NUCLEAR ENGINEERING
TEXAS A&M UNIVERSITY

NSUF Nuclear Science User Facilities

In Situ Irradiation and Molten Salt Corrosion

at Texas A&M University Accelerator Laboratory

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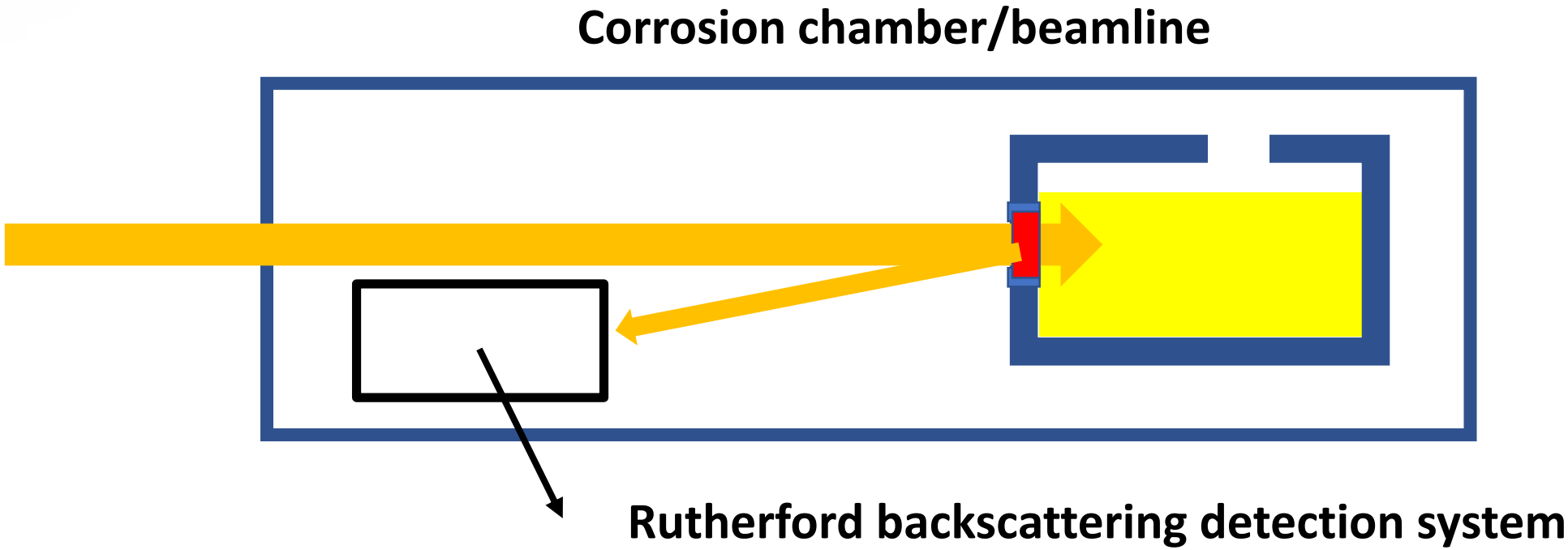
The purpose and motivation behind the new capability

- Corrosion testing for molten salt reactor applications requires the capability to show the synergistic effect between corrosion and irradiation.
- Corrosion + irradiation testing requires the capability of in situ characterization.
- The characterization needs to provide quantitative information about corrosion susceptibility.
- Characterization needs to be high-resolution.
- Corrosion behaviors at the early stage are of particular interest.

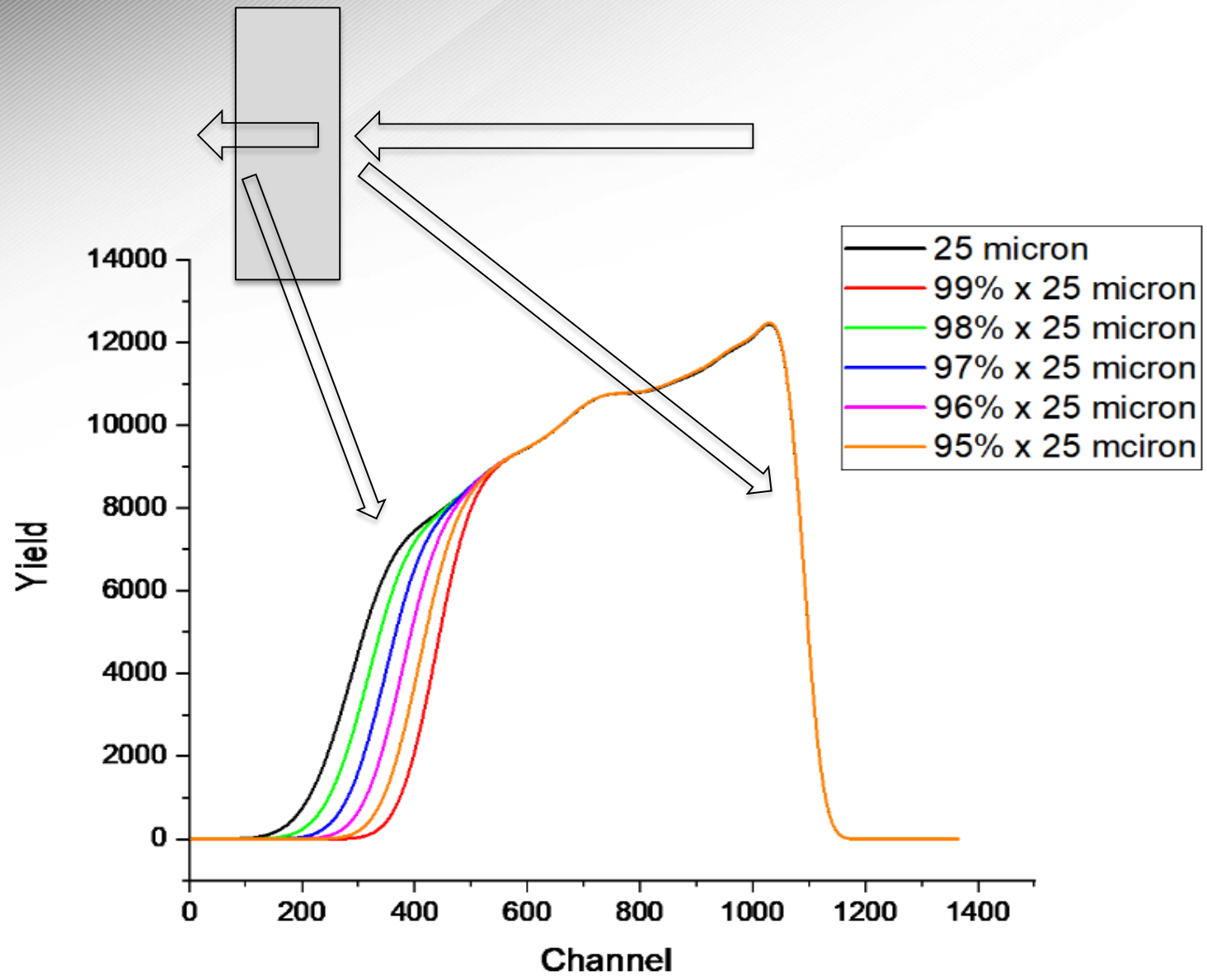
Design principles

- Using a 3 MeV proton beam to irradiate and penetrate through a thin film, introducing radiation damage into both the film and the salt.

- A small fraction of the proton beam is backscattered, and their energy is utilized to characterize the film thickness.

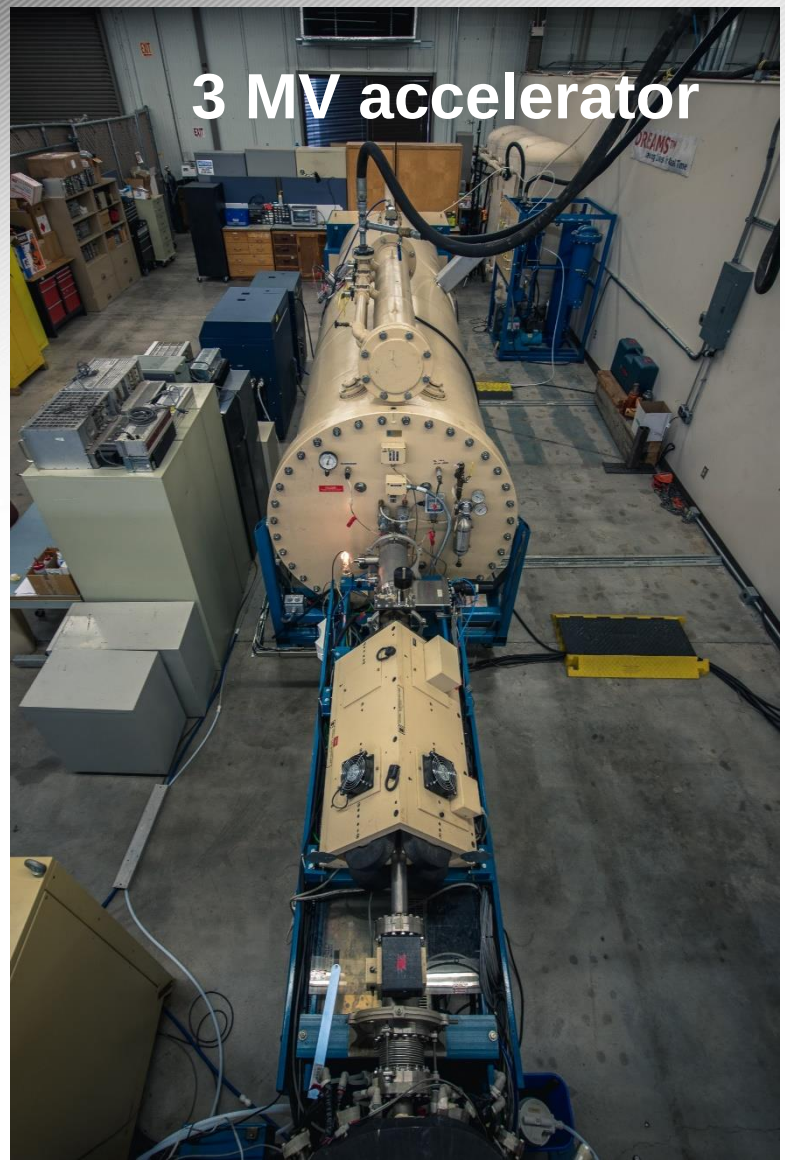


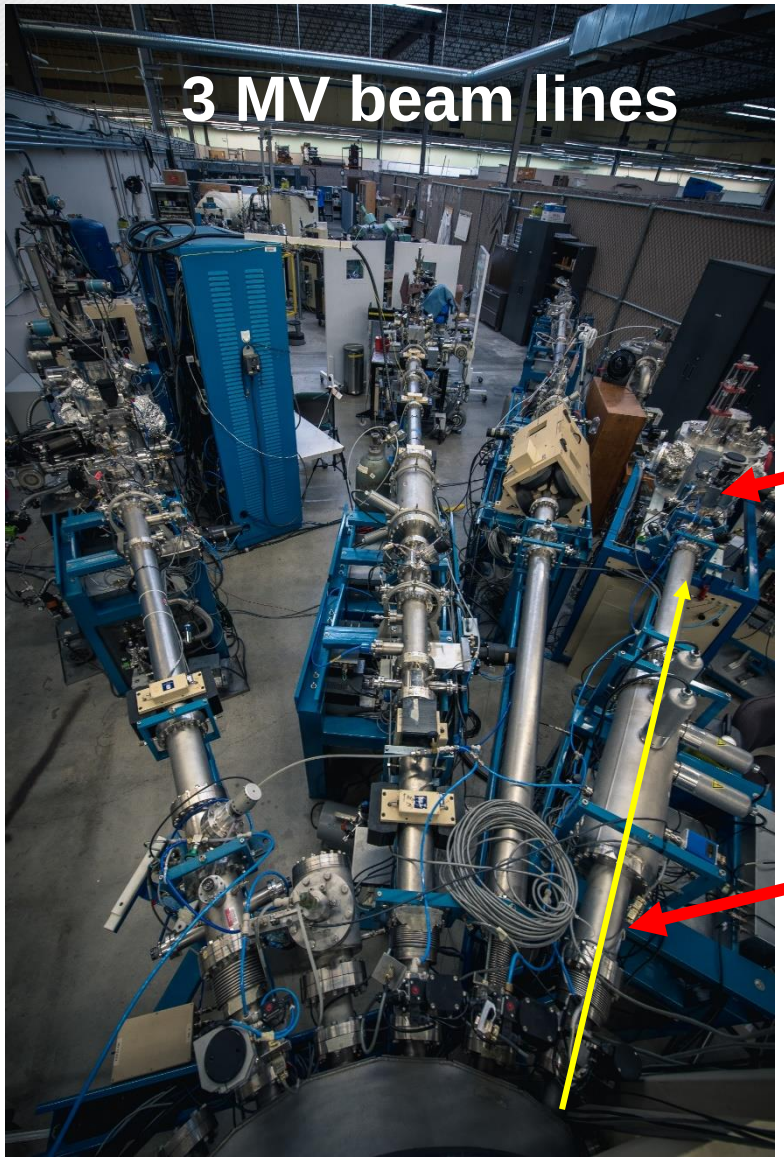
Rutherford Backscattering



A semiconductor detector for the backscattered proton beam can achieve an energy resolution of 15 to 20 keV, corresponding to a thickness resolution of 50 nm.

Design principles

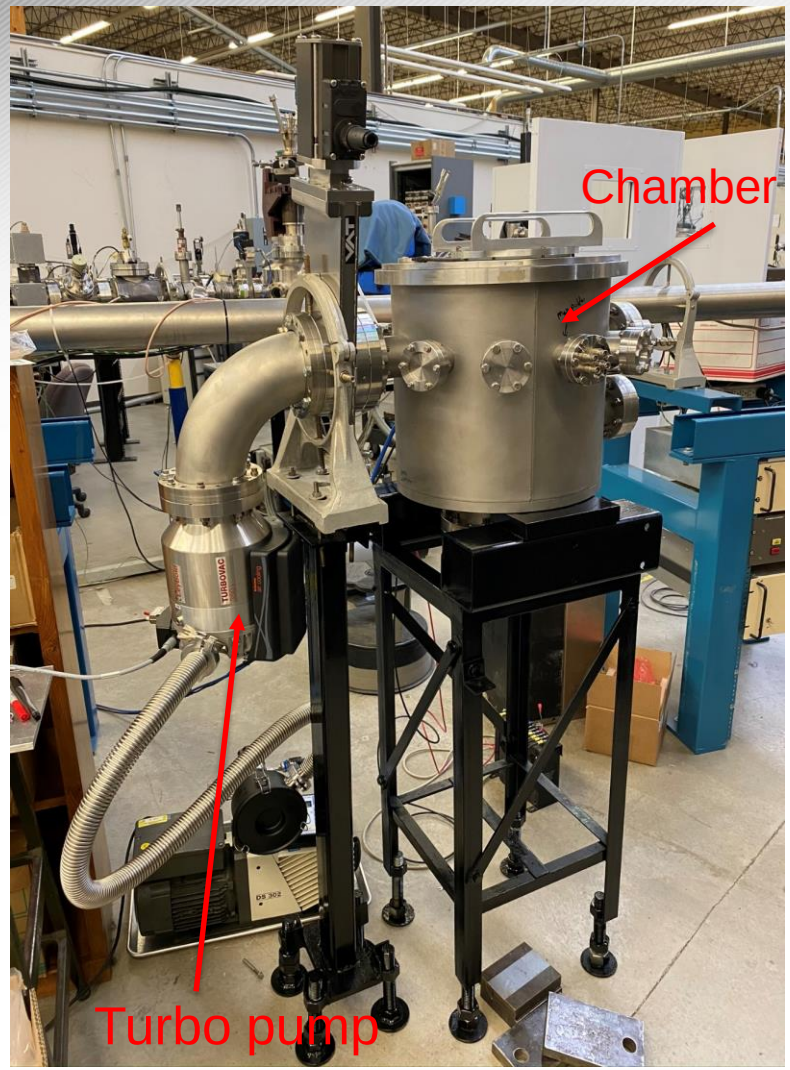


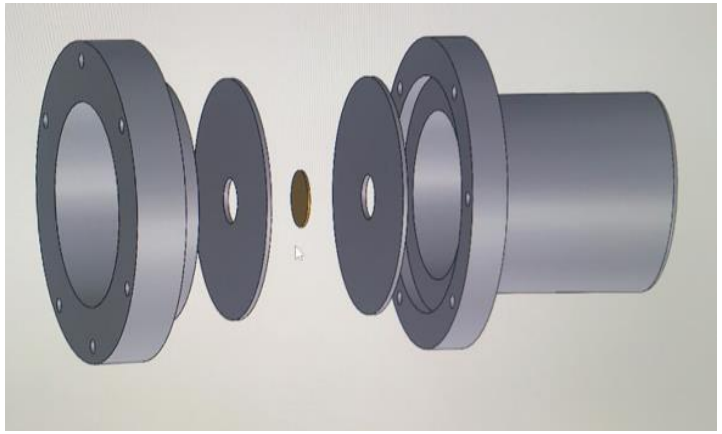


The chamber for proton irradiation, molten salt corrosion, and in situ characterization.

Beam line for 3 MeV proton

Early design





Challenges

- Salt purification
- Corrosion cell design and thin film loading
- Heater design for uniform heating (critical)
- Beam model (rastering, focused, defocused) and the impact on temperature & stress of the film (critical)
- Collecting backscattering spectra in front of a heated cell (500 to 700°C) (challenging)

Salt purification

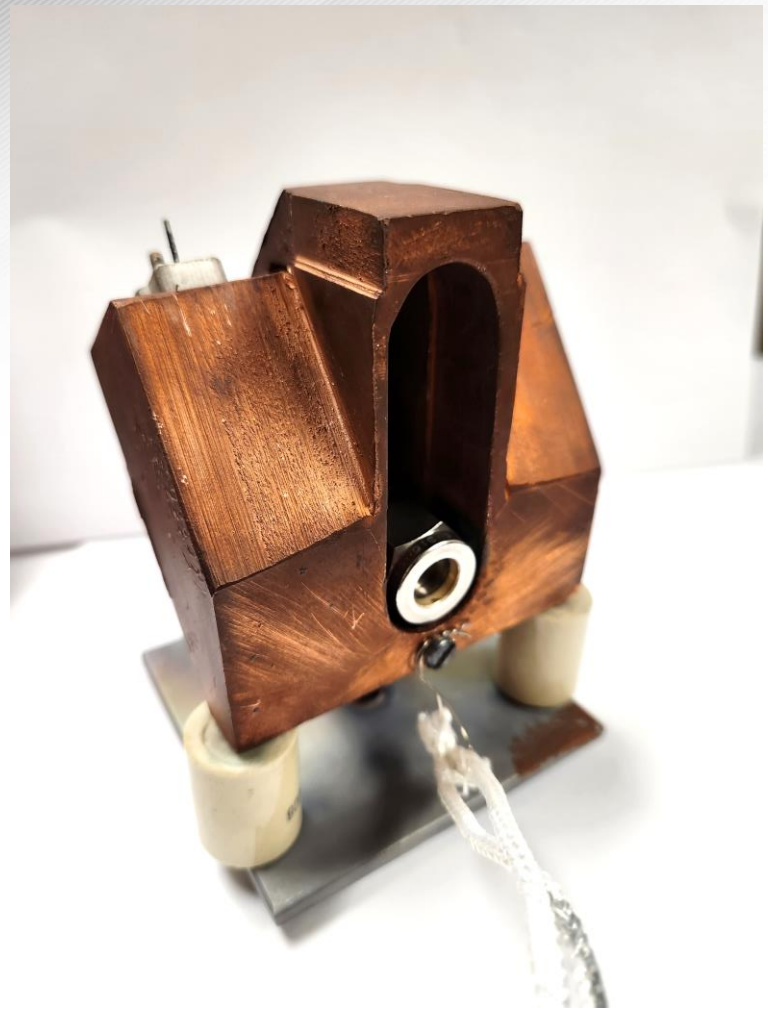
The fluorination step removes nonmetal impurities

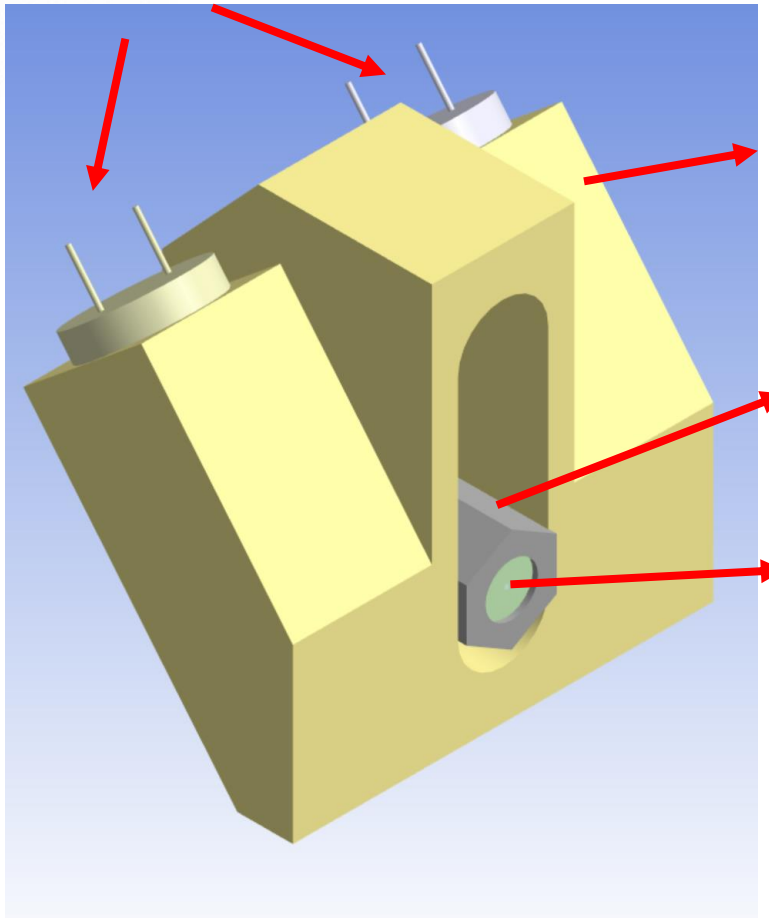
$$\mathbf{MeOH_{(d)} + HF_{(g)} \rightleftharpoons MeF_{2\,(d)} + H_2O_{(g)}.}$$

The reduction step eliminates metal impurities and excess fluorine

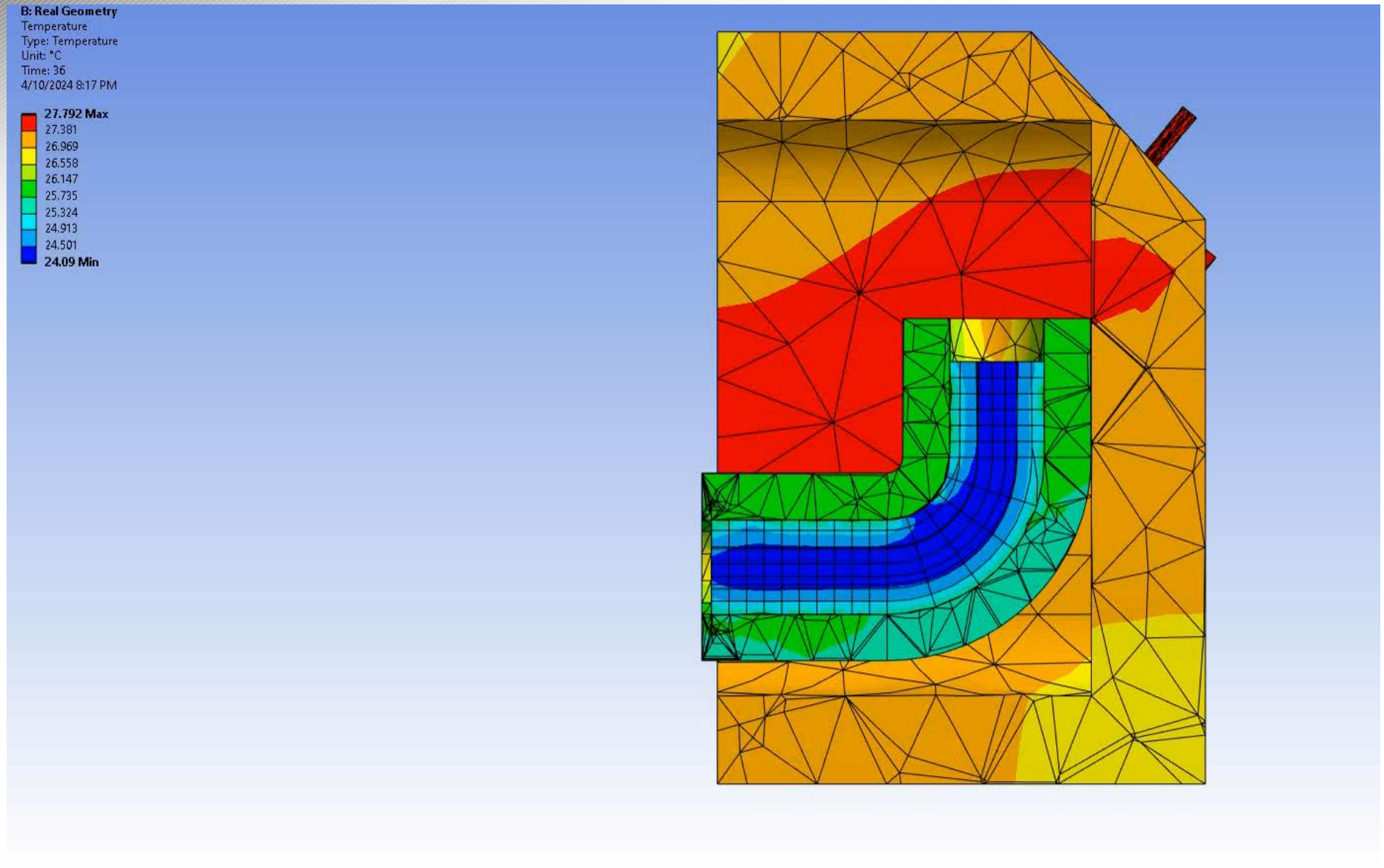
$$\mathbf{MeF_{2\,(d)} + H_{2\,(g)} \rightleftharpoons Me_{(s)} + 2HF_{(g)}.}$$

Optimized corrosion cell

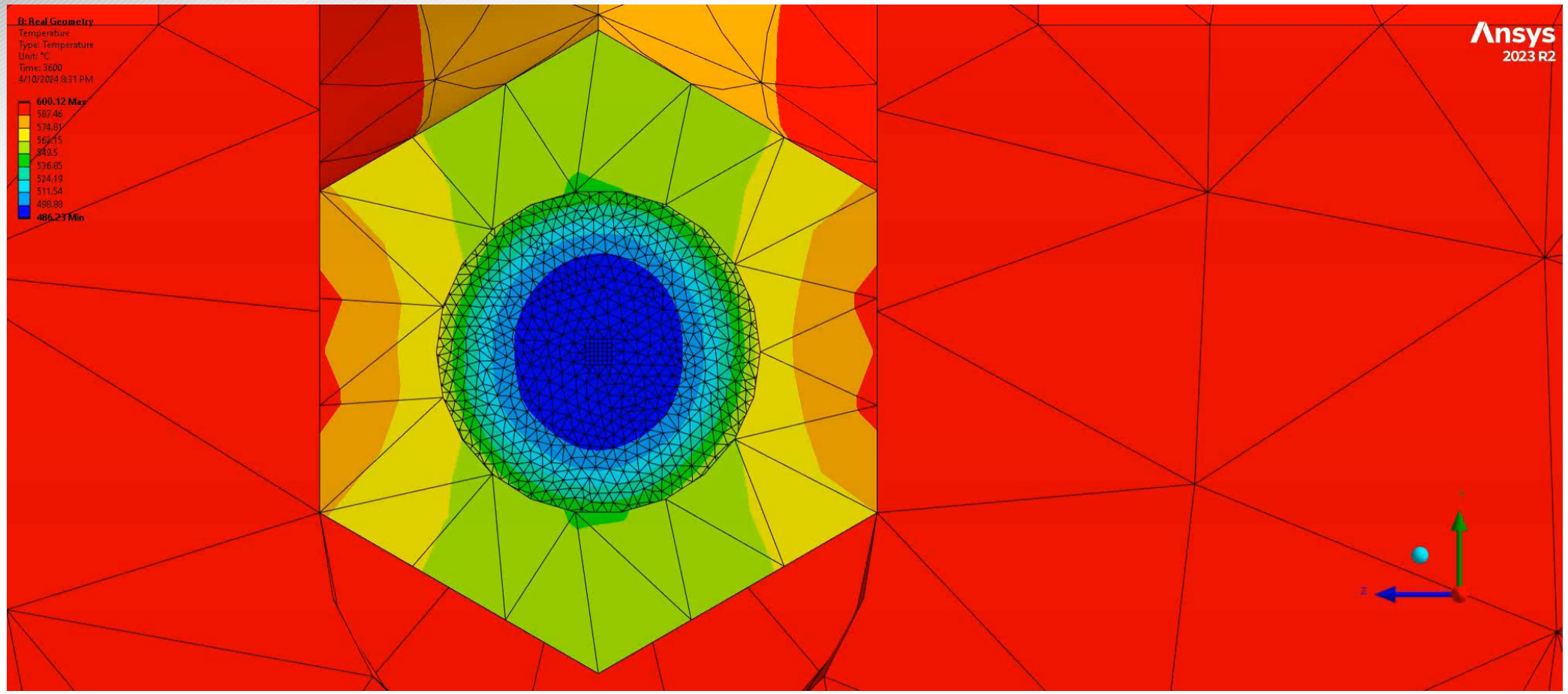




Molten salt melting (without beam)



Beam heating by a focused 3 MeV proton beam (1mm×1mm)



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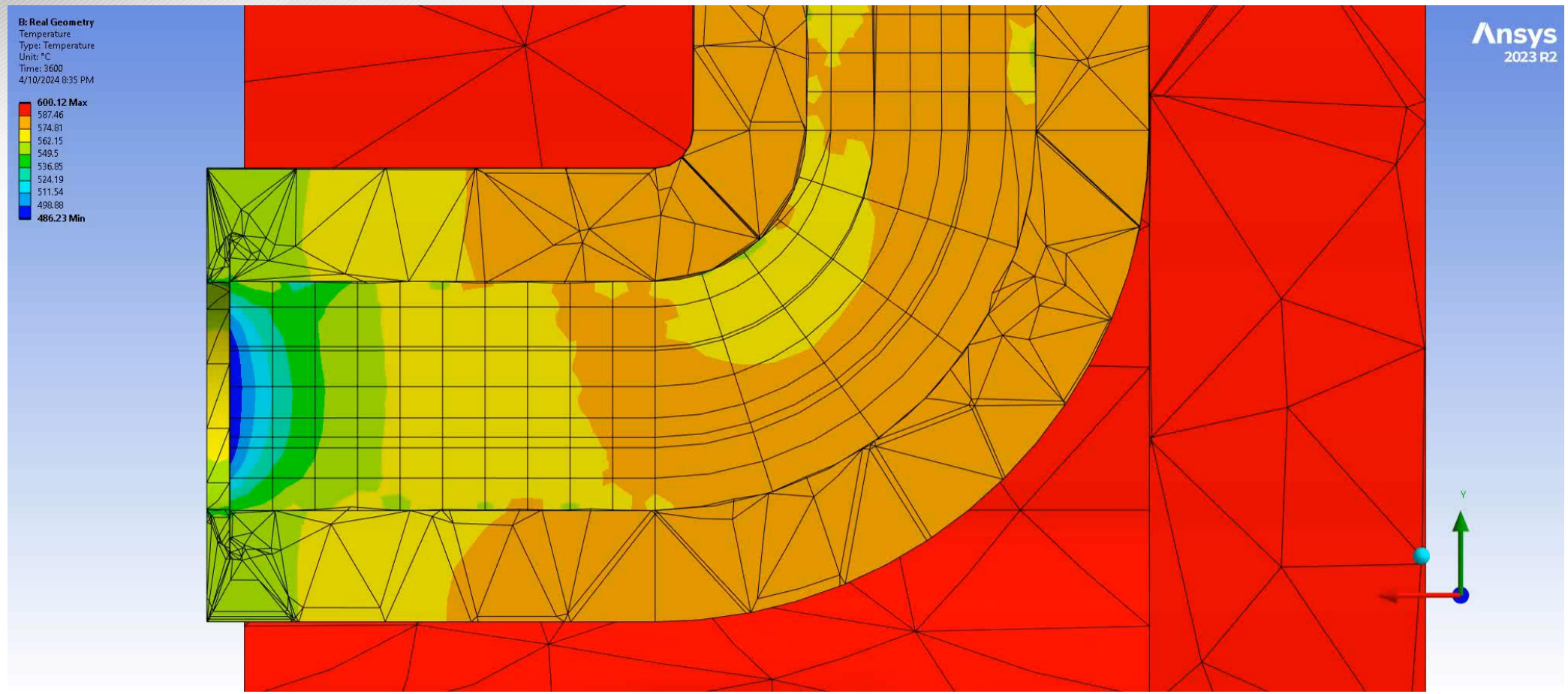
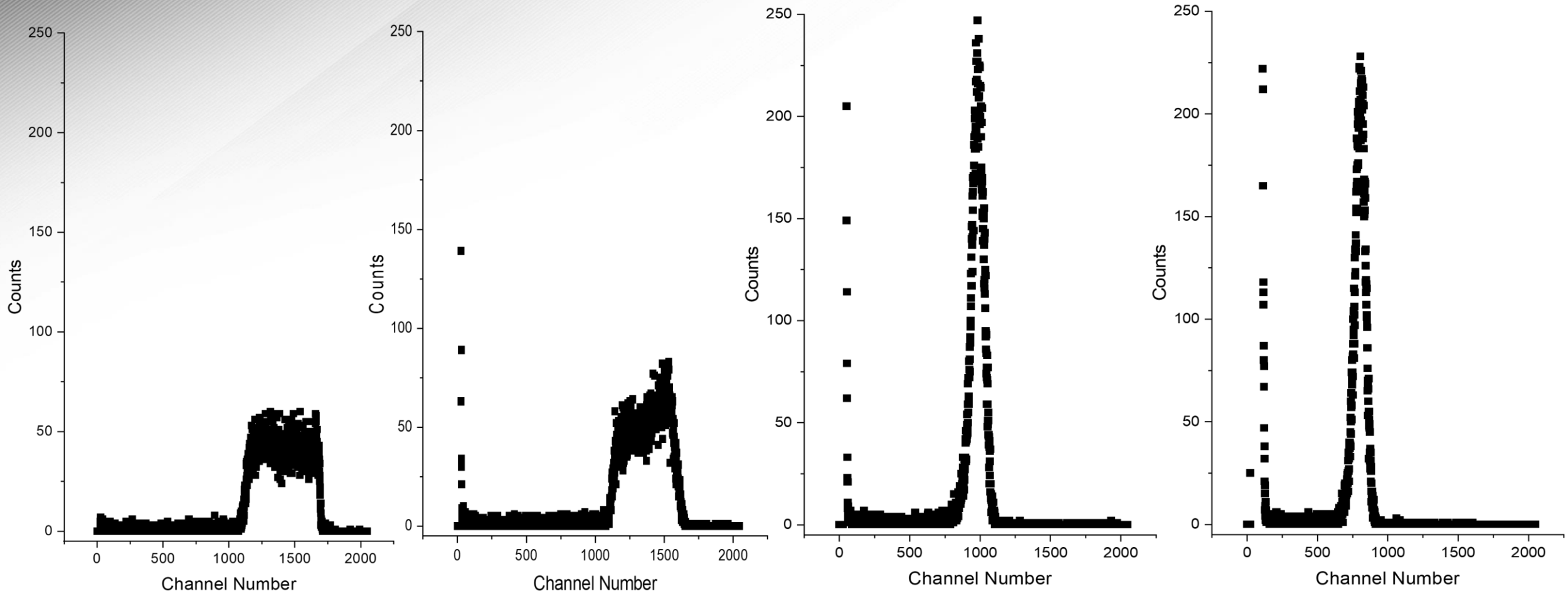


lessons and best practices

- Multiple heaters are needed to uniformly melt salts (i.e., FLiNaK).
- Heater temperature should exceed the melting point sufficiently for uniform melting. Our current design requires at least 100°C higher.
- A focused proton beam creates a focused hot spot on the thin film, with a large temperature gradient and large thermal stress.
- A rastering beam at high frequency (i.e., 550 Hz) over an area larger than the thin film is needed to avoid pitfalls and artifacts.
- If the heating temperature is low and a focused beam is used, a large cavity (without salt) is produced immediately behind the thin film, causing no corrosion.

The backscattering detector fails in the corrosion chamber



Increasing temperature

The detector fails at a temperature > ~60°C.

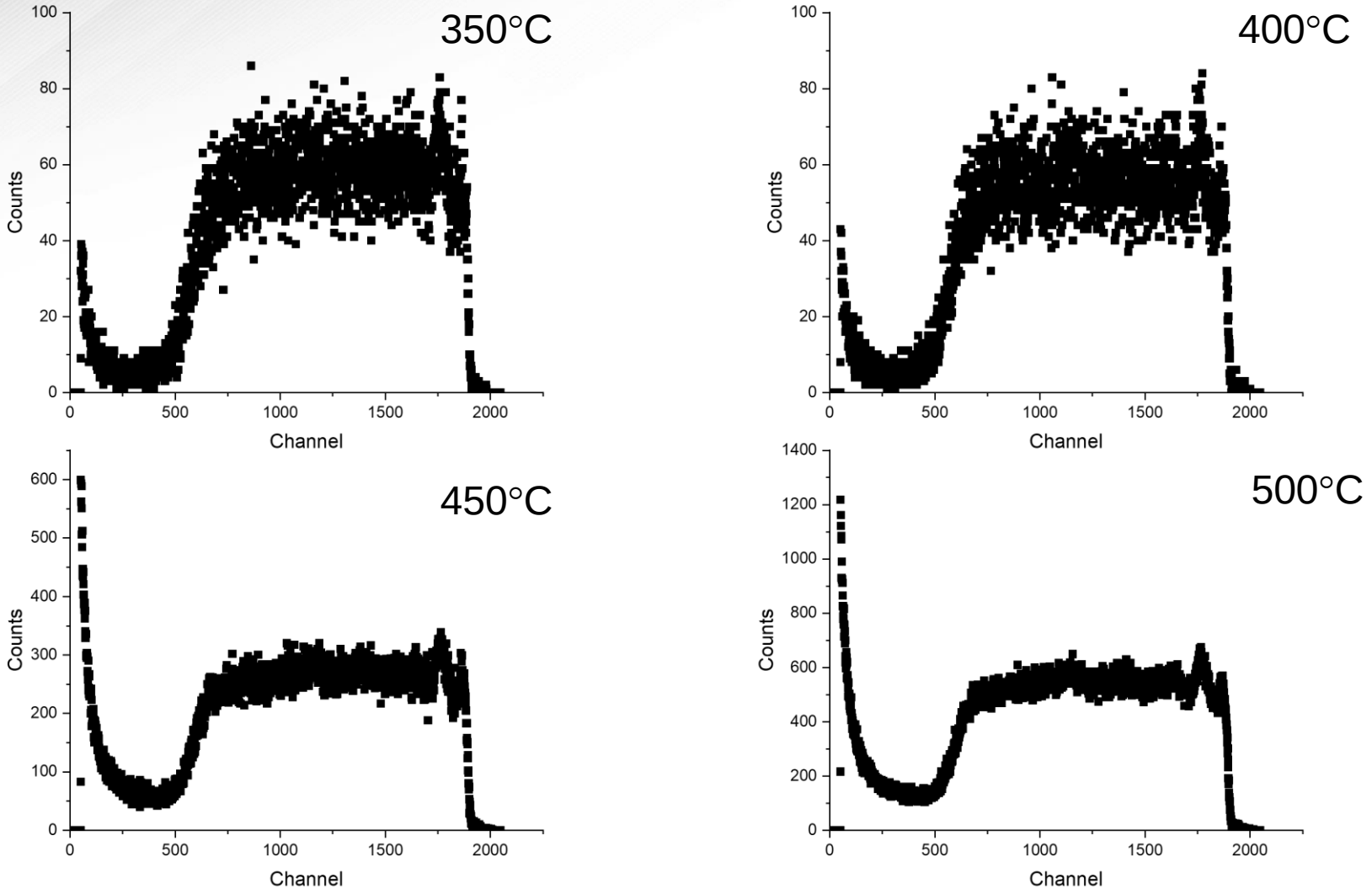
Solution to mitigate the heating effect on the detector

Using aluminum foil as a thermal shield:

For a high-temperature backscattering spectrometry detection system, we use aluminum foil as a thermal shield around the semiconductor detector. The key is to block heat from the cell and to block light from the cell's blackbody radiation. The foil also blocks the thermal electrons emitted from the corrosion cell. The aluminum foil cannot attach to the detector system and needs to be self-suspended.

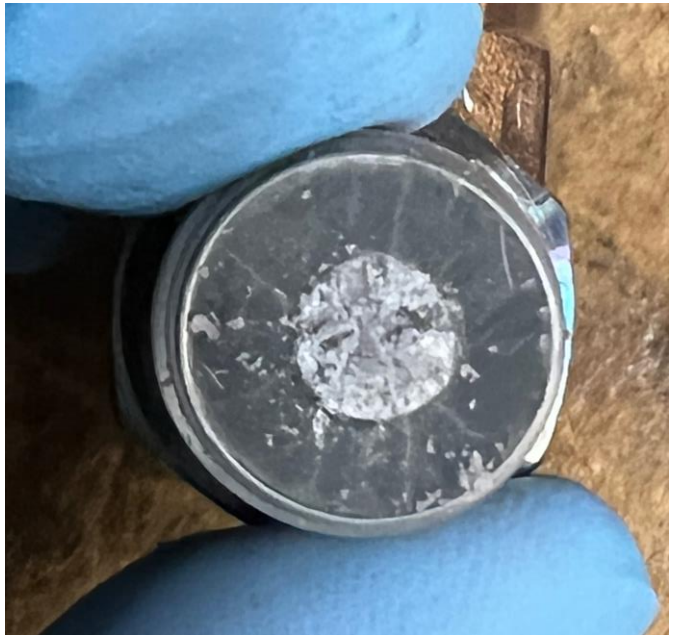
Detector heating issue solved

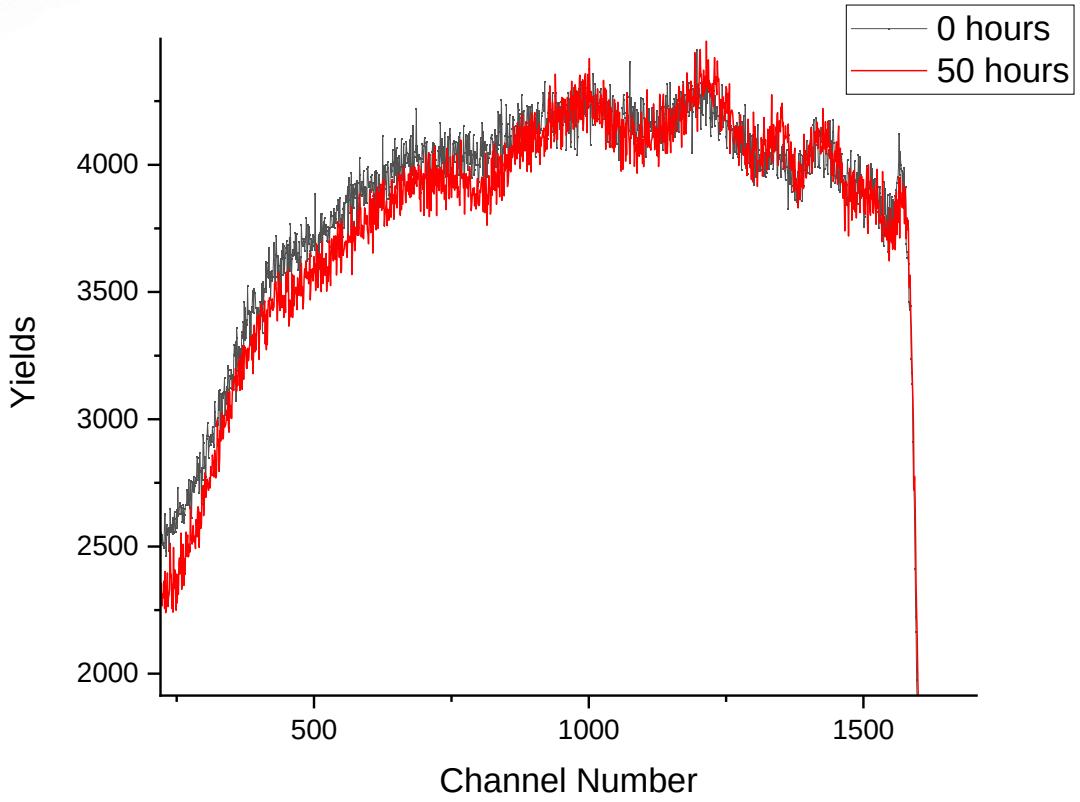
Backscattering spectra of 25-micron 316L heated to various temperatures.



Corrosion of 316 at 550°C

Proton (H^+): 3.5 MeV
Temp: 550°C (salt)
Beam Current: 10 nA
Time: 50 hours
Sample: 17.5 µm 316 SS





Summary

- It is feasible to combine proton irradiation, molten salt corrosion, and in situ characterization to study corrosion resistance of thin films.
- It is critical to use multiple heaters to create uniform melting.
- It is critical to avoid a focused beam, which causes overheating in the film center and introduces thermal stress.
- A rastering beam should be used.
- Collecting energy of backscattering spectra is not trivial at high temperatures, requiring additional thermal shielding around the detector.