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# Summary of Blast Shield and Material Testing for Development of Solid Debris Collection at the National Ignition Facility (NIF)

D. A. Shaughnessy, J. M. Gostic, K. J. Moody, P. M. Grant, L. A. Lewis, I. D. Hutcheon

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## Disclaimer



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# Summary of Blast Shield and Material Testing for Development of Solid Debris Collection at the National Ignition Facility (NIF)

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Chemical Sciences Division

The ability to collect solid debris from the target chamber following a NIF shot has application for both capsule diagnostics, particularly for fuel-ablator mix, and measuring cross sections relevant to the Stockpile Stewardship program and nuclear astrophysics. Simulations have shown that doping the capsule with up to $10^{15}$ atoms of an impurity not otherwise found in the capsule does not affect its performance (see D.A. Shaughnessy *et al*., "Evaluation of the RADCHEM Diagnostic as an Assessment of Fuel-Ablator Mix and Fuel Rho R", LLNL Technical Report LLNL-TR-472595, 2011). The dopant is an element that will undergo nuclear activations during the NIF implosion, forming radioactive species that can be collected and measured after extraction from the target chamber. For diagnostics, deuteron or alpha induced reactions can be used to probe the fuel-ablator mix (see D.A. Shaughnessy *et al.,* "Alternate Alpha Induced Reactions for NIF Radiochemistry", LLNL Technical Report LLNL-TR-424893, 2010.) For measuring neutron cross sections, the dopant should be something that is sensitive to the 14 MeV neutrons produced through the fusion of deuterium and tritium.

Developing the collector is a challenge due to the extreme environment of the NIF chamber. The collector surface is exposed to a large photon flux from x-rays and unconverted laser light before it is exposed to a debris wind that is formed from vaporized material from the target chamber center. The photons will ablate the collector surface to some extent, possibly impeding the debris from reaching the collector and sticking. In addition, the collector itself must be mechanically strong enough to withstand the large amount of energy it will be exposed to, and it should be something that will be easy to count and chemically process. In order to select the best material for the collector, a variety of different metals have been tested in the NIF chamber. They were exposed to high-energy laser shots in order to evaluate their post-shot surface characterization, morphology, degree of melt, and their ability to retain debris from the chamber center.

The first set of samples consisted of 1 mm thick pieces of aluminum that had been fielded in the chamber as blast shields protecting the neutron activation diagnostic. Ten of these pieces were fielded at the equator and one was fielded on the pole. The shields were analyzed using a combination of scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), x-ray fluorescence (XRF), neutron activation analysis (NAA) and chemical leaching followed by mass spectrometry. On each shield, gold debris originating from the gold houlraum was observed, as well as large quantities of debris that were present in the center of the target chamber at the time of the shot (i.e., stainless steel, indium, copper, etc.) Debris was visible in the SEM as large blobs or splats of material that had encountered the surface of the aluminum and stuck. The aluminum itself had obviously melted and condensed, and some of the large debris splats arrived after the surface had already hardened. Melt depth was

determined by cross sectioning the pieces and measuring the melted surface layers via SEM. After the SEM analysis was completed, the pieces were sent for NAA at the USGS reactor and were analyzed by U. Greife at the Colorado School of Mines. The NAA showed that the majority of gold mass present on the shields was not in the form of large blobs and splats, but was present as small particulates that had most likely formed as condensed vapor. Further analysis showed that the gold was entrained in the melted aluminum surface layers and did not extend down into the bulk of the aluminum. Once the gold mass was accounted for from the NAA, it was determined that the aluminum fielded at the equator was collecting a fraction of the total gold hohlraum mass equivalent to 120% ± 10% of the solid angle subtended by the shield. The attached presentation has more information on the results of the aluminum blast shield analysis. In addition to the information given in the presentation, the surfaces of the shields have been chemically leached and submitted for mass spectrometric analysis. The results from that analysis are expected to arrive after the due date of this report and will be written up at a later time.

Based on the results of the aluminum blast shield analysis, it was determined that additional materials needed to be tested as potential collectors in the NIF chamber. 1-2 mm thick pieces of tantalum, niobium, vanadium, silver, titanium, molybdenum, and graphite foil were fielded in the Wedge Range Filter (WRF) mount at a distance of 50 cm from target chamber center during the shock timing campaign. The pieces were subsequently removed and analyzed in a similar fashion to the aluminum shields. As of this writing, the pieces are still under analysis, but initial results indicate that gold debris was collected on the various materials. Currently, the pieces are being cross-sectioned so that the melt depths of each material can be compared. In addition, NAA and/or mass spectrometry will be performed in order to determine the total gold mass that was collected on each surface. This information will help us down-select the best material(s) to use as a collector based on collection efficiency and mechanical strength. It appears in the SEM images that molybdenum was splattered out of the collector onto the neighboring metallic pieces, which may mean it is too soft to use as a collector at NIF. The other materials have to be evaluated as well. Some pieces were fielded at the pole as opposed to the equator; those pieces have much more damage from high velocity particles and shrapnel than pieces fielded at the equator. The equator pieces seem to have a uniformly deposited layer of debris as compared to the pits and craters seen in the polar pieces. Although capsule debris has not yet been identified at either position due to the small mass of measurable germanium in the capsules (high sensitivity analysis is in process), it seems that fielding a collector at the equator would have a higher probability of collecting the debris we are interested in. For our purposes, a larger quantity of high-Z material would need to be doped into the capsule for us to make a meaningful measurement.

In addition to the various metals, we also fielded a pairing of tantalum pieces where one was polished and the other surface was not polished. Based on our initial results, the polished surfaces appear to have collected more material than the unpolished surfaces by a factor of two. The reasons for this are being examined, but it is likely that the unpolished surfaces ablated more material and prevented the debris from reaching the metal surface. This implies that any future collectors should have surfaces polished to within ± 1 micron surface finish regardless of the metal used. These same samples were also radiation counted approximately 24 hours after the shot occurred (DT capsule). Gamma radiation from neutron activated

hohlraum gold was observed and using a simplified model of the hohlraum geometry resulted in > 50% solid angle collection efficiency for the polished tantalum surface. This was the first example of activated, solid debris collection from the NIF chamber following a shot.

The last set of samples that were fielded were ride-along pieces that were placed behind a debris wind measurement in the nose cone of the DIM. The cone was open to the chamber and the debris wind measurement consisted of several tabs fielded on the circumference of the inside of the nose cone, leaving an open line of sight from the nose cone opening into the cone itself. Tantalum and vanadium were fielded (separately) behind the debris wind tabs in an effort to look for gold collection. The purpose of this experiment was to determine if having a conical shape or tube as the collector opening would somehow enhance the amount of debris collected. Initial examination of the tantalum and vanadium foils show that no gold was collected on the back foil, however there appears to be high-Z material on the inner surface of the cone. The cones have been sectioned and we are in the process of analyzing the identity of the material inside to determine if it is either gold or ablated tantalum or vanadium. From our initial results, it appears that using a conical geometry did not enhance the collection of chamber debris, but the pieces are still under analysis.

The next step for turning our work into an actual diagnostic tool at NIF is to increase the surface area of the collector. This can be accomplished by fielding more pieces in the chamber during a given shot, or designing a larger area mount similar to the WRF that covers a larger solid angle. Once our analyses of the various samples are complete, we will be able to select the best collector material.

Attached presentation: "Development of Radiochemical Diagnostics at NIF through Collection of Solid and Gaseous Debris"

# Development of Radiochemical Diagnostics at NIF Through Collection of Solid and Gaseous Debris

**Fortner Meeting**
**June 23, 2011**

**Julie Gostic and Dawn Shaughnessy**

**for the NIF Radiochemistry Collaboration**
**Chemical Sciences Division / Physical and Life Sciences**

Lawrence Livermore National Laboratory • National Ignition Facility & Photon Science

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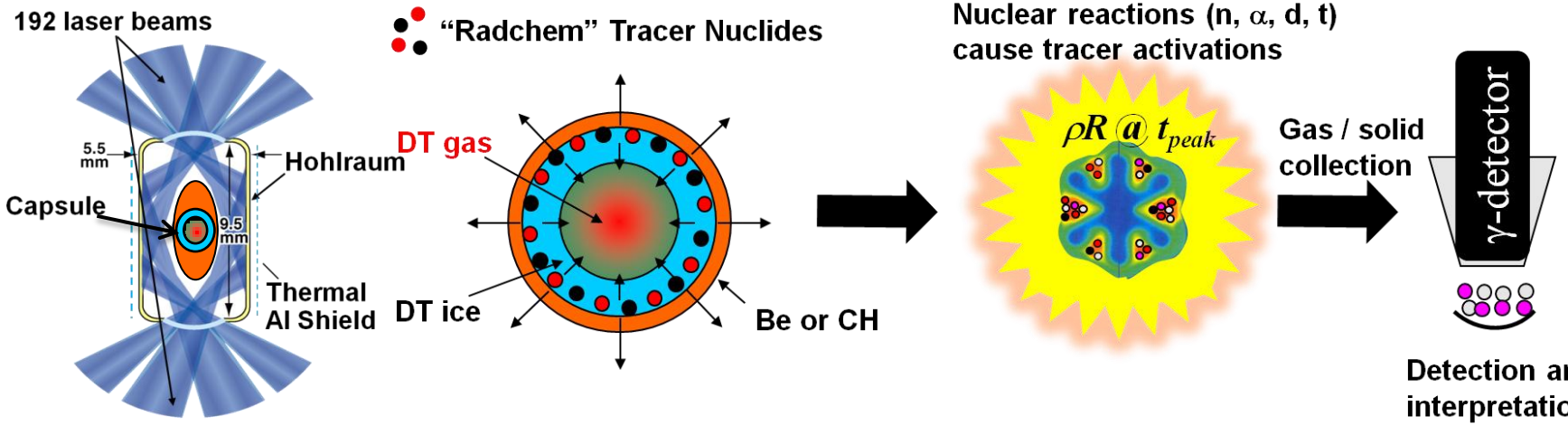
# Collection of the activated target debris after implosion for radiochemical diagnostics are under development.




**Longest range – probes remaining shell.**

$(^{124}Xe + n \rightarrow {}^{123}Xe + 2n)$

*$E_{th}$ = 8.7 MeV 1.4 barn (14.5 MeV)*

$(^{124}Xe + n \rightarrow {}^{125}Xe + \gamma)$

*low $E_{th}$ 0.01 barn (1 MeV) 0.002 barn (5 MeV)*

**Intermediate range – probes inner shell.**

$(^{127}I + d \rightarrow {}^{127}Xe + 2n)$, $^{79}Br(d,2n)^{79}Kr$

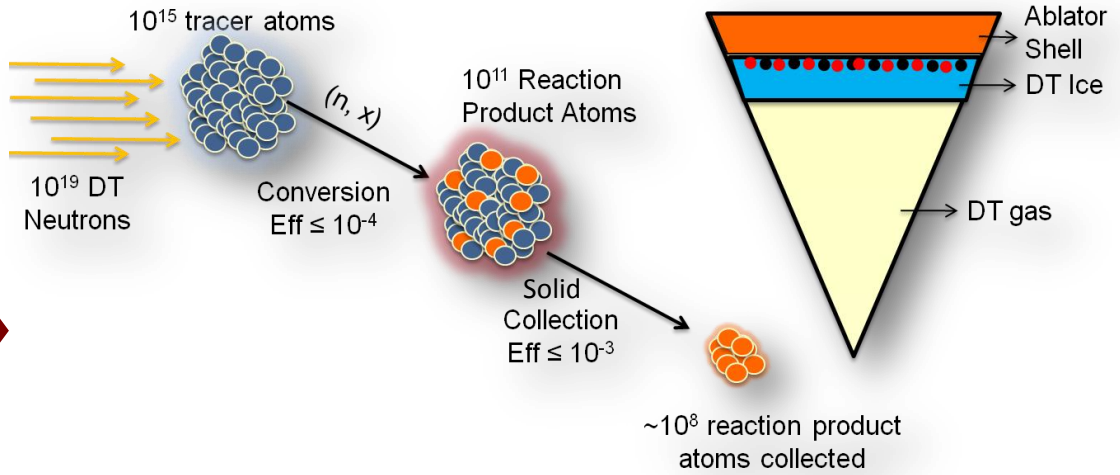
*$E_{th}$ = 4.2 MeV 0.4 barn (10 MeV)*

**Shortest range – probes hot spot region.**

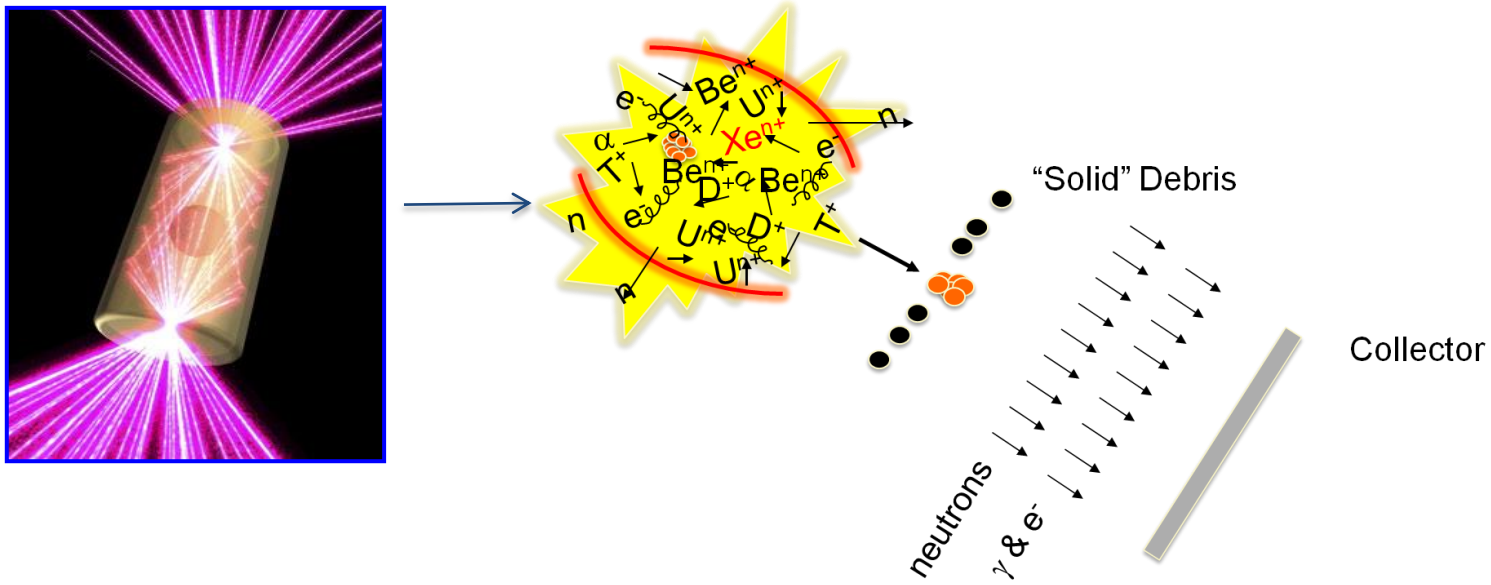
$(^{18}O + \alpha \rightarrow {}^{21}Ne + n)$

*$E_{th}$ = 1.8 MeV 0.2 barn (3.5 MeV)*

Based on simulations, only $10^{15}$ target atoms can be loaded into the capsule without interfering with implosion performance.



# Collection efficiency must be optimized based on the specific elements loaded into the capsule.

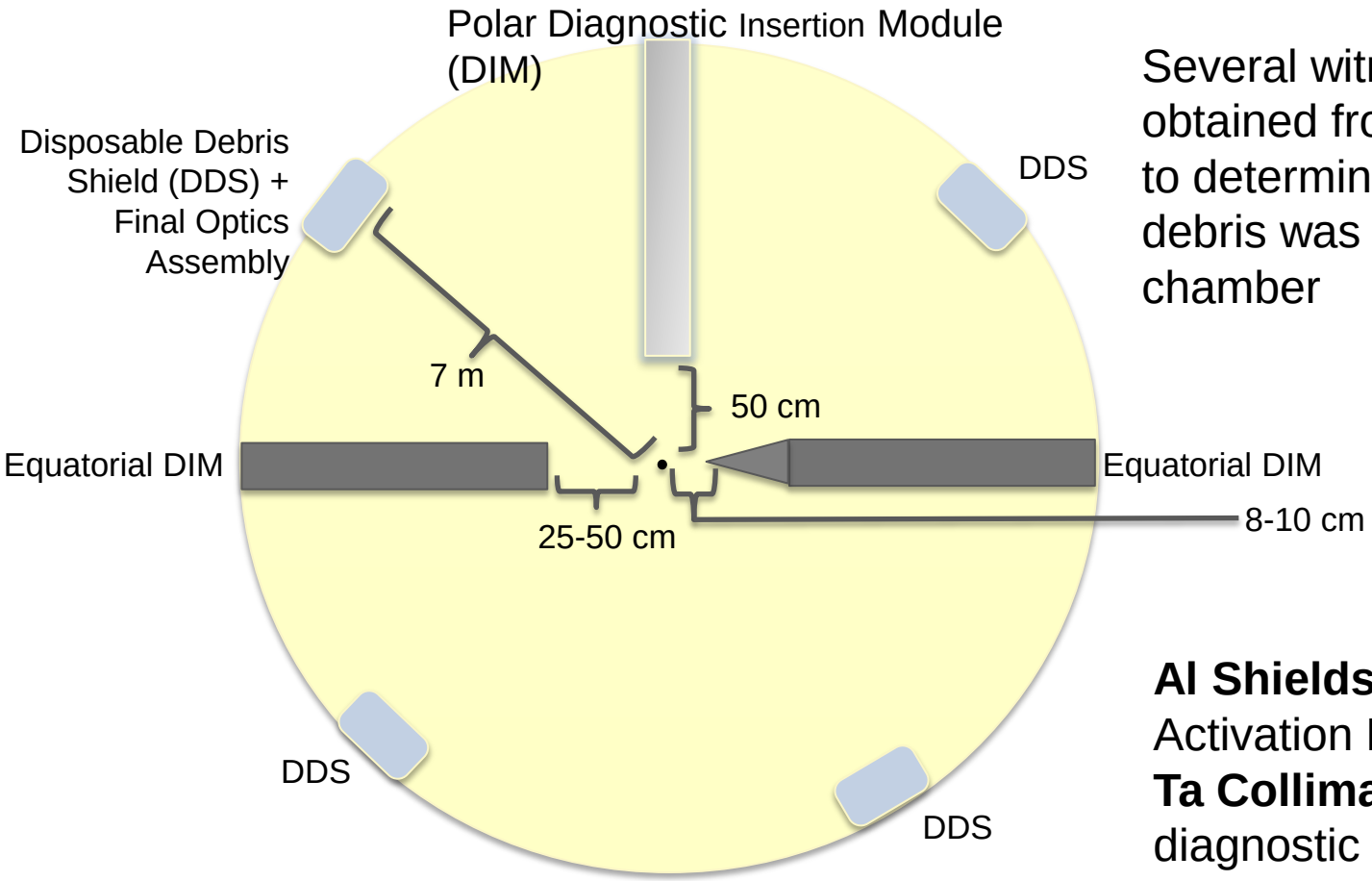



- For solid collection, debris from target chamber center (TCC) must be identified.
- There may be a directionality of the debris based on the hohlraum design.
- We must evaluate optimal collector materials and how far away they must be from TCC.
  - Sample loss due to ablation of the material must be minimized
- Once these issues are resolved, the geometry of the collection device must be evaluated
  - Many engineering "lessons-learned" are available through other nuclear diagnostics that are currently being commissioned.

# Solid Collection Efforts at NIF

The story so far…on a limited budget.

# Our initial focus has been on determining what type of debris is generated inside the target chamber.




Several witness plates were obtained from different areas to determine what type of debris was generated in the chamber

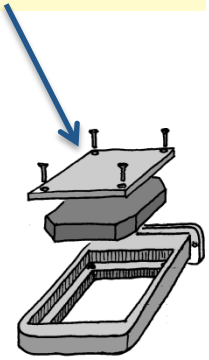
**Al Shields:** covered Neutron Activation Diagnostic
**Ta Collimators**: part of x-ray diagnostic assembly
**Glass DDS**: covered the final optics assembly

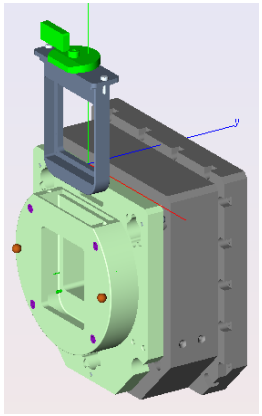
Drawing Not to Scale, Side View

Chamber diameter is ~10 meters

# Several aluminum blast shields have been analyzed to characterize chamber debris.

Protected Neutron Activation Diagnostic

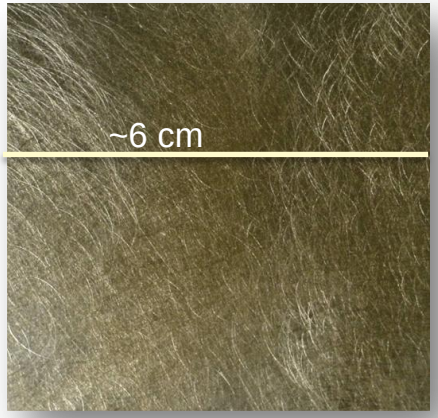




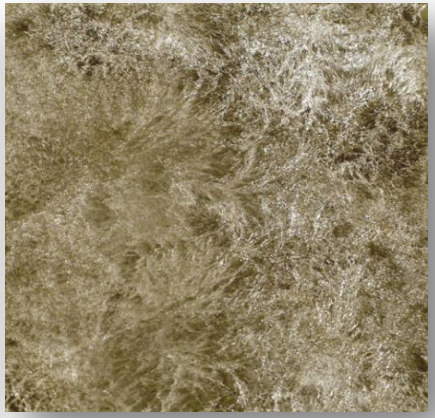
Analyzed Al witness plates (n =11) that were fielded 25 – 50 cm from Target Chamber Center during the Fall 2009 campaign for shot energies ranging from 50 kJ – 1 MJ.

Equatorial DIM Mounts, 25 cm from TCC

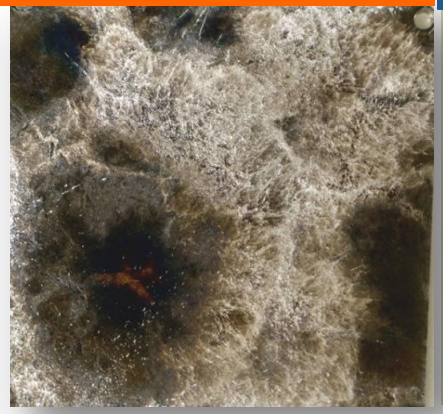
Polar DIM Mount, 50 cm from TCC




46 kJ, $10^9$ neutrons



689 kJ, $10^9$ neutrons



836 kJ, $10^9$ neutrons


567 kJ, $10^{10}$ neutrons

Exploding Pusher, No hohlraum

Cryogenic D-D capsules with a Au hohlraum

# The surface of each shield was examined by secondary electron microscopy (SEM).

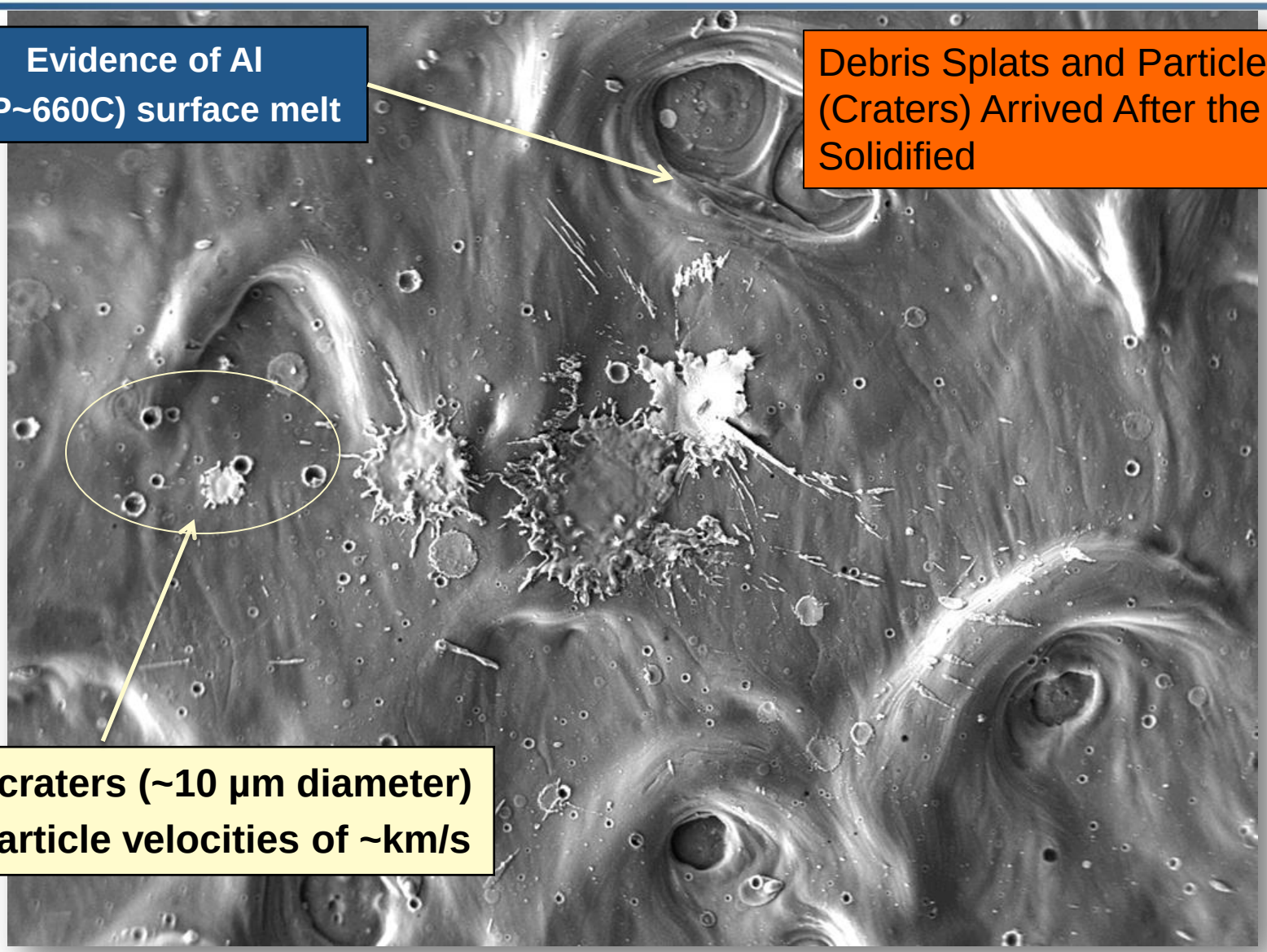



Image field of view = 300 μm

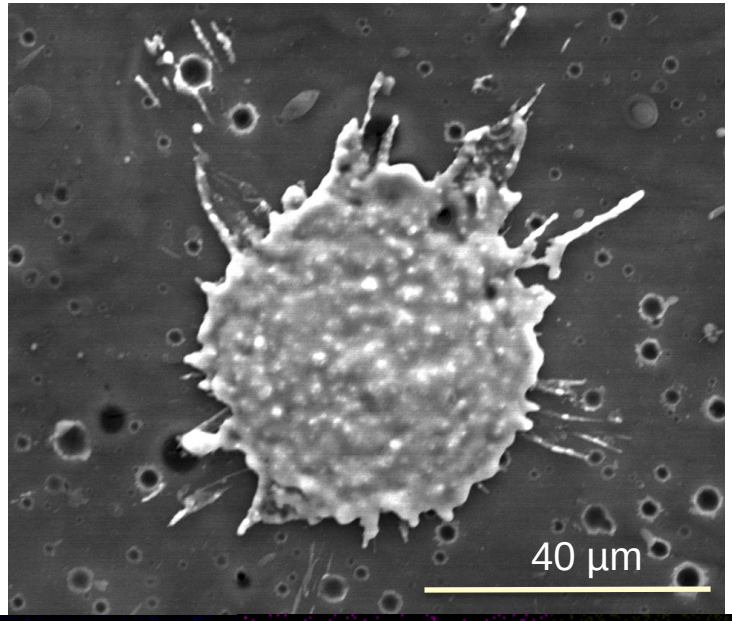
# Elements identified by Energy Dispersive Spectroscopy (EDS) were traced to structural components inside the target chamber.

| | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | Ar |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| Fr | Ra | Ac | | | | | | | | | | | | | | |

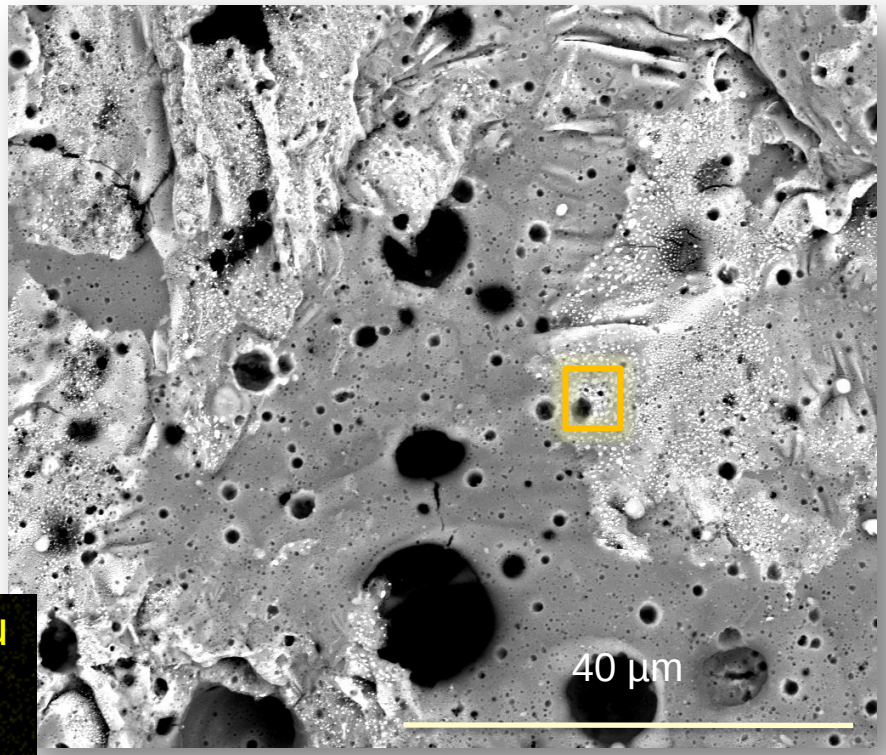
| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
| Th | Pa | U | Np | Pu | Am | Cm | Bk | Cf | Es | Fm | Md | No | Lr |

Au and Ge are not used in the chamber and are only associated with the target assembly.

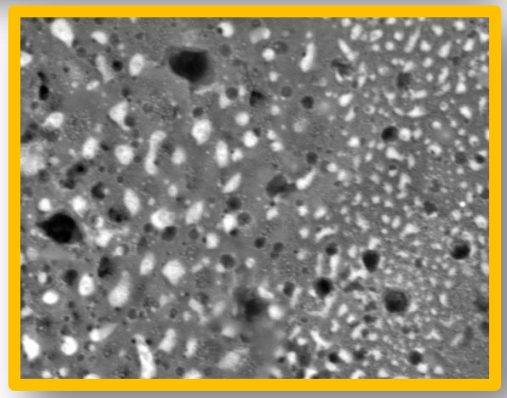
# Gold debris from target center was identified.



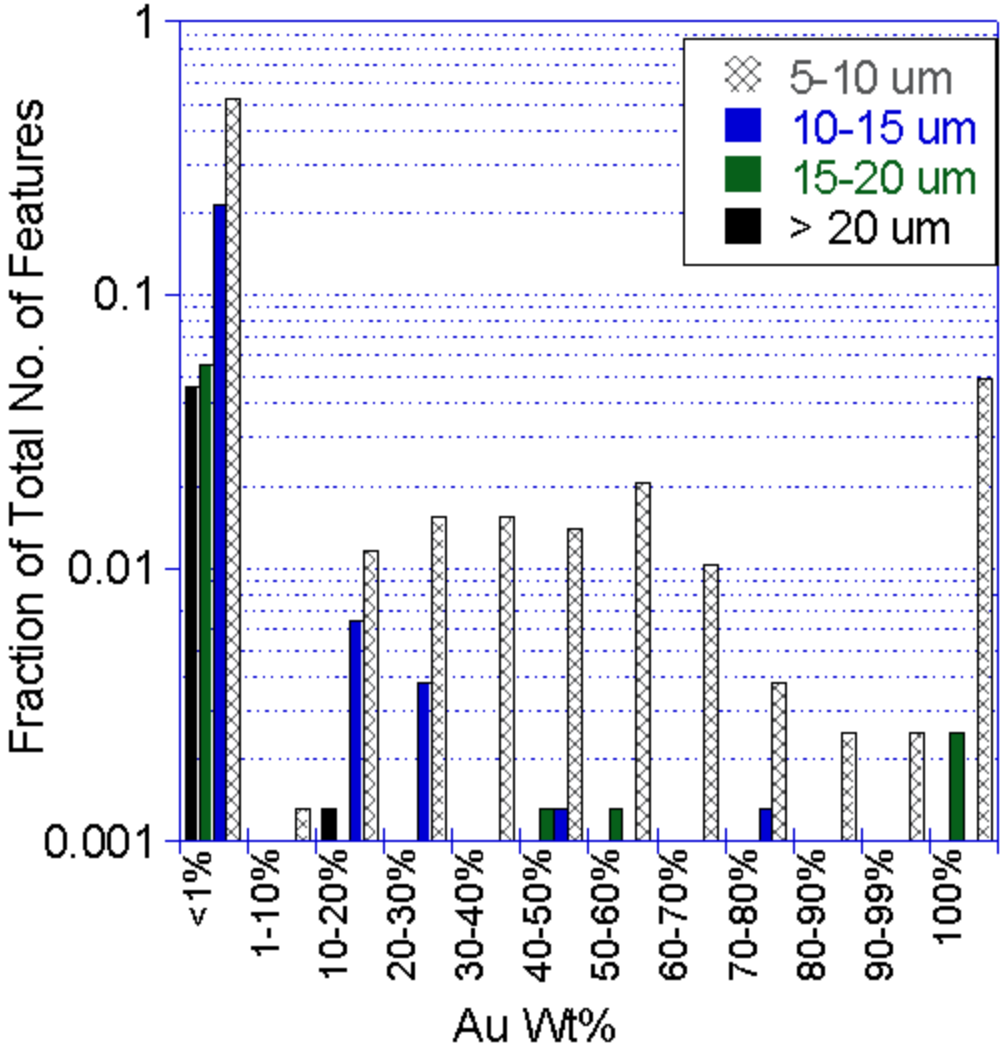




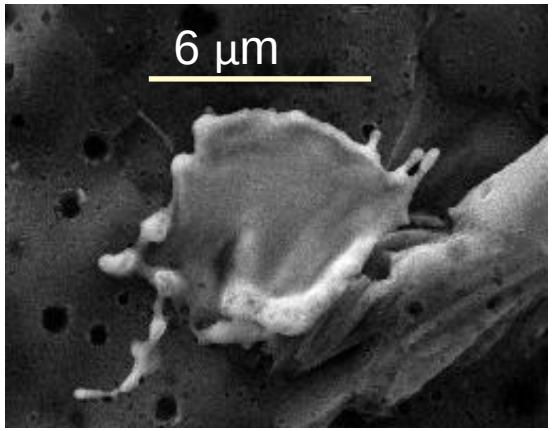

- Unique elemental signature – usually incorporated with Si and Cu
- These sub-micron Au features contribute little in terms of mass collection but may be of interest in terms of chemical/geometric fractionation.

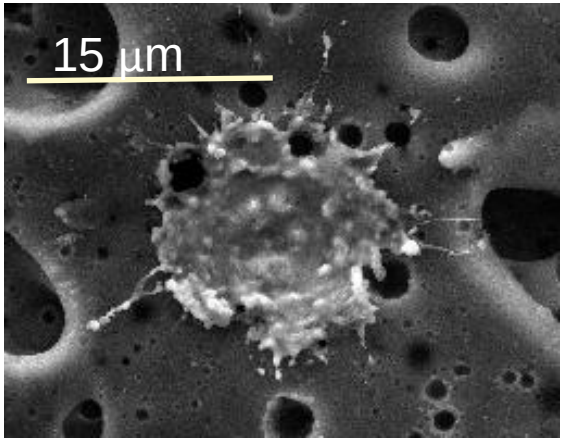


# SEM and Energy Dispersive Spectroscopy (EDS) indicate differences in Au content based on size.

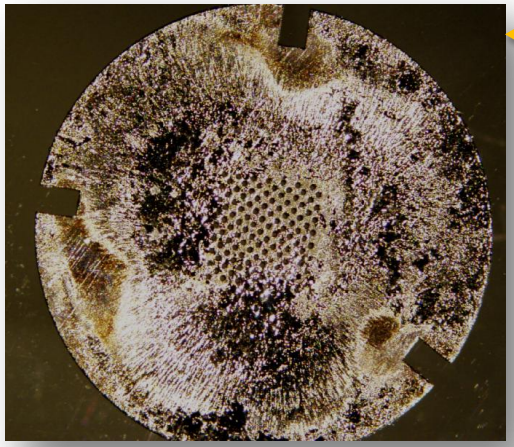



- The majority of the features contain little to no Au and are smaller than 10 μm.

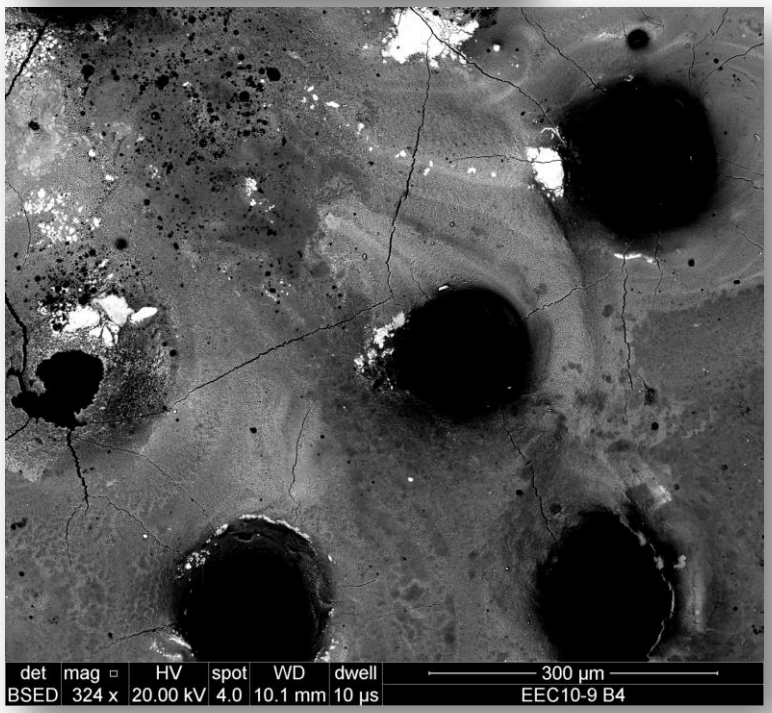


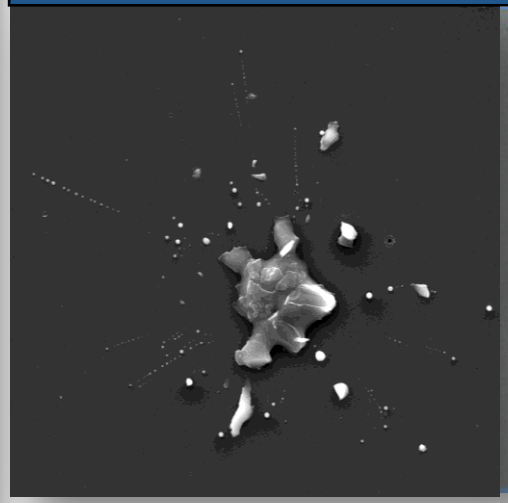
# Analysis of materials both close to TCC and at the chamber wall allow for optimized collection distance.

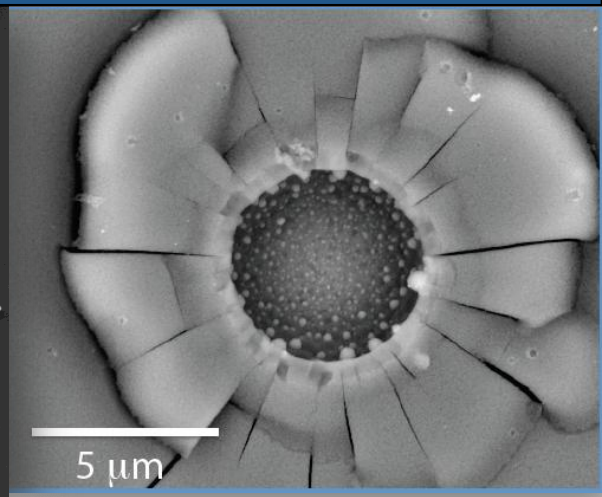


- This Ta collimator (75 µm thick) was fielded on the end of the GXD, 80 mm from TCC.
- Melt region on front side is mainly stainless steel ($MP_{SS}$~1500C) from the holding ring; there is also evidence of Ta melt ($MP_{Ta}$= 2980ºC)

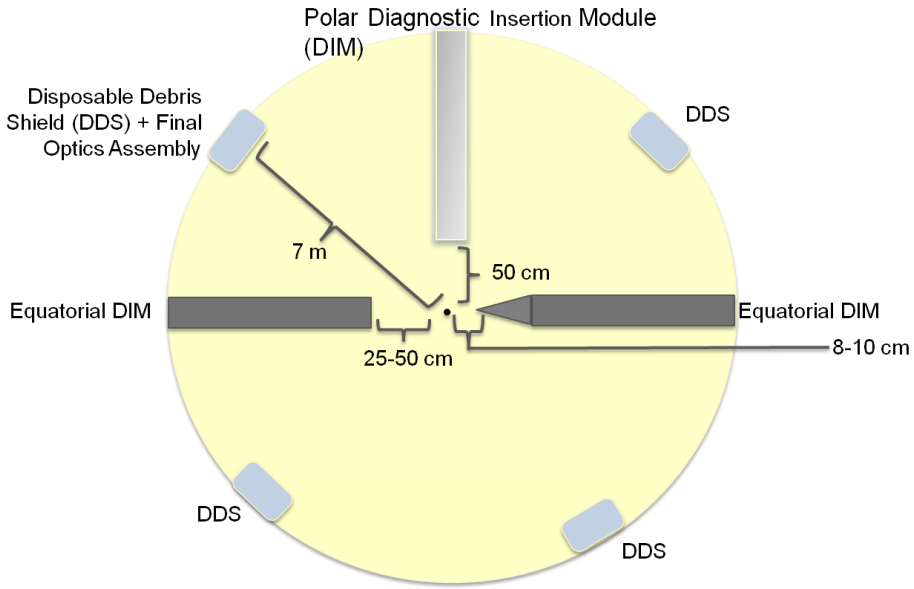



**Particle impact craters and molten splats were found on the DDS (7 meters from TCC), but at much lower concentrations.**





# Gold has been identified on all three shield types.




## Percent of 4π Covered

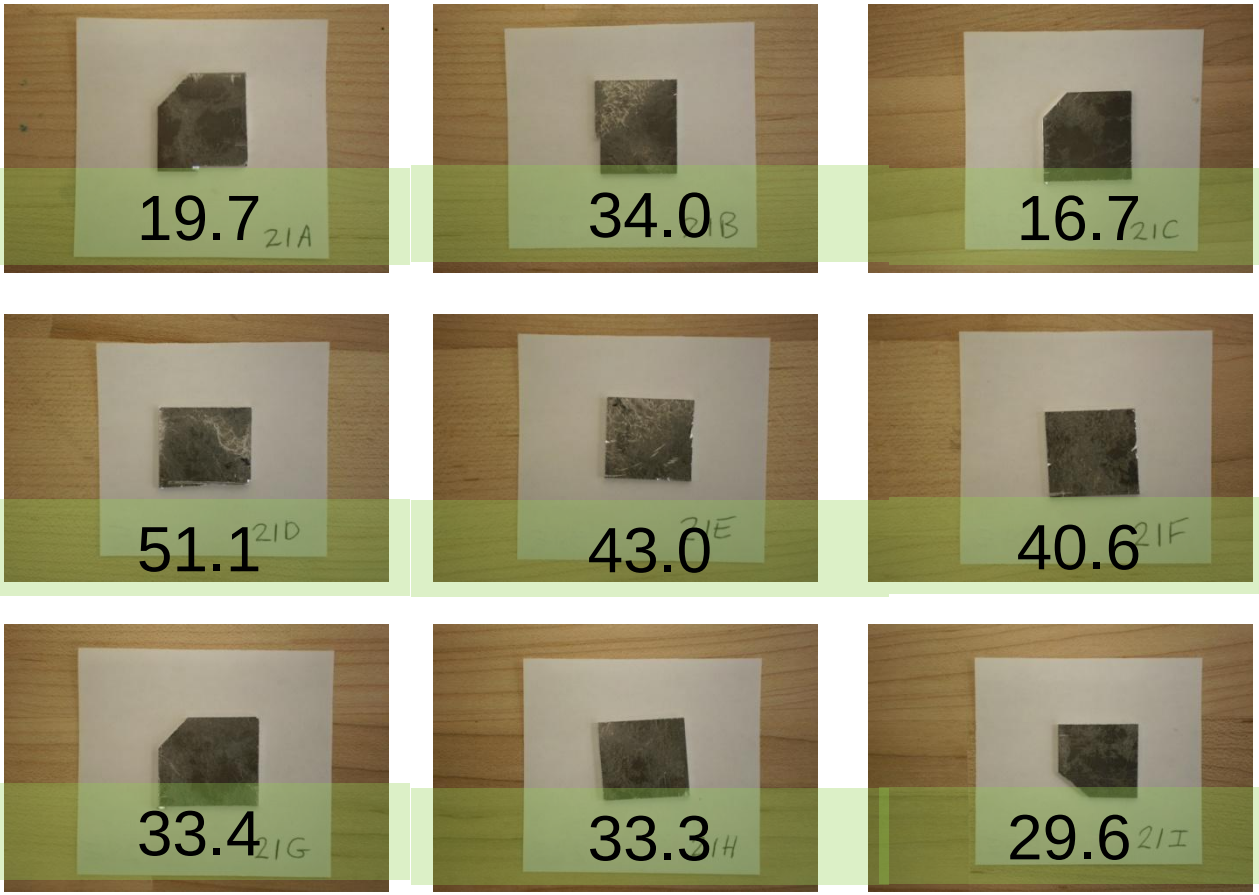
Ta Disk: 0.1 - 0.2%
Al Shields: 0.1-0.4%
DDS: 0.02%

## Solid Angle Estimate

- Gold hohlraum mass 118.48 mg
- Assuming homogeneous distribution and complete mixing, we would expect 121 µg Au to be deposited on the Al shield
- Preliminary estimates determined that < 1µg Au was collected ($\varepsilon_{collection}$ < 3%).
- This is being verified using neutron activation analysis and mass spectrometry.

This method is not sensitive to atomic deposition (sub-micron features).

# Gold distribution (μg Au/g Al shield) across the Al shield as determined by neutron activation analysis.




| | |
|---|---|
| Total Mass of Au found | 343 μg |
| Solid Angle Predictions | 296 μg |
| Collection Efficiency (4π) | 0.2% covered |
| Actual Collection Efficiency | 120% |

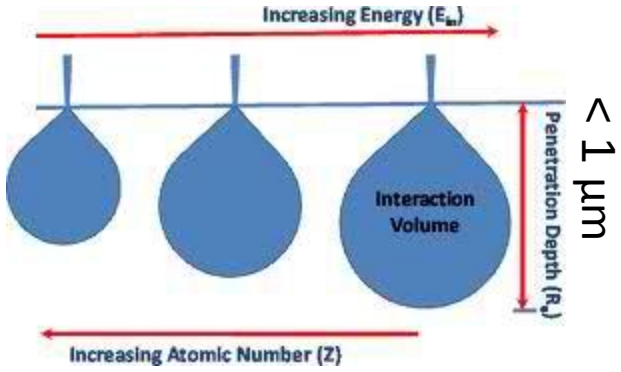
Data collected by our CSM collaborators at the USGS TRIGA Reactor, Colorado

| Shot ID | Laser Energy | Distance from TCC (cm) | Original Mass of Au (g) |
|---|---|---|---|
| Au Hohlraum, D-D cryogenic | 574 kJ | 50 | 0.11848 |

# Why do we see this difference?

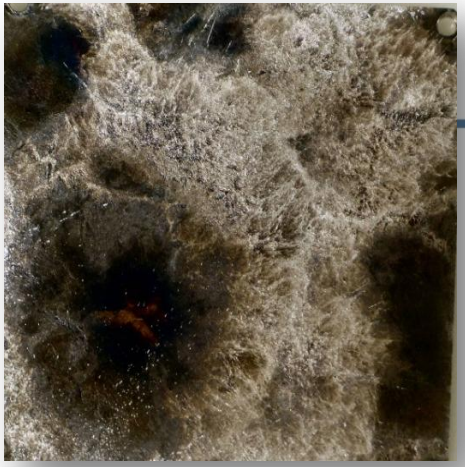
- The coupling of Secondary Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) provide elemental information about the first few 100 nm of the surface layer
- Neutron activation analysis provides elemental information across the total volume of the material
- The Al metal was sufficiently heated to melt the surface
- Evidence of molten debris hitting the surface after the metal cooled, but the interaction timeline of the debris wind is unclear

Interaction Volume Electron Beam

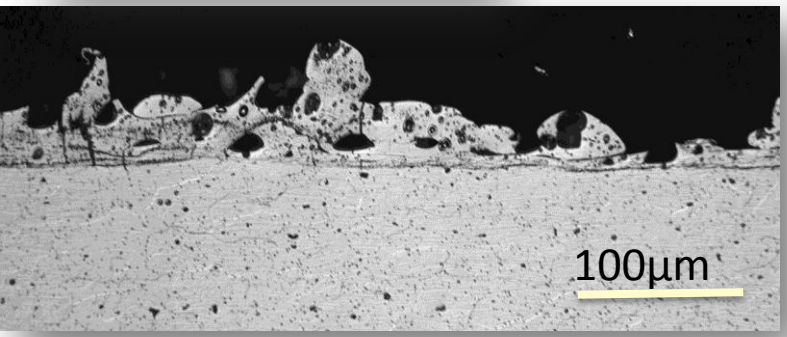


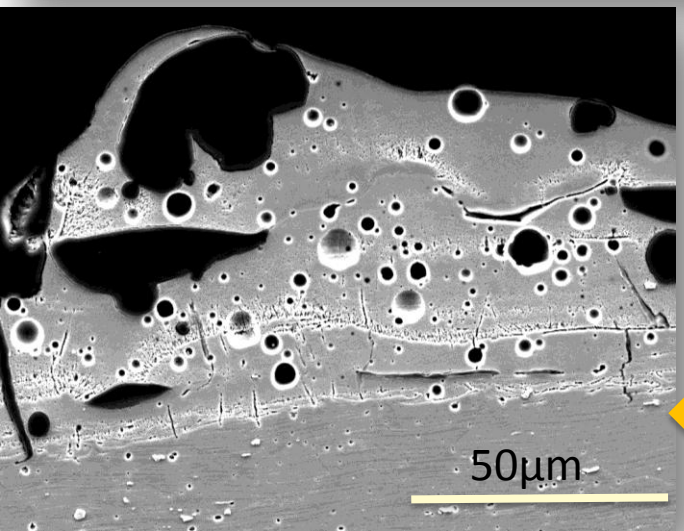

Next step is to determine the depth of penetration of the Au using XRF and chemical etching of the surface.

# Loss of collected debris must be evaluated.



- Al shields were not available for characterization pre-shot.
- There is no pre- and post-shot surface data comparison




| Laser Energy (kJ) | Distance to TCC (cm) | Al Thickness (mm) | Average Melt Depth (µm) |
|---|---|---|---|
| 568 | 50 | 1.20 | n/a |
| 837 | 50 | 1.18 | 14 ± 5 |
| 689 | 25 | 1.18 | 29 ± 10 |
| 729 | 25 | 1.18 | 43 ± 12 |
| 831 | 25 | 1.21 | 32 ± 14 |
| 836 | 25 | 1.22 | 15 ± 7 |

The Al surfaces were melted and then re-condensed or ablated.

# Experiments in 2010 focused on evaluating witness plates at various distances from TCC

- Debris associated with the target chamber has been identified
  - Au has been the primary focus because it can be directly linked to the target assembly
- The solid angle of our various witness plates was 0.02 to 0.4% of 4π
  - We expected ~ 300 μg of Au to be present on the Al shield
  - We see the Au but it may be dispersed throughout the metal
  - We have not identified Ge, mainly because of the high levels of Mn impurities in the Al
    - Interference for gamma spectrometry analysis after NA analysis.
- We have looked at various witness plates at different distances from TCC
  - A “Goldie-Locks” approach – not too close, not too far
    - Ta collimators (8 cm from TCC) observations: melting, warping, and significant spalling, but we do observed Au debris.
    - DDS glass shields (7 m from TCC) observations: particle s and molten debris are present, but in lower concentration)
  - We observed physical differences between the polar and equatorial plates (but we one had 1 polar plate)

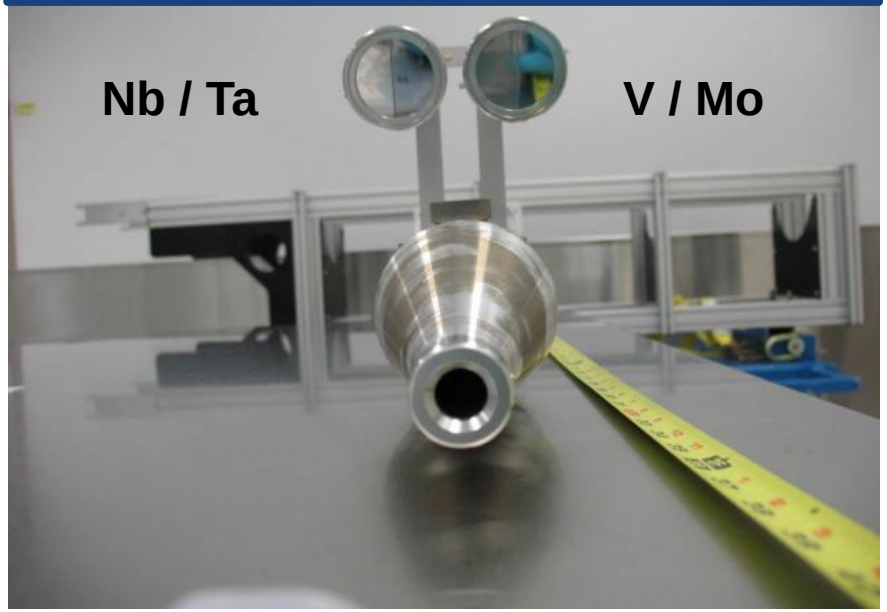
# Solid Collection Efforts at NIF

Cool science can happen at NIF, if you are patient….

# Several materials were recently fielded as shot ride-alongs to test their feasibility as collectors

- Combinations of Nb, Ta, V, Mo, Ti and graphite foil were fielded using existing wedge range filter mounts that attach to the DIMs
- Three shots fielded 5/23 - 6/03, plastic Ge-doped capsules, Au hohlraum, laser energy from 940 – 1300 kJ
- Samples will be evaluated for morphology changes, Au collection, Ge collection, surface melt, ablation depth, etc.
- Data will be used to down-select the best collection materials

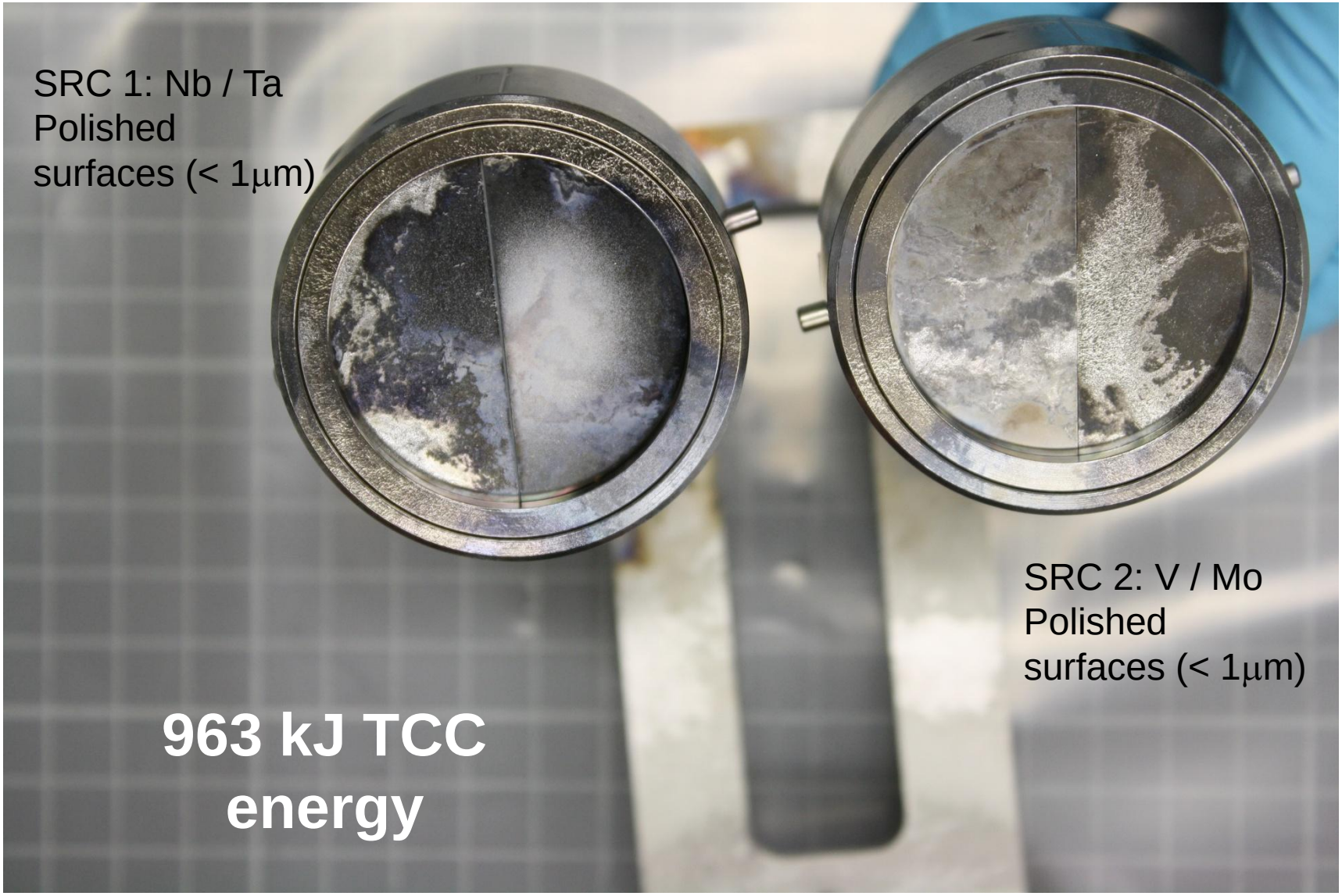
**Preshot assembly**



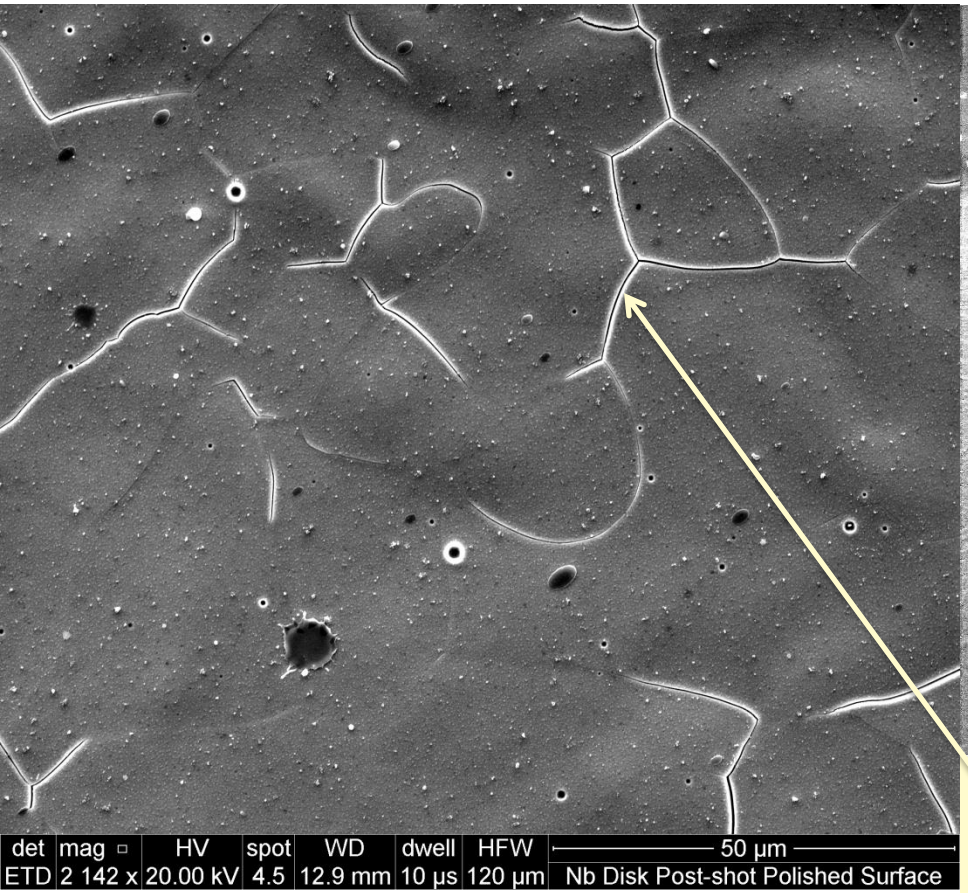

**Postshot assembly**

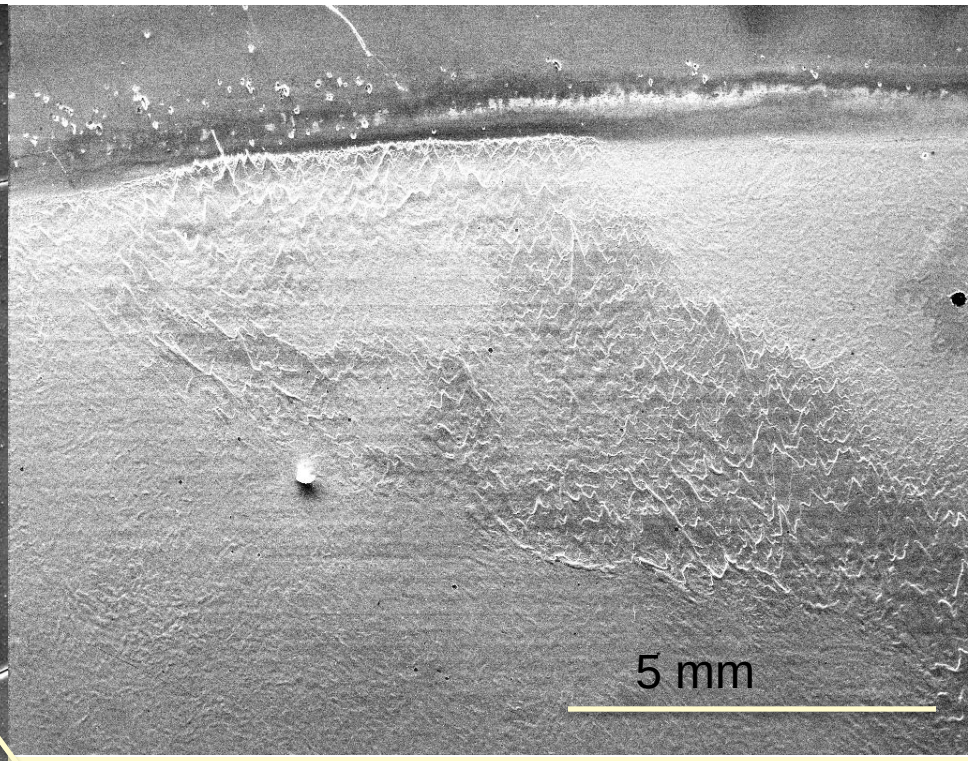
**963 kJ TCC energy**

# (90,78) DIM samples, 50 cm from TCC, IT_3_Shock_123_S20 (postshot assembly from TD Factory)



# The Nb disk fielded on the 90-78 (Equator) appears to have sustained melt damage on one side, suggesting that debris wind exposure was not uniform.

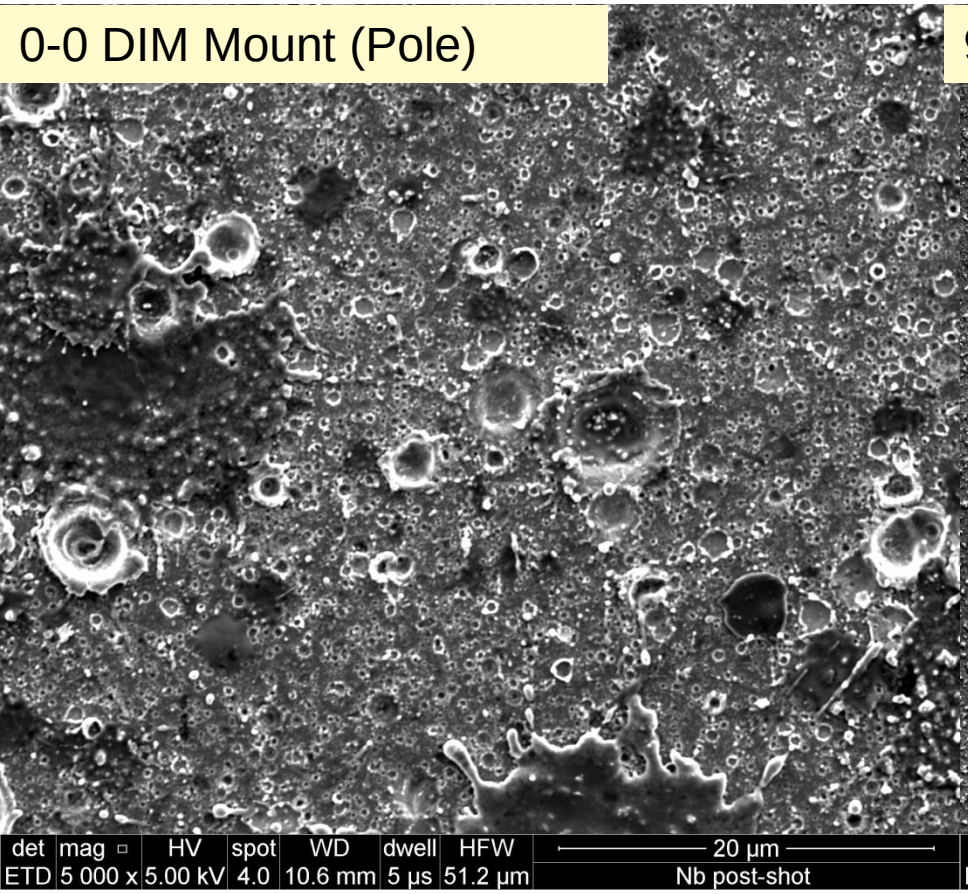


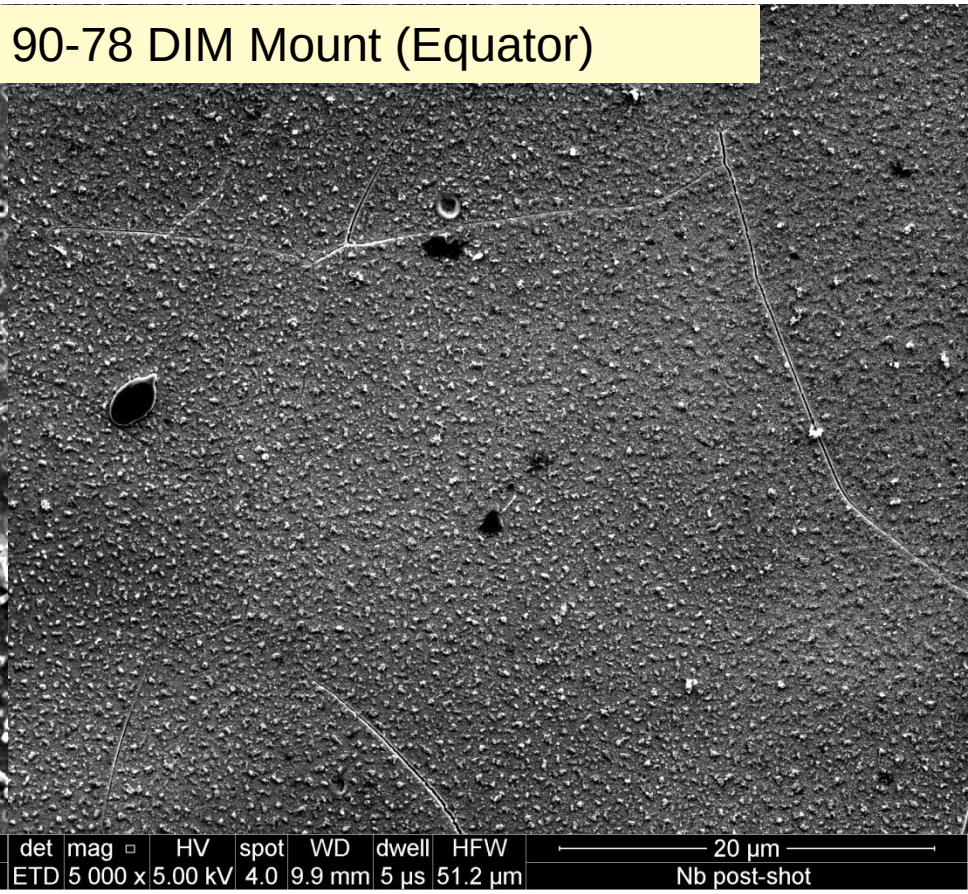

The “glow” around the cracks is due to surface charging effects.

# There are clear physical differences between the plates fielded at the Polar vs. Equatorial Mounts

0-0 DIM Mount (Pole)

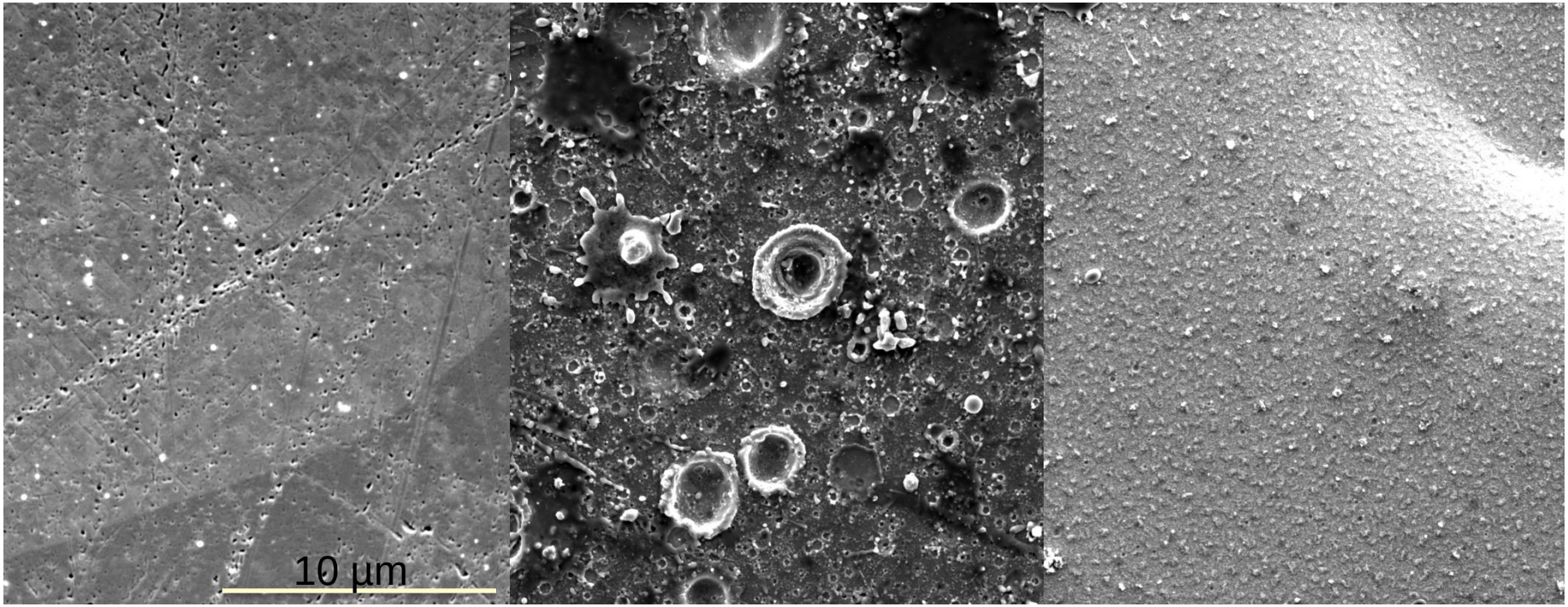



90-78 DIM Mount (Equator)




The Nb disk fielded at the pole has a high density of craters and large splats. The Nb disk fielded at the equator has less craters, which are smaller in diameter, and less large debris. There is a high density of very small (< 1µm) features on the disks.

# Surface Comparison of Pre-shot, Post-Shot Polar and Equator Nb Plates.

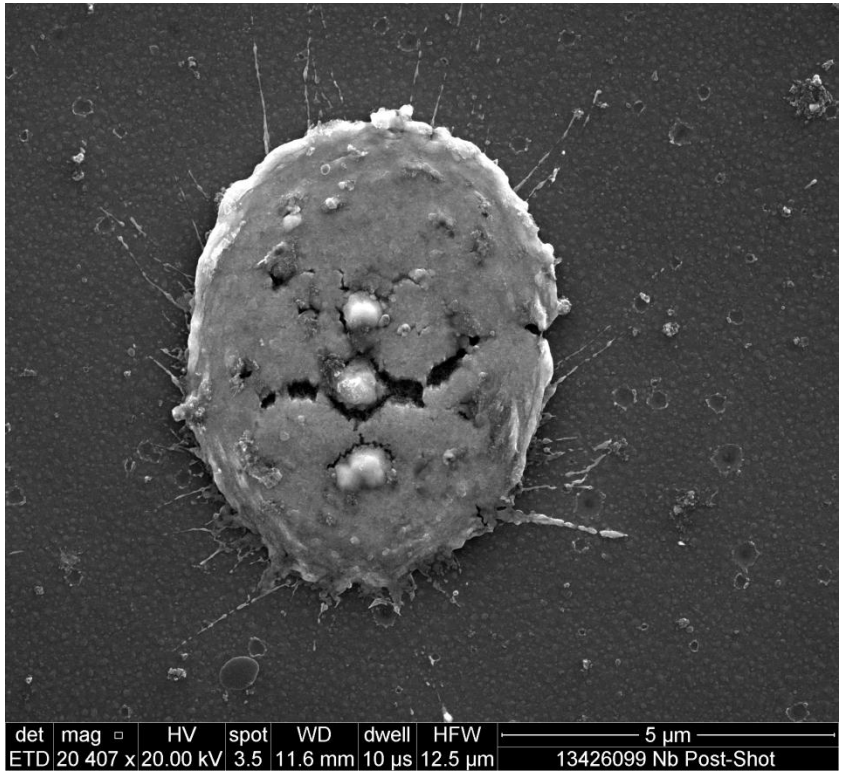


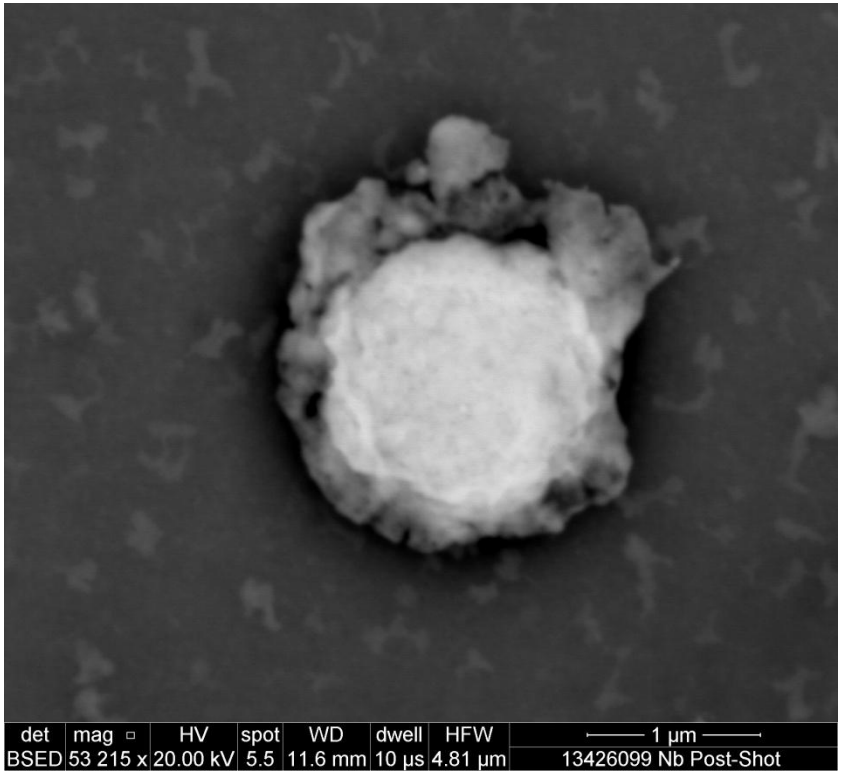

Pre-shot

Post-shot (0-0, Polar)

Post-shot (90-78, Equator)

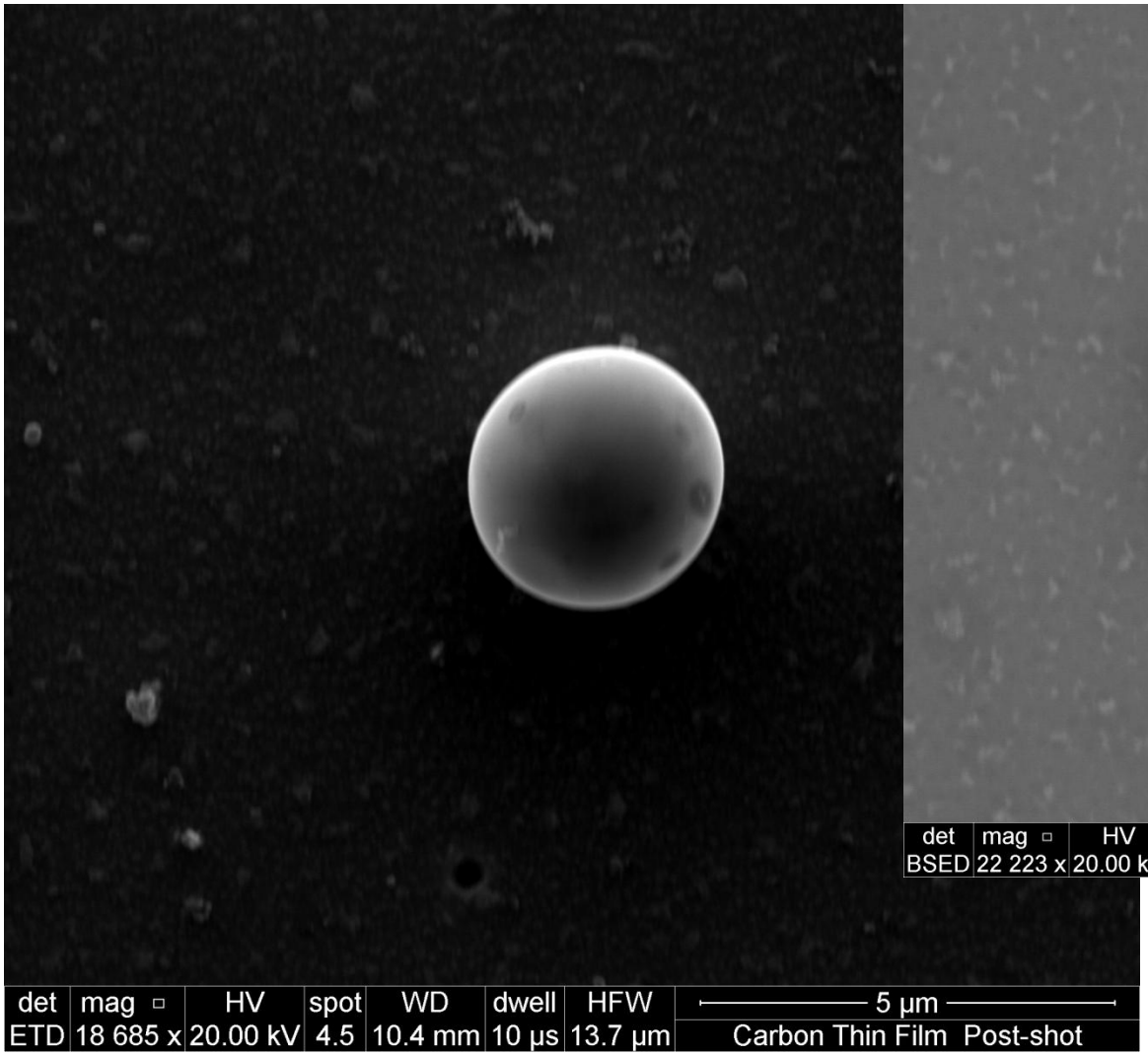
# Metal half-disks are currently being characterized.

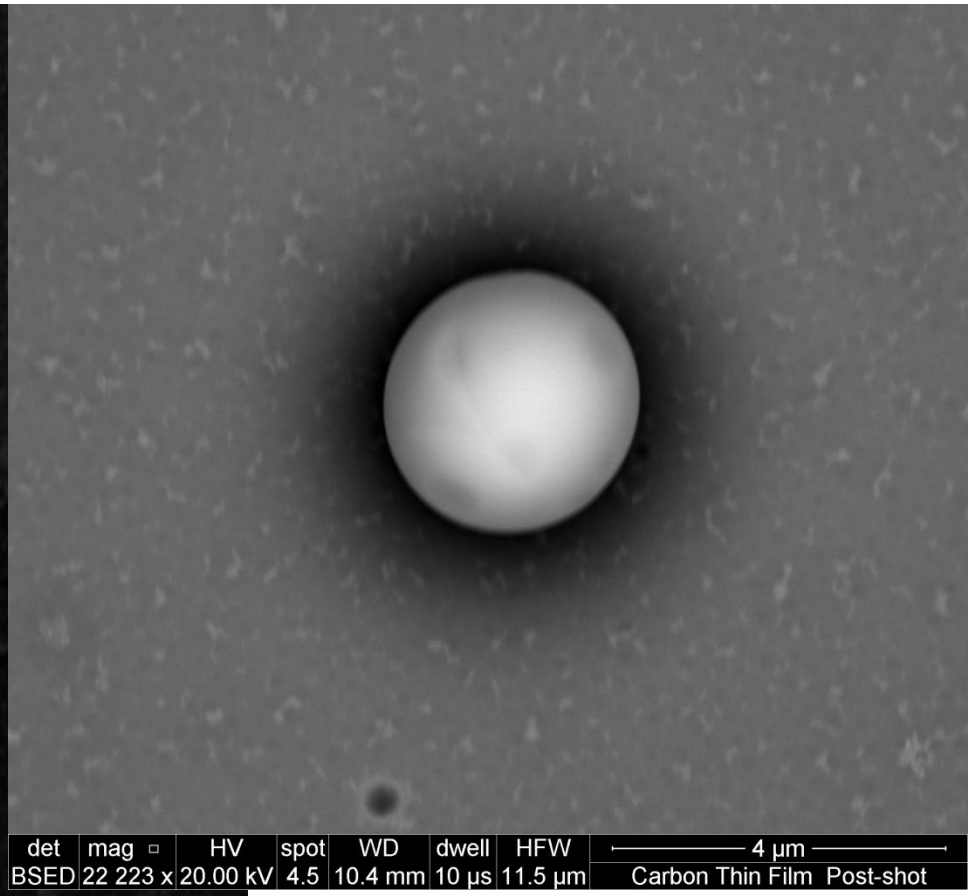



Results are very preliminary (samples received 6/14/2011). Gold has been identified in small features < 5 µm. There is also evidence of many sub-micron features that contain Au.

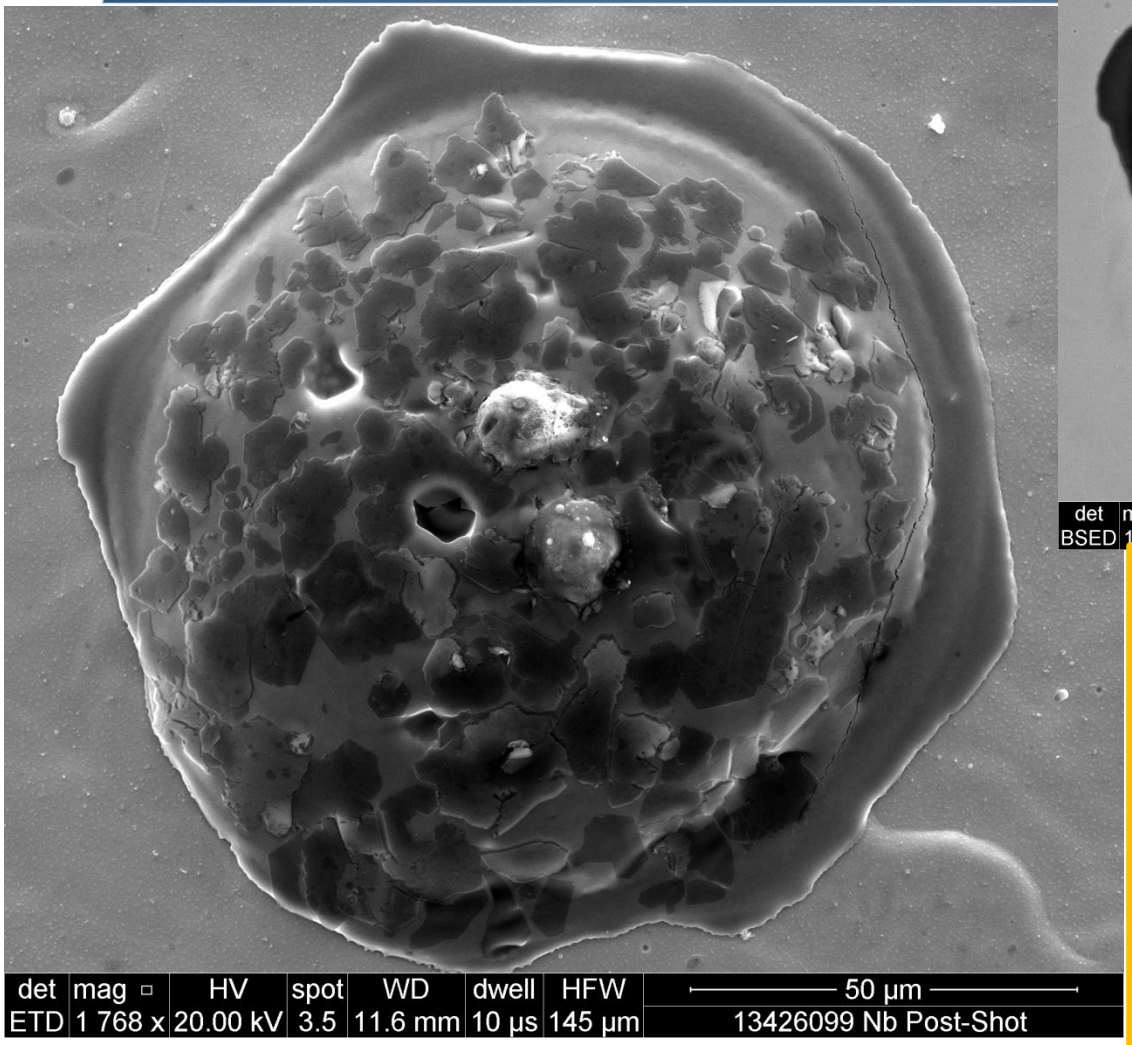
# Additional Features of particular interest are Au spherioles. Several were identified, the one displayed below is the largest that was found.

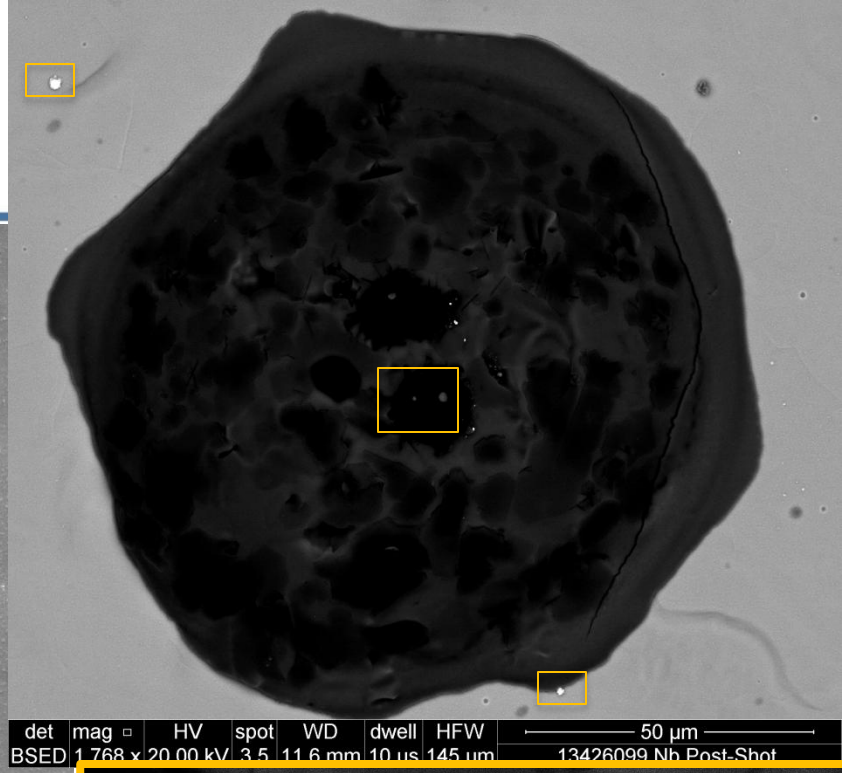


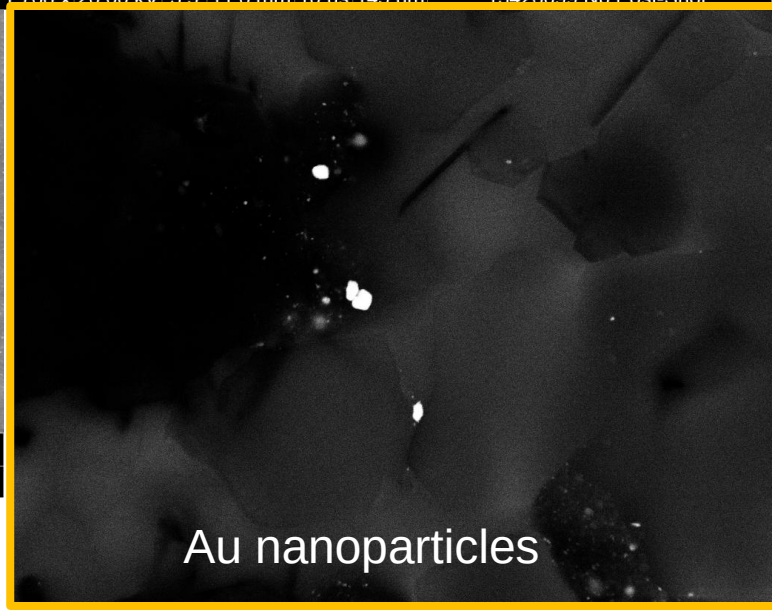




Notice in the backscatter image (above) the small Au features surrounding the sphere.

# Aluminum Oxide Feature



# Solid Collection Efforts at NIF

If you build it, they will come….

# Current activities and next steps for a ride-along diagnostic (as supported presently)

- Need to identify whether Ge from the capsule is being collected along with hohlraum Au
  - Al shields and material test samples will be surface leached and neutron activation (collaboration with Colorado School of Mines) will be used to look for Ge
- Material test samples will be analyzed
  - Non-destructive analyses include SEM, EDS, profilometry, neutron activation analysis, XRF
  - Destructive analyses include dissolution, surface leaching, mass spectrometry
  - Evaluate collection of hohlraum and capsule material
- New mounting cap is being made at CSM to determine if there is enhanced collection via conical or flat collectors; shots are being negotiated
- Design a new DIM mount that takes the place of WRF (WRF does not operate > $10^{14}$ n yield); increase solid angle while mounted on the snout (operates simultaneous with x-ray diagnostics)

# How can we turn ride-along solid collection into measurement of radiochemical cross sections?

- With dedicated shots, we can design a larger collector that uses the entire DIM (i.e., x-ray diagnostics are not fielded) and is not limited to the snout
- Requirements for development and outstanding issues (activities in red have the longest lead times):
  - Fielding an experiment or diagnostic at NIF requires an engineer (RI)
    - If WCI is committed to this effort, I have been promised that NIF will assign an RI to solid collection
  - Capsule fab – how to dope capsules with yttrium or other elements of interest?
    - Alex Hamza has ideas on how to put yttrium in capsules, but it is an R&D effort that needs to start soon
    - Second-order neutron measurements require (small amounts) of radioactivity in the capsule; Alex Hamza said this capability will be available in a few months
  - Design modeling
    - Charlie Cerjan provided simulations used for initial sensitivity studies
    - Additional simulations are required to set design requirements on the actual capsule
    - We have not studied packages on the outside of the hohlraum, but John Perkins has offered to run these

# How can we turn ride-along solid collection into measurement of radiochemical cross sections?

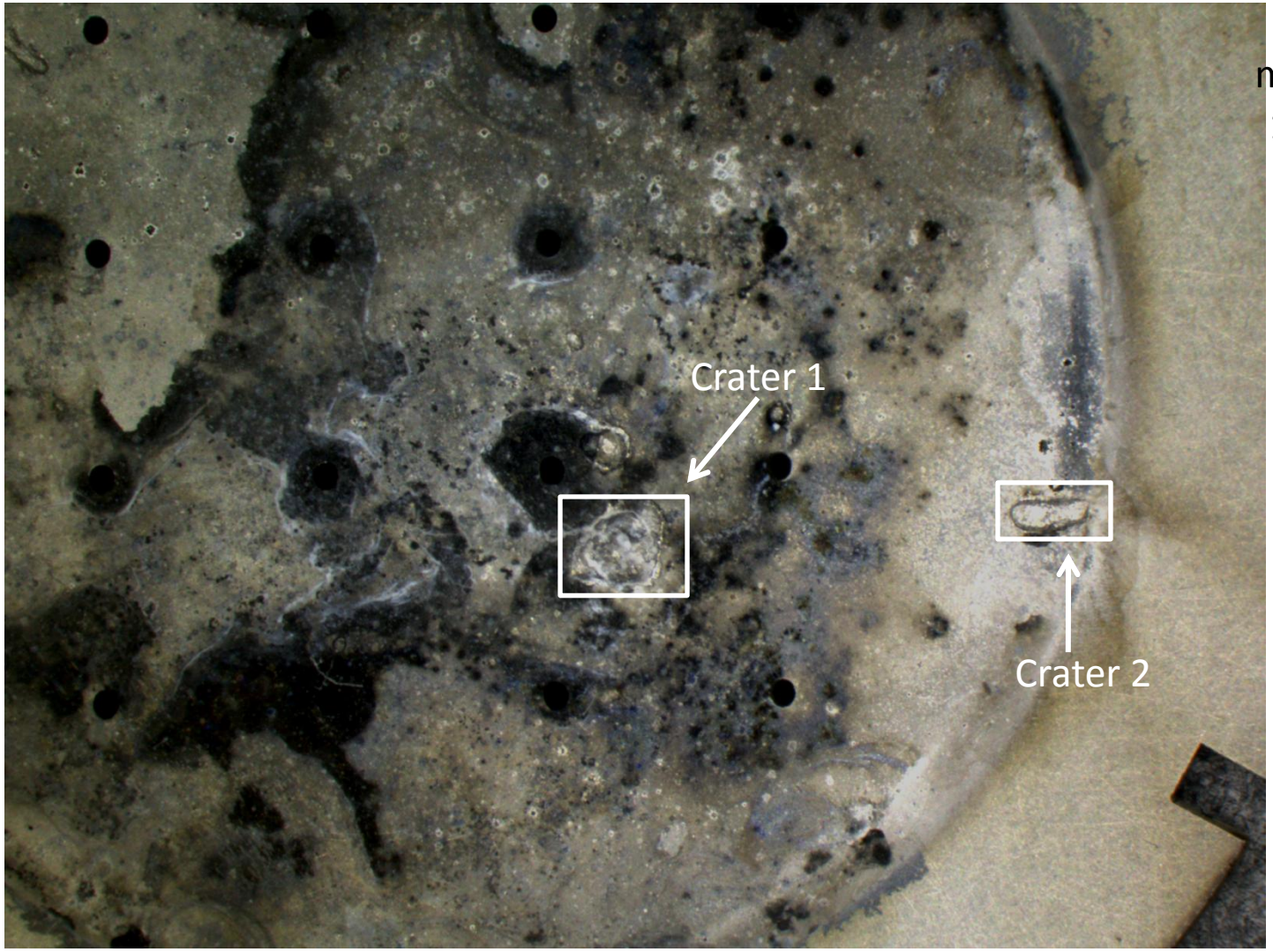
- Requirements for development and outstanding issues (activities in red have the longest lead times):
  - A few more ride-along shots are needed to finalize collector material and geometry
    - These are currently being negotiated
  - Design, fabrication, and testing of a large area collector based on results from ride-along studies
    - This requires a dedicated engineer (preferably one in the NIF system)
  - Gaseous radiochemical sample collection is coming on line shortly as well – additional personnel will be needed to support solid collection simultaneously
    - Metallurgist
    - Additional radiochemistry postdoc (we have a candidate)

# NIF Radchem Collection Collaborators

- D. Shaughnessy (LLNL)
- I. Hutcheon (LLNL)
- L. Lewis (LLNL, UCB)
- K. Moody (LLNL)
- P. Grant (LLNL)
- E. Ramon (LLNL)
- K. Harward (LLNL)
- E. Tereshatov (LLNL)
- D. Eder (LLNL)
- D. Schneider (LLNL)
- C. Velsko (LLNL)
- D. Jedlovec (LLNL)
- C. Cerjan (LLNL)
- A. Riddle (LLNL)
- K. Fournier (LLNL)
- R. Zacharias (LLNL)
- J. Dzenitis (LLNL)
- W. Stoeffl (LLNL)
- U. Greife (CSM)
- R. Larson (CSM)
- G. Grimm (LANL)
- R. Rundberg (LANL)
- A. Hayes-Sterbenz (LANL)
- L. Linden-Levy (LLNL)
- M. Stoyer (LLNL)
- R. Hoffman (LLNL)
- L. Berstein (LLNL)
- J. Dzenitis (LLNL)
- R. Fortner (LLNL)

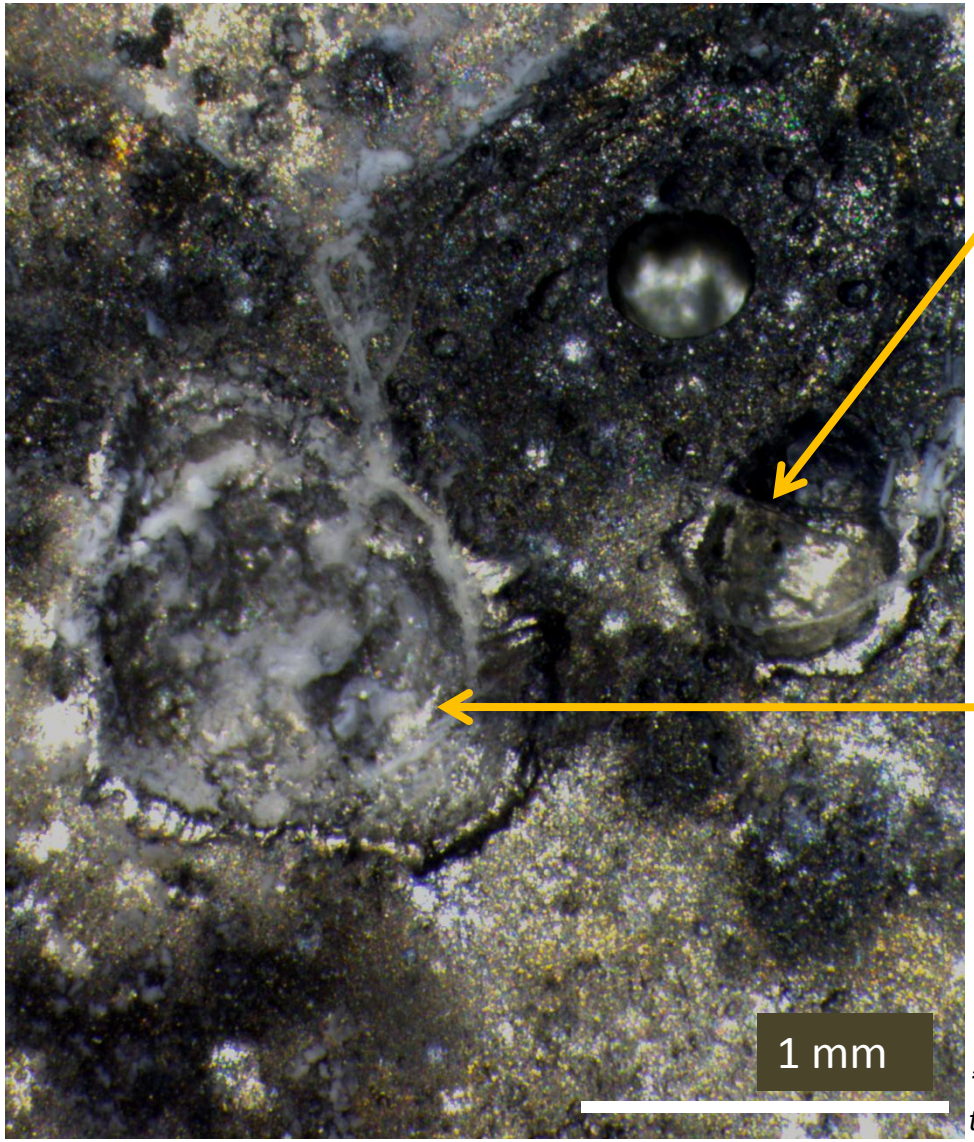
We would also like to thank the Los Alamos National Laboratory (LANL) Solid Collection team for their joint efforts and look forward to collaborating with them this year.

There are many craters and splats on the surface of the Ta collimator facing TCC; however, the foil sustained significant damage.




Surface profile measurements of Crater 1 and Crater 2 indicate that the impact of the debris caused the Ta metal to warp (i.e. there is evidence of spall on the back surface of the foil).

This is an optical image of the large crater next to one of the collimator holes. The debris within the crater is similar to the debris found in multiple areas on the surface.

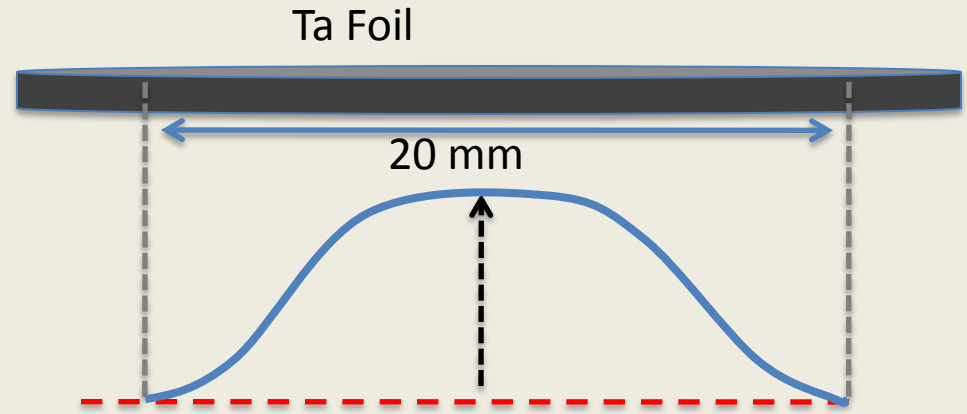



This smaller crater is filled with the same debris, but there is no deformation evident on the back surface. Dimensions are 0.64 x 0.53 mm. Depth estimation is 51 µm.*

This crater corresponds to the deformation observed on the back surface of collimator disk. Dimensions of the crater are 1.13 x 1.44 mm. This crater is approximately 225 µm deep.*

**Profile estimates are difficult due to surface roughness; therefore, depth measurements were collected with a petrographic microscope.*

A surface profile of the forward collimator was performed using a DEKTAK 3030 profilometer. A 14x20 mm region was scanned to determine if there was any noticeable warping of the disk.




Schematic of Surface Profile Trace

0 or reference point

Vertical (height) displacement

Horizontal displacement

The forward collimator disk is slightly bowed in the center, with the maximum height displacement of 57 ± 7 µm. There may be some side warping, but this cannot be verified with the current technique (maximum horizontal scan length is 25 mm). Warping is also seen on the rear collimator and pin hole array.