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Permeation Experiments at SNL-CA

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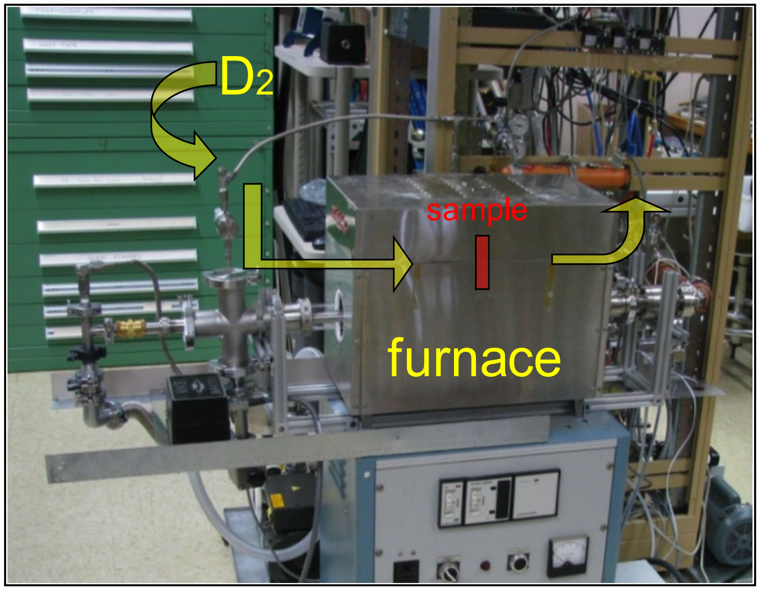
Permeation Experiments at Sandia California

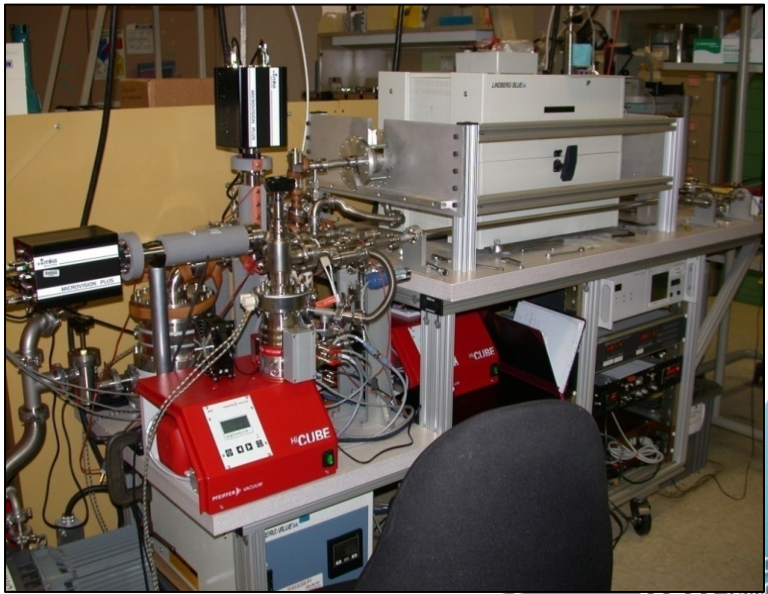
- **Deuterium gas driven permeation capabilities in use at SNL**
  - 1st generation (150 < T < 500 °C) used stainless steel construction (VCR seals), evacuated quartz outer tube to reduce D_2 bypass, and low flow to prevent surface contamination
  - 2nd generation (50 < T < 1150 °C) uses Al_2O_3 construction and soft, pressure loaded seals for brittle specimens (funded by "Work For Others" program to measure SiC permeation barriers for fusion blankets)

ULTRAMET ADVANCED MATERIALS SOLUTIONS

$P_{SiC} < 10^{-12}$ mol H^2 m^{-1} s^{-1} $MPa^{-0.5}$



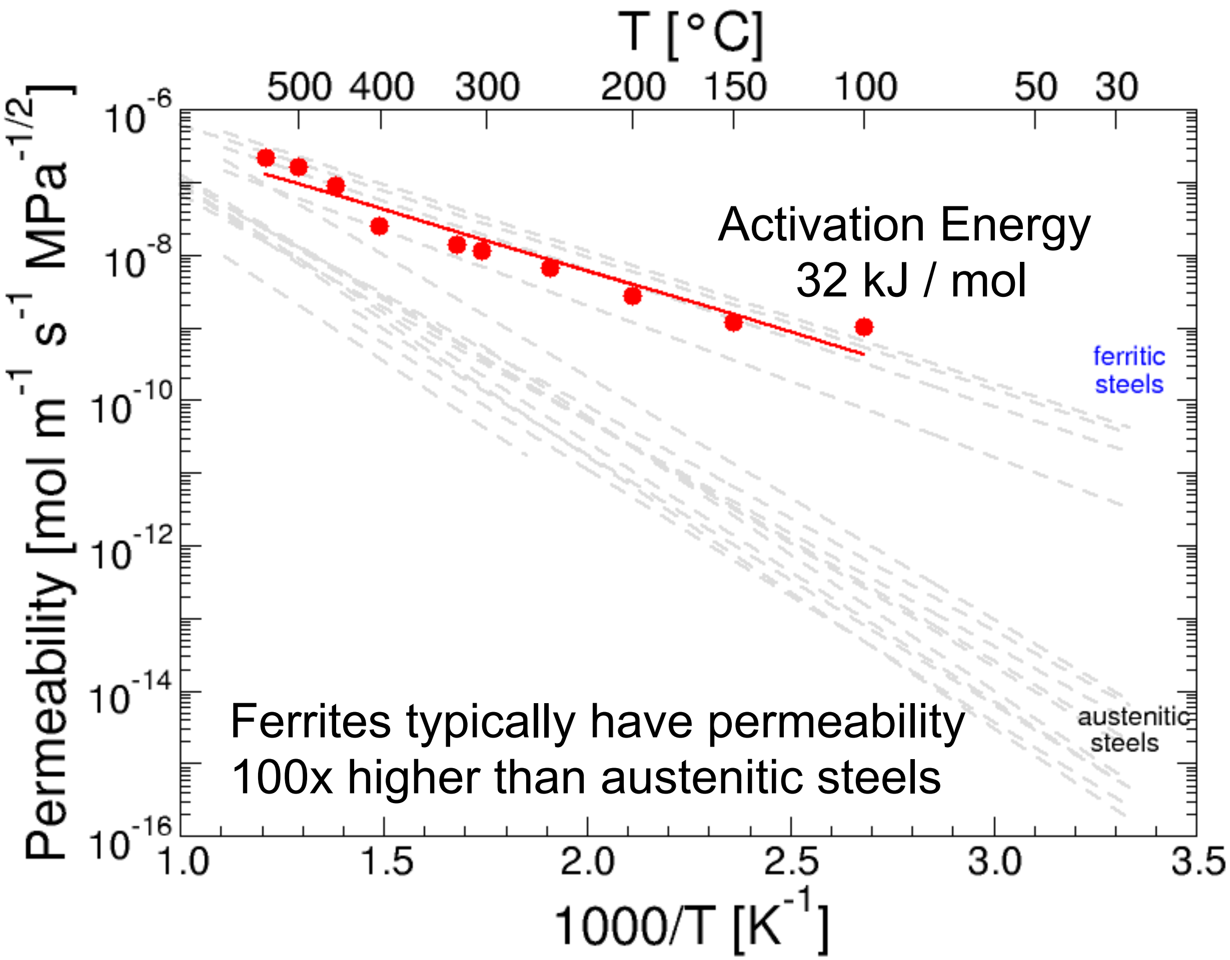
1st Generation System



2nd Generation System

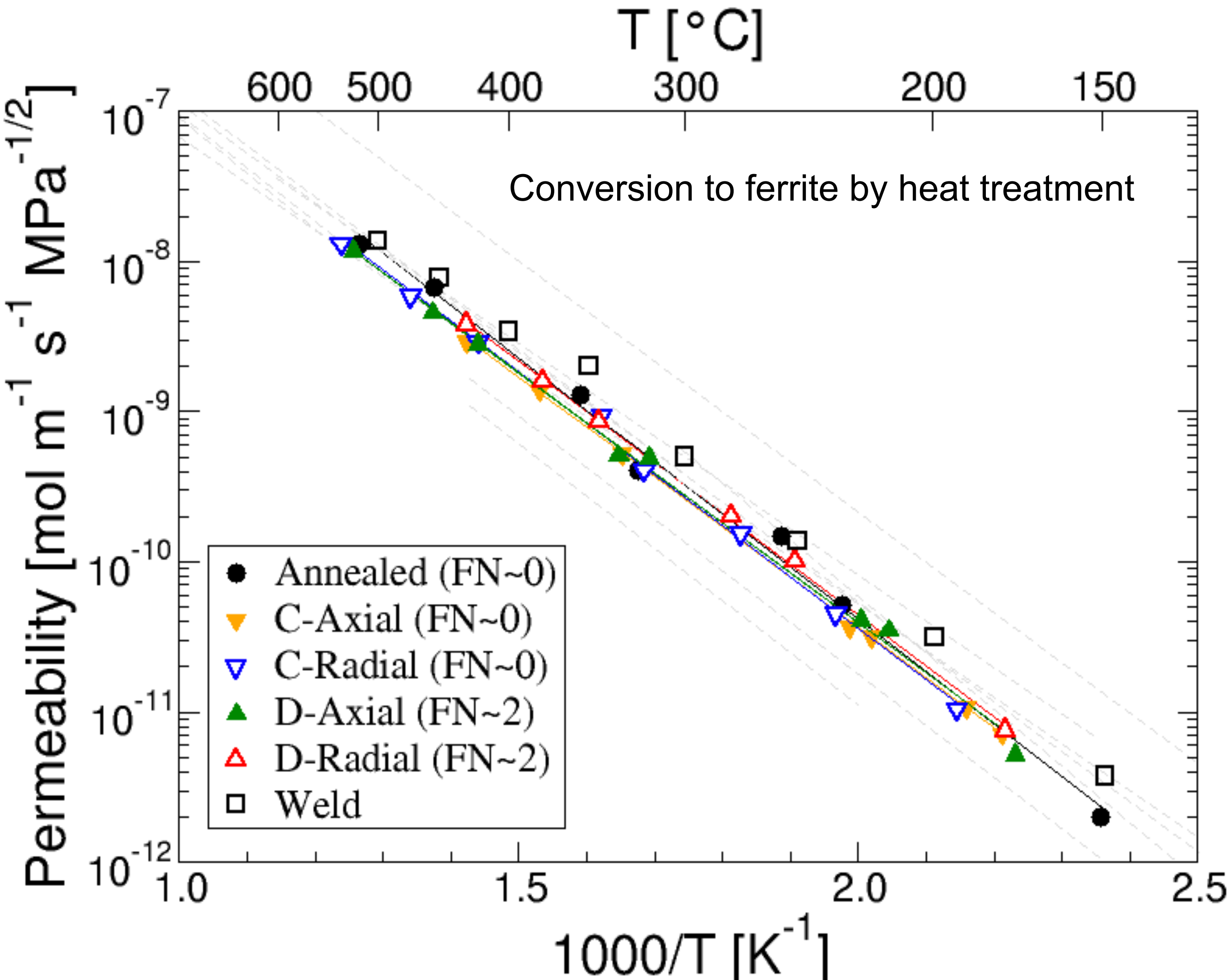
Gas Permeation of 4130X Steel (fully ferritic)

- **4130X permeation measured in the low temperature permeation system**



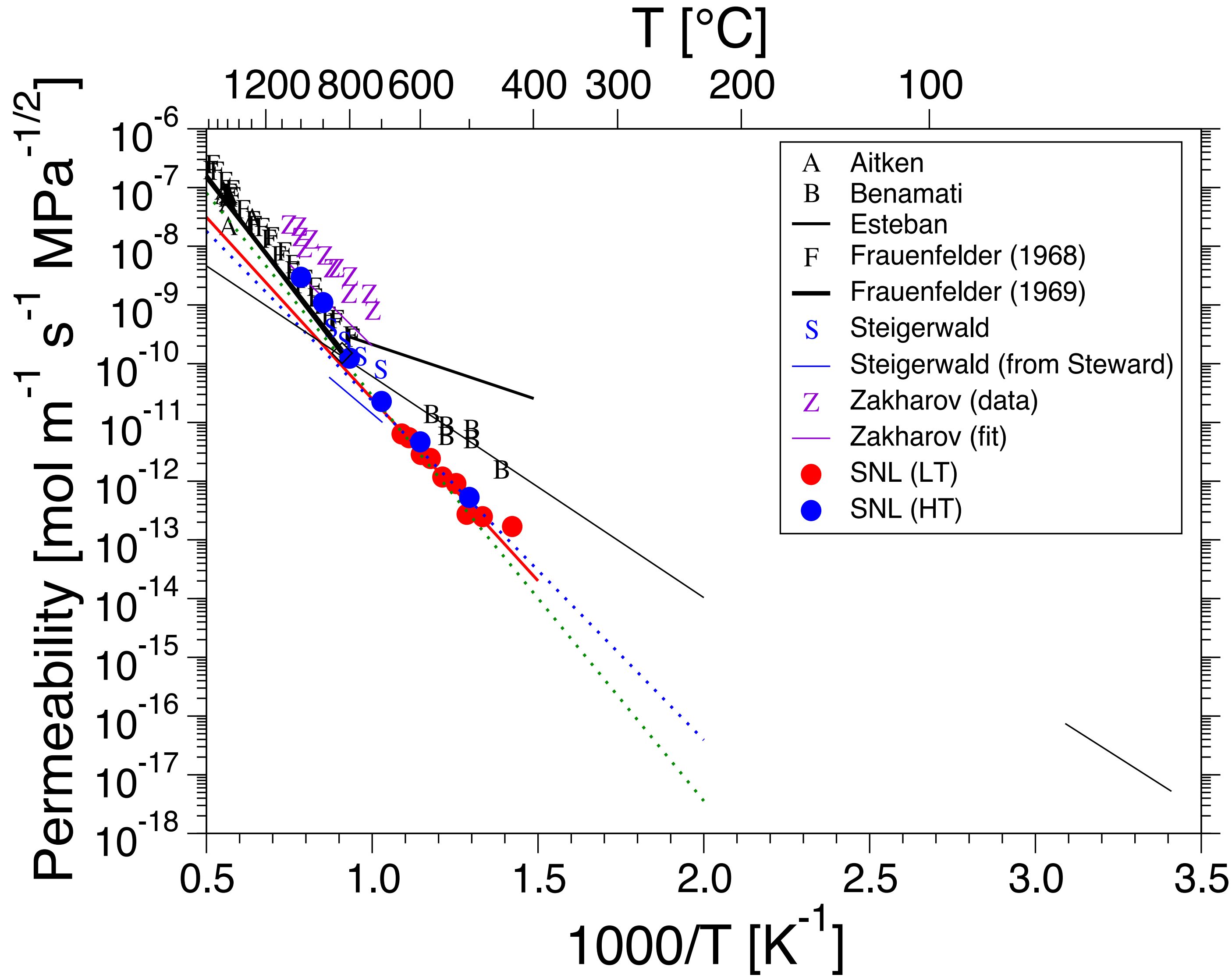
Gas Permeation of Austenitic Steel (and welds)

- **Example of influence of ferrite on permeation (21-6-9 steel)**



Gas Permeation in Tungsten

- **Permeation in tungsten foil in good agreement with literature values. SNL experiments performed to lower temperatures and in two systems.**



ITER Grade Tungsten & VPS-Tungsten

- **Experiments in August and September 2013 were challenging due to brittle samples (grain structure elongated through the foils)**
  - JA: 3× 50 µm thick ITER grade W (sized for use in HiFIT)
  - JA: 1× 730 µm thick ITER grade W
  - US: 2× 500 µm thick ITER grade W
- **Mounting of three samples in the low temperature (LT) system (Cu gaskets)**
  - 50 µm (2-side polish) fractured during pump down
  - 500 µm (upstream polish) fractured along the copper gaskets
  - 500 µm (not polished) would not pump down
- **Mounting of two samples in the high temperature (HT) system (Grafoil gaskets)**
  - 50 µm (2-side polish) fractured during pump down
  - 730 µm (2-side polish) mounted and used for permeation calibration
- **Equipment issues identified**
  - Downstream rough pump on the LT system repaired
  - Leaking o-ring on the butterfly valve found (used to control downstream pressure on the LT system)
  - Gas bottle leak found on the LT system (repaired)
  - Deuterium bottle pressure regulator replaced (HT system)

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  - JA: 3x 50 µm ITER grade W (sized for use in HiFIT)
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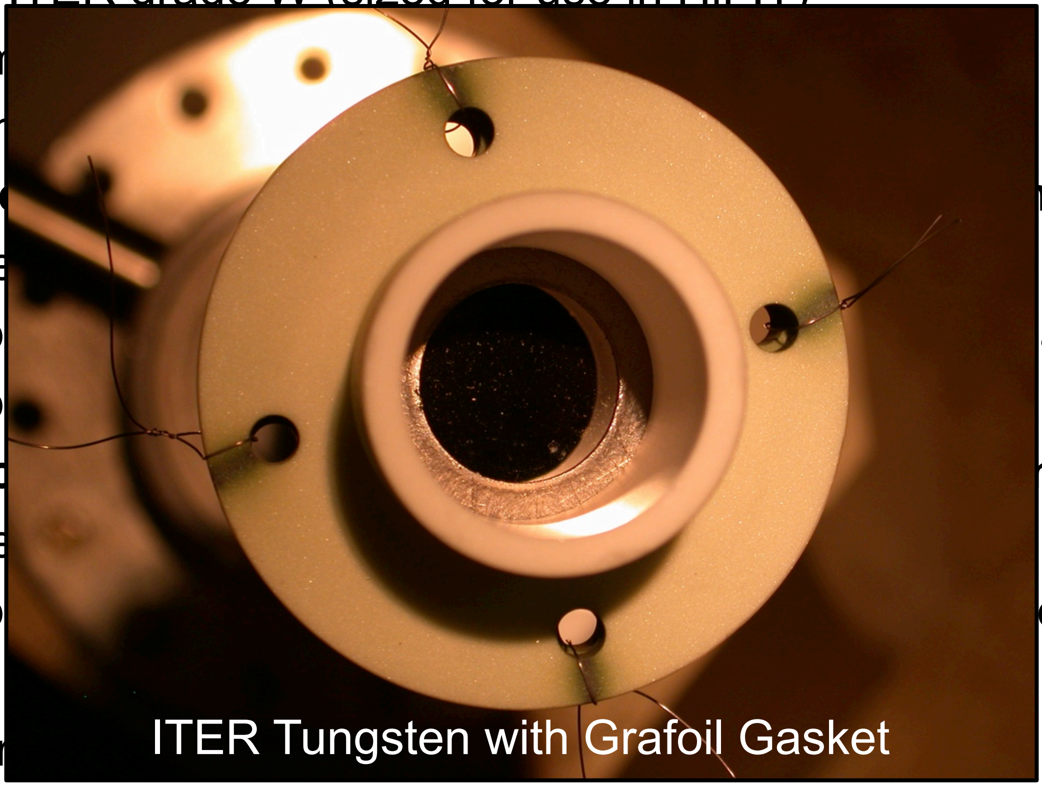
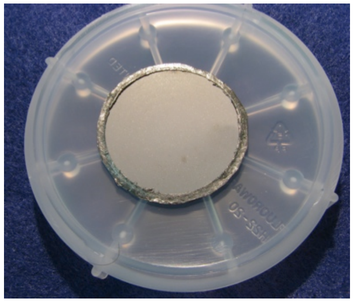
  - US: 2x 500 µm
- **Mounting of thr… m (Cu gaskets)**
  - 50 µm sample
  - 500 µm samp… askets
  - 500 µm samp
- **Mounting of two… n (Grafoil gaskets)**
  - 50 µm sample
  - 730 µm samp… on calibration
- **Equipment issu**
  - Downstream
  - Leaking o-ring on the butterfly valve found (used to control downstream pressure on the LT system)
  - Gas bottle leak found on the LT system (repaired)
  - Upstream deuterium bottle pressure regulator replaced (HT system)



ITER Tungsten with Grafoil Gasket

ITER Grade Tungsten / VPS-Tungsten

- **US FY14 fusion funding delay was compounded by several issues in 2014**
  - While our lab moves in 2013 didn't directly affect the permeation equipment, the relocation of laboratories forced a complete revision of our safety documentation (along with a new layer of engineered safety).
  - Funding for non-fusion permeation work was also delayed (January – June)
  - Problem with facility power led to 3 outages; difficult to plan for multi-week permeation experiments
- **Additional samples were fabricated**
  - JA: 2× 500 μm thick VPS-W on 500 μm thick F82H
  - US: 7× 500 μm thick ITER grade W
  - US: 4× 500 μm thick VPS-W on 500 μm thick F82H (being polished)



- **Mounting of seven samples in the high temperature (HT) system (new centering)**
  - 500 μm VPS-W / 500 μm F82H (2-side polish) fractured under loading
  - 730 μm (2-side polish) fractured under loading (previously ok)
  - 430 μm (2-side polish) fractured under loading
  - 1 mm (upstream polish) fractured (loading & o-ring / alumina tube change)
  - 500 μm (upstream polish) fractured after system baking and T ramp to 500 °C
  - 500 μm (upstream polish) with fresh Grafoil fractured after T ramp to 550 °C

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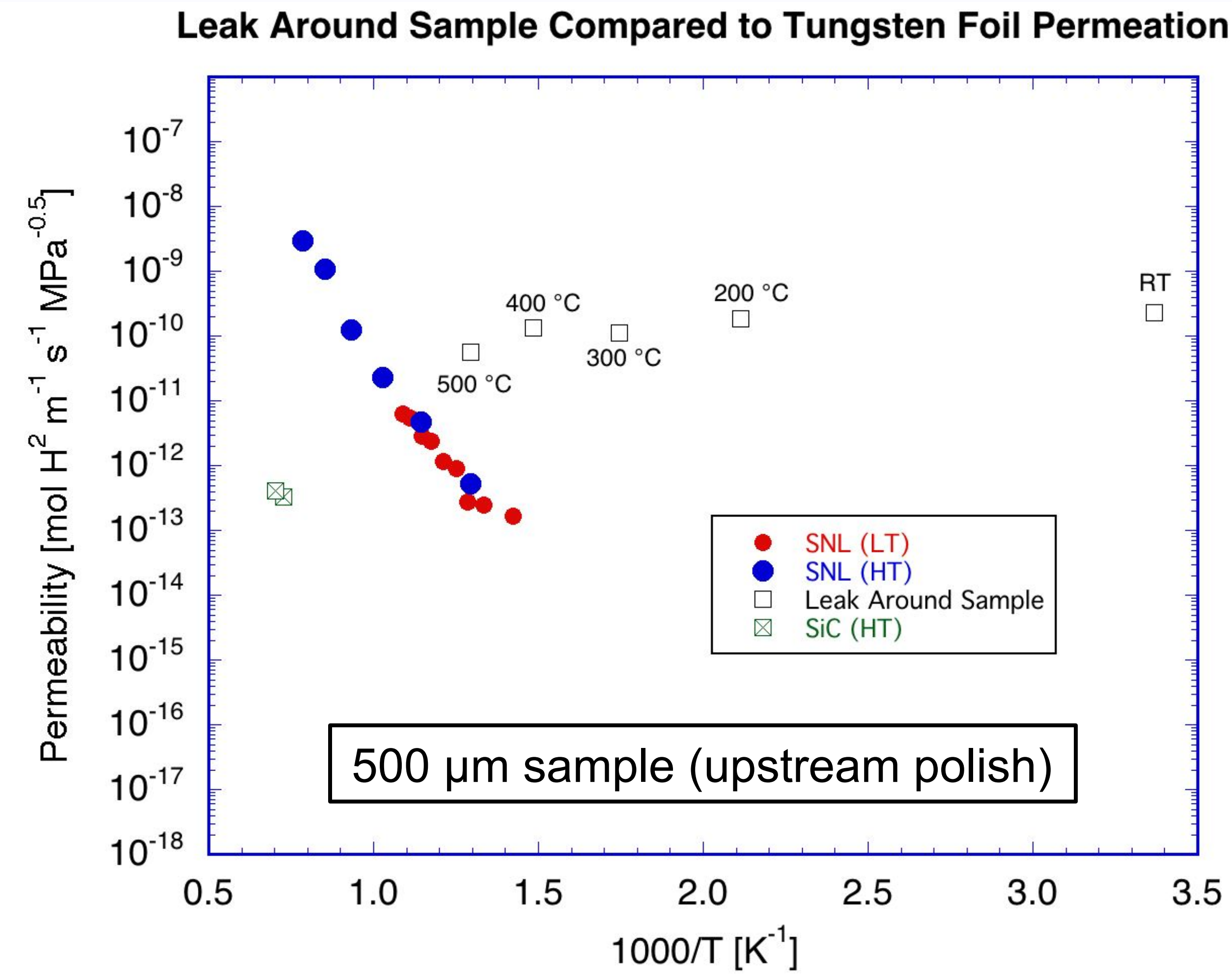
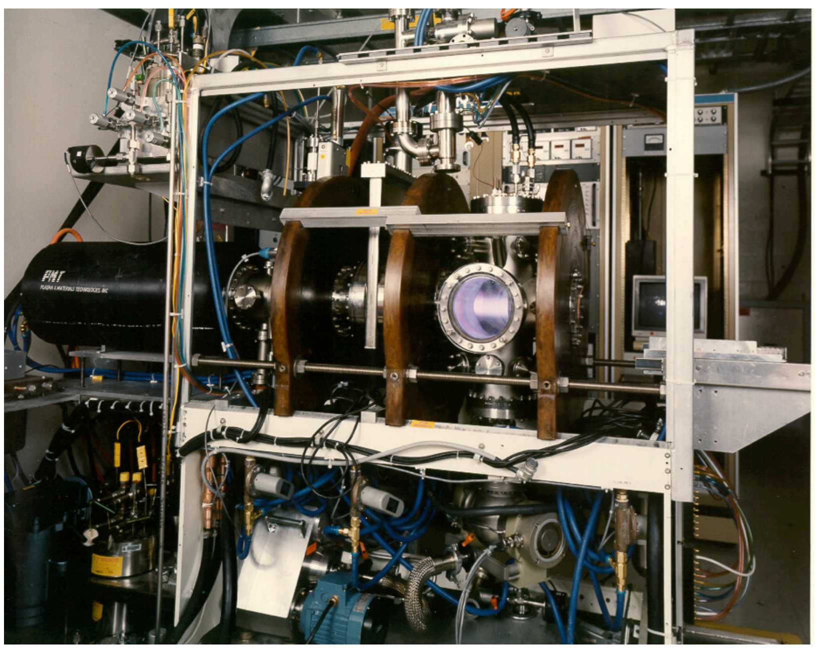
Leak rate change with increasing temperature

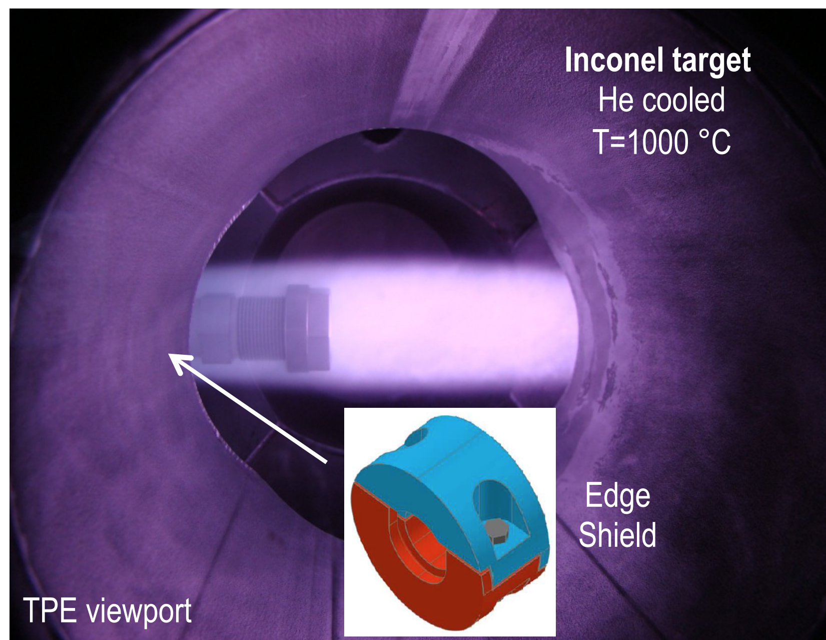


- **Estimate of thermal expansion in hot zone ➔ 0.054 inch expansion. Grafoil gaskets are 0.030 inch thick each must compress due to rigid loading structure**
- **Presently a 960 µm sample (2-side polish) is mounted and will be T ramped without loading**

Tritium Plasma Permeation Measurements

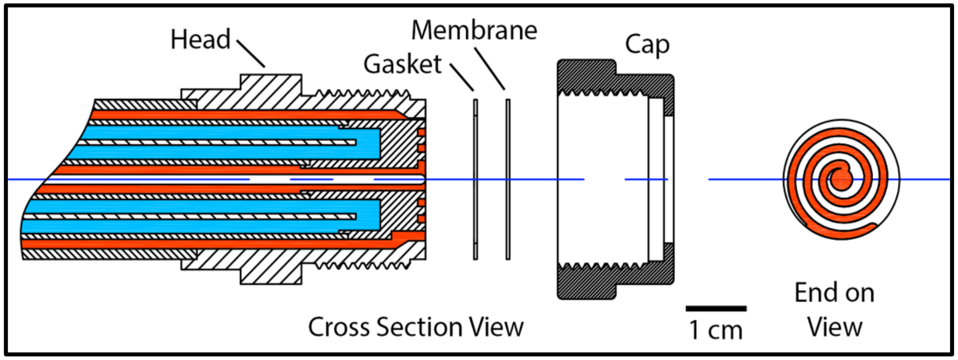
- **Initial experiments using a high temperature tritium retention stage are in progress on TPE (using deuterium)**
  - Stable operation at 800 °C for 1 hour (1000 °C for shorter times)
  - Feedback control of He flow to be implemented to reduce thermal ramp up time (now ~ 30 minutes)
- **Experience on this stage and from gas permeation experiments at SNL-CA aided in the design of a permeation membrane holder**





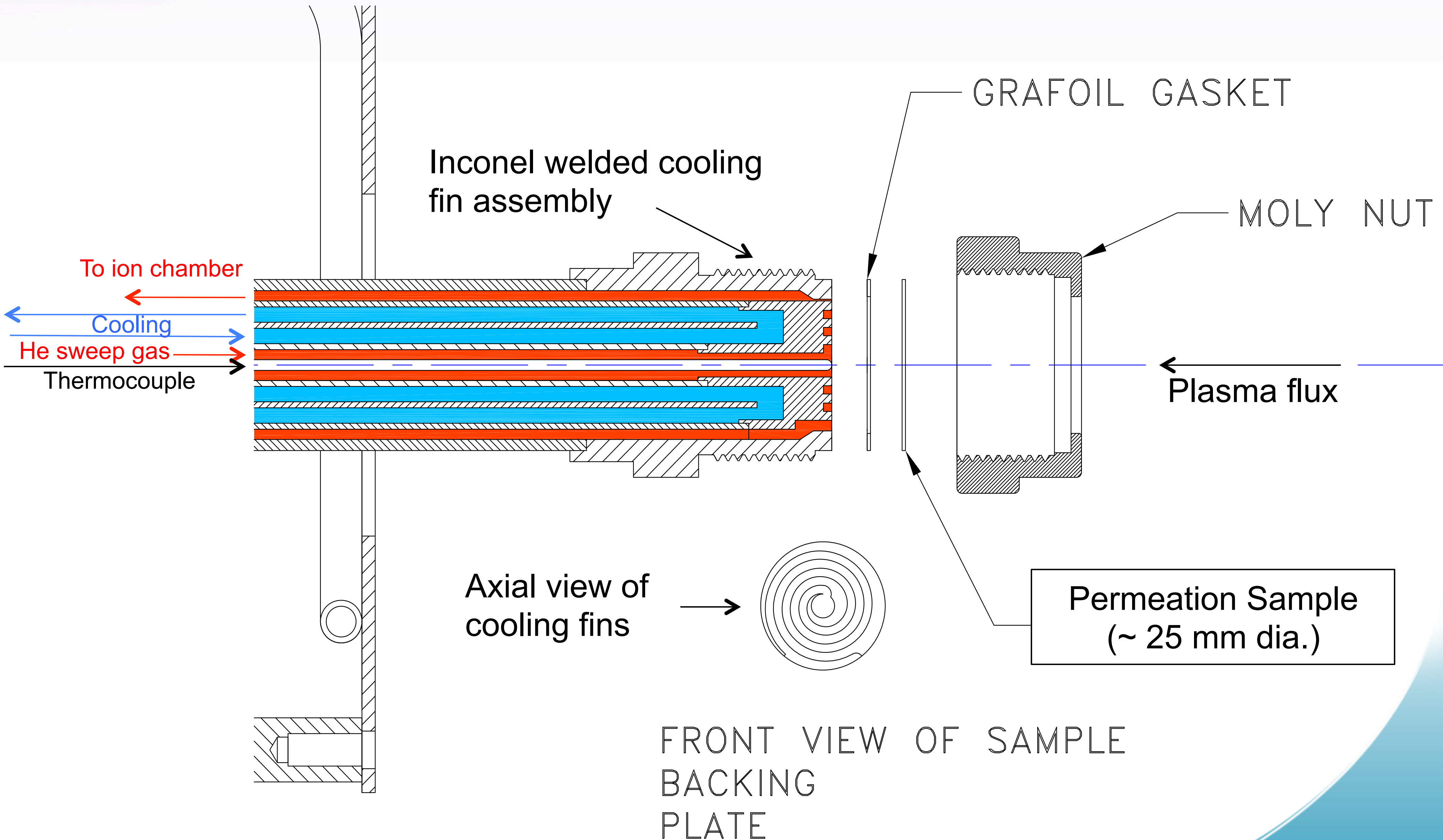
Design of Tritium Permeation Experiment

- **New permeation membrane holder is ready for integration in TPE**
  - He carrier gas to capture permeating D/T between cooling fins and sample
  - Membrane sealing has been demonstrated to 1000 °C
  - High pressure bellows controls axial position of spiral cooling fin
  - Modeling: He carrier gas flow shows low p drop; thermal transport through He gas up to maximum TPE flux



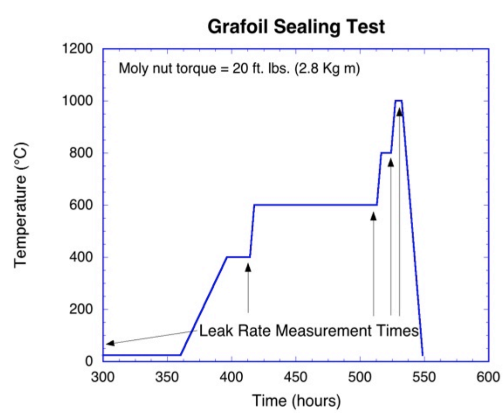
Membrane Holder Photos

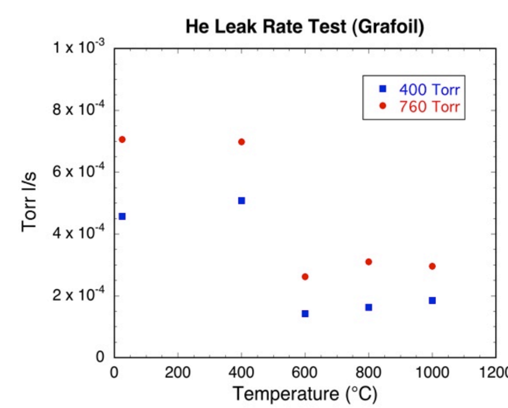
Expanded View of Sample Region



Design Considerations

- **Estimate of permeation flux taken from HiFIT* (ion beam permeation)**
  - Observed 10^{10} D $cm^{-2}s^{-1}$ to 10^{13} D $cm^{-2}s^{-1}$
  - Account for TPE membrane size (8×) and 4-10× thicker membranes
  - Account for TPE 100× higher flux, but use of 1% T
  - Implies TPE permeating current of 2.4×10^{-10} Ci/s to 2.4×10^{-7} Ci/s
  - For a 1000 sccm helium carrier gas flow and 1000 cm^3 ion chamber, this implies 1.5×10^{-5} Ci/m^3 to 1.5×10^{-2} Ci/m^3 (60 s resonance time)
  - Fits within the 4 decade range of a controller (1 μCi/m^3 to 10000 μCi/m^3)
- **Sealing tests have been used to demonstrate acceptable leak He leak rate and sequestration of He by the TPE pumping system**
  - Max leak < 0.5% of TPE D_2 fueling
  - Ionization of He: $He^+/D^+ \sim 6\times10^{-5}$
- **Break through times can be strongly affected by trapping**
- **Brittle membranes require careful design to protect the TPE pumping system**

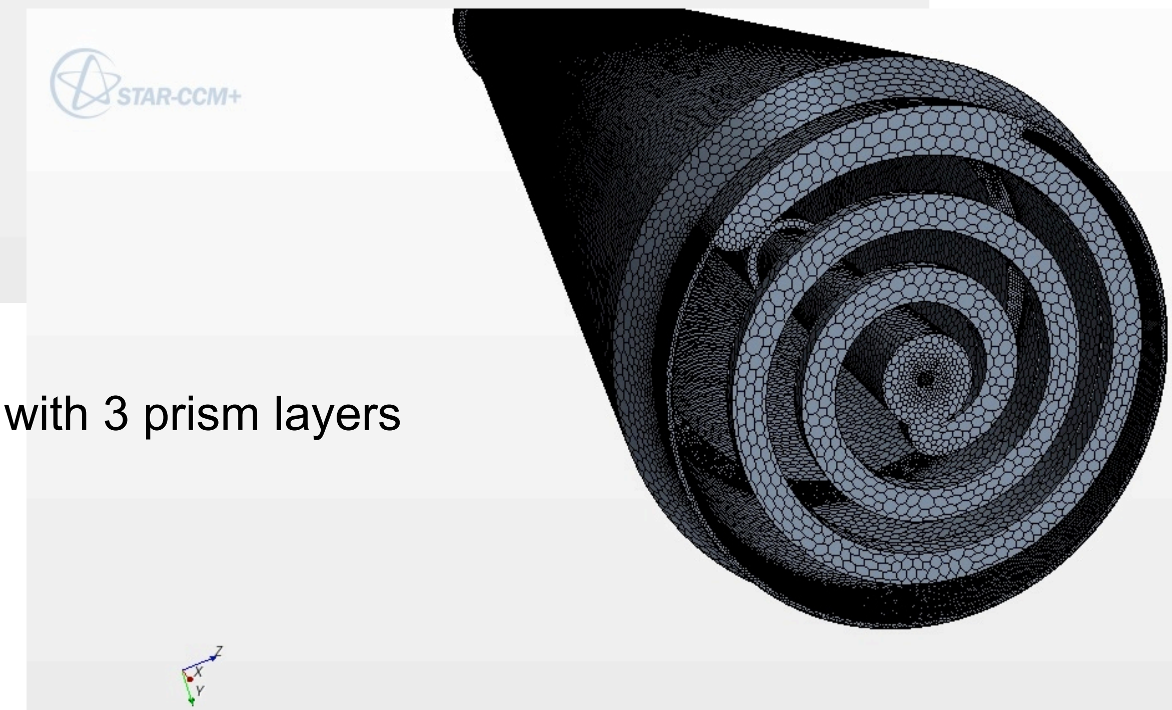




*H.T. Lee, et. al., J. Nucl. Mater. 390-391 (2011) S696.

Modeling of Membrane Holder

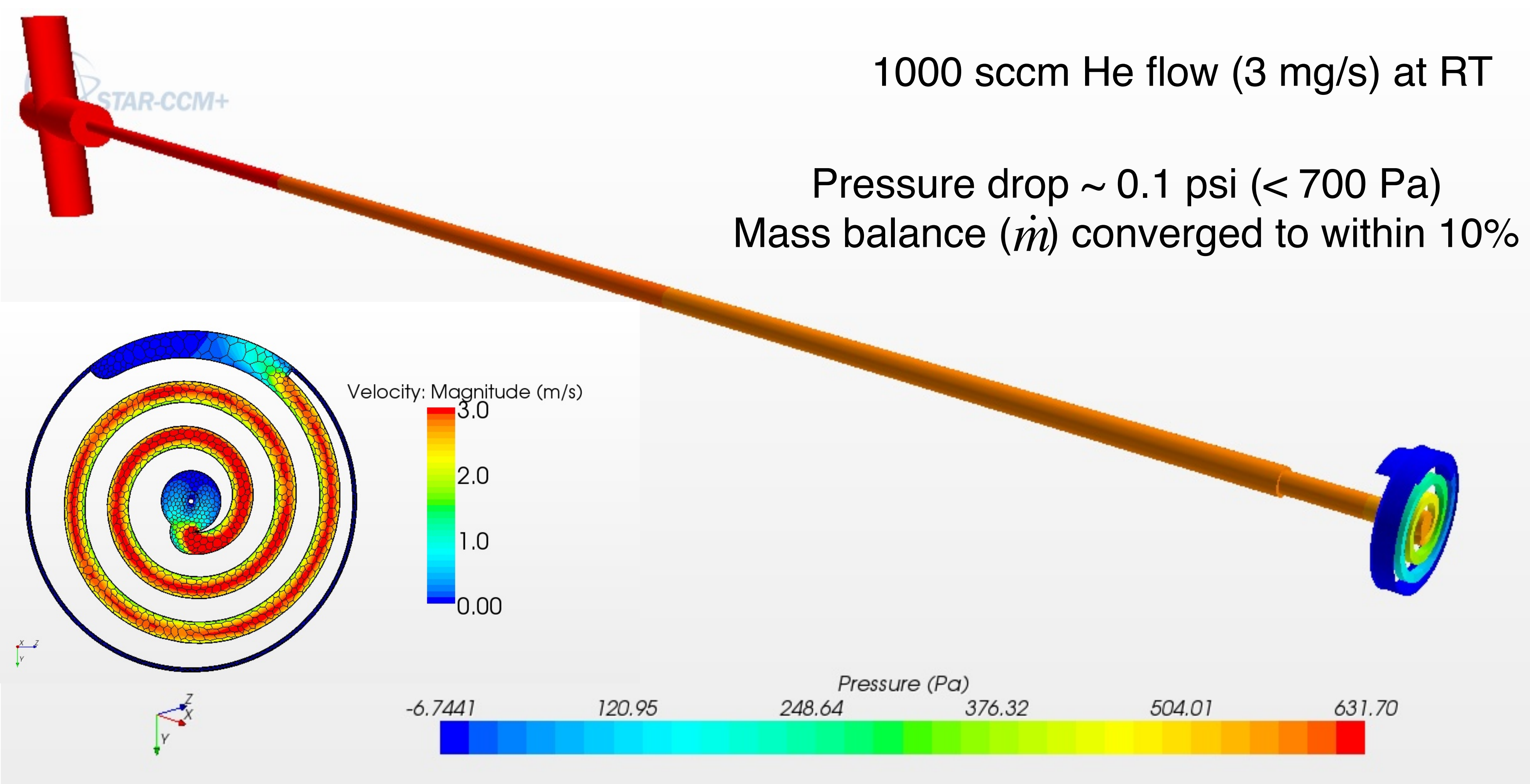
- Helium carrier gas flow and heat transfer were modeled using computational fluid dynamics (STAR-CCM+)
- Low conductance of He inlet and spiral fin structure motivated flow simulation



631 k polyhedral cells with 3 prism layers

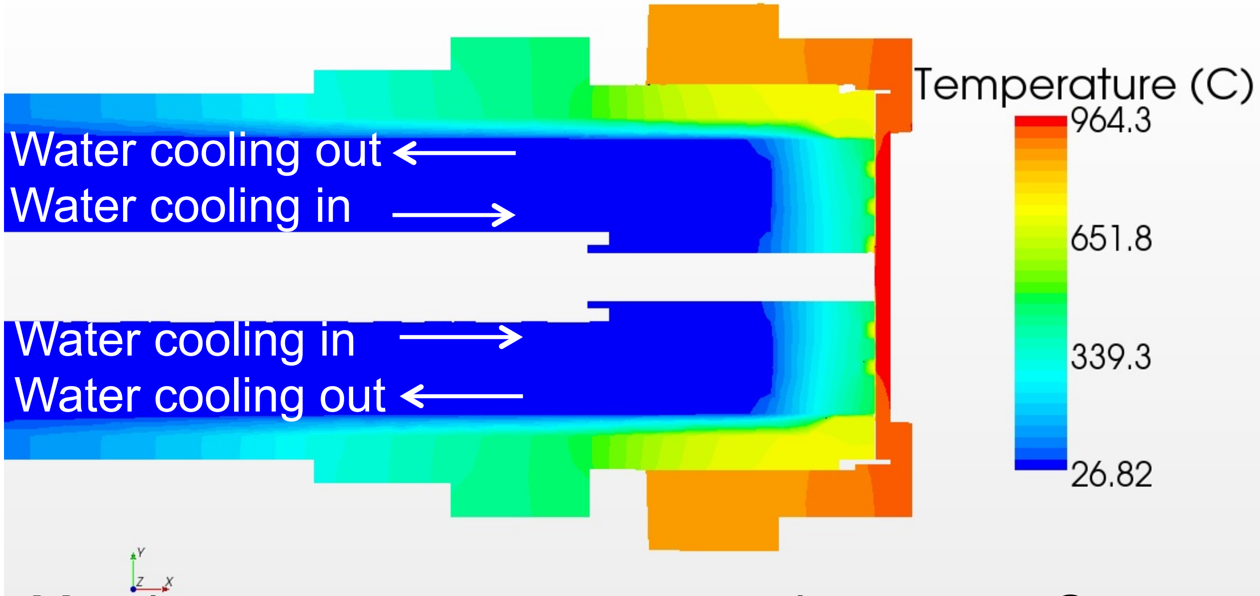
Minimal Pressure Drop Realized

- **Smooth flow pattern observed through the spiral fin (< 3 m/s)**



Thermal Transport Modeling

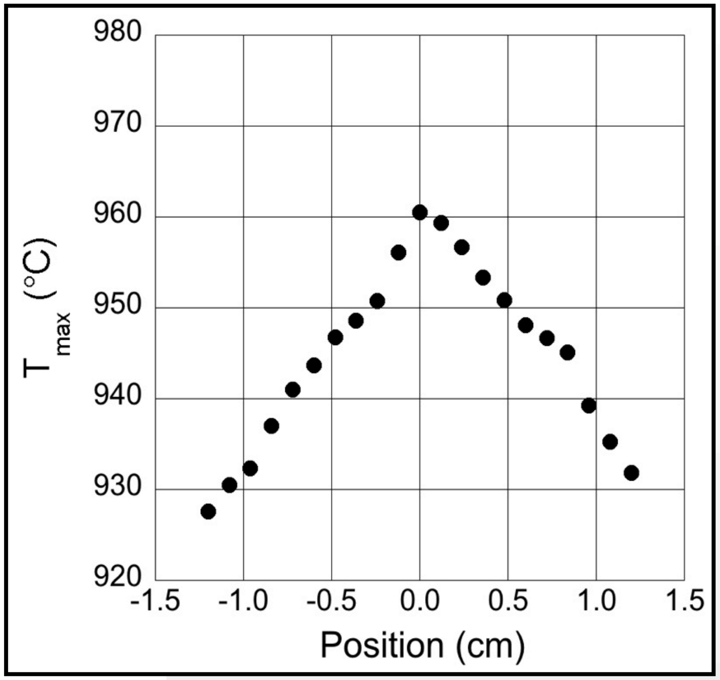
- **Reference case used is the highest TPE ion flux (2.5×10^{22} D $m^{-2}s^{-1}$) and maximum sample bias (-200 V) ➔ q" = 0.8 MW/m^2**
- **Radiation is included from surfaces to a 300 K background**
- **Molybdenum cap**
  - Thread engagement area and 10^{-3} m^2K/W contact resistance
- **Membrane**
  - Membrane - cooling fin gap set to 75 μm
  - Membrane thickness = 1 mm
- **Water cooling**
  - 10 gpm (0.63 kg/s)



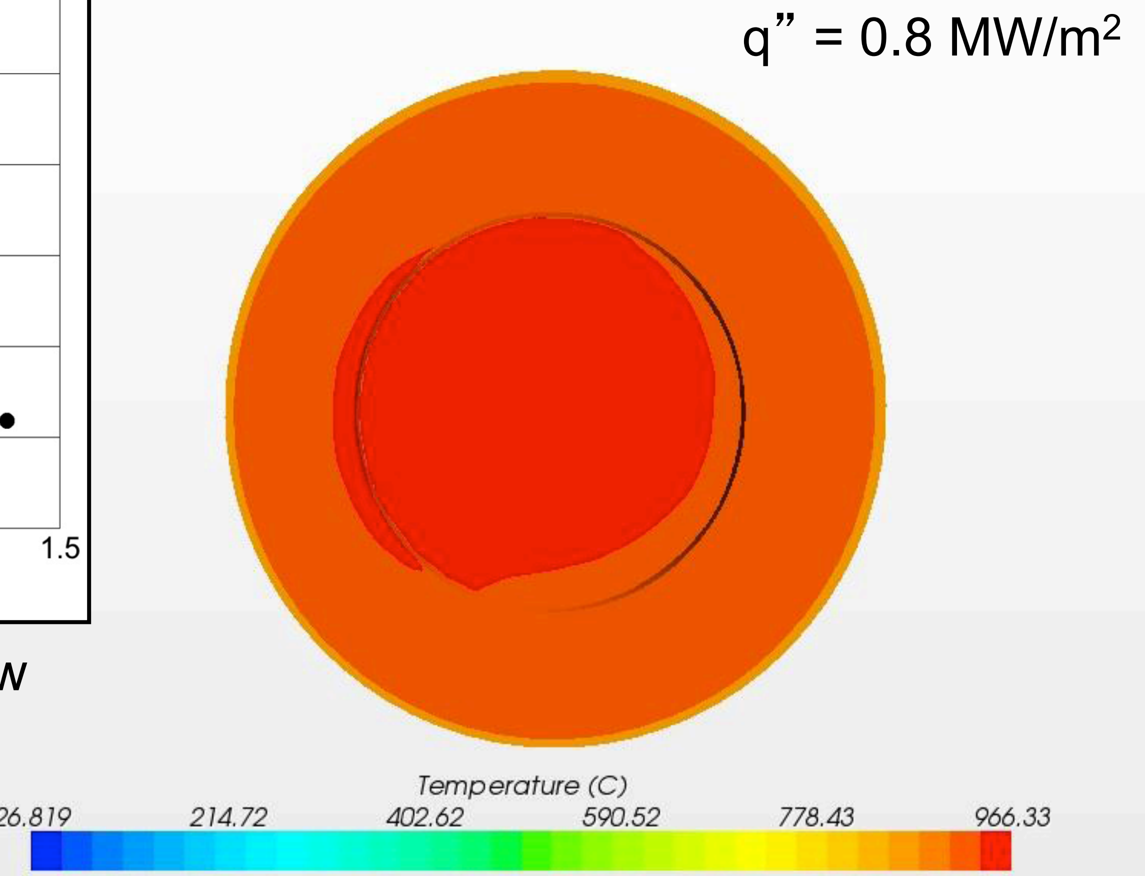
Membrane temperature approaches 1000 °C

Thermal Transport Modeling (continued)

- **End view of membrane shows asymmetry due to outer return channel for the He carrier gas**

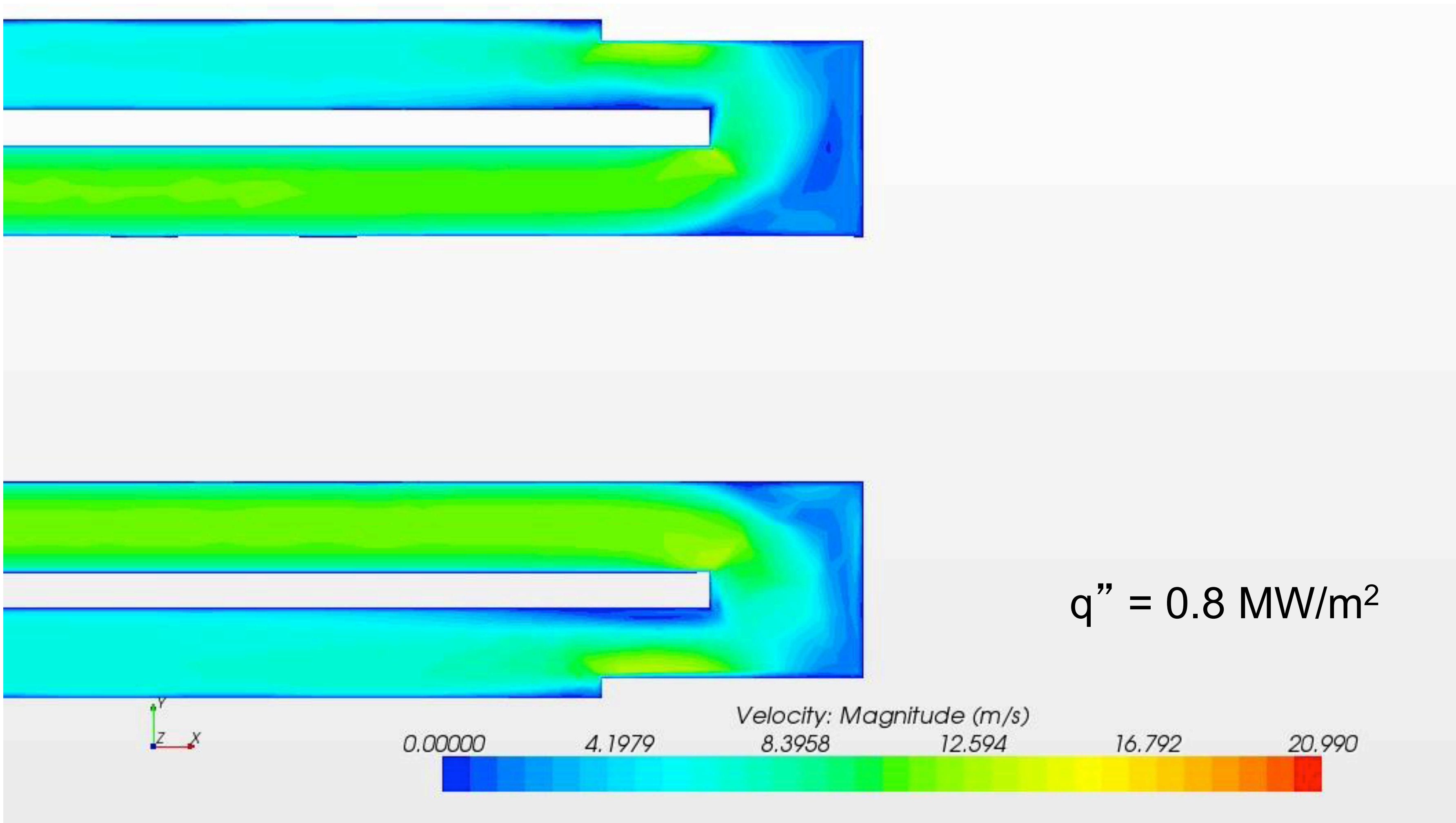


Line scan 200 μm below membrane surface



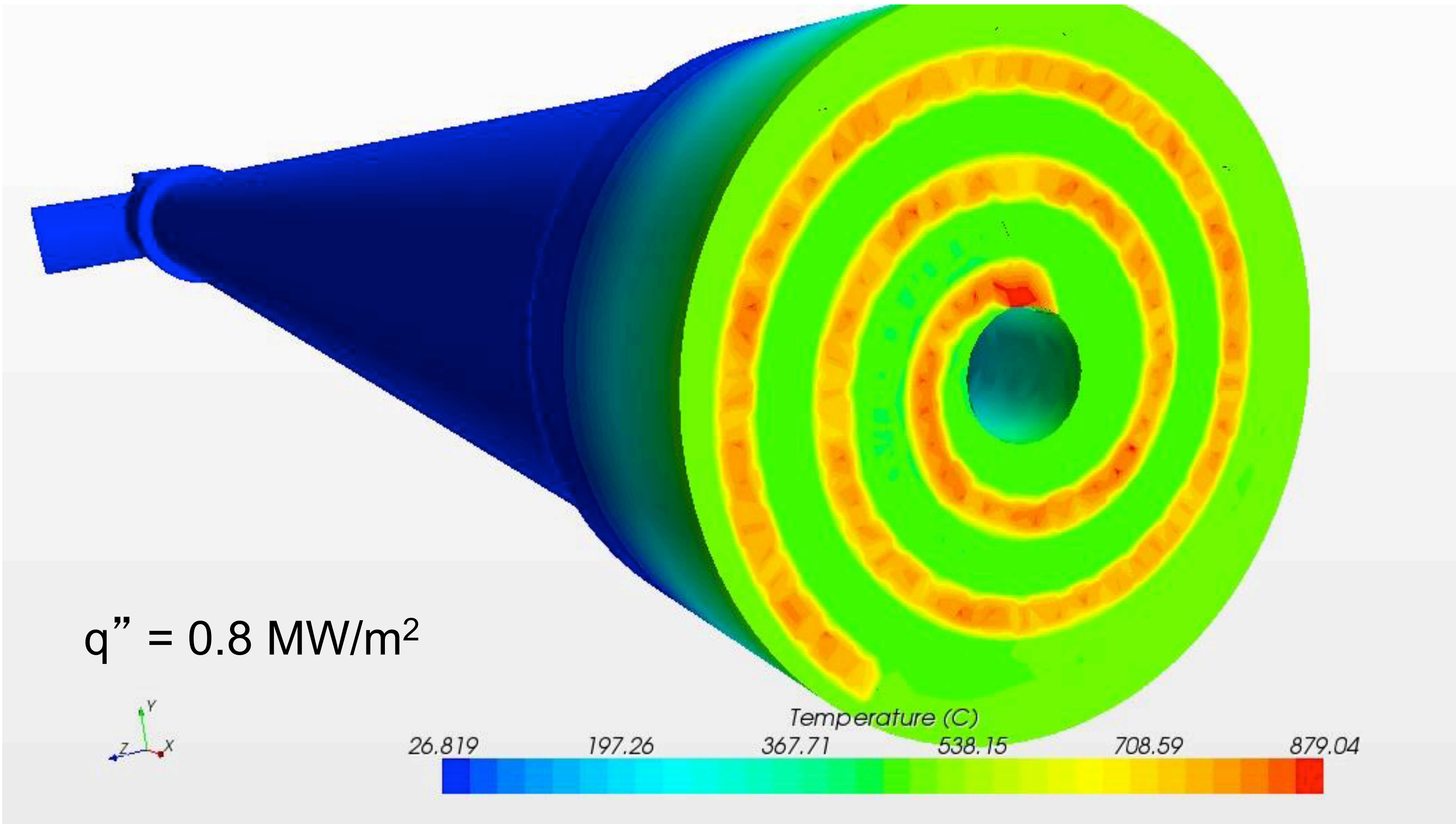
Water Cooling Effective for 0.8 MW/m²

- **Velocity distribution shows water velocity reaching 12-15 m/s with some stagnation in corners**



Thermal Modeling Showing He Temperature

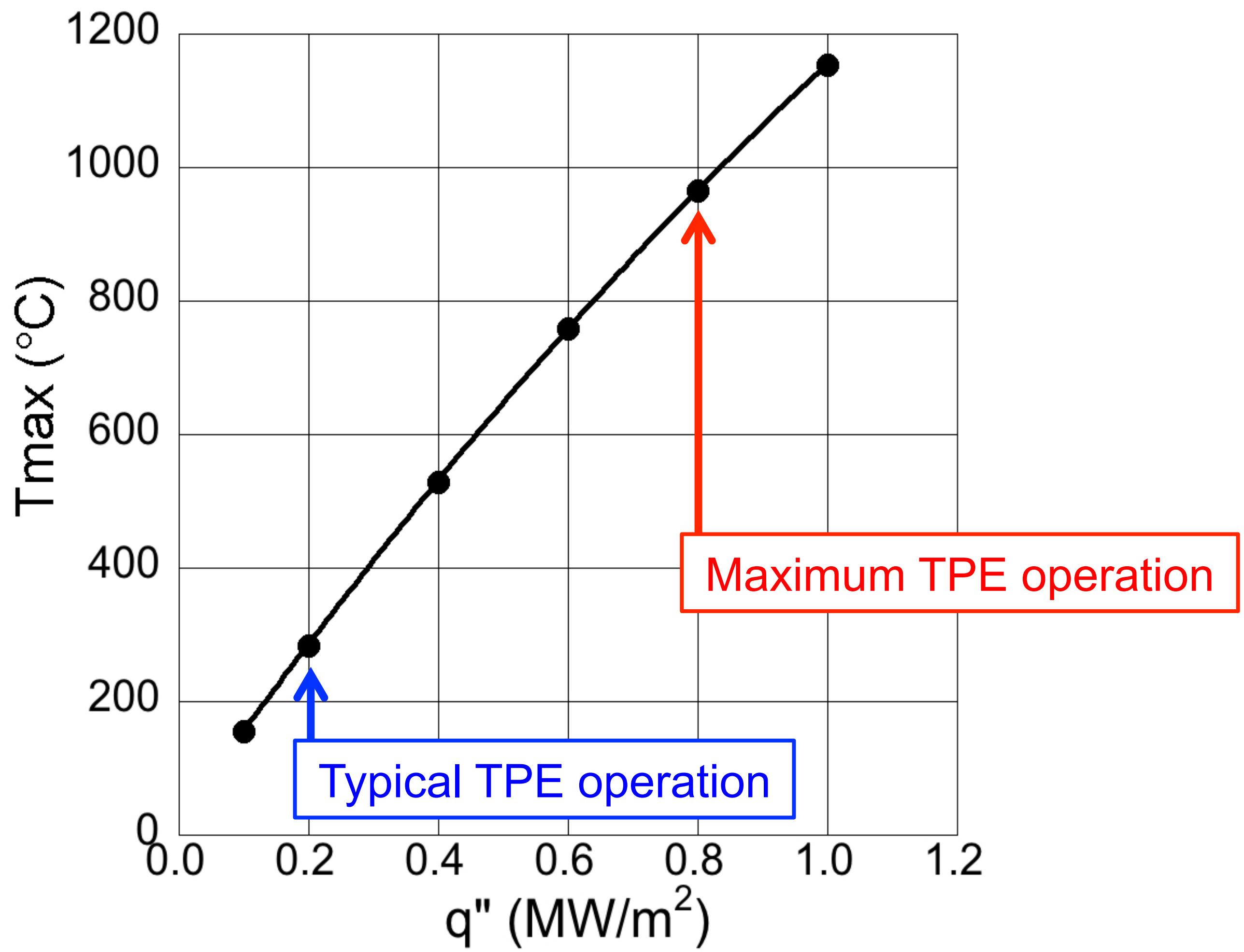
- **Heating of helium carrier gas is clearly visible in the CFD simulation**



Membrane Temperature Scaling With q”

PSI Science Center

- **Scaling of the membrane temperature (200 µm below the heated surface) indicates significant margin for water cooled operation**



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Summary

- Gas driven deuterium permeation experiments for PHENIX have been challenging due to the microstructure of ITER grade tungsten.
- Several hardware issues were addressed and techniques to improve sealing are in progress. Fallback: reproduce permeation results from previously measured materials.
- We have designed and fabricated a novel tritium permeation membrane holder for integration in TPE.
- Cooling can be controlled by varying fluid flow and positioning of a spiral fin behind the membrane under test. Heating is solely from the incident plasma flux.
- Sealing tests have demonstrated adequate helium leak rates up to temperatures of 1000 ºC.
- Flow modeling indicates a minimal helium pressure drop (< 700 Pa).
- Thermal modeling shows good heat removal even up to peak TPE ion fluxes (membrane temperature ~ 1000 ºC, with surface temperature variation of only +/- 2%.

Integration into TPE will require updating safety documents and interlocks for overpressure concerns

Extra