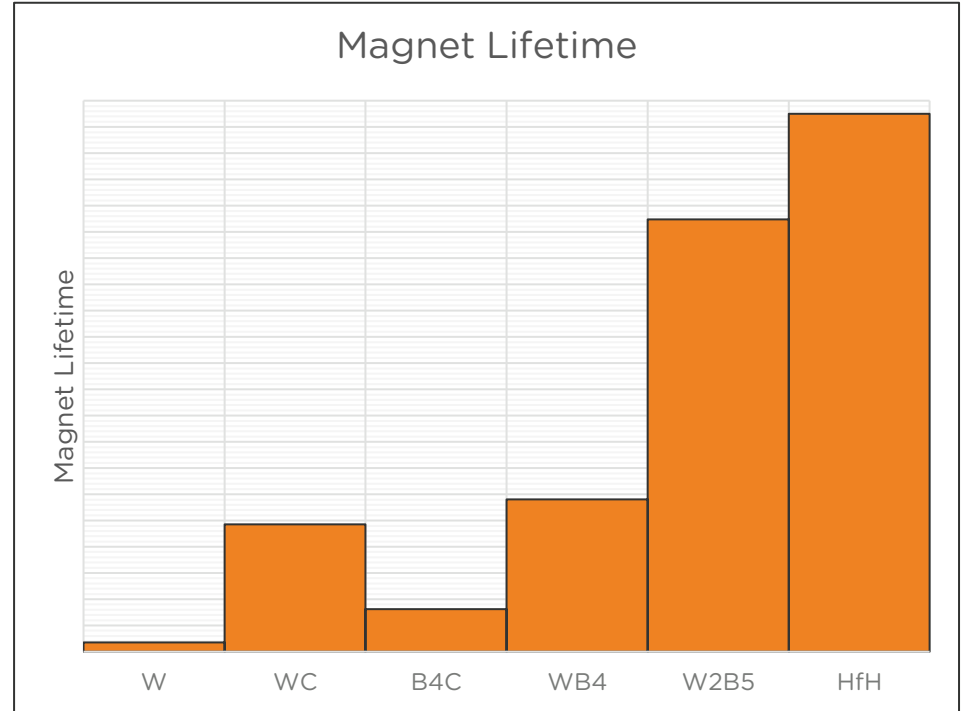


# This HfH project is providing the material basis towards an advanced shield solution for tokamak devices

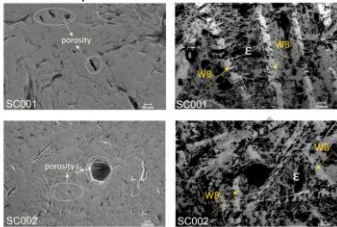
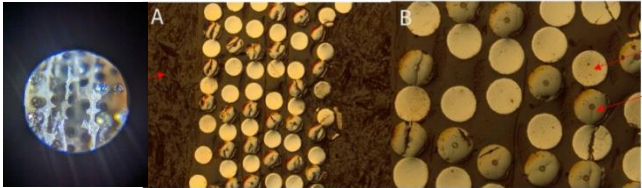
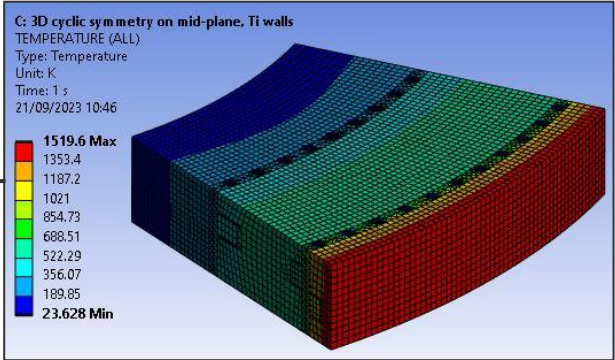
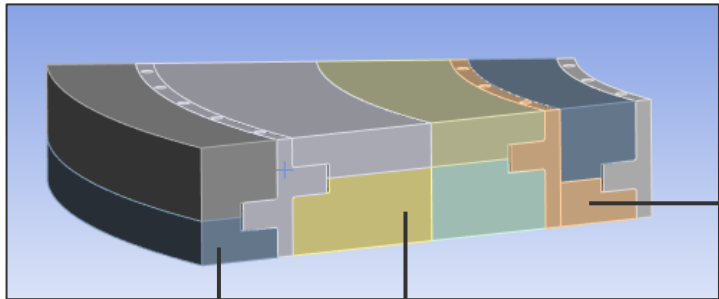


# This HfH project is providing the material basis towards an advanced shield solution for tokamak devices

- Tokamak Energy's Neutronics calculations show Hafnium Hydride has the potential to greatly increase the lifetime of HTS magnets in compact devices.
- Hafnium Hydride is optimal when used in a smalls shielding layer close to the magnet in combination other advanced shield materials



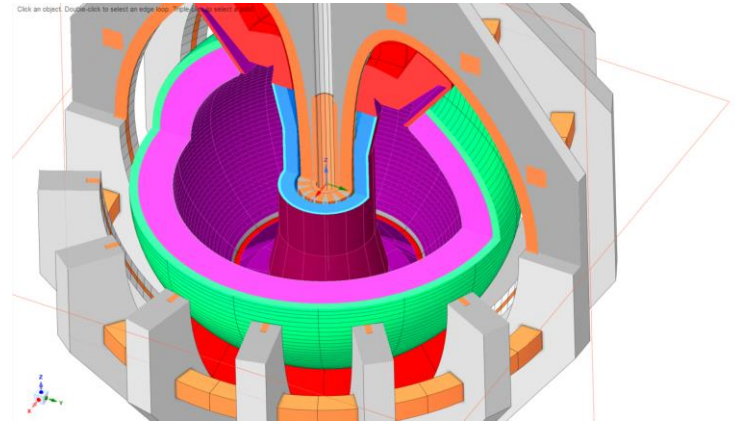
# This HfH project is providing the material basis towards an advanced shield solution for tokamak devices



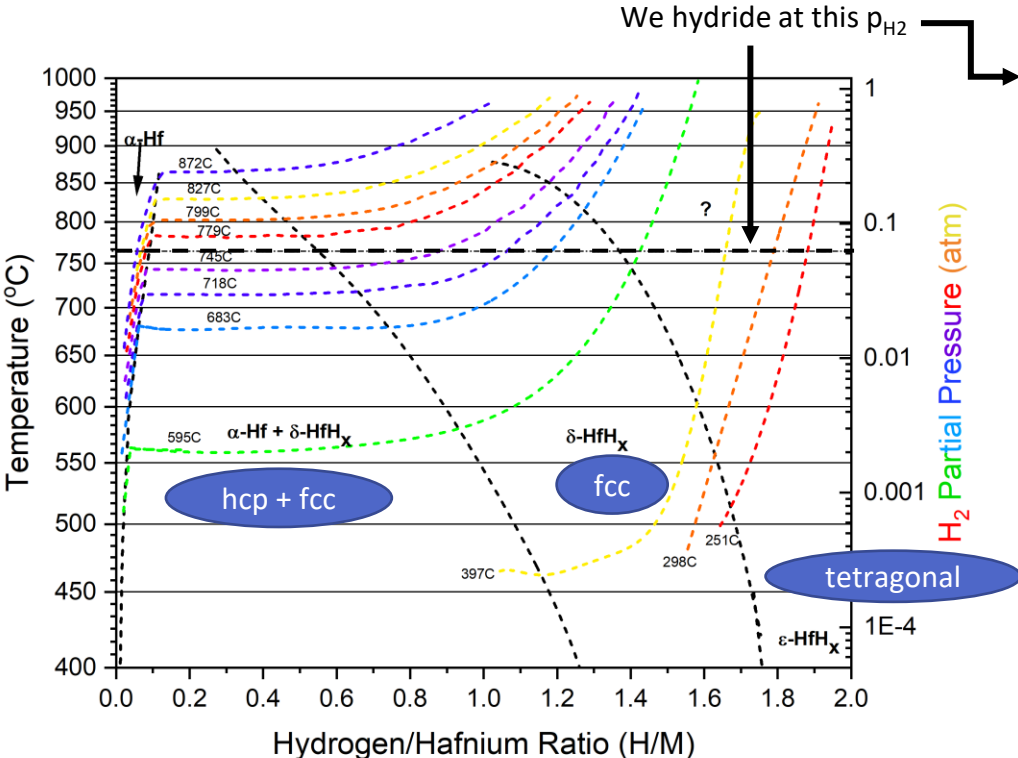


# Hafnium Hydride is becoming a key part of the shield strategy towards commercial tokamak devices

- Hafnium hydride can either be used to increase the lifetime of the HTS magnets which will decrease the total down-time which will decrease the levelized cost of electricity (LCOE)
- Alternatively, a more effective inboard shield can decrease the overall size of a fusion power plant which will decrease the levelized cost of electricity (LCOE).
- Tokamak Energy has an existing relationship with Imperial College London which we utilized for increasing our understanding of HfH
- Tokamak Energy's existing relationship with Bangor University was also utilized to gain an understanding on the Hydrogen accommodation within the lattice structure complementing previous literature



# Thermodynamics of hafnium hydride (HfH<sub>x</sub>)

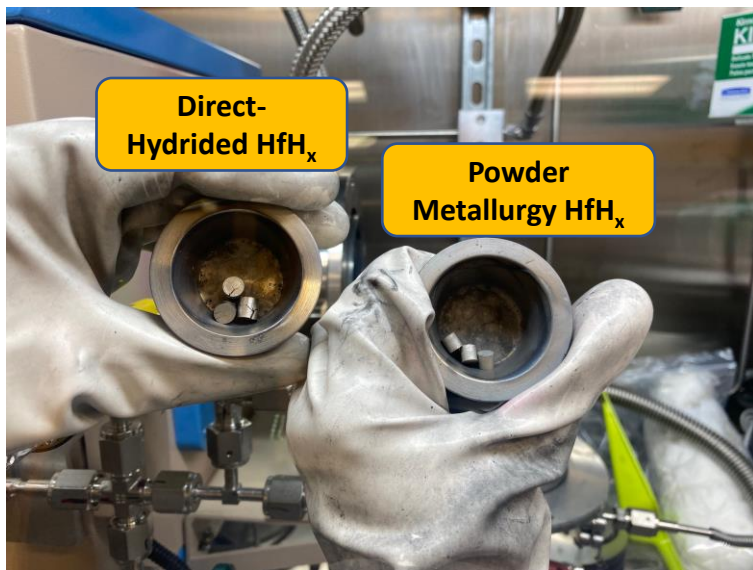


We look at where the isotherms intersect this line to choose a temperature to achieve a specific H/M

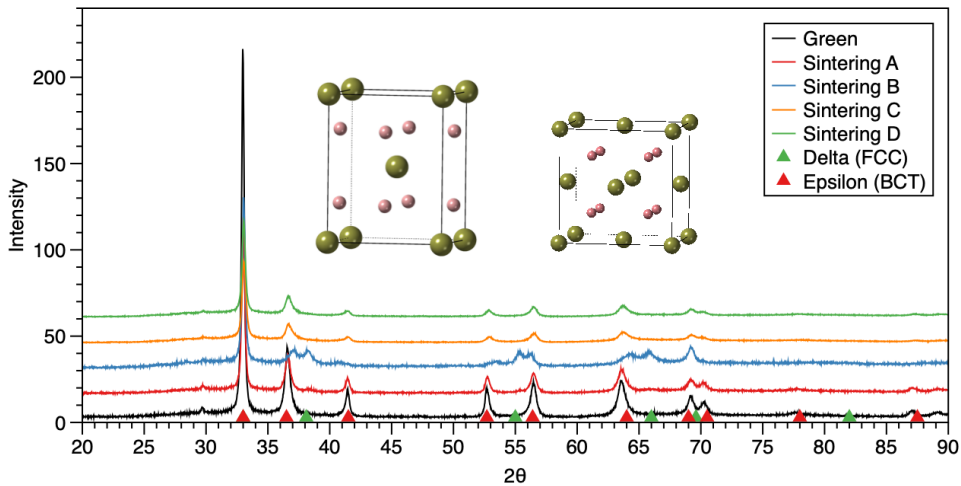
We fully hydride metals to form a single-phase metal hydride crystal structure

# Hafnium Hydride Fabrication

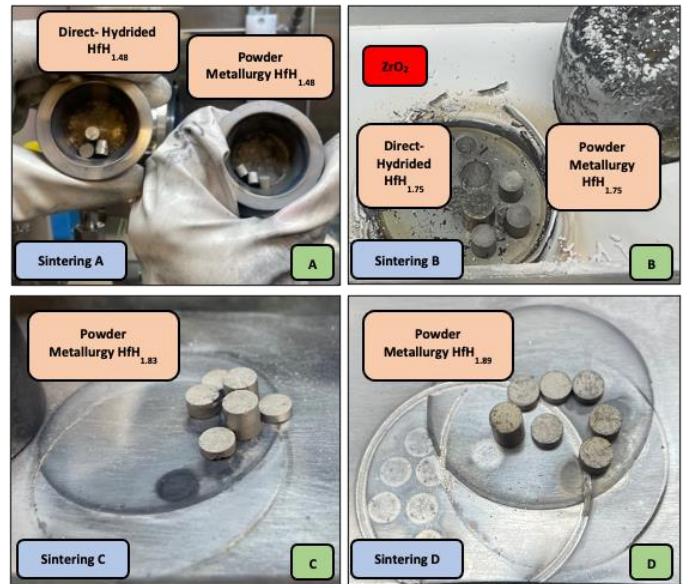
- We hydrided Hf feedstock material to  $\text{HfH}_{1.5}$ . Hf metal obtained from Kurt J Lesker (99.9% pure, excluding Zr. 2.96% Zr).
- Direct hydriding was difficult- significant amount of cracking
- Prepared pellets using a powder metallurgy process in a Ar glovebox and put through a single sinter+hydride cycle (patent pending) using a 1600°C furnace



# Single phase HfH<sub>x</sub> was achieved at various sintering temps

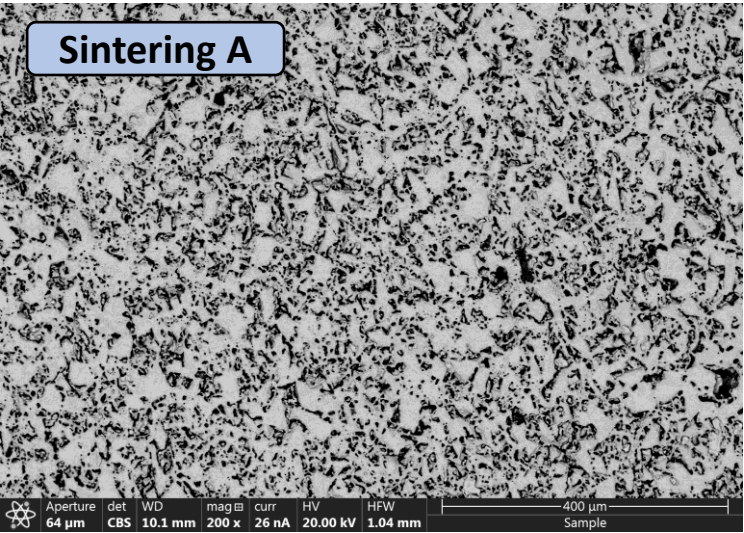


Sample	Theoretical Density (g/cm <sup>3</sup> ) [2, 3, 4, 5]	Density	Phase	H/Hf
Green Pellet	11.47	70.95%	Epsilon	1.52
Sintering A	11.47	71.80%	Epsilon	1.48
Sintering B	11.52	80.79%	Delta + Epsilon	1.76
Sintering C	11.38	84.08%	Epsilon	1.83
Sintering D	11.38	90.91%	Epsilon	1.89
Sintering E for Irradiation	11.38	90.46%	Epsilon	2.0

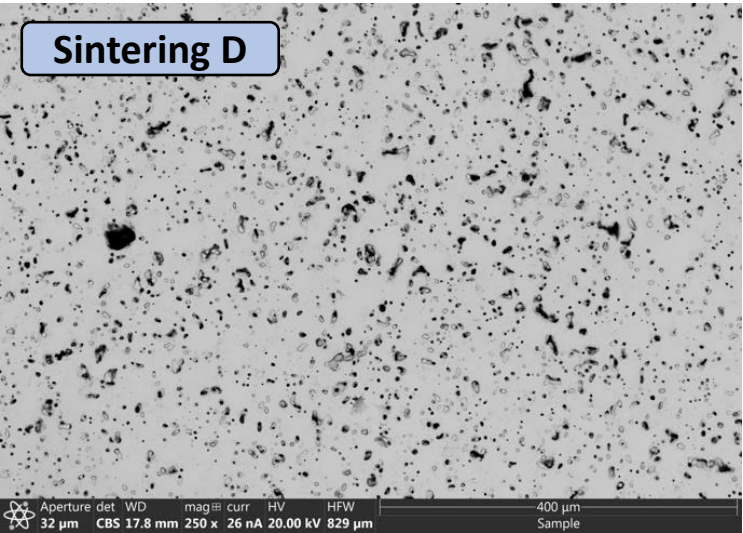




# Scanning electron microscopy confirmed density increase with sintering temperature



71.80% dense

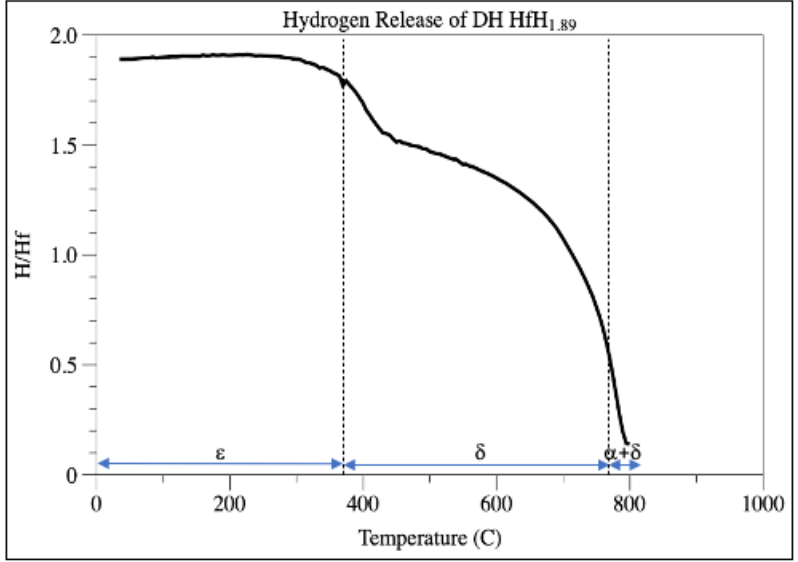
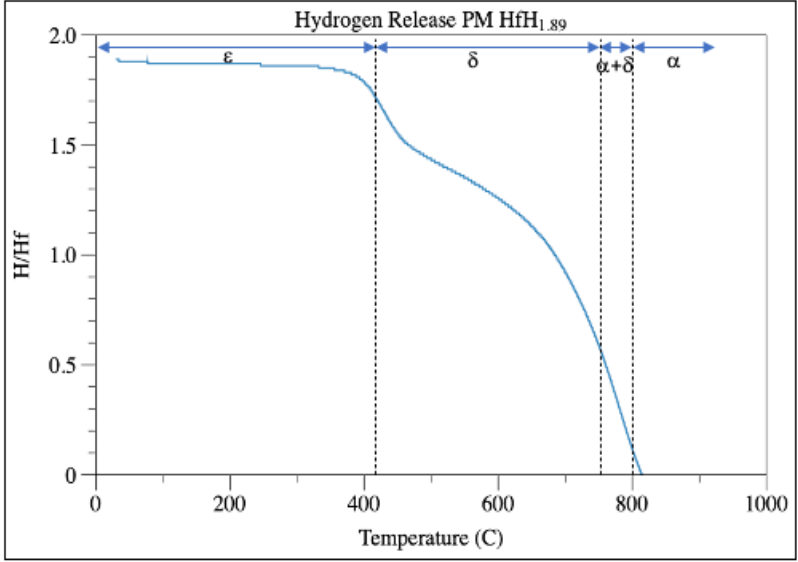


90.91% dense



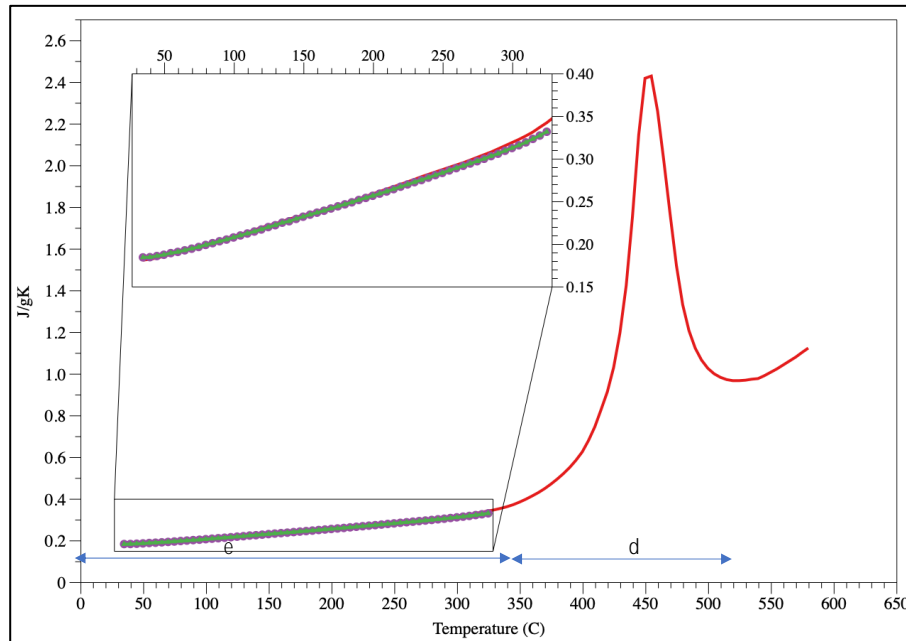
# Dehydrating measurements

- Hydrogen desorption as a function of temperature was measured using a Netzsch simultaneous thermal analyzer (STA) Model 449 F3 Jupiter.
- This analysis method measures mass change from thermal effects, which in these samples is assumed to be the desorption of hydrogen.
- The STA results show that the powder metallurgy sample remains in the epsilon phase up to a higher temperature (~415°C)



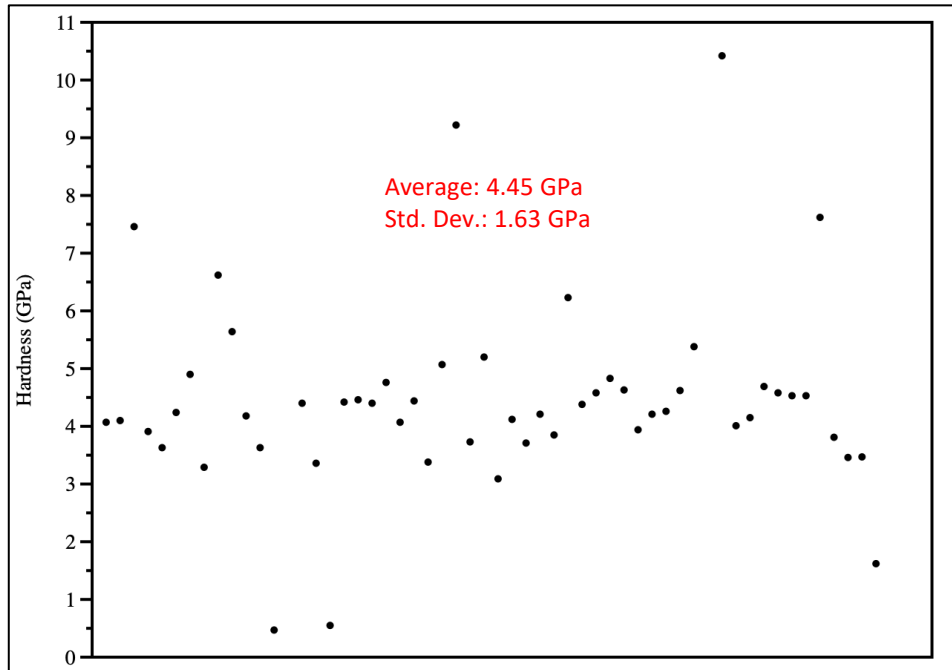
# Heat Capacity

- Heat capacity measurements were acquired by differential scanning calorimetry (DSC) using a Netzsch DSC Model 404 F1 Pegasus.
- Heat capacity rose at a consistent rate within the epsilon phase (up to 330°C). As the sample changes from epsilon phase to delta phase at higher temperature, a Bredig transition was observed



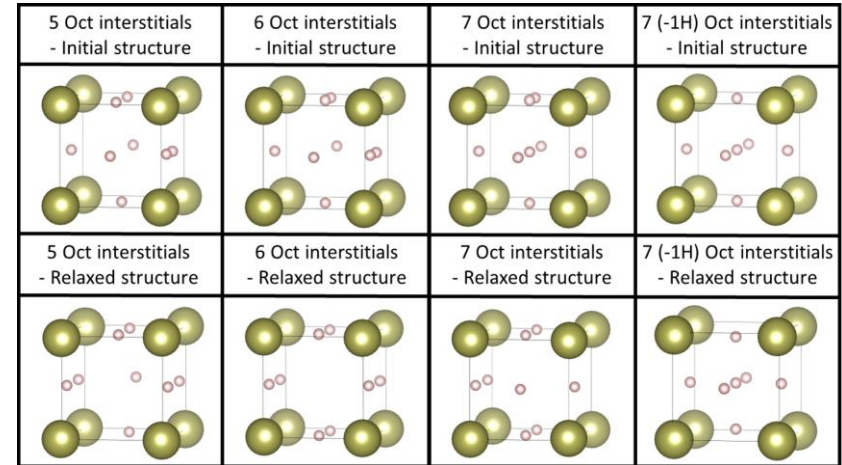
# Hardness measurements

- Nanoindentation tests were performed on the HfH<sub>1.89</sub> samples with a Keysight G200 Nanoindenter using a diamond Berkovich tip to a final displacement of 400 nm with a constant strain rate of 0.05 s<sup>-1</sup>. Continuous stiffness measurements (CSM) were applied at a frequency of 45 Hz with 2 nm displacement amplitude. Hardness measurements were determined using the Oliver-Pharr method.



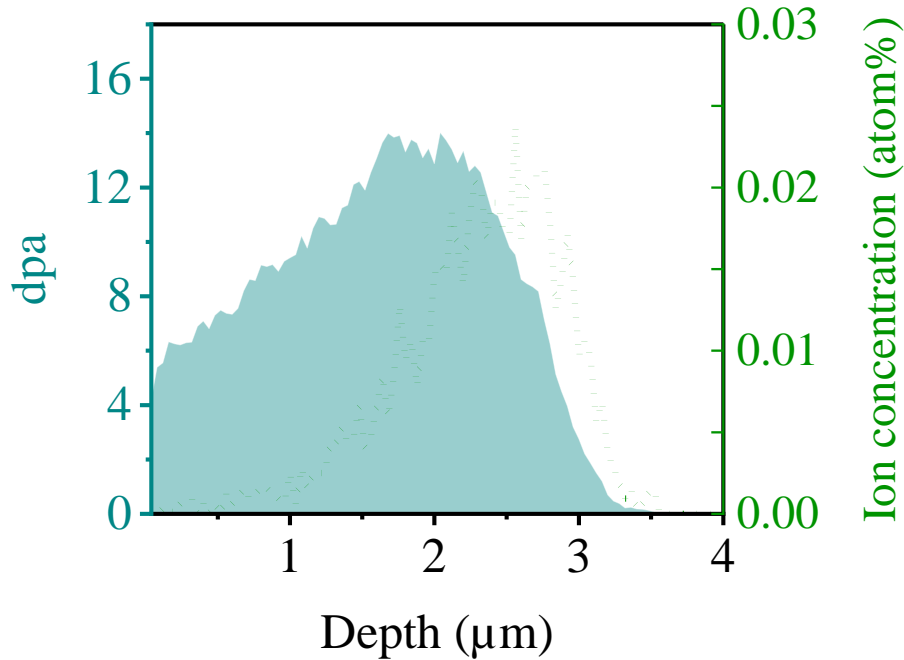
## Hydrogen accommodation was simulated to model the interactions between hydrogen interstitials and monovacancies.

- Density functional theory was used to simulate hydrogen accommodation in  $\epsilon\text{-HfH}_x$
- Hydrogen atoms were found to preferentially occupy the tetrahedral holes in the crystal structure
- The presence of monovacancies, and the stabilizing effects of a hydrogen atom positioned on a lattice site, leads to a shift in preferred interstitial position
- The extent of the hydrogen clustering that can be achieved in a  $\epsilon\text{-HfH}_x$  monovacancy was not fully explored



The initial and fully relaxed structures for the DFT calculations. The top row illustrates a selection of the explored arrangements with varying numbers of interstitials placed on octahedral sites. The bottom row highlights that only the final arrangement (right) demonstrated octahedral interstitials in the fully relaxed system.

## Hafnium Hydride samples sent to Imperial were irradiated with 20MeV Au Ions for preliminary analysis

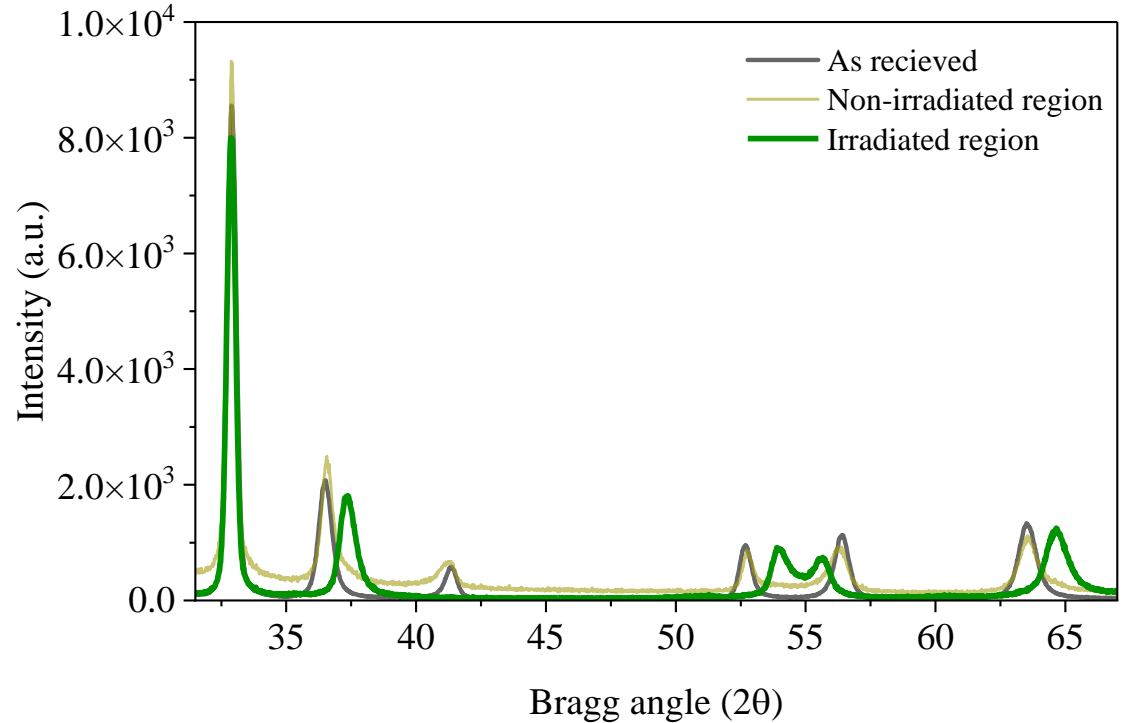


- Gold ion irradiation campaign of  $\epsilon$ -HfH<sub>2</sub> using 20 MeV Au ions at DCF accelerator. (SRIM calculations shown)
- Samples were masked, allowing the irradiated region to the non-irradiated region to be compared under the same conditions



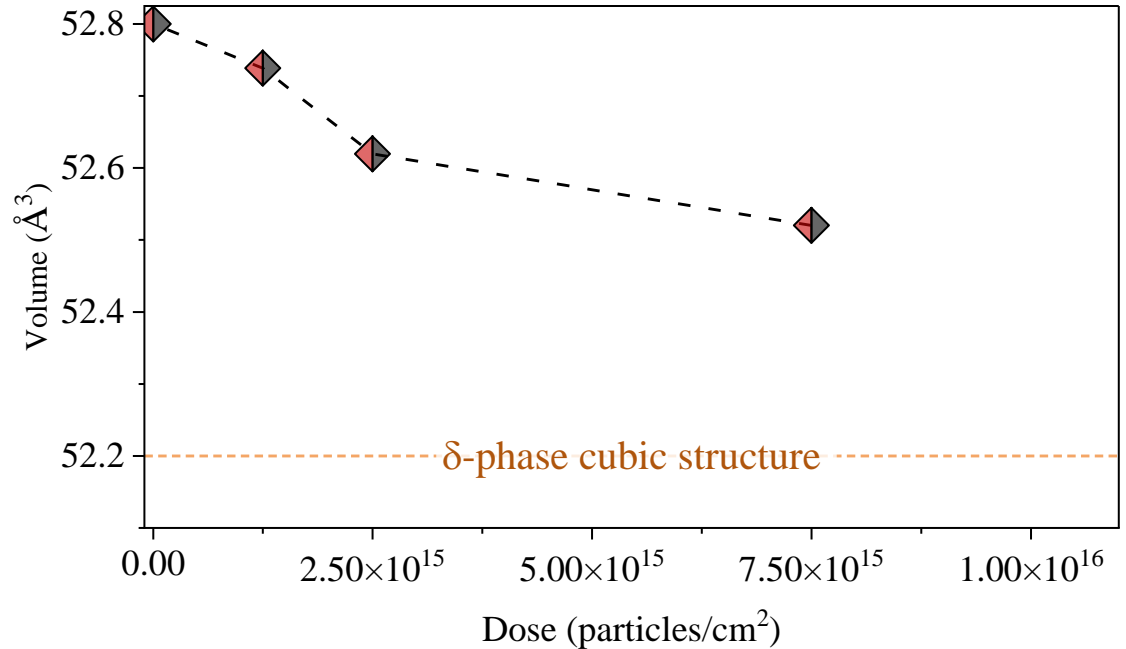
## Ion irradiation on $\epsilon$ -HfH<sub>x</sub> shows a phase change towards $\delta$ -HfH<sub>x</sub>

- XRD pattern by grazing incidence. To probe only the damaged region (~2 micron)
- Irradiated region (green) shows a crystal structure change from tetragonal (epsilon) phase towards a cubic (delta) phase. The non-irradiated region (gold) is unchanged, so the effect is not from beam heating.



## Ion irradiation on $\epsilon$ -HfH<sub>x</sub> shows a phase change towards $\delta$ -HfH<sub>x</sub>

- The change in structure is a strong function of the dose.
- The initial tetragonal phase moving more towards a cubic structure
- Small amounts of the  $\delta$ -HfH<sub>1.6</sub> phase is forming indicating hydrogen loss as a result of irradiation.



## $\epsilon$ -HfH<sub>x</sub> shows good agreement with literature on thermal diffusivity

- Thermal property experiments are being ran on the Hf-H samples using laser flash analysis
- Initial results on epsilon phase show good agreement with literature.
- Experiments in future will probe delta-phase to higher temperature than reported in the literature.

