Lawrence Livermore National Laboratory

Recent Advances in the Fabrication of Very Thick, Multistepped, Iron and Tantalum Films for EOS Targets

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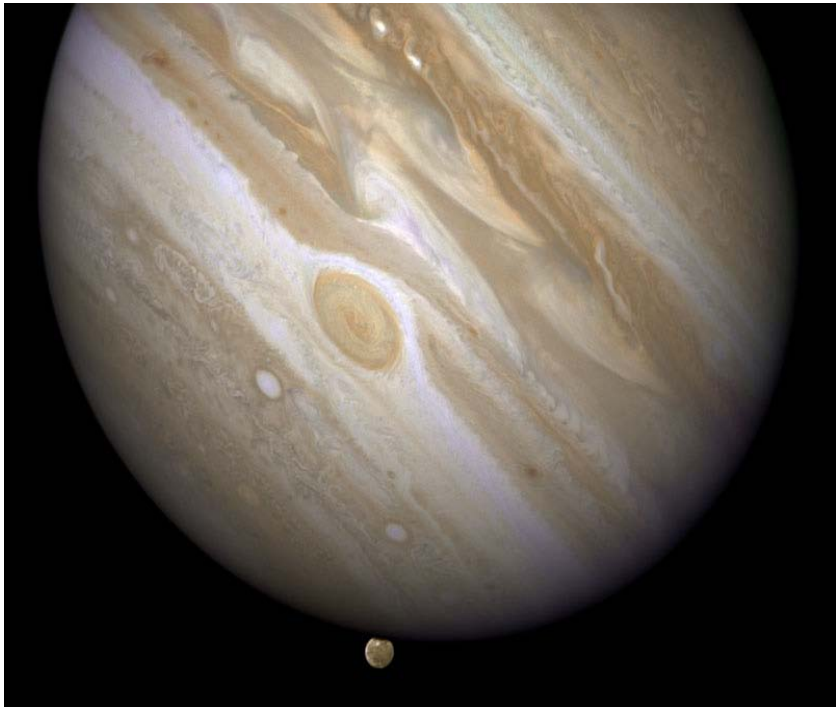
Outline

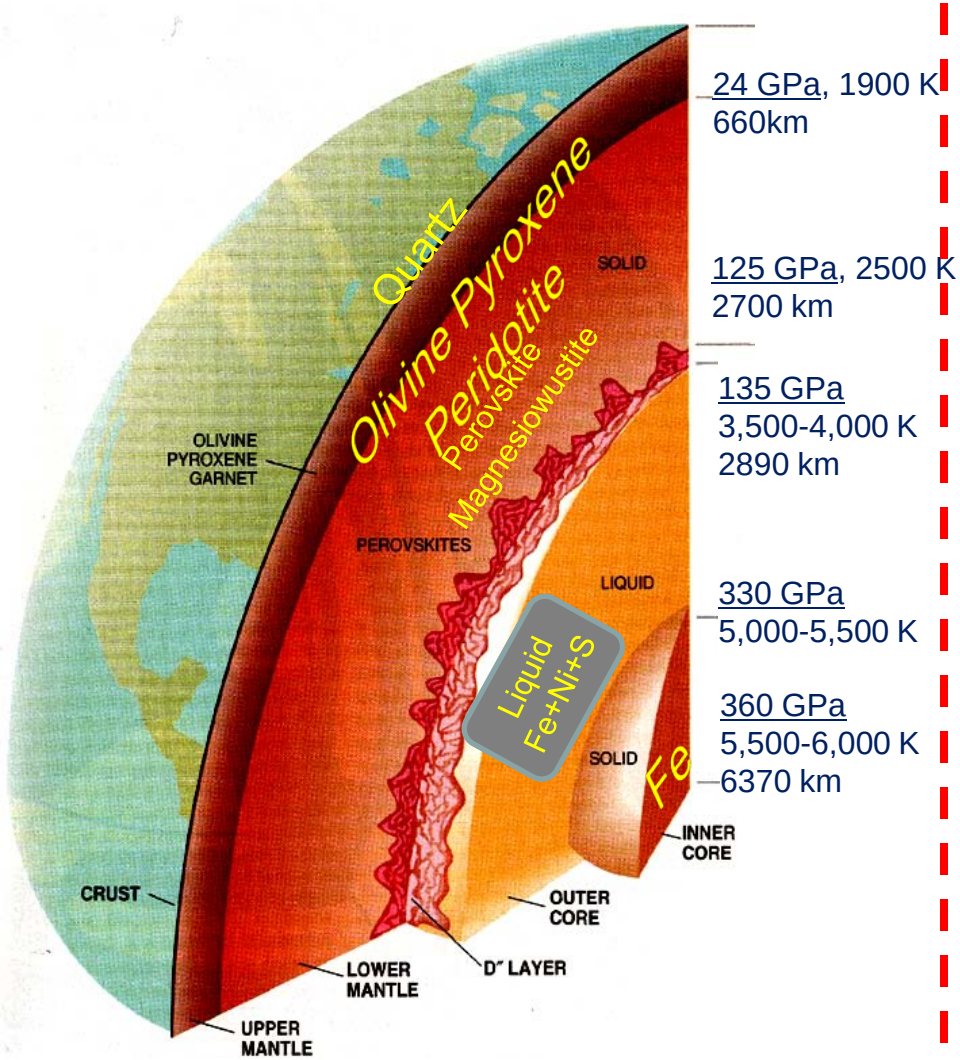
- Introduction
- Thick Stepped Iron films for EOS targets on NIF
- Thick Stepped Tantalum films for EOS targets on NIF
- Very thick Tantalum films for EOS targets on Z.
- Conclusions

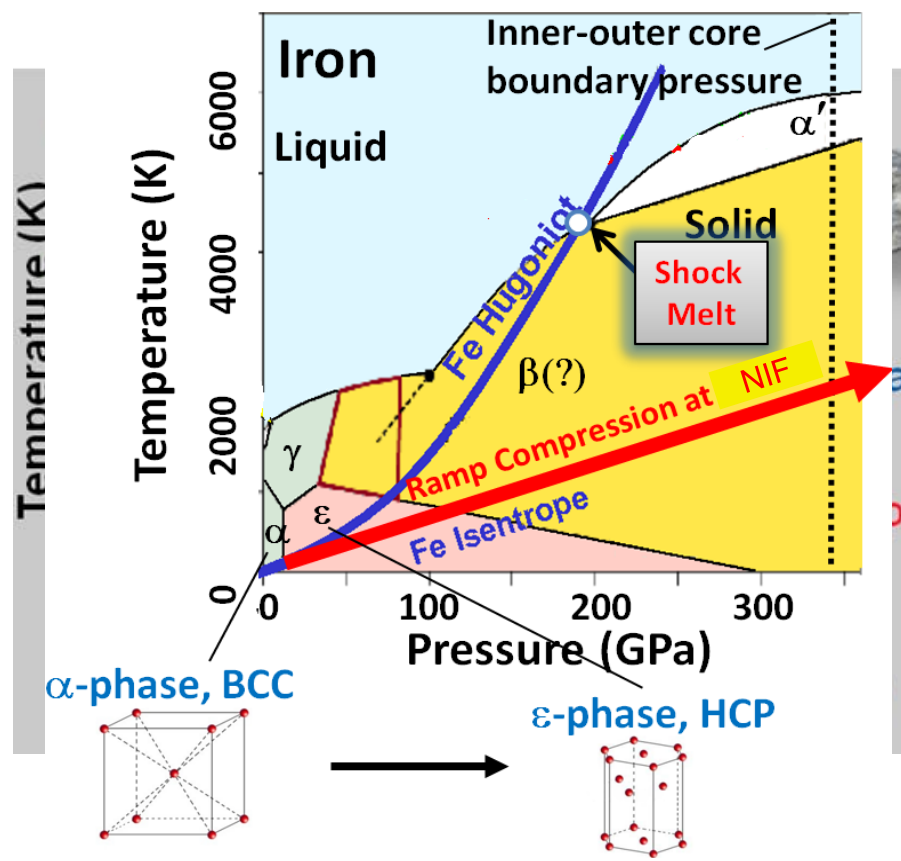
Using Iron Targets on NIF to Answer Fundamental Questions about Our Solar System and Beyond

- What is the nature of the iron core at the center of Earth and other terrestrial planets?
- What is the interior structure of Jupiter and the other giant planets?
- How did Jupiter and the giant planets form and what are the implications for the origin and evolution of the solar system?



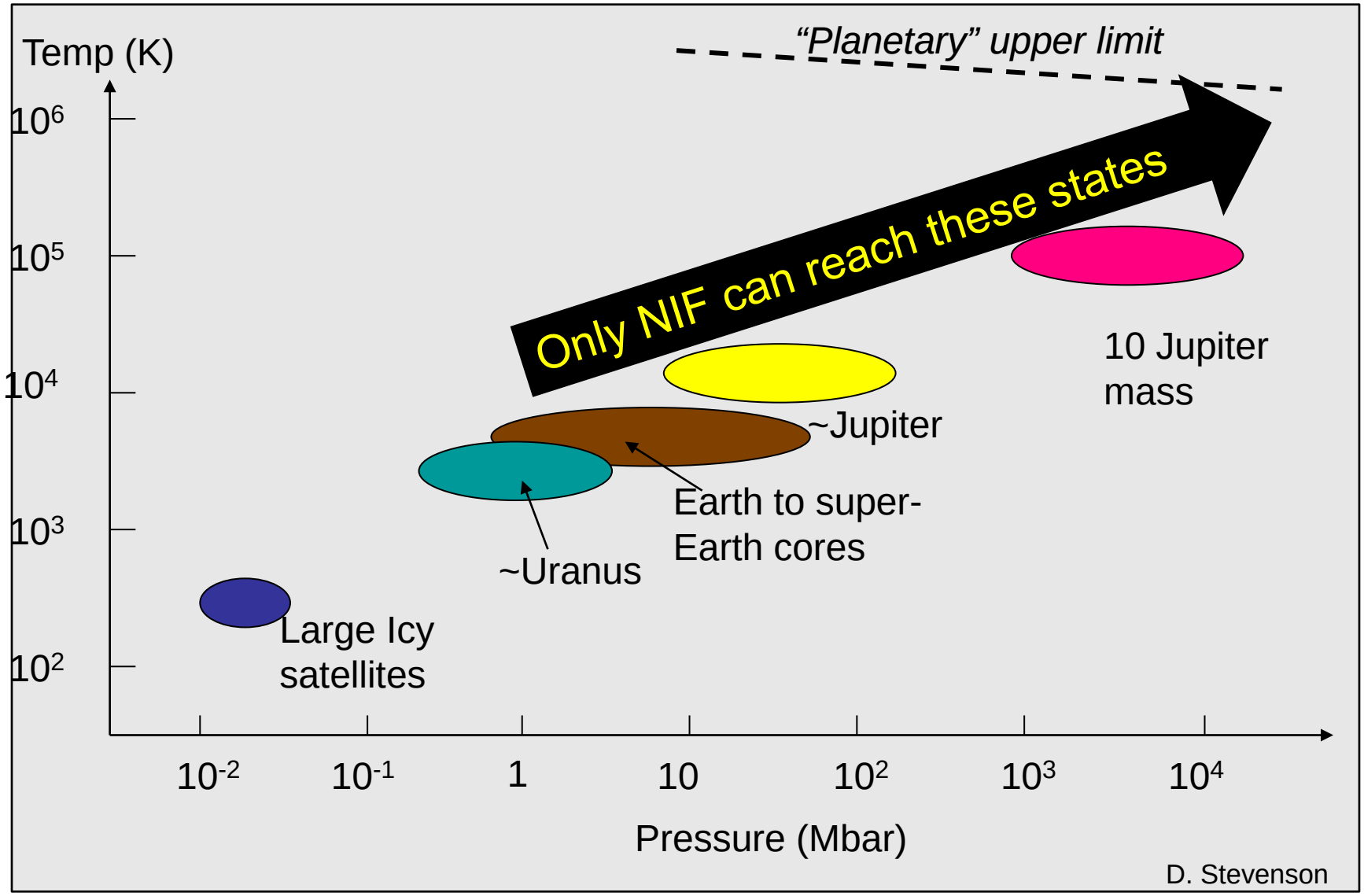
Ramp Compression of Iron to Very High Pressures: A New Window into Planetary Interiors



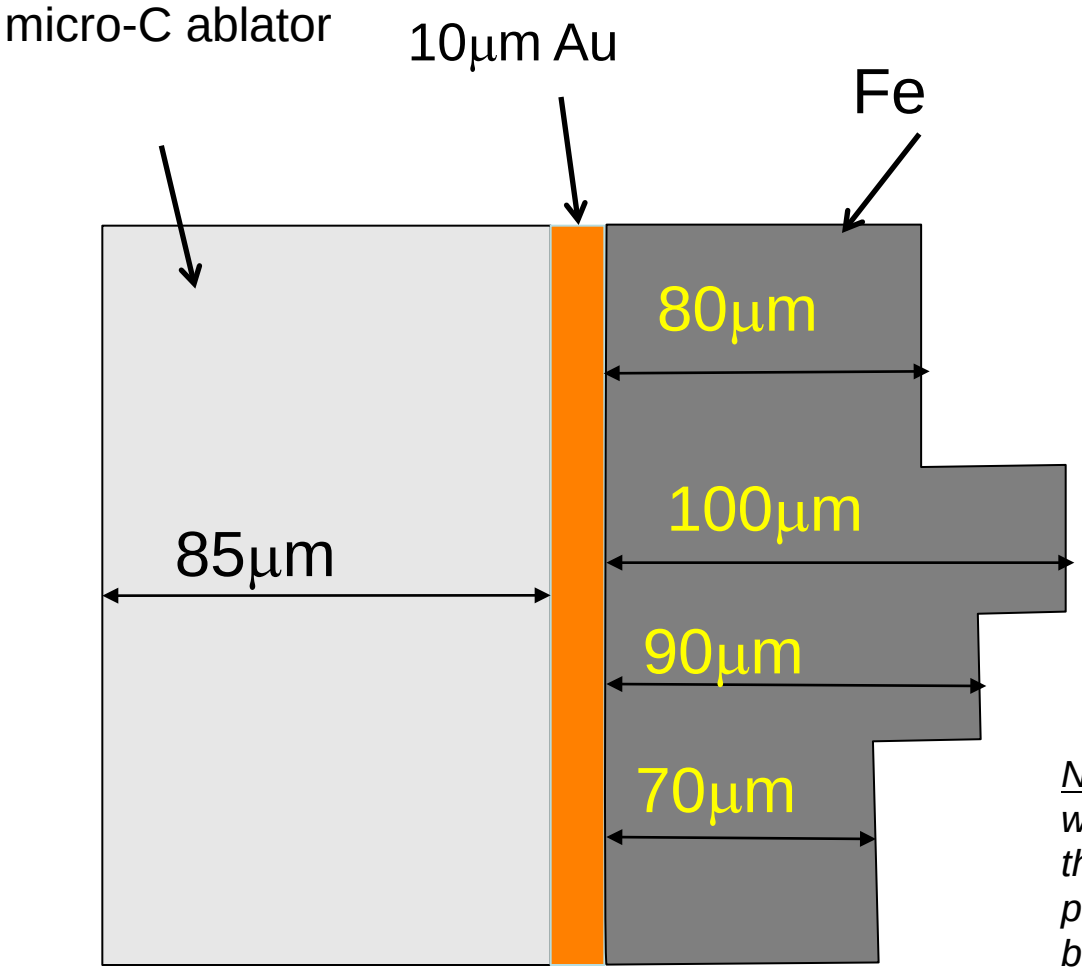


Tom Duffy

Understanding planetary structure requires knowledge of the Equation of State (EOS) at extreme conditions



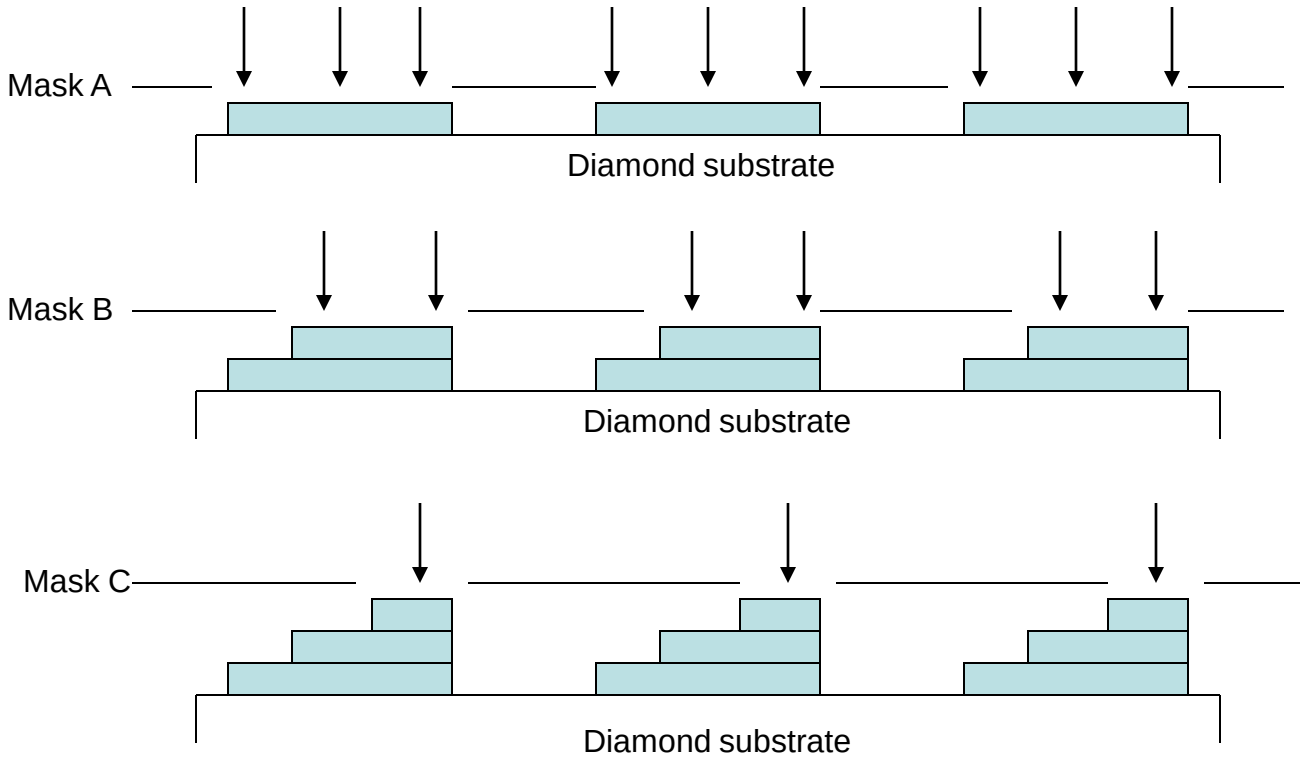
The following target design was requested from us to enable measurements of the stress-density response on Fe up to 20Mbar on NIF



<u>Note</u>: The absolute Iron thicknesses were significantly greater than any films that we had been asked to fabricate previously, so film stress and adhesion become more of a challenge.

Our Method to Produce Micron-Scale Steps

Utilizes a basic masking technique utilizing precisely machined blades to shield selected areas from depo flux, and create small steps.



Note: The depo system is vented and the blades readjusted for each of the three steps.

Deposition /Fabrication Considerations and Boundary Conditions for the Iron Deposition

- For practical purposes we need relatively high depo rates, which entails the “substrate-to-sputter source” distance is relatively small (< 3”).

- We have found previously that varying the sputter gas pressure has less of an impact on film stress under the above conditions.

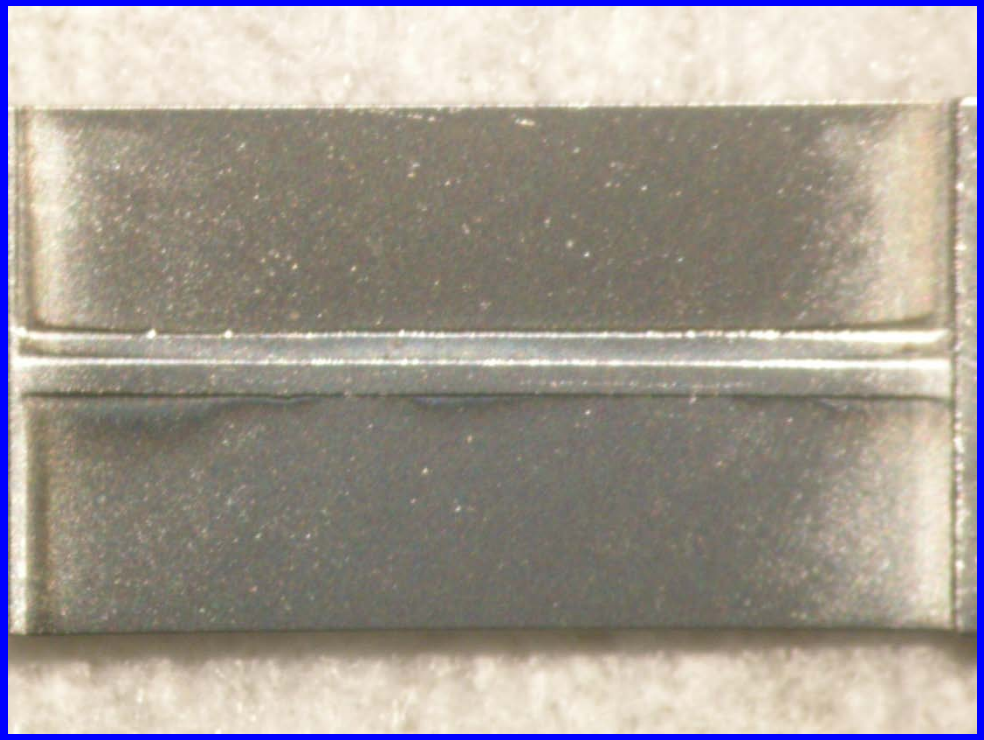
- So we employ a small negative bias of (low energy ion bombardment) to control stress and promote adhesion. For example, -40 V for the Iron is enough to add energy without inducing significant resputtering.

- We also utilize thermal effects, through an external heater and the heat from the sputter source. Typical depo temp is ~210 C.

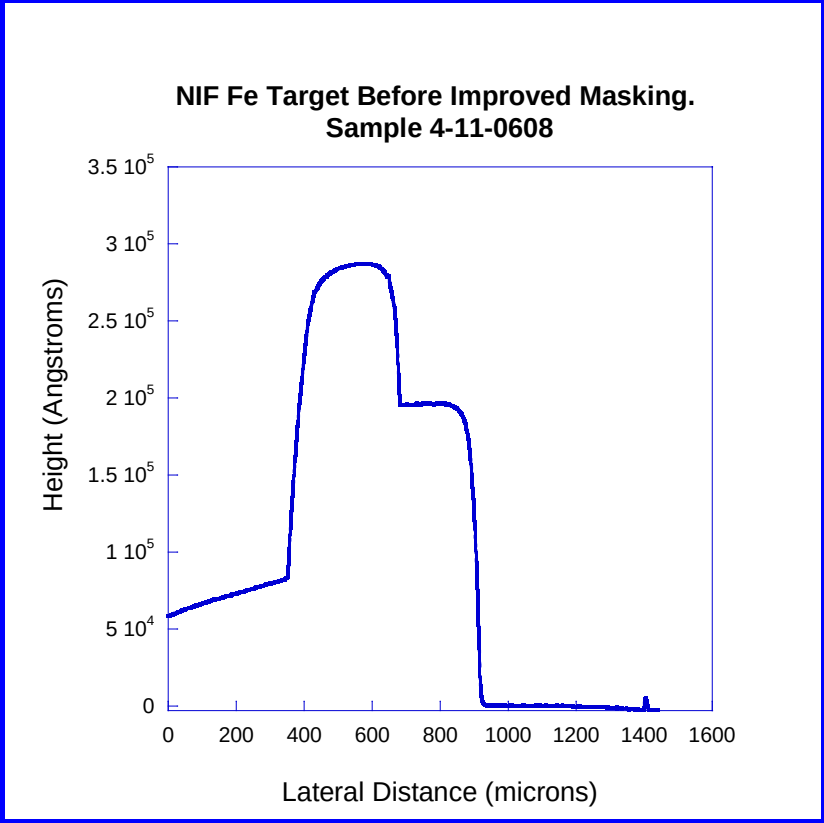
- Also note that because the Iron targets are magnetic, we need thinner targets. We can get about 40-something microns out a target with our current sputter sources.

We were able to fabricate the very thick stepped Fe targets

Fe target (top view)

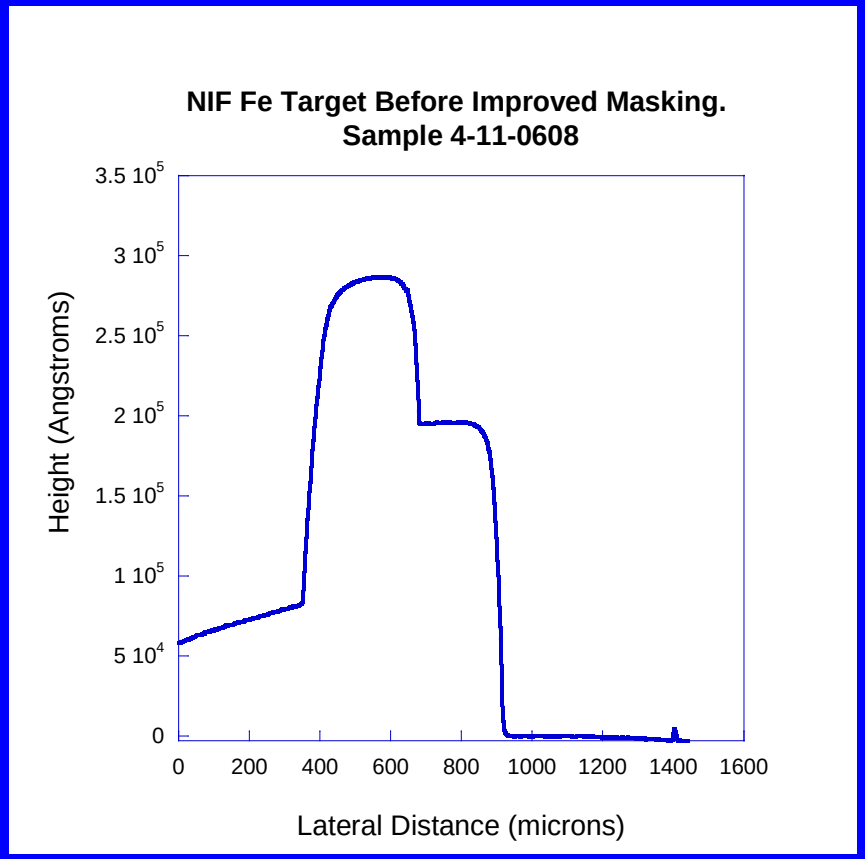


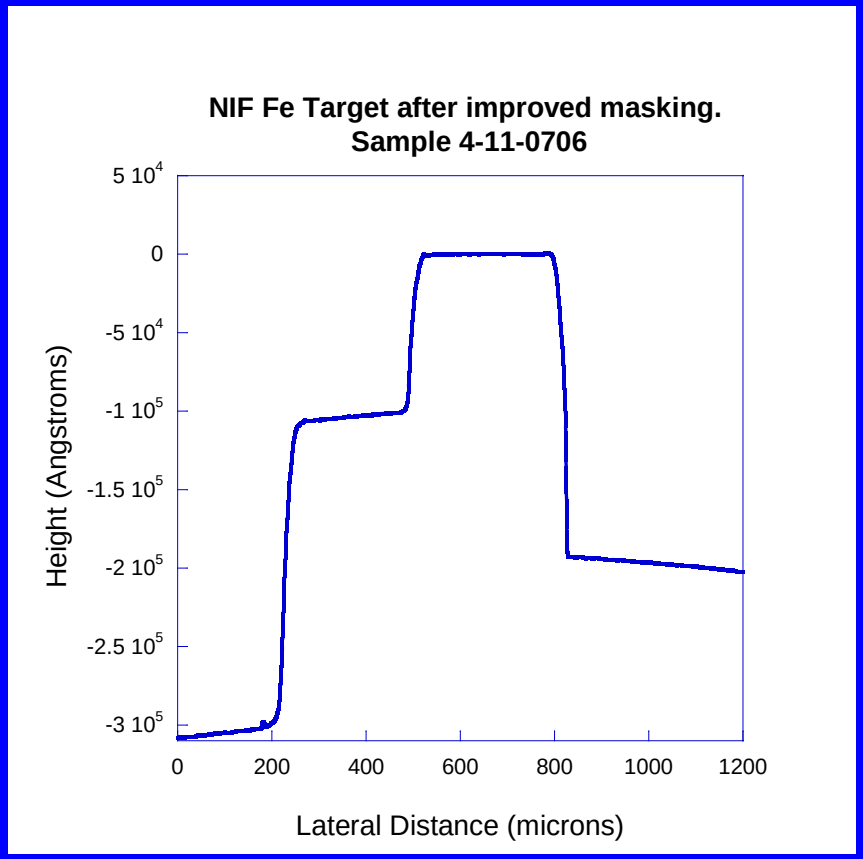
The steps looked decent but there was more rolloff than expected.



Anecdotal observation: We observed more rolloff for Fe using very similar masking hardware and substrate fixturing than we did for Tantalum.

We modified our mask fixturing geometry and were able to improve the rolloff considerably

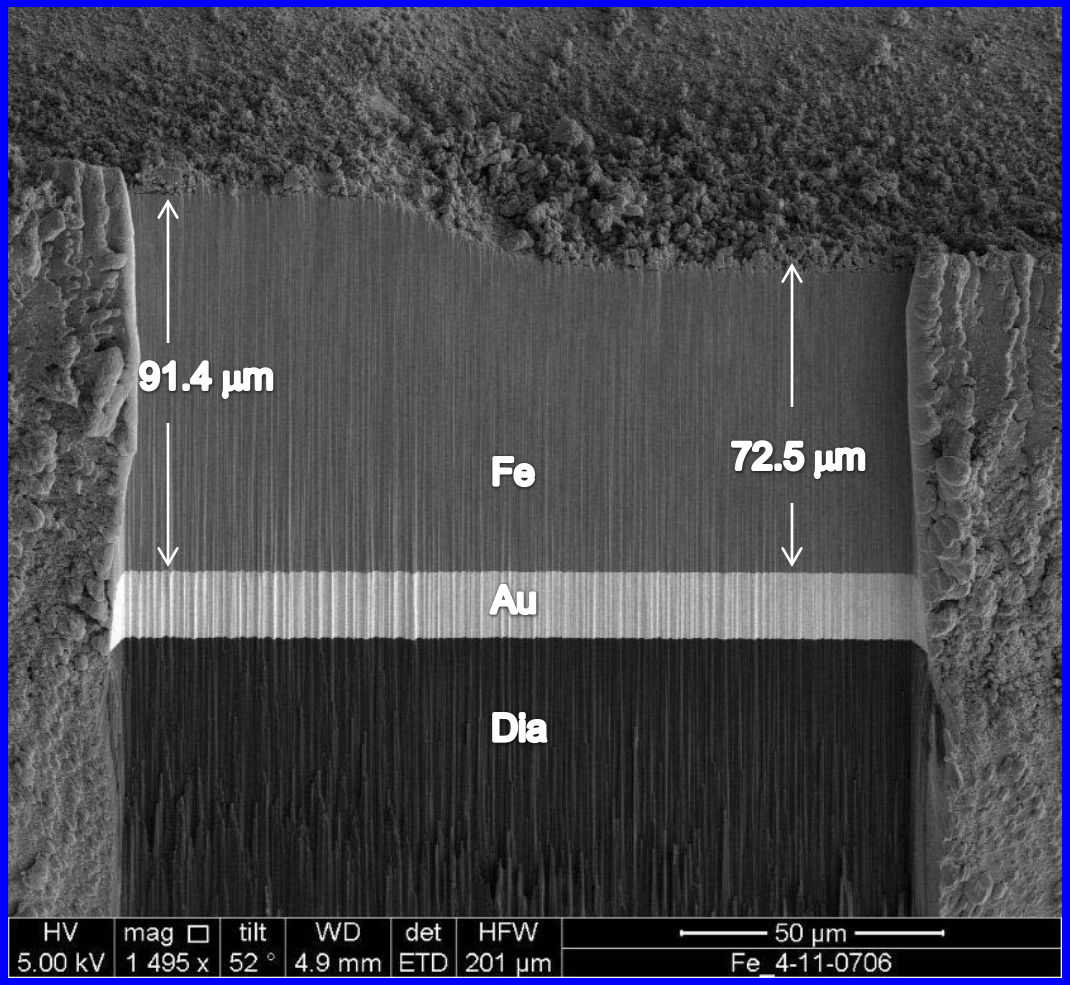




Estimated Rolloff ~ 96 microns

Estimated Rolloff ~ 36 microns

X-section using a FIB for sample prep and an SEM for imaging



Fe microstructure consistent with columnar grains with a small lateral dimension (nominally ~0.5 microns)

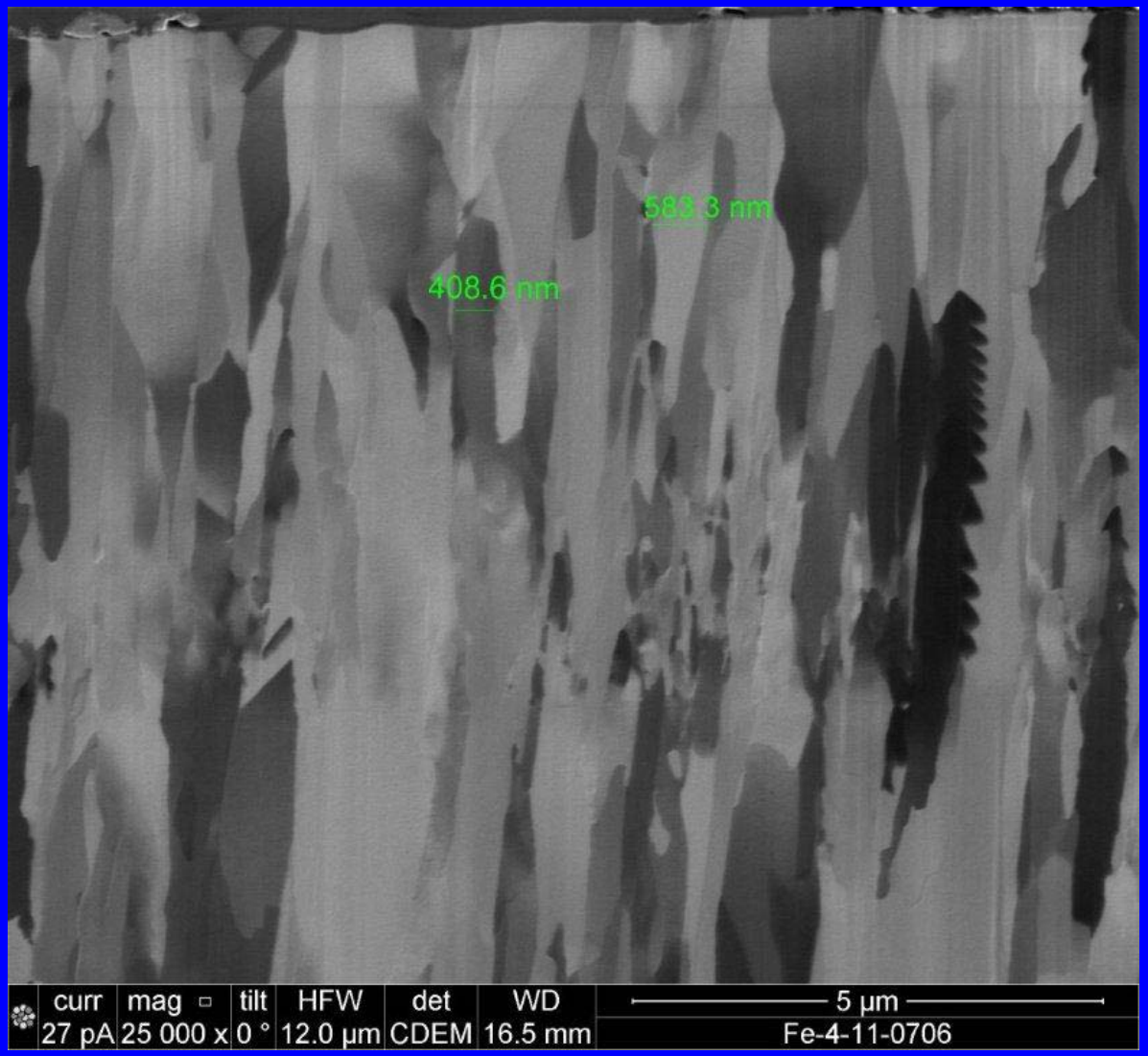
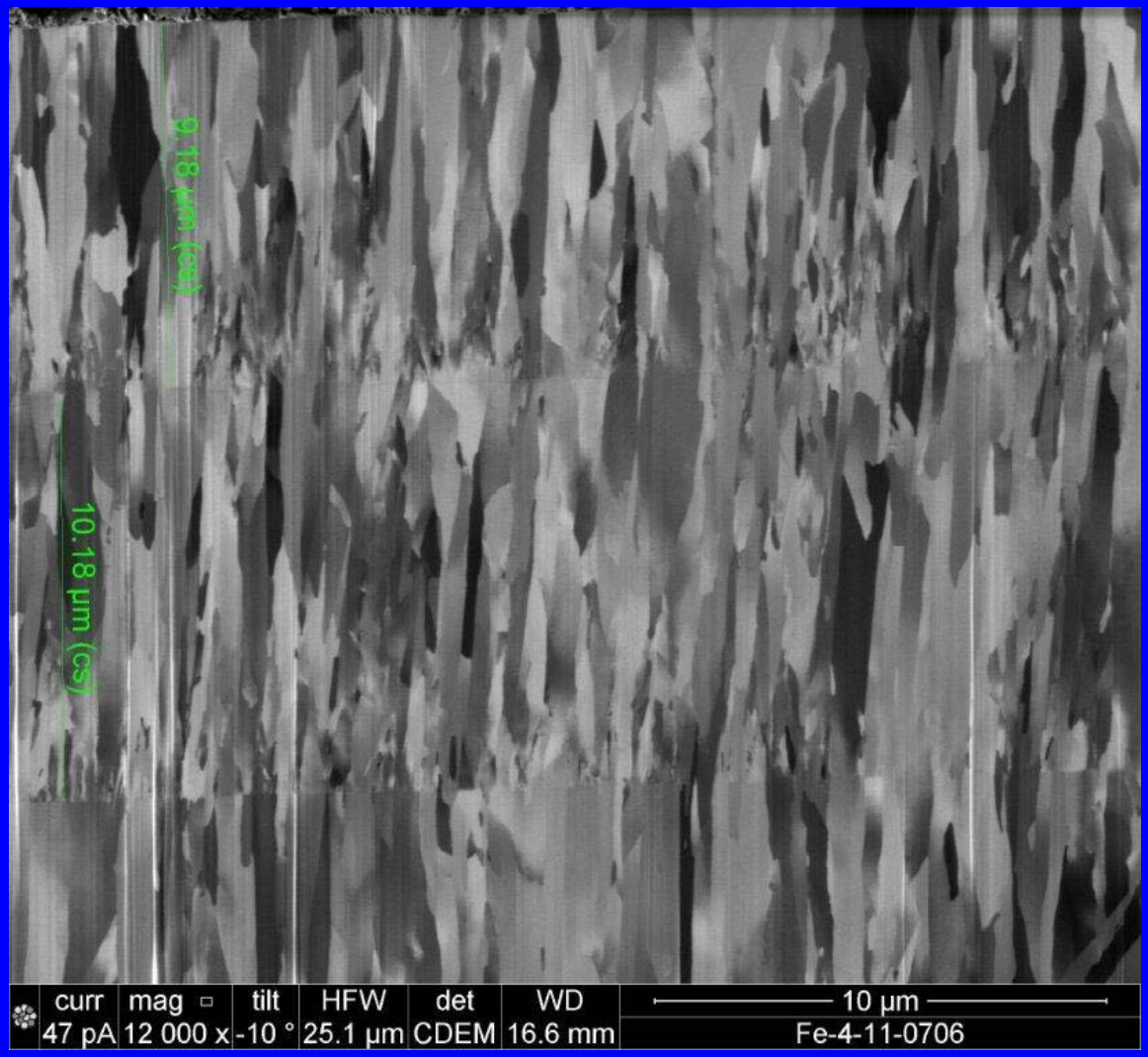


Image obtained with ion beam imaging (as opposed to electron imaging)

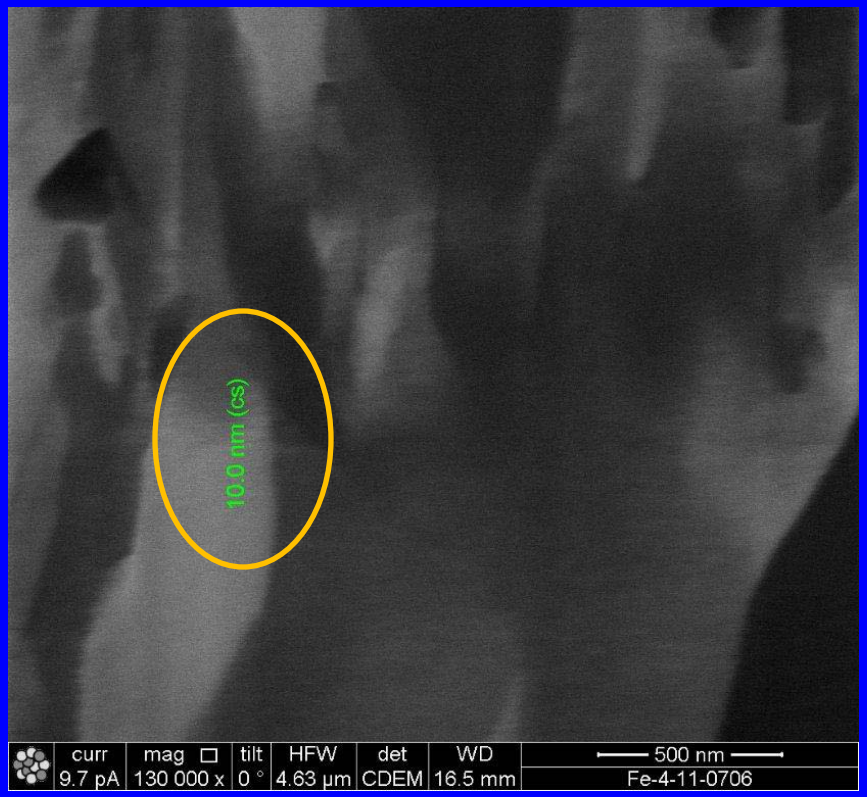
In this and other images, we can observe where the venting occurs.

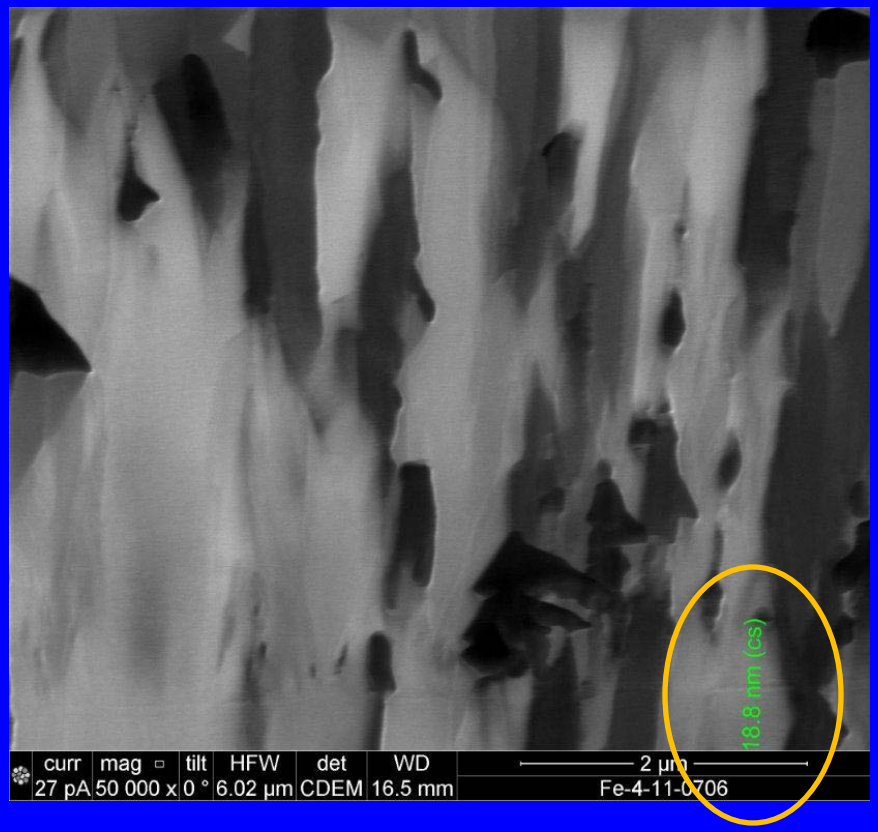
Venting occurred here



Interesting how little the venting appears to impact the microstructure?

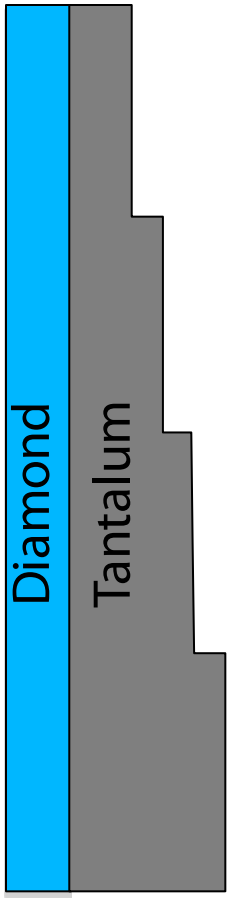
Difficult to estimate the thickness of the oxide layer, but we can put an upper bound of approximately 10-20 nm.

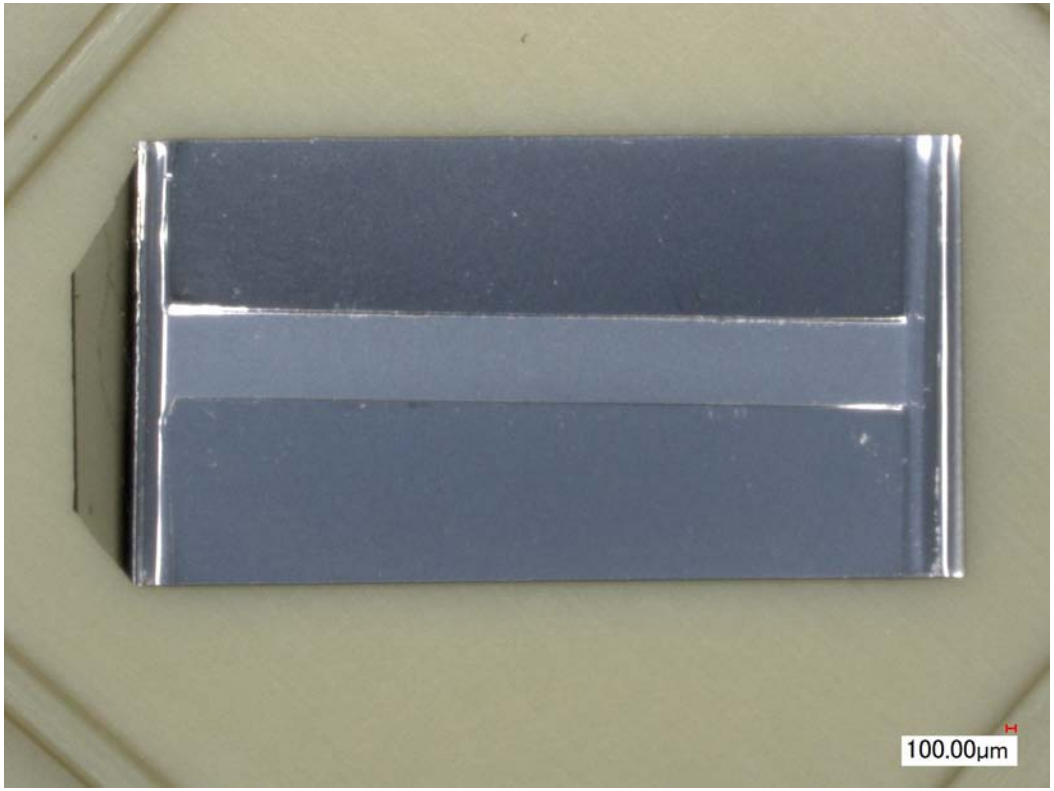




One interesting observation is that the oxide layer appears to have a minor effect on the grain growth
(which may suggest it is a thinner than 10-20 nm?)

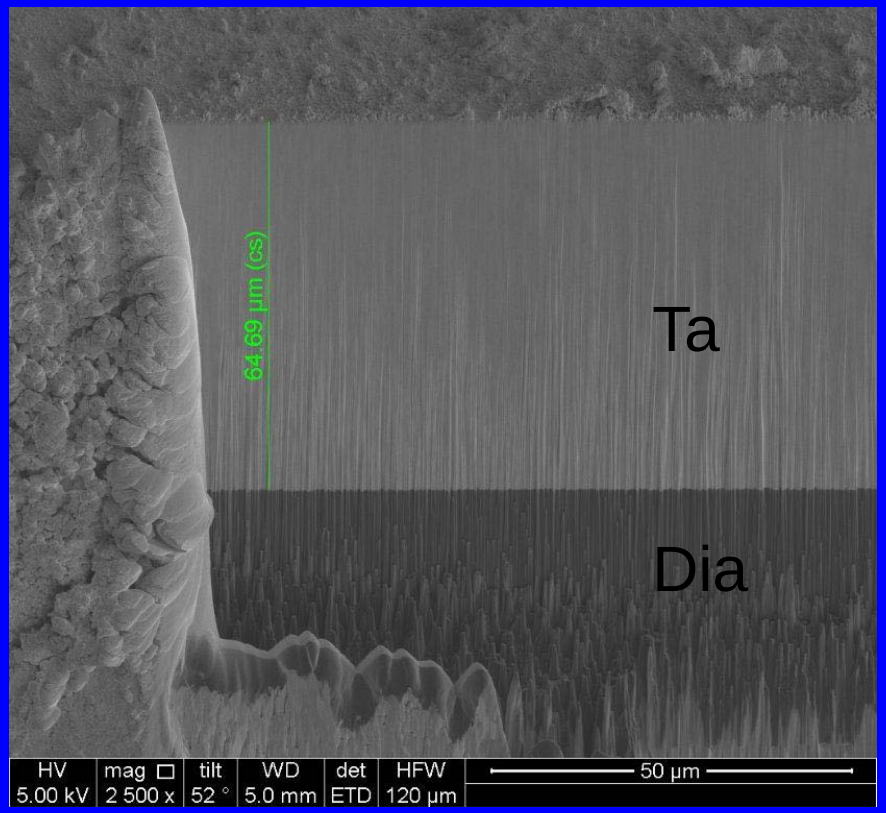
Thick Tantalum targets are also of significant interest for EOS experiments at NIF*

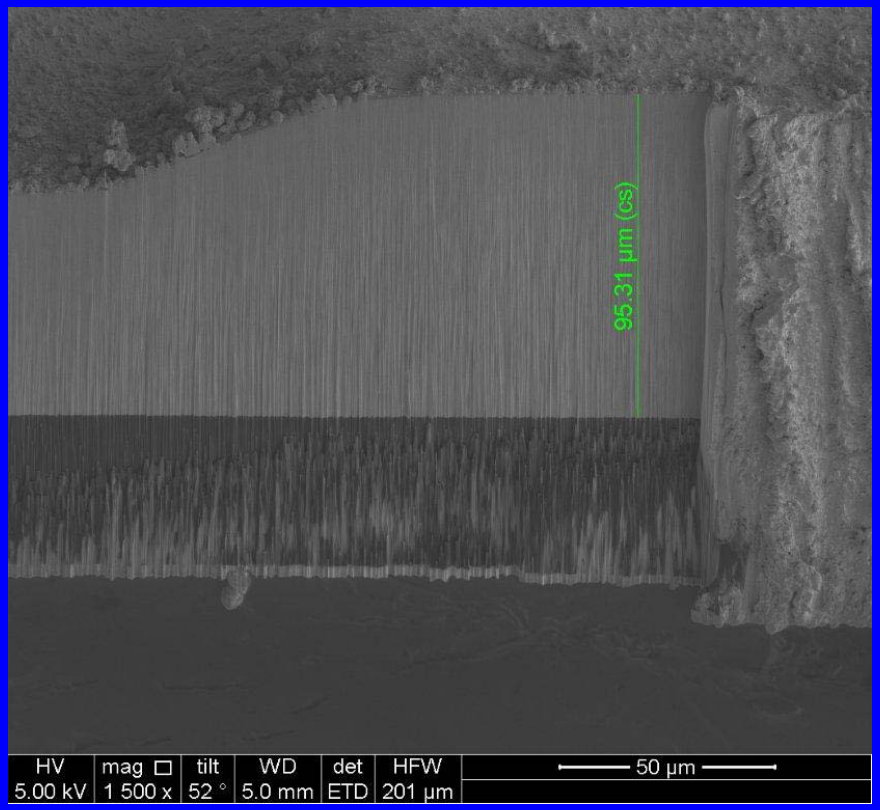




*Some of the reasons why cannot be discussed at this forum

X-section of a stepped Tantalum targets





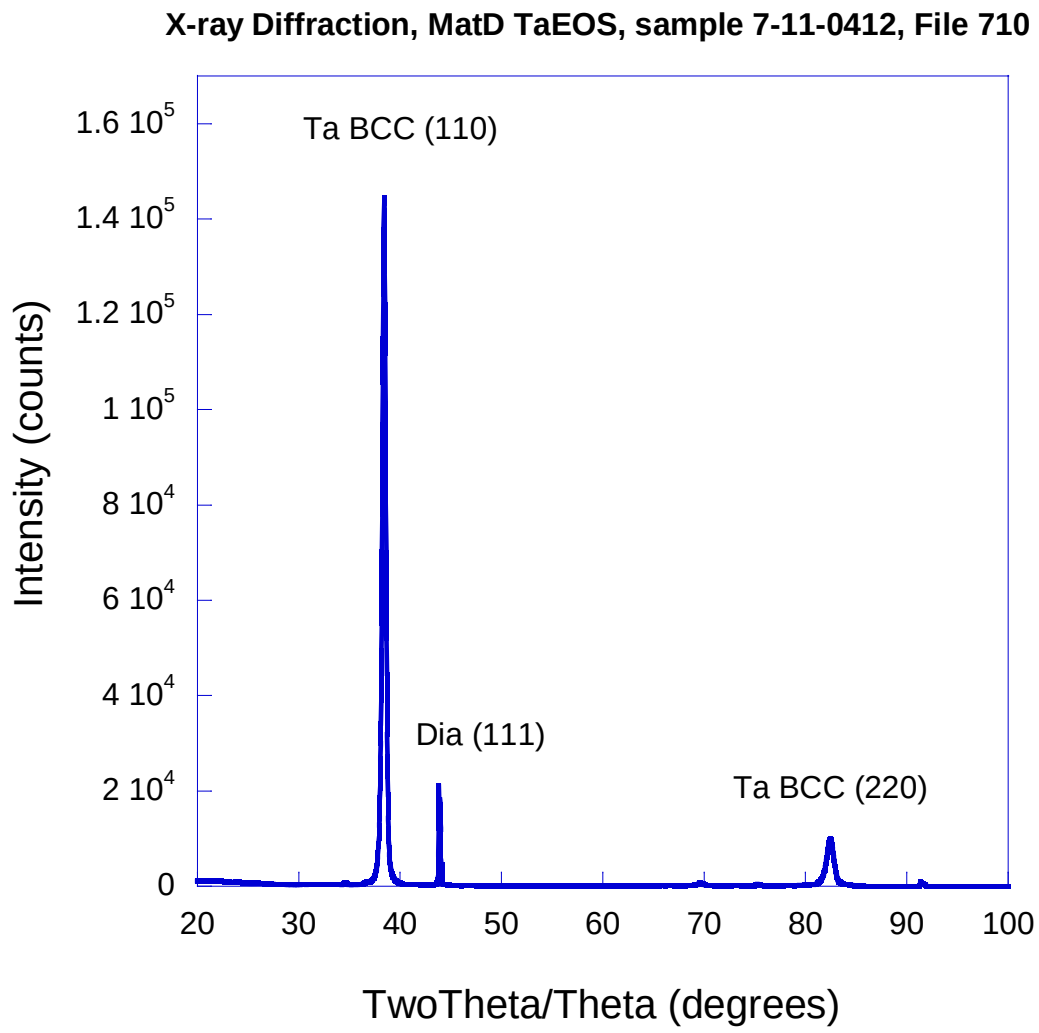
Ta base layer = 64.7 microns
(Goal was 65.0 microns)

Thickness of Ta base layer plus all three Ta steps = 95.3 microns
(Goal was 95.0 microns)

Note: Even though we have a number of instances where we came close to meeting the specifications, our consistency/yield could still be improved.

X-Ray Diffraction of the Tantalum Films on Diamond substrates.

- Important to do XRD since BCC phase not a given when depositing films
- Our Ta shows a strong (110) preferred orientation
- Need to have a substrate temp > 400 C to get BCC phase



We have been asked to sputter deposit very thick Tantalum films for upcoming experiments on the Z-machine.

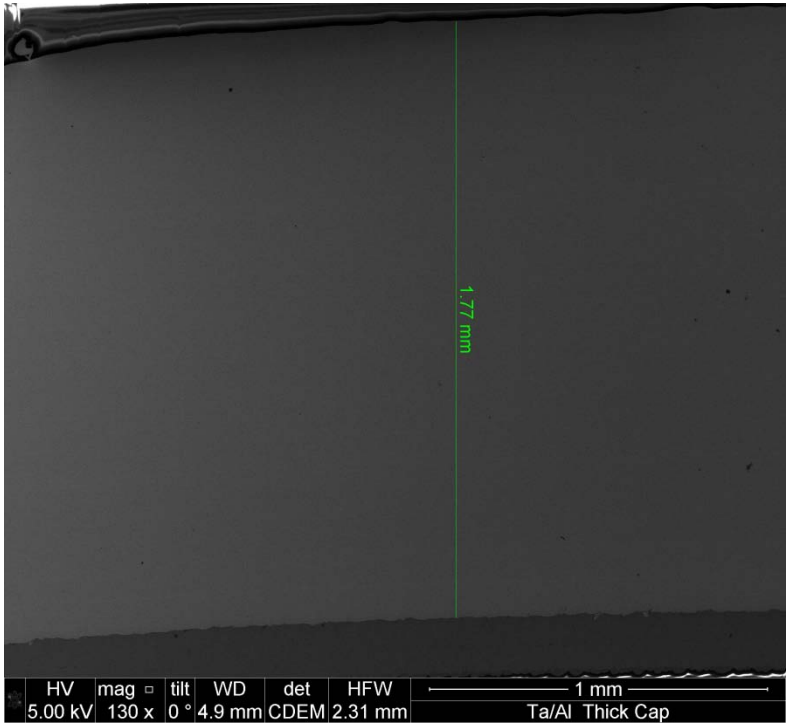
- They want to compare results across platforms and do not want microstructure differences to be a variable (i.e., want ultra thick film targets with microstructures similar to what was fielded on platforms like NIF and Omega)

- Design being fine tuned but will be ≥ 1 mm thick (1000 μm's!).

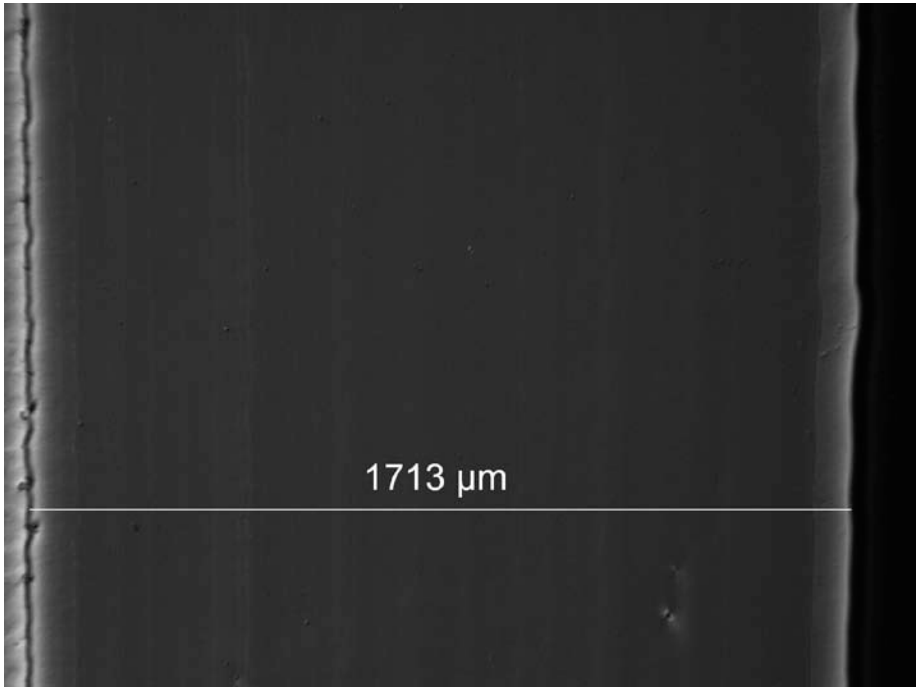
- Film will be freestanding (or on an aluminum substrate)

We believe we have deposited the thickest sputtered Tantalum film that has been achieved.

From SEM



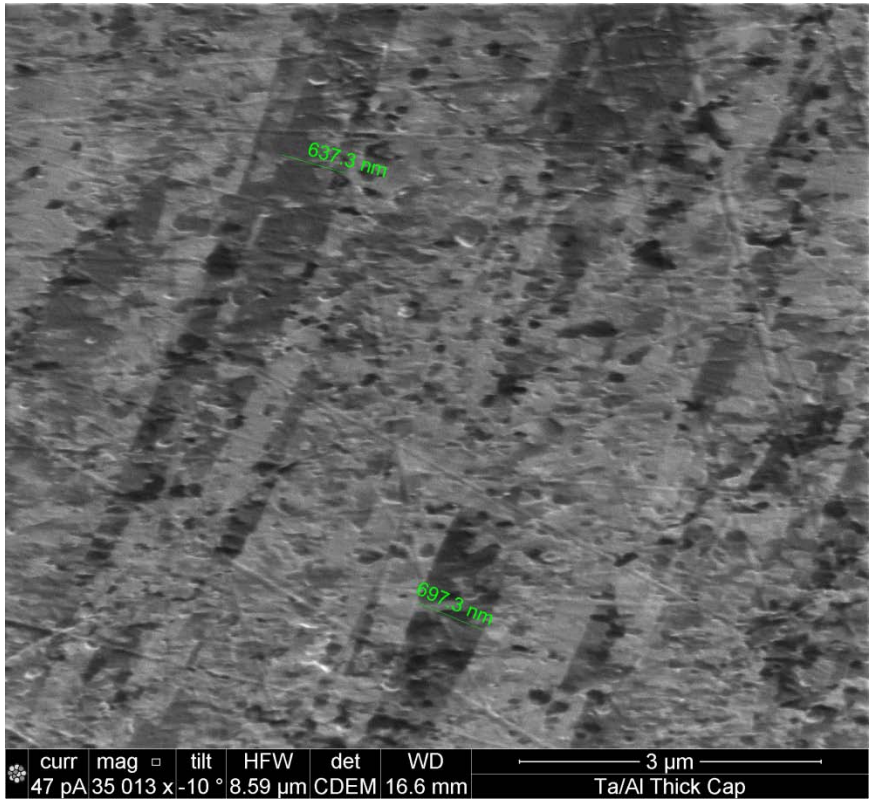
From Optical Microscopy



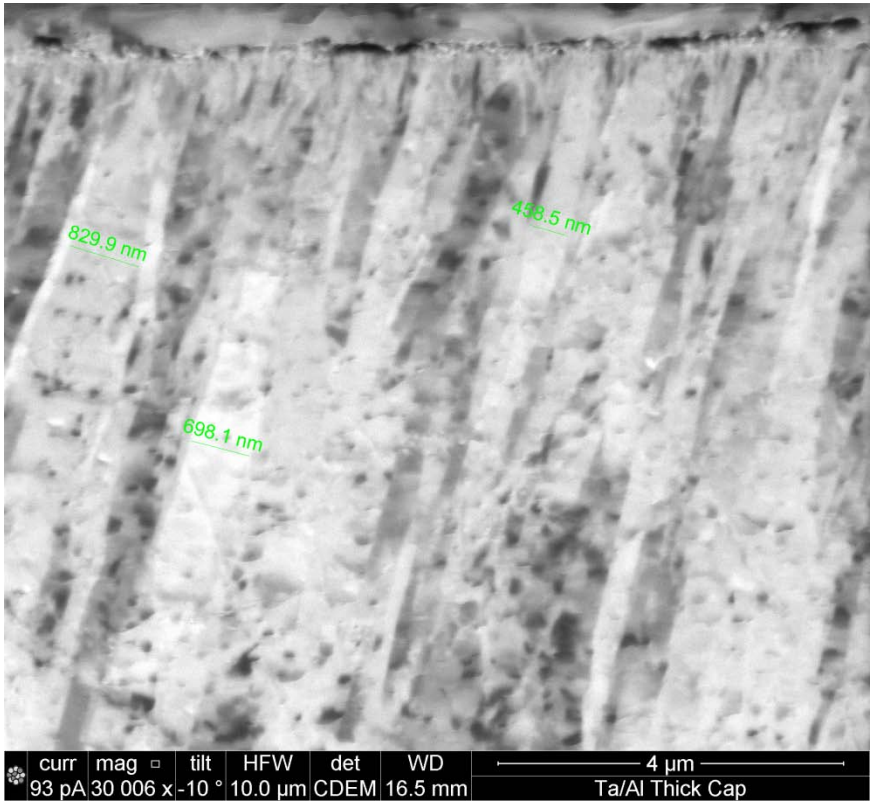
- Sectioned from the cover for our sample holder assembly.
- Ta film measures 1.75 mm thick!
- We believe the primary reason is that we are able to achieve a low film stress; for example, we measured ~26 MPa on a much thinner sputtered Tantalum film.

Not seeing the grain size change to much in this very thick film

Near substrate

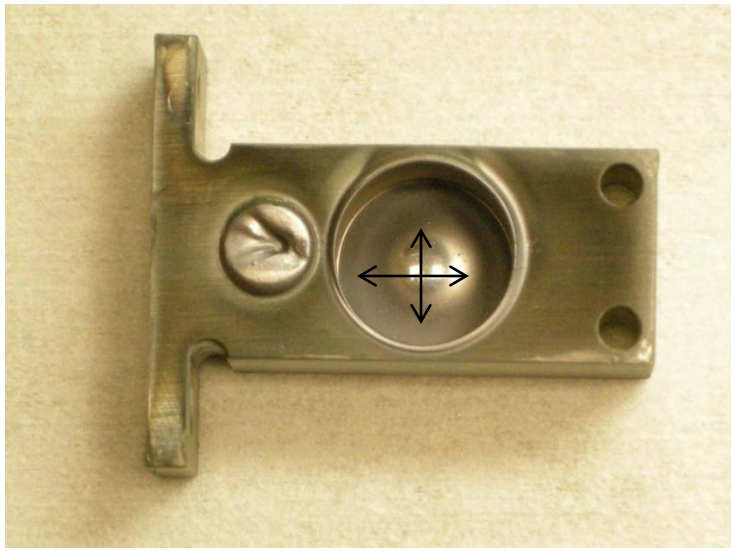


Near surface



- Grain size seems to be approx. ≤ 0.7 µm in either case
- Film was interrupted/vented a number of times, so this may help maintain the small grain size during film growth.

Have sputtered ~1 mm thick Ta films inside a ~1 cm deep hole



- Were able to deposit within a relatively deep hole. Hole was 1 cm diam by 1 cm deep
- Did observe nonuniformity in the coating thickness within the hole . . .

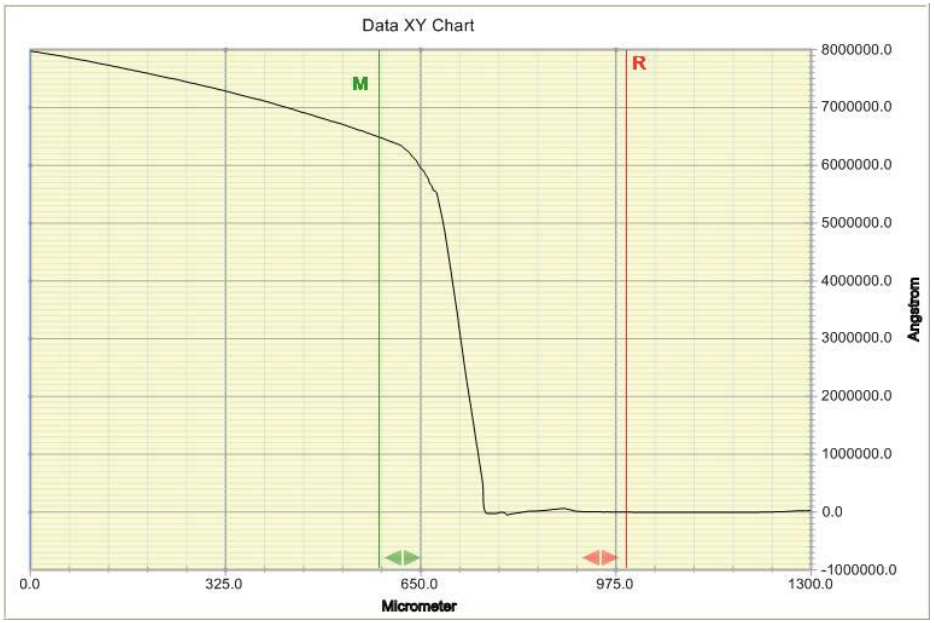
↔

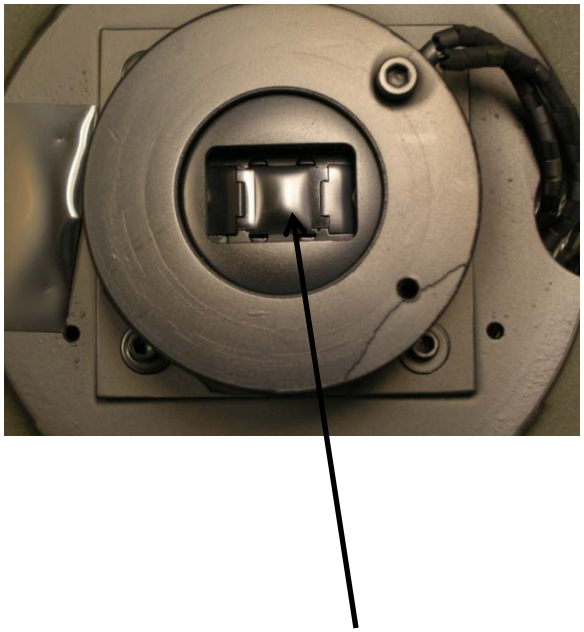
+0.4 cm	+0.2 cm	0 (center)	-0.2 cm	-0.4 cm
-0.47 mm	-0.12 mm	0	-0.12 mm	-0.41 mm

↕

+0.4 cm	+0.2 cm	0 (center)	-0.2 cm	-0.4 cm
-0.30 mm	-0.16 mm	0	-0.16	-0.30 mm

Have sputter deposited ~700 micron thick Ta films with good adherence, and then was able to remove the substrate to create a freestanding Tantalum film





Very Thick
Ta on Al

Conclusions

- We have successfully sputter deposited thick, multistepped Iron and Tantalum films for past and future EOS experiments on NIF.
- We have also successfully sputter deposited very thick Tantalum films for future experiments on Z.