

# Post Irradiation Examination of an Alloy 718 Beam Window

Stuart A. Maloy<sup>1</sup>,

Hong Bach<sup>2</sup>, Tarik A. Saleh<sup>1</sup>, Tobias J. Romero<sup>2</sup>,

Osman Anderoglu<sup>1</sup>, Bulent H. Sencer<sup>3</sup>

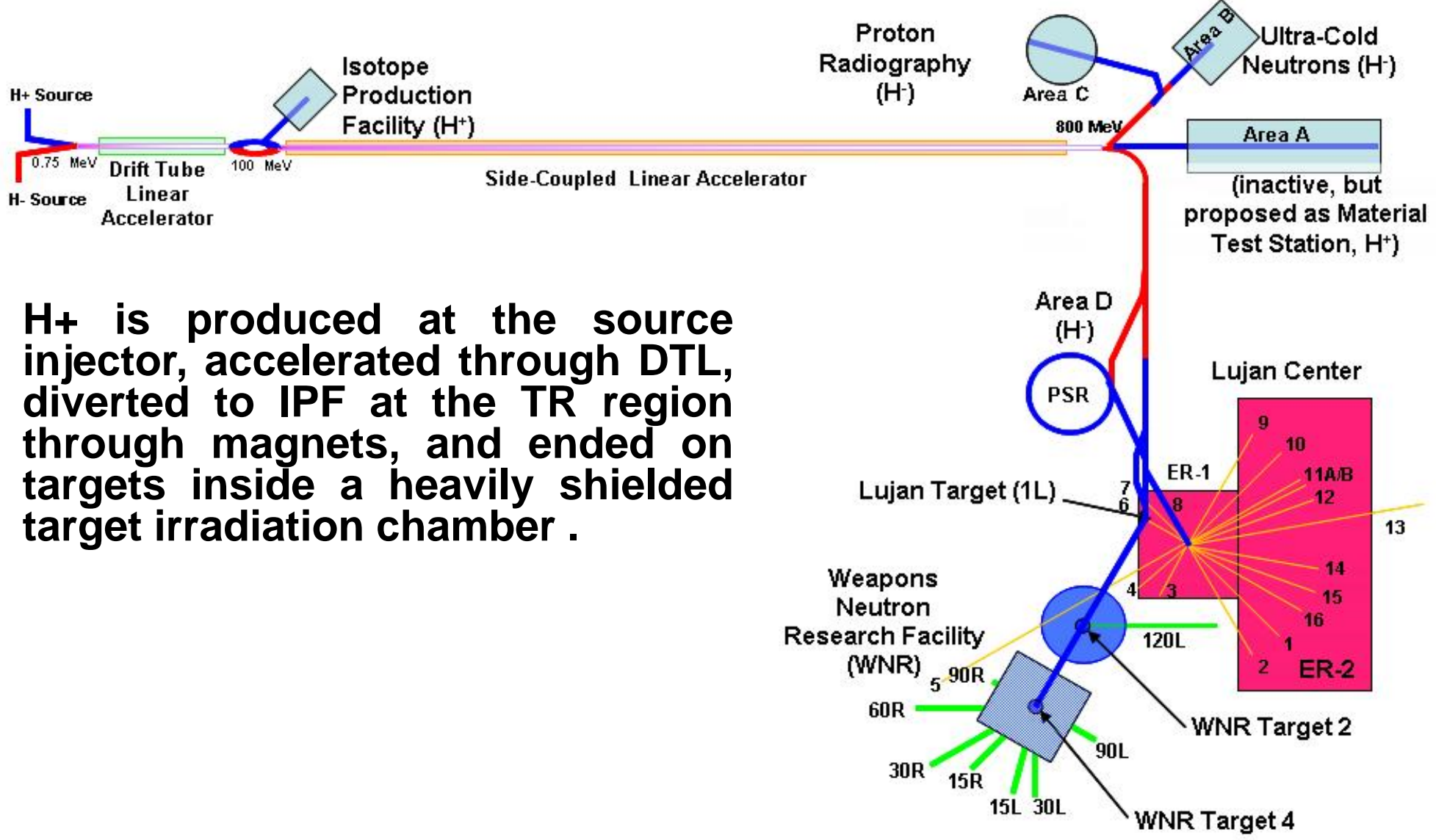
1. Materials Science and Technology Division -Los Alamos National Laboratory
2. Chemistry Division -Los Alamos National Laboratory
3. Idaho National Laboratory

(May 21, 2014)

# Outline

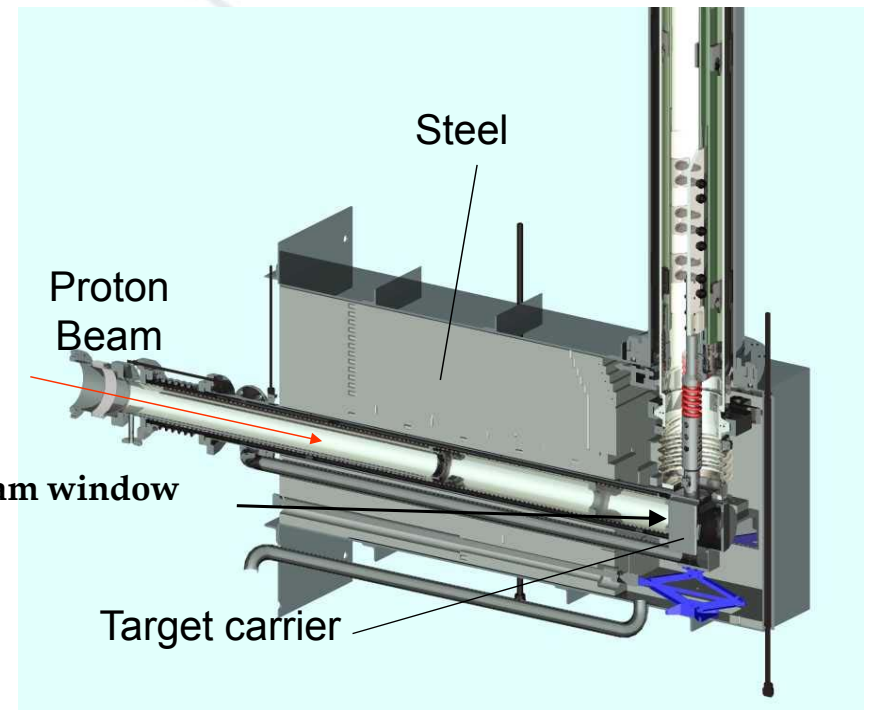
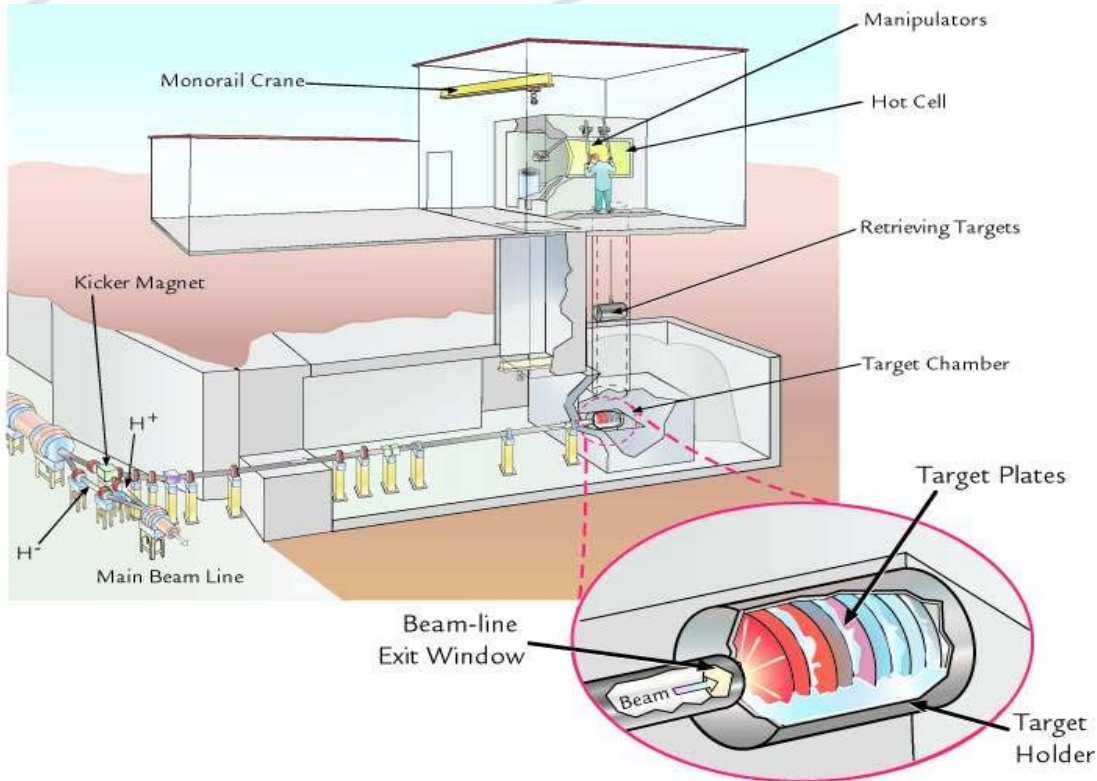
- Isotope Production Facility
- Cutting of Window and Dimensional Measurements
- Calculation of Dose and Irradiation Temperature
- Shear Punch Testing
  - Trepanning of Specimens
  - Dose/Irradiation Temperature vs. Location
  - Shear Punch Testing and Data
- TEM Analysis
- Comparison to previous test results
- Summary

# Isotope Production Facility - LANSCE



H<sup>+</sup> is produced at the source injector, accelerated through DTL, diverted to IPF at the TR region through magnets, and ended on targets inside a heavily shielded target irradiation chamber .

# Isotope Production Facility and Beam Window



The proton beam is delivered via a vacuum beam pipe. Inconel 718 beam window isolates the beam pipe (under vacuum) and the target irradiation chamber (15 psig of cooling water).

Present design limit for window is 5 years and would like to increase that limit through analyzing properties after 5 years of operation.

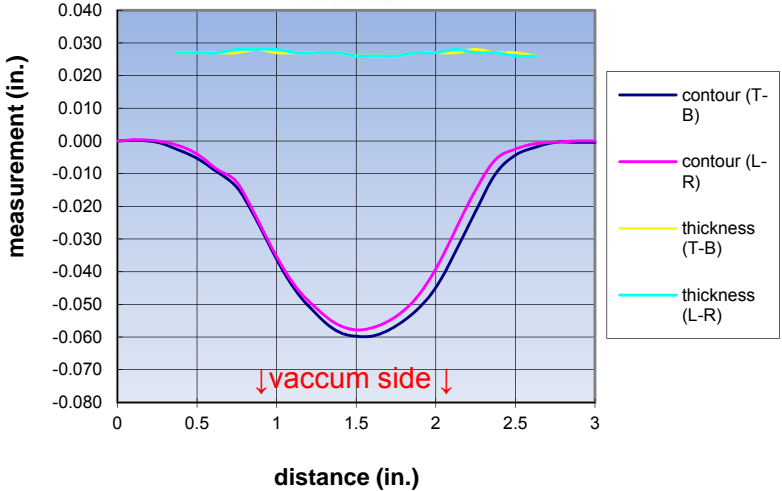
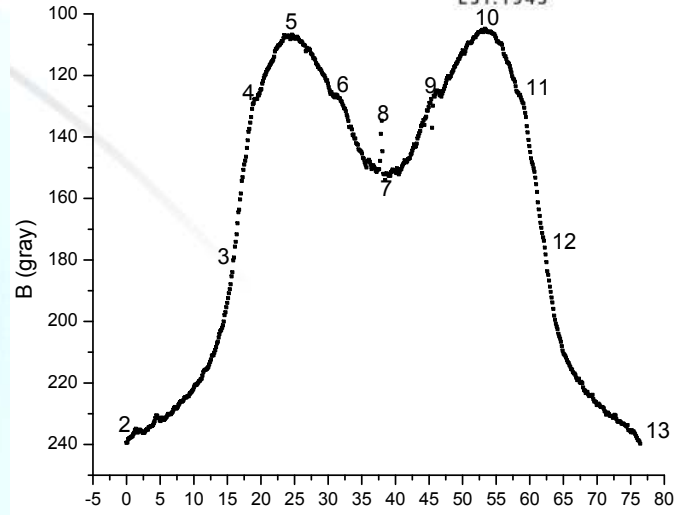
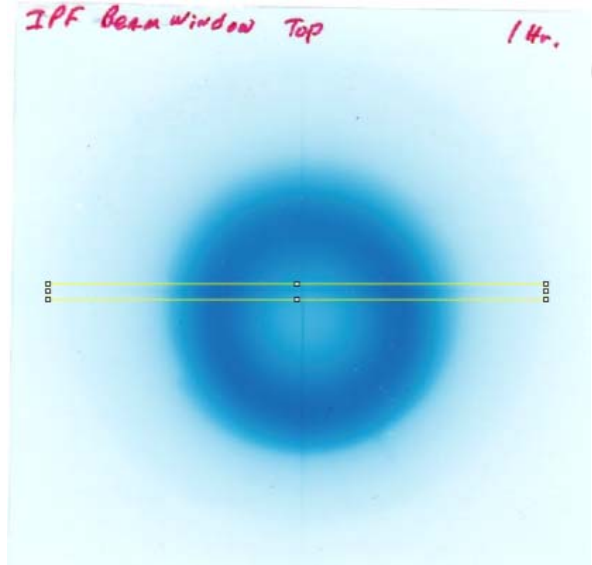
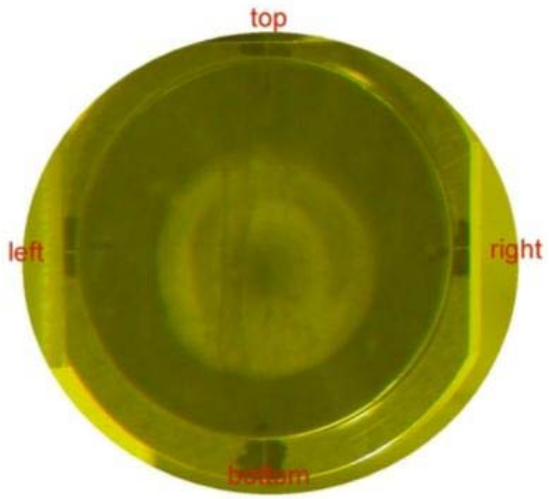
# Cutting off the Window



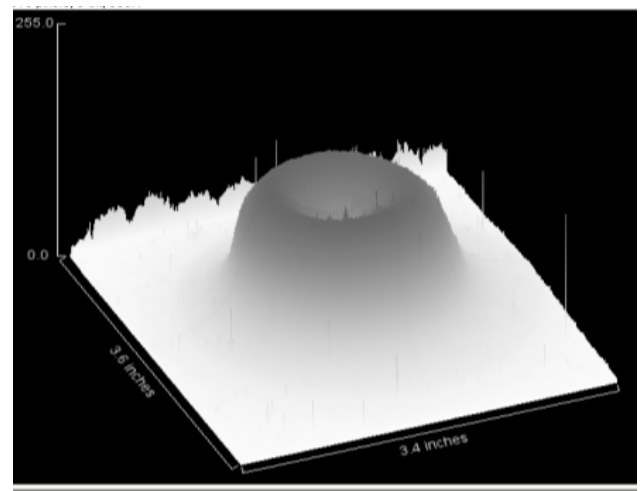
Window was cut from beam tube remotely in the corridor of the CMR Wing 9 hot cells

Then, the window was placed into an individual hot cell for analysis and sample preparation.

# Dimensional and Dose Measurements on Window



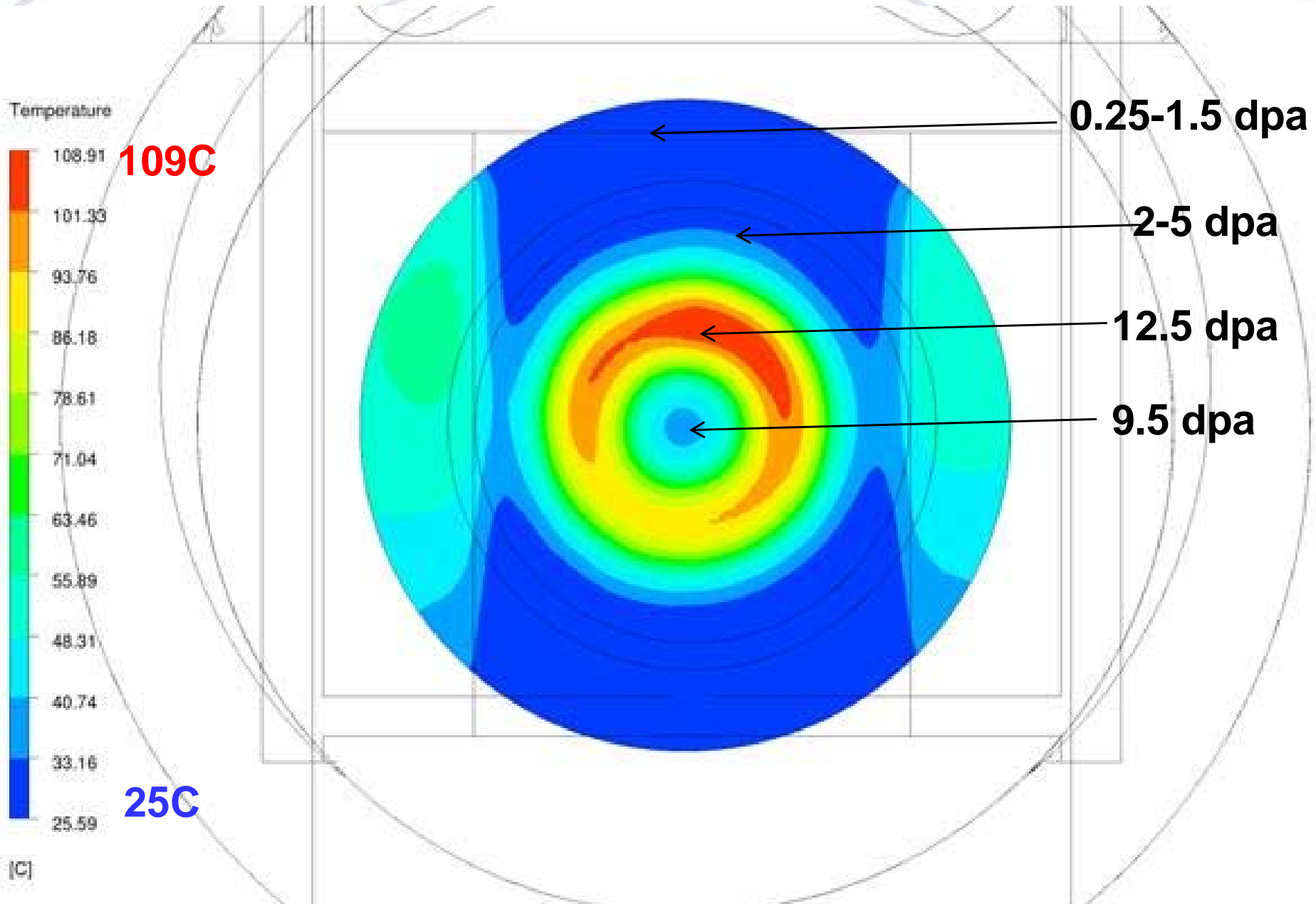
GAFCHROMIC HD-810 dosimetry film was used to measure the absorbed dose of high energy photons from the activated beam window.



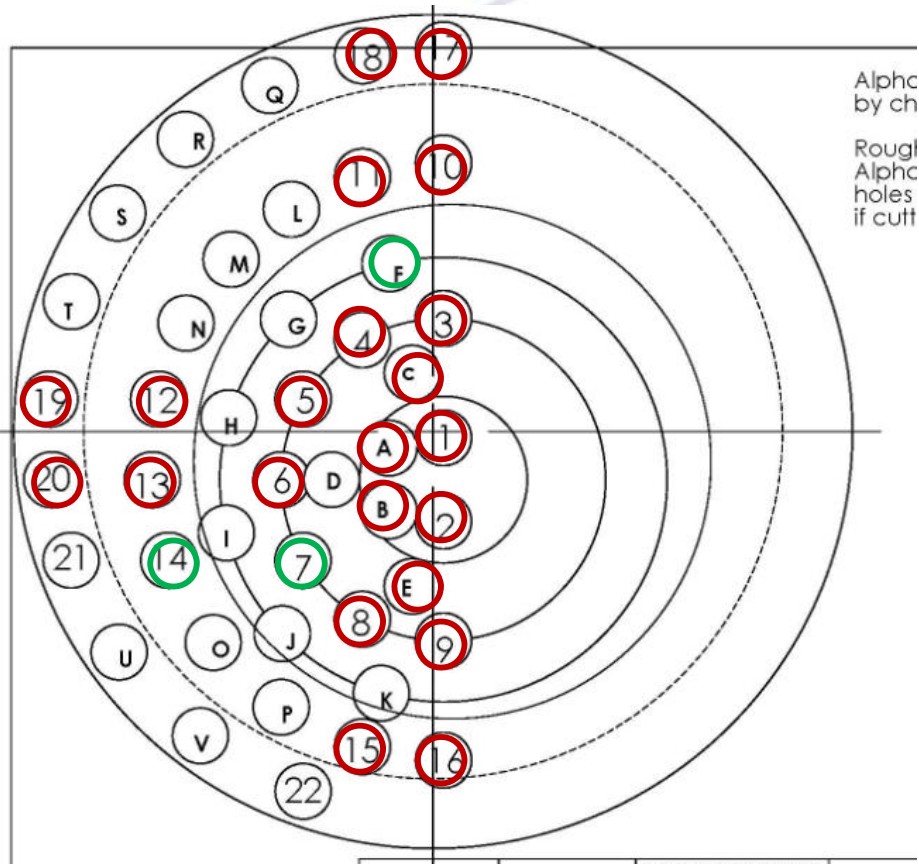
Beam window deformed 1.5mm into the vacuum side.

Rastered beam profile shows a Gaussian distribution and the highest dose region corresponds to the darkest blue region on the Gafchromic film.

# Temperature and Dose Map



# Cutting and Shear Punch Testing Plan



**Samples Tested**

**Samples Cut but Untested**

**Black Circles were not cut**

- Beam profile was superimposed on the window to determine the cutting plan as a function of radiation dose (dpa).
- 3-mm OD samples were cut with a Mill machine. A total of 3 cutting bits were spent to cut out 20 numerical samples (1-20) and 5 alphabetical samples (A, B, C, E, and F).
- Cut-out samples were polished and thinned from on both sides to 0.254 mm thickness.
- The shear punch testing for the following samples were completed as a function of radiation dose (dpa):
  - 2 controls samples of unirradiated Inconel 718
  - 1-6, 8, 9, 10-13, 15-16, 17-18, 19-20, A-C, and E



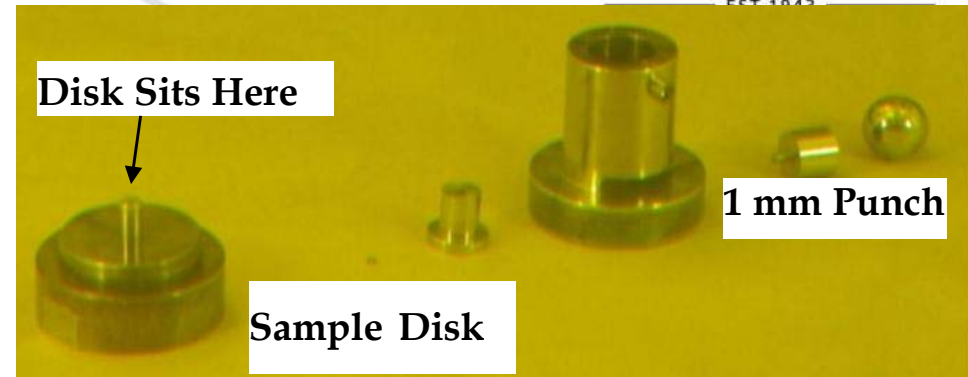
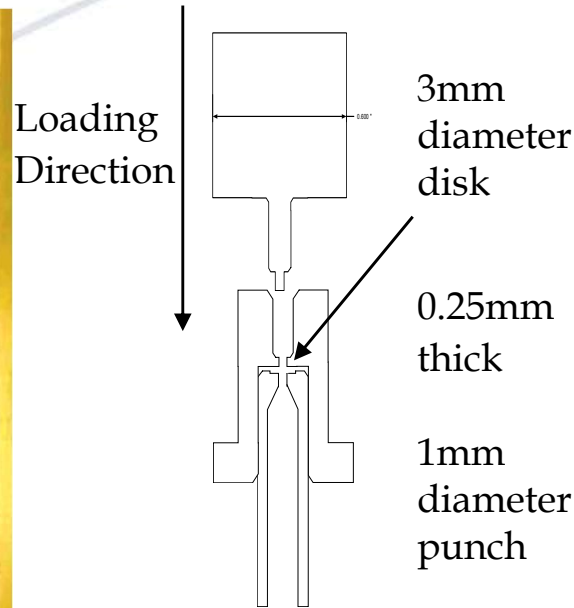
# Trepanning 3 mm Diameter Samples from Window



# Shear Punch Testing Equipment at CMR Hot Cell



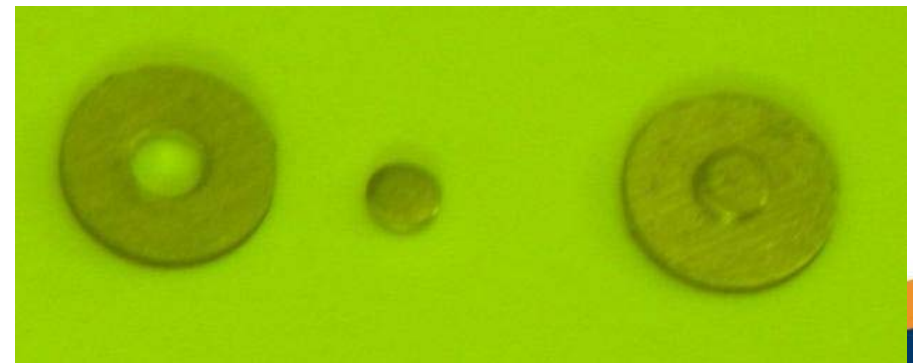
Shear Punch Set-up



- Performed 25 shear punch tests on 3 mm diameter specimens.
- Tested at initial strain rate of  $5 \times 10^{-4}$ /s.
- Tested at in ultra high purity argon.

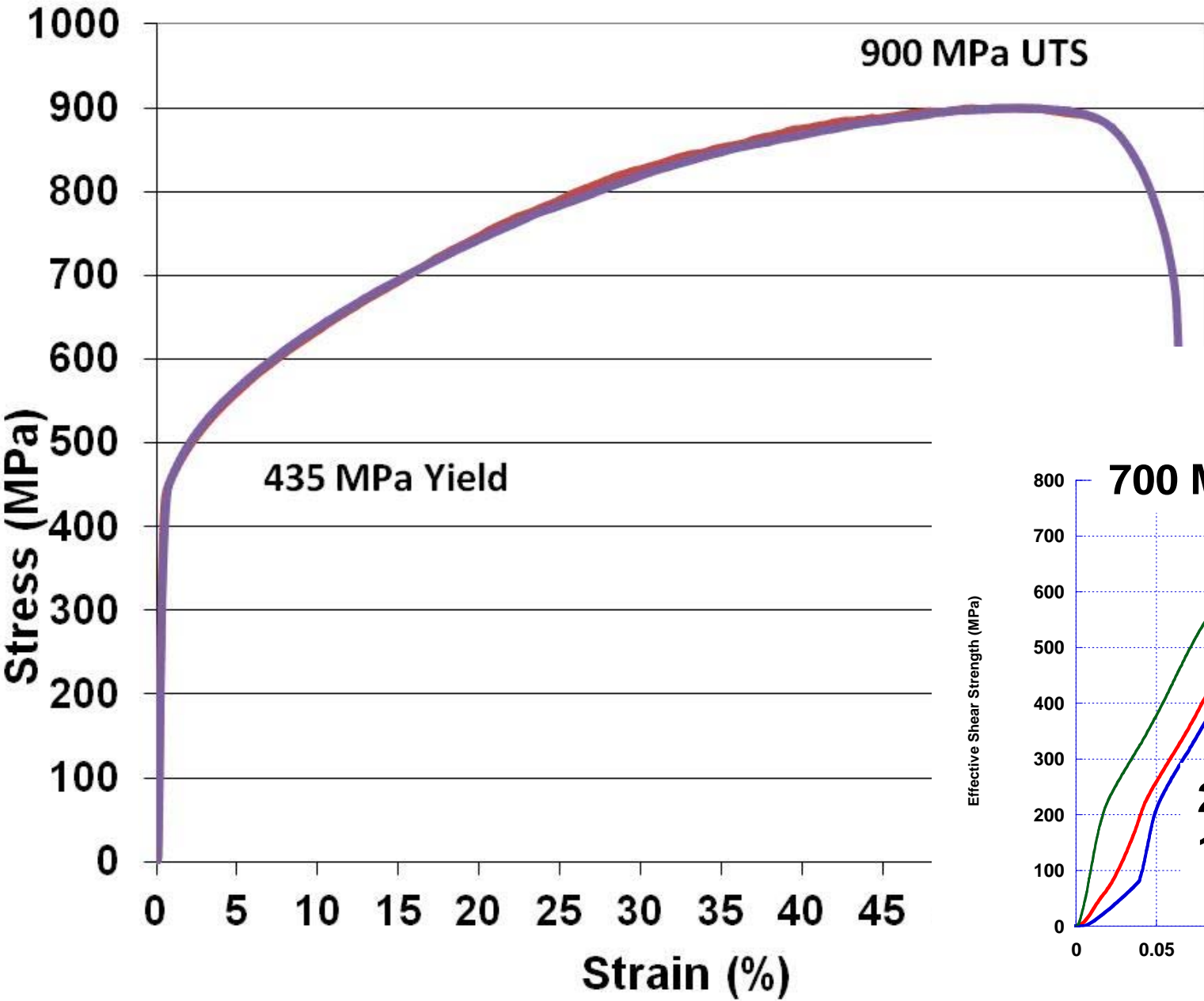


Loading sequence



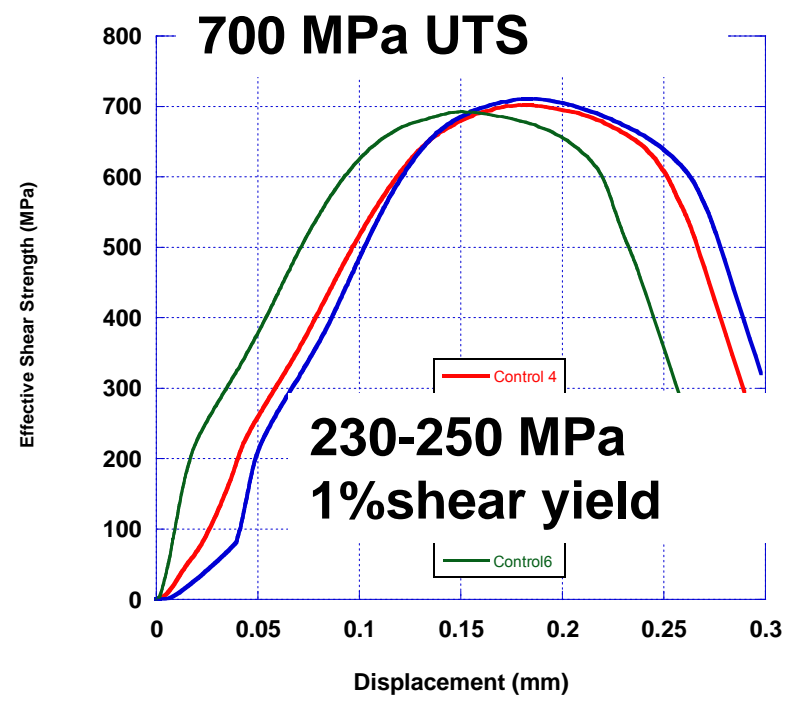
Typical shear punch specimen

# Control Material Tensile Tests vs Shear Punch

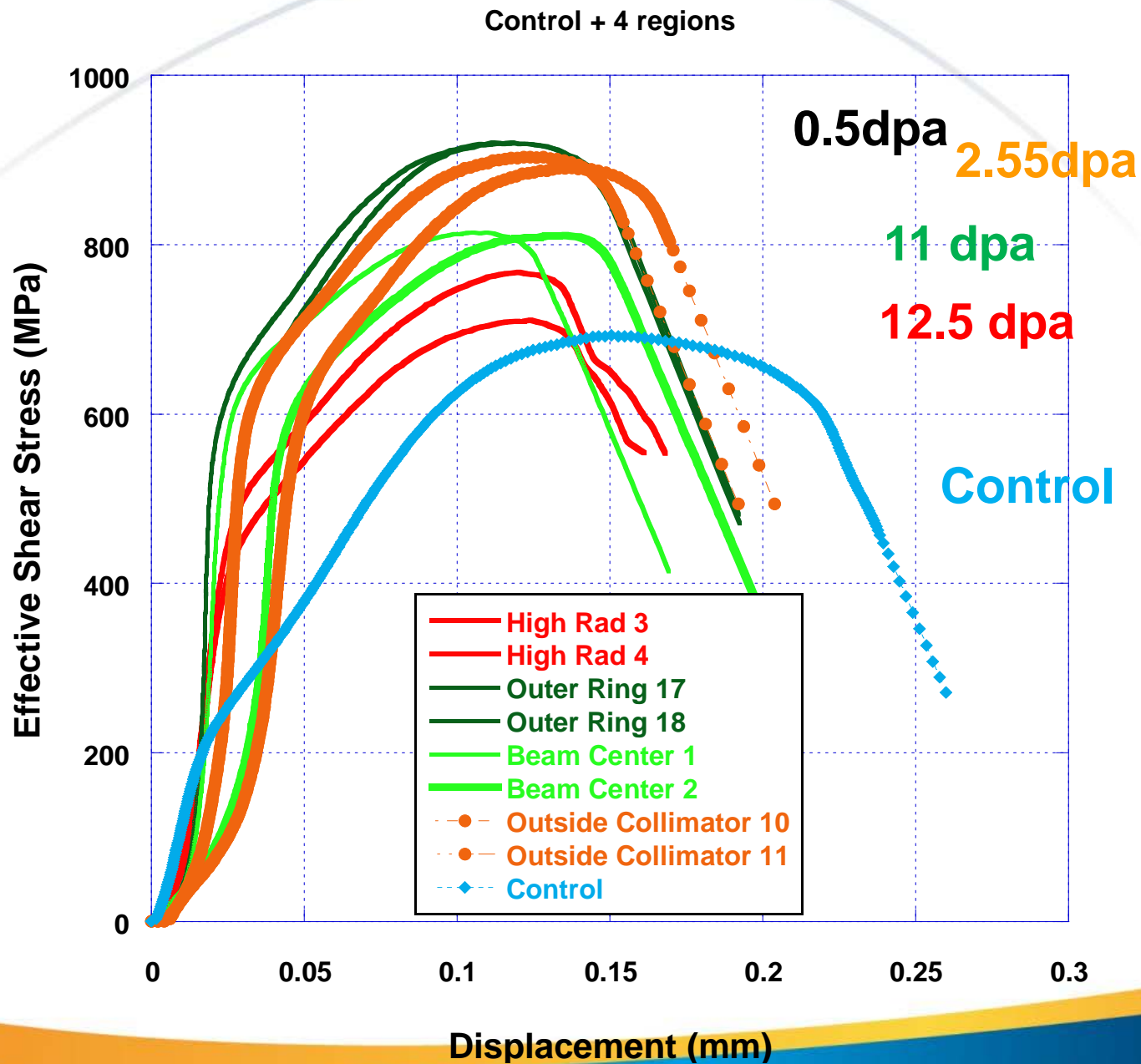


- UTS correlation - 1.28
- Yield Correlation - 1.77
- Lit values 1.4, 1.73

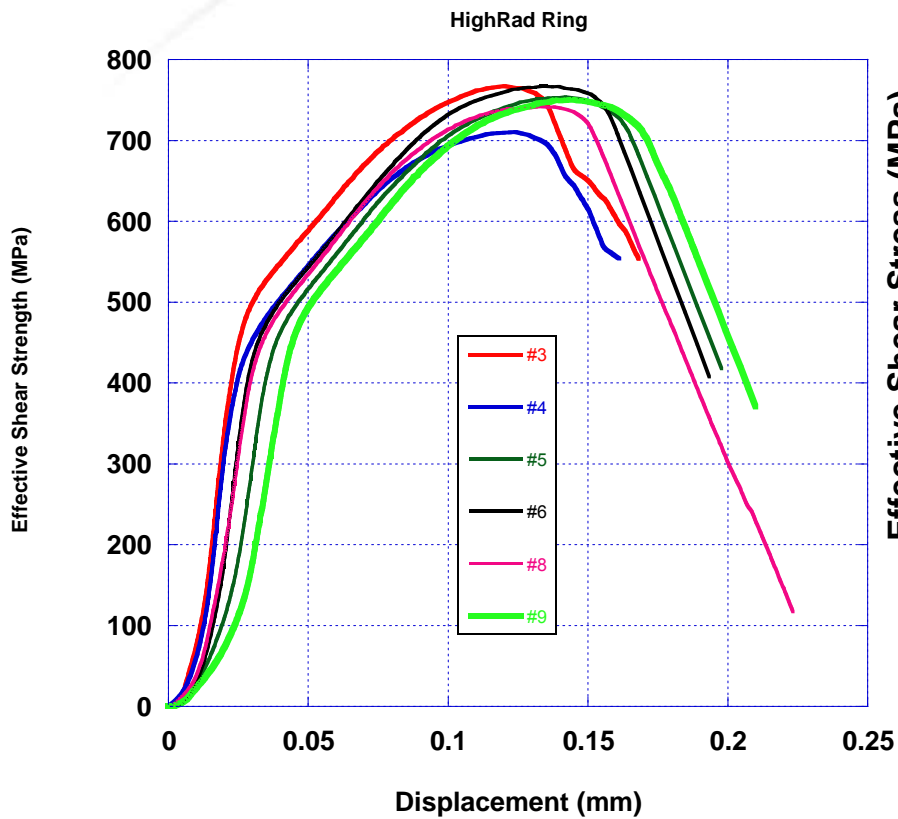
Toloczko&Kurtz



# Shear Punch, Outer to Inner

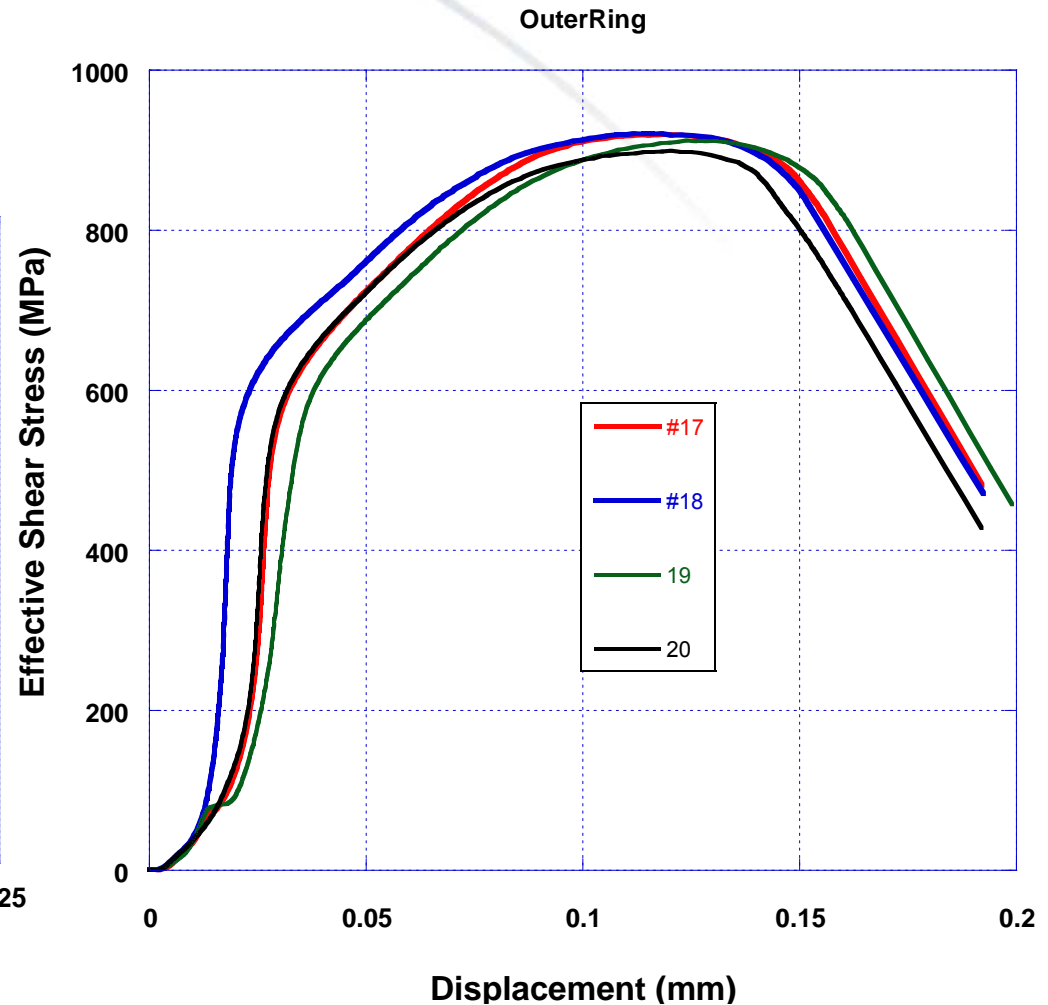


# Comparison of Shear Stress/displacement Curves



**Dose = 12.5 dpa**

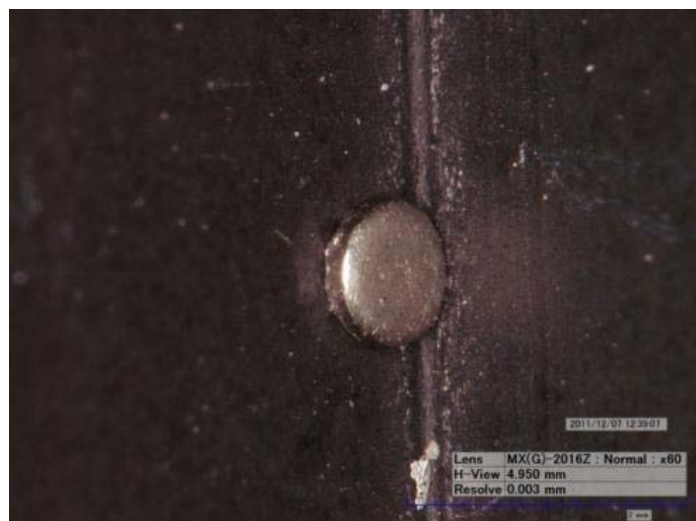
**Tirr= 109C**



**Dose = 0.5 dpa**

**Tirr=50C**

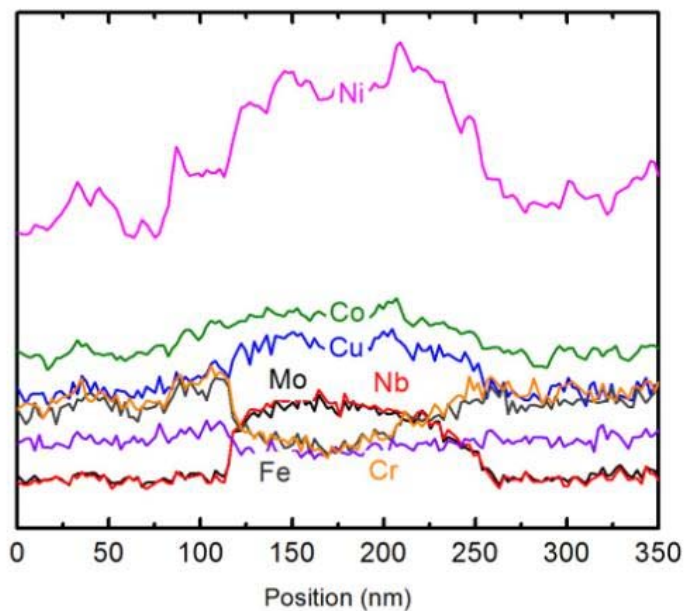
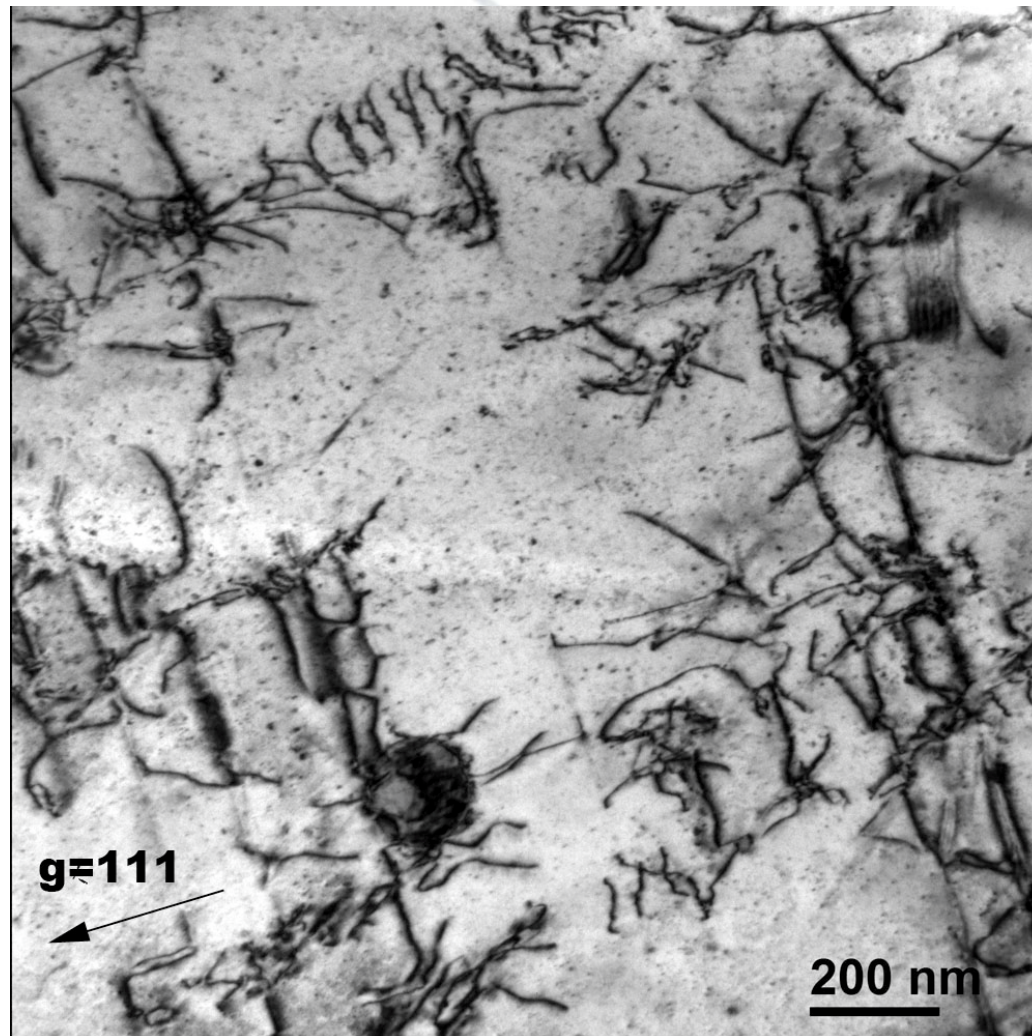
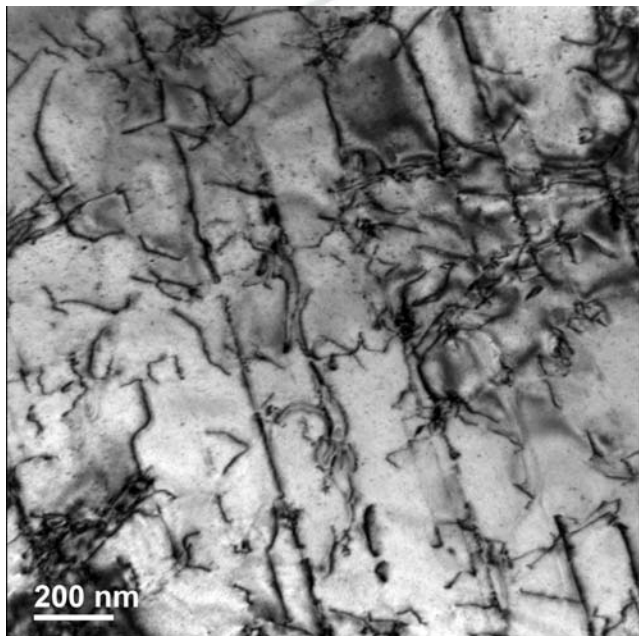
# Optical Images of Exit side of Shear Punch Specimens



**Control Material**

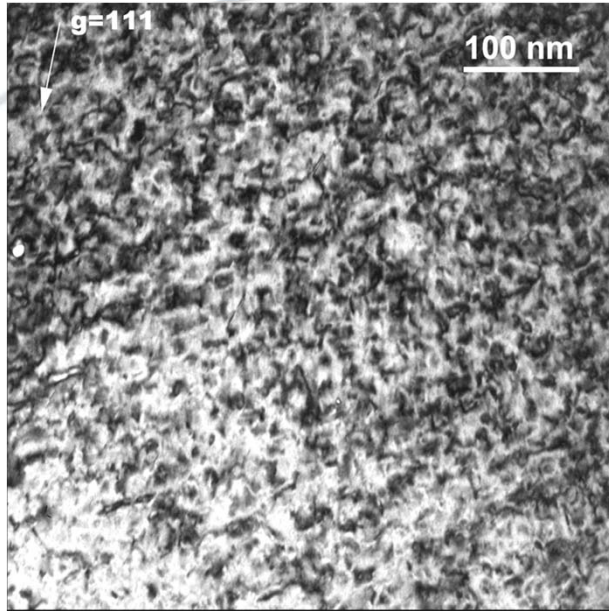
**Dose = 12.5 dpa**

# Inconel 718 Solution Annealed- unirradiated

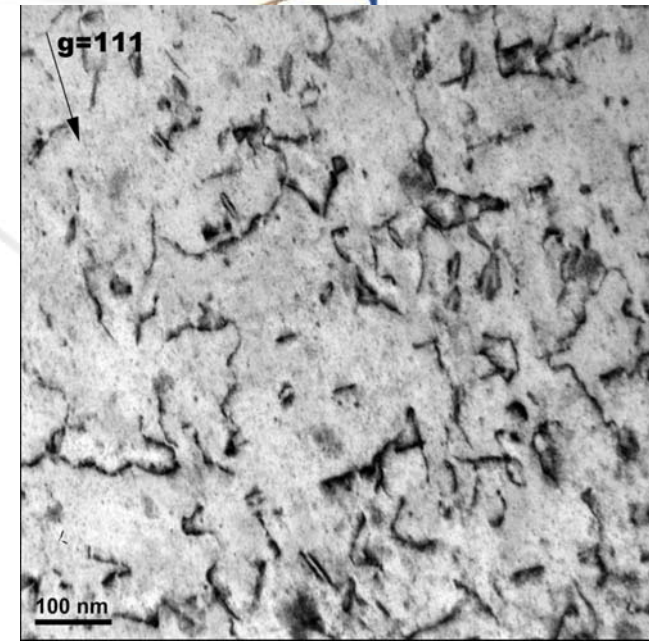
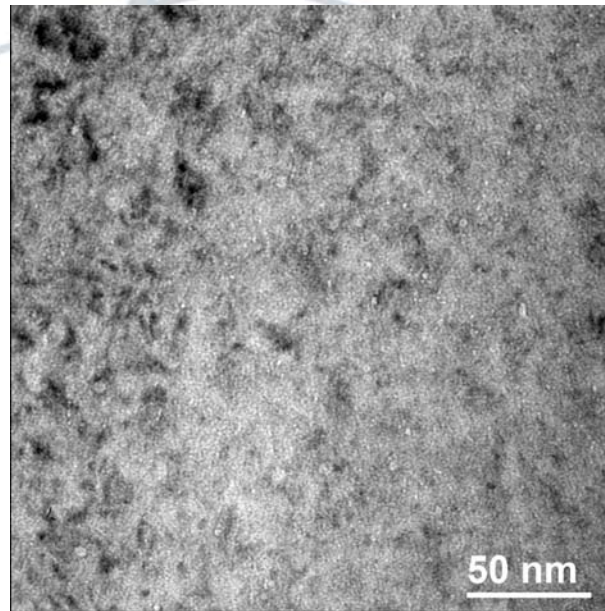


■ Bright field TEM images showing dislocations and some precipitates

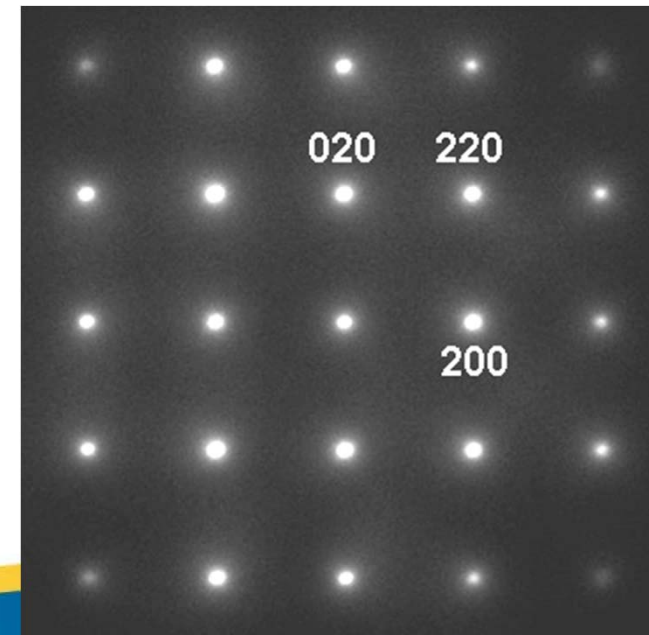
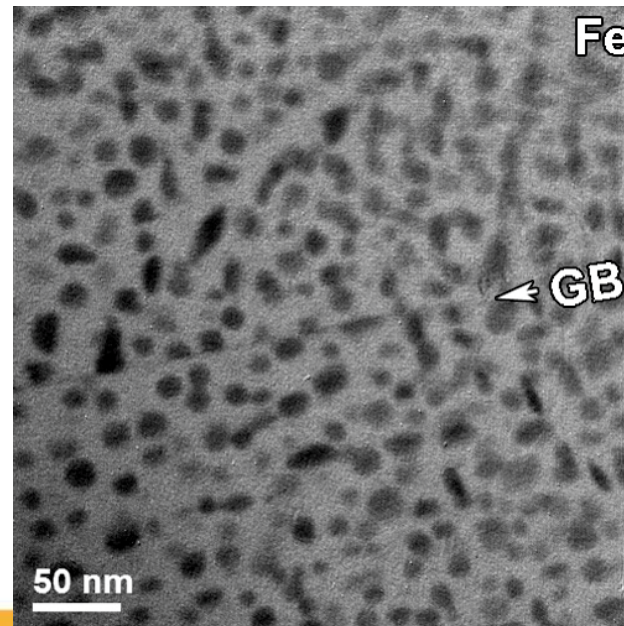
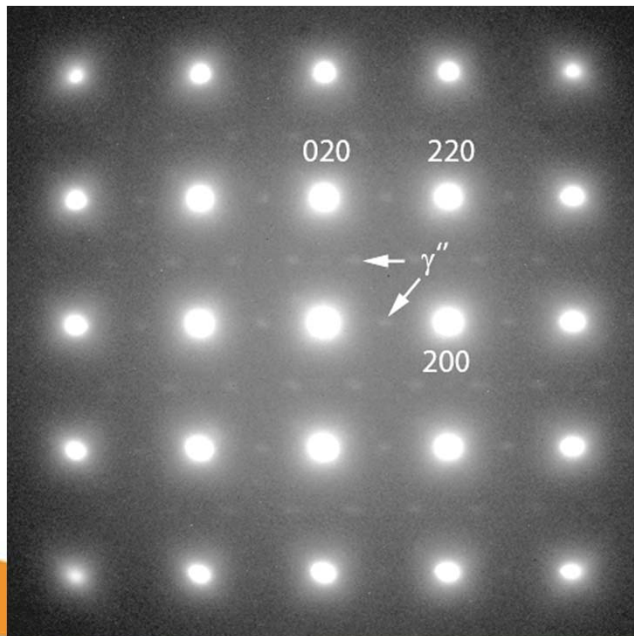
# TEM Analysis of Irradiated Alloy 718 Samples



0.5 dpa at 50°C



12.5 dpa at 109°C

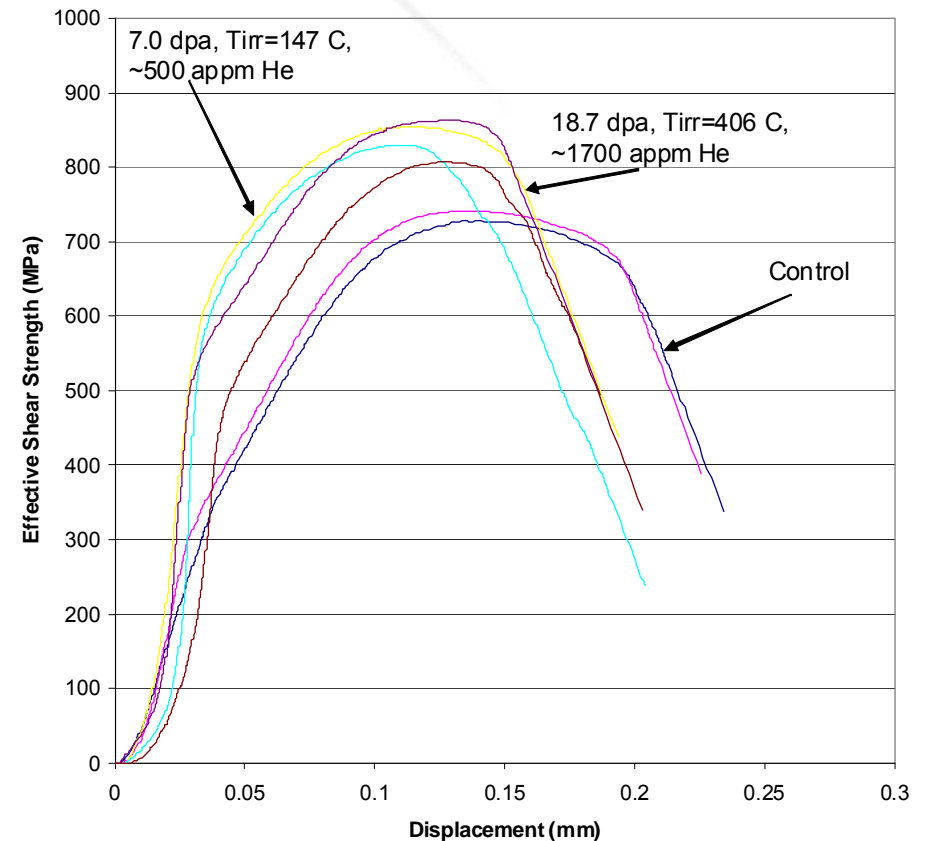
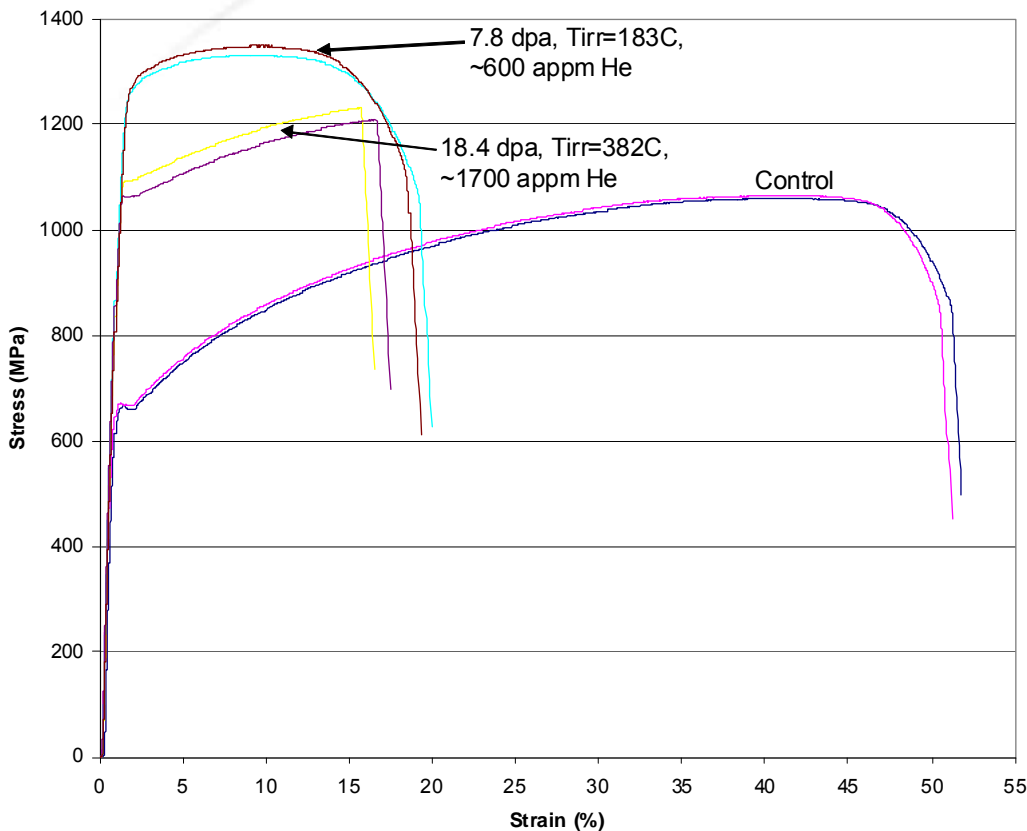




# Previous Results on Proton Irradiated Annealed 718 SPF

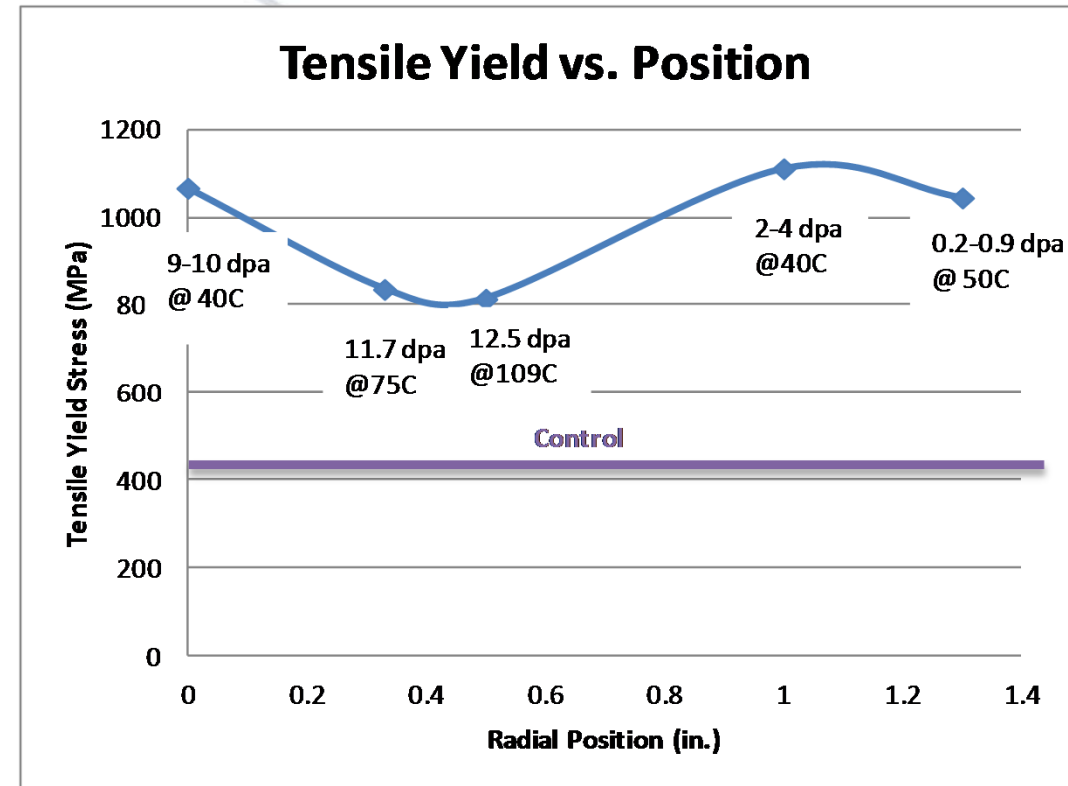
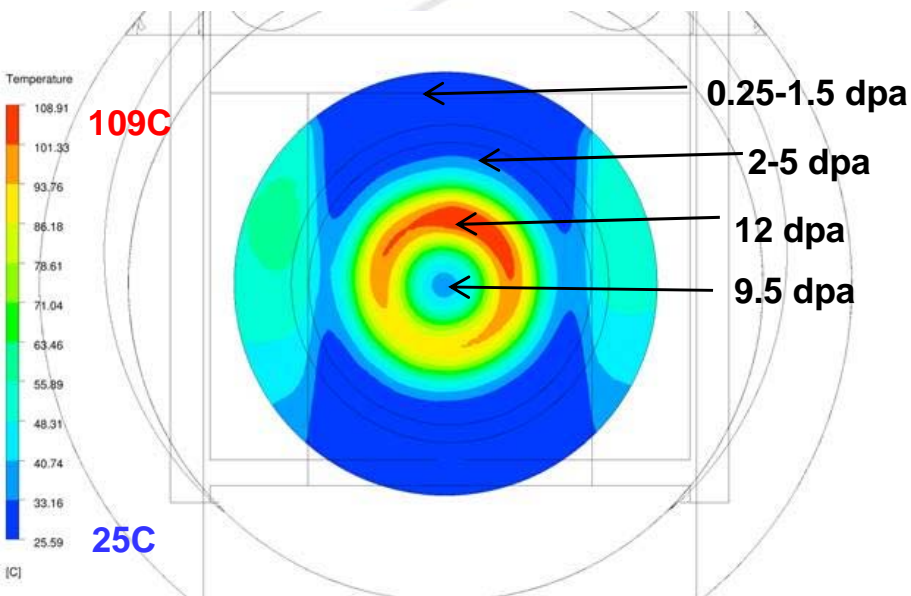
STIP-II Inc 718 SPF Shear Punch Tests

STIP-II Inc718 SPF Tensile Tests



- Inconel 718 SPF samples irradiated in STIP-II
- Samples show good retention of ductility even with much higher helium levels

# Summary of Tensile Results



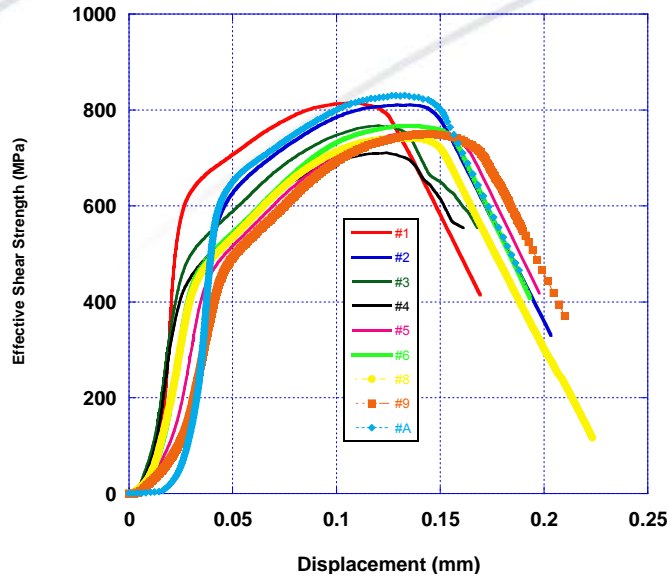
- All samples display ductility in both yield vs UTS and optically.
- Samples taken in outer ring and outside collimator have a higher yield and UTS than control or high radiation dose samples.
- Increased hardening appears to be a combination of increased defect density, bubble density and second phase precipitation
- From these results we are confident to push lifetime out to ~17 dpa
- Further analysis required to understand bubble formation in low dose samples.

## Comparison to previous tensile results

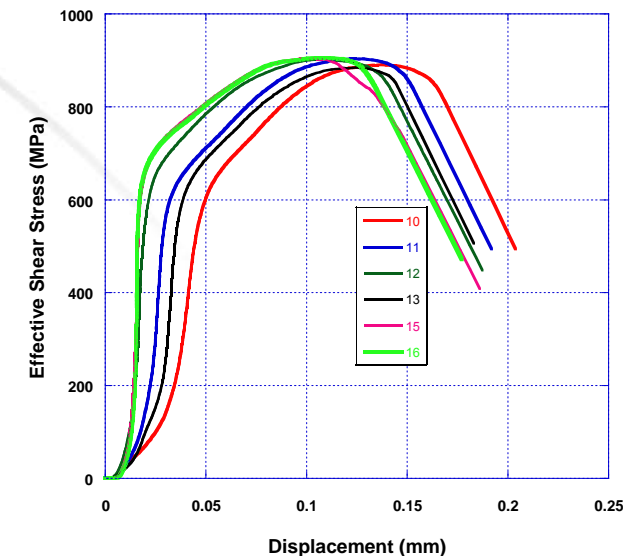
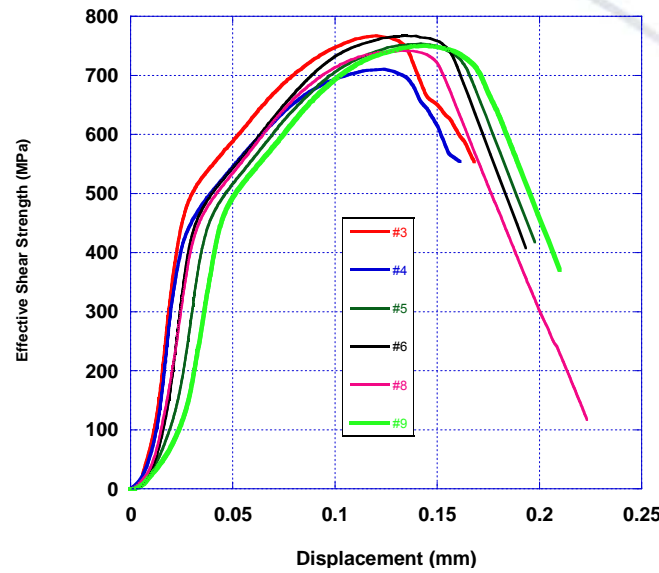
- Farrell et al. (03) shows increase from 350 MPa to 900 MPa for irradiation at 50-100C for a dose of 1.2 dpa
- STIP-II shows increase from 600 to 1200 MPa at 183C and 600 to 1100 MPa at 382C to doses of 7.8 and 18 dpa respectively
- IPF data shows increase from 450 to 1100 MPa at 9 dpa and 850 MPa at 11-12 dpa. Also an increase to 1100 MPa is observed at 2-4 dpa.

# Shear Punch Results

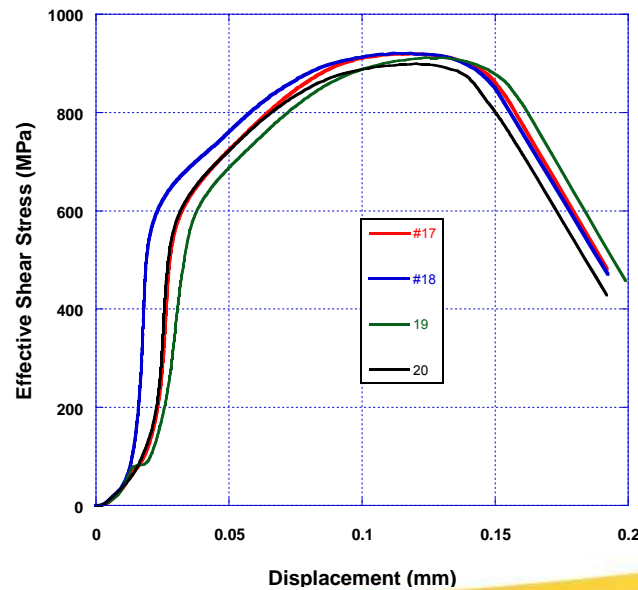
Almost All Middle Range



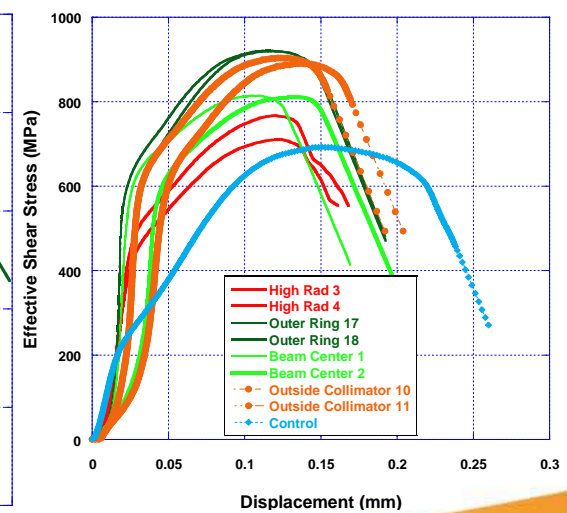
HighRad Ring

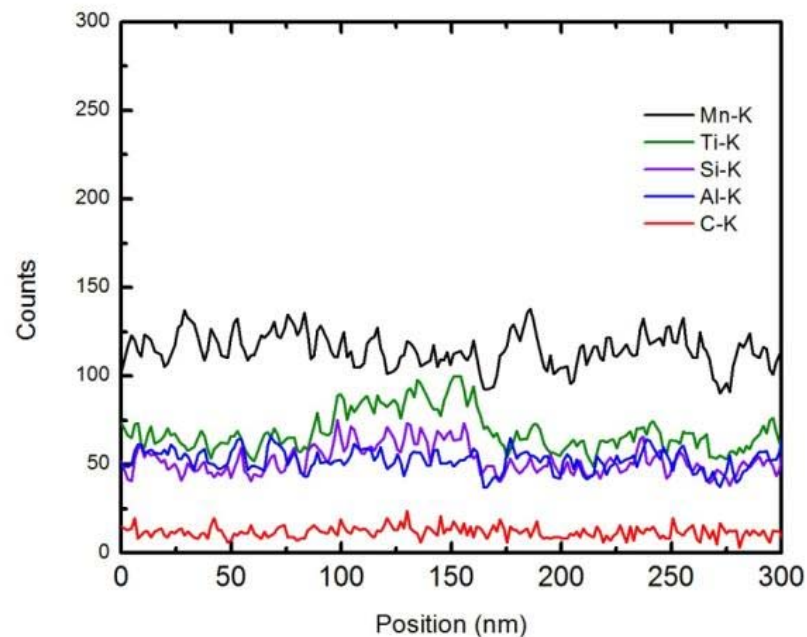
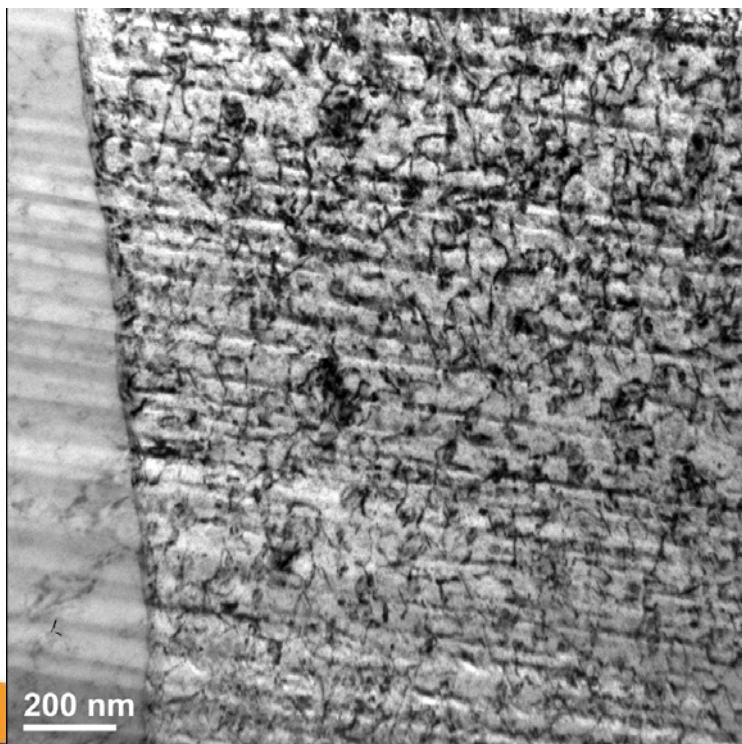
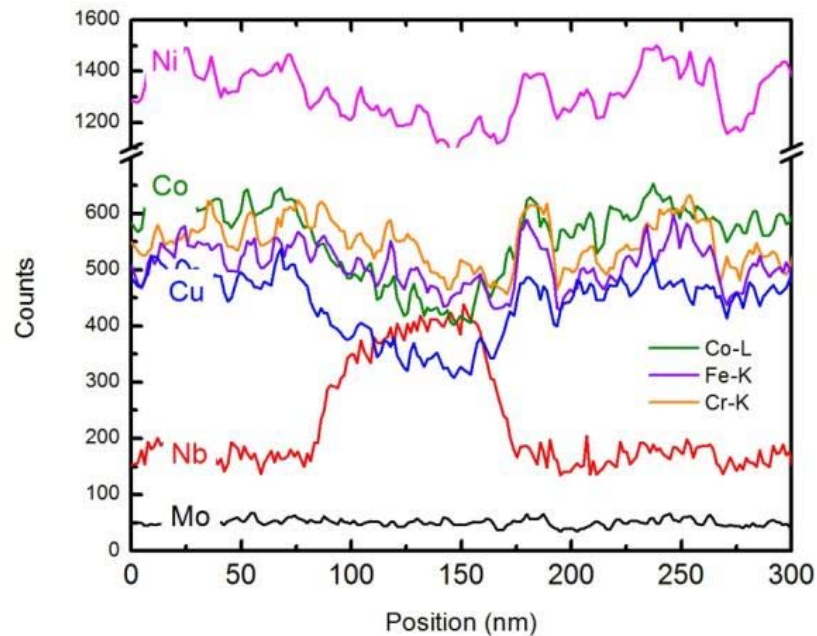
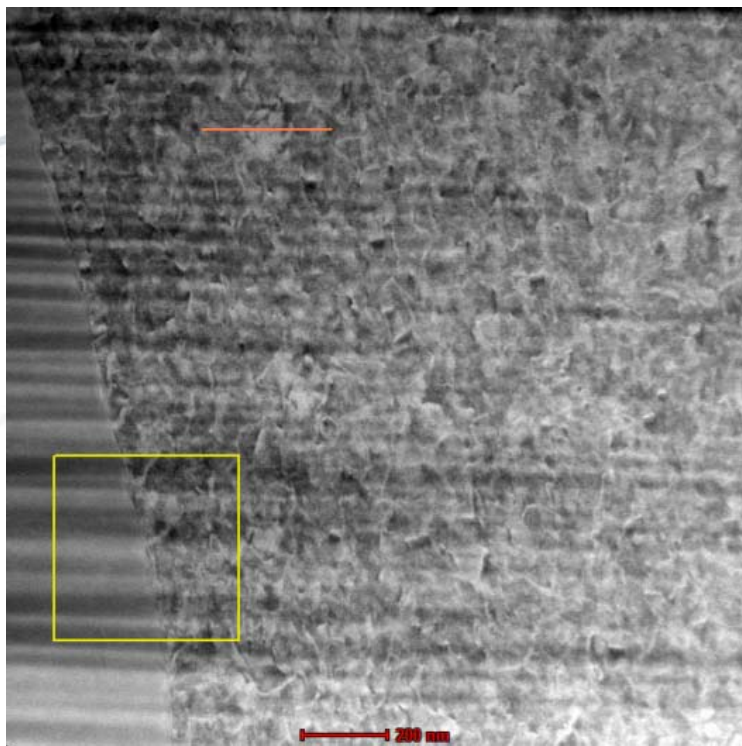


OuterRing



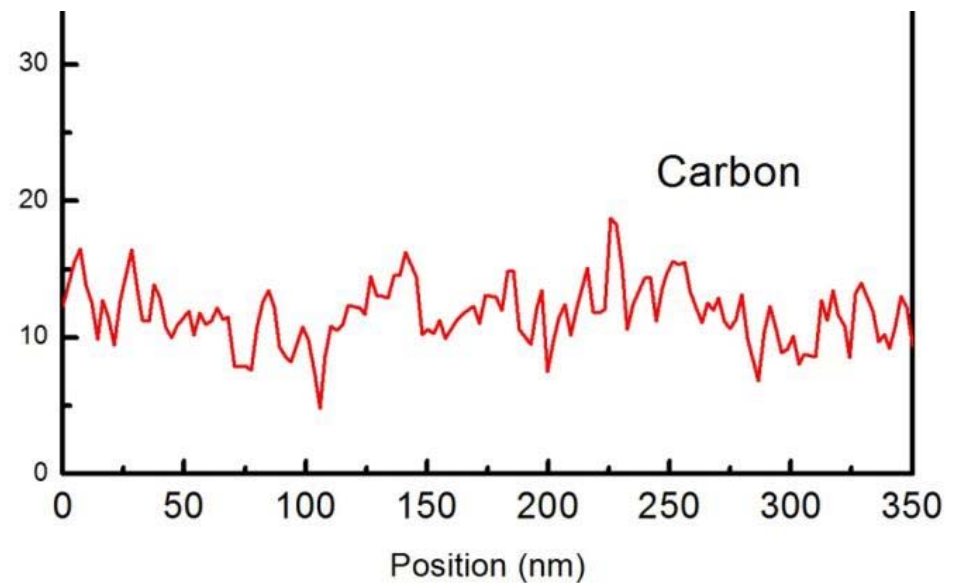
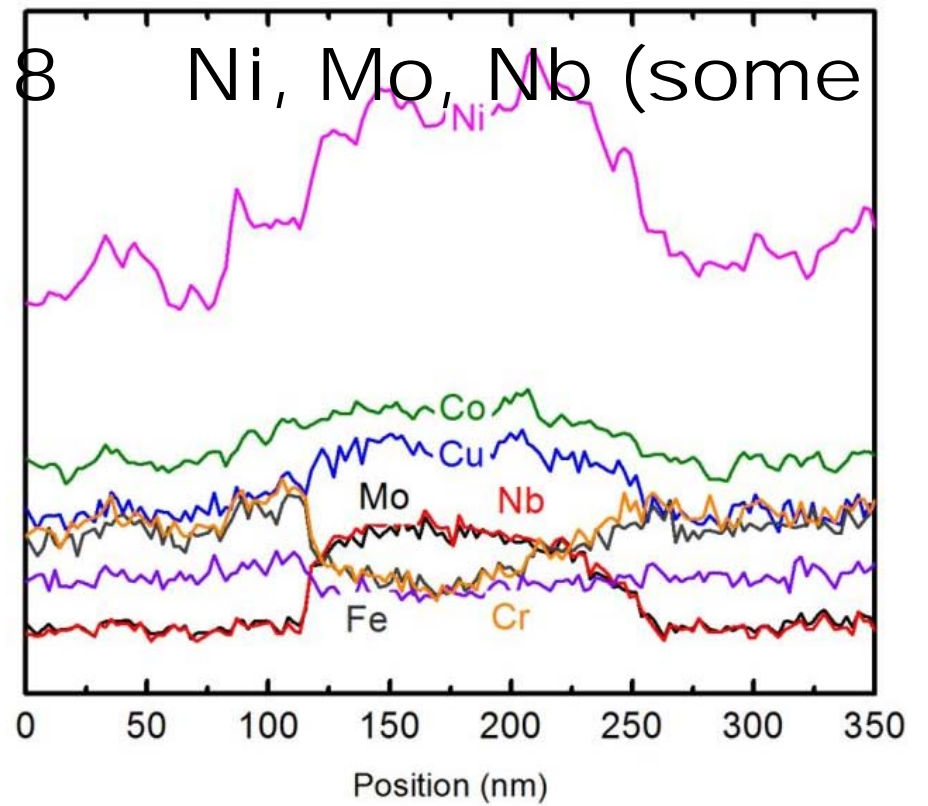
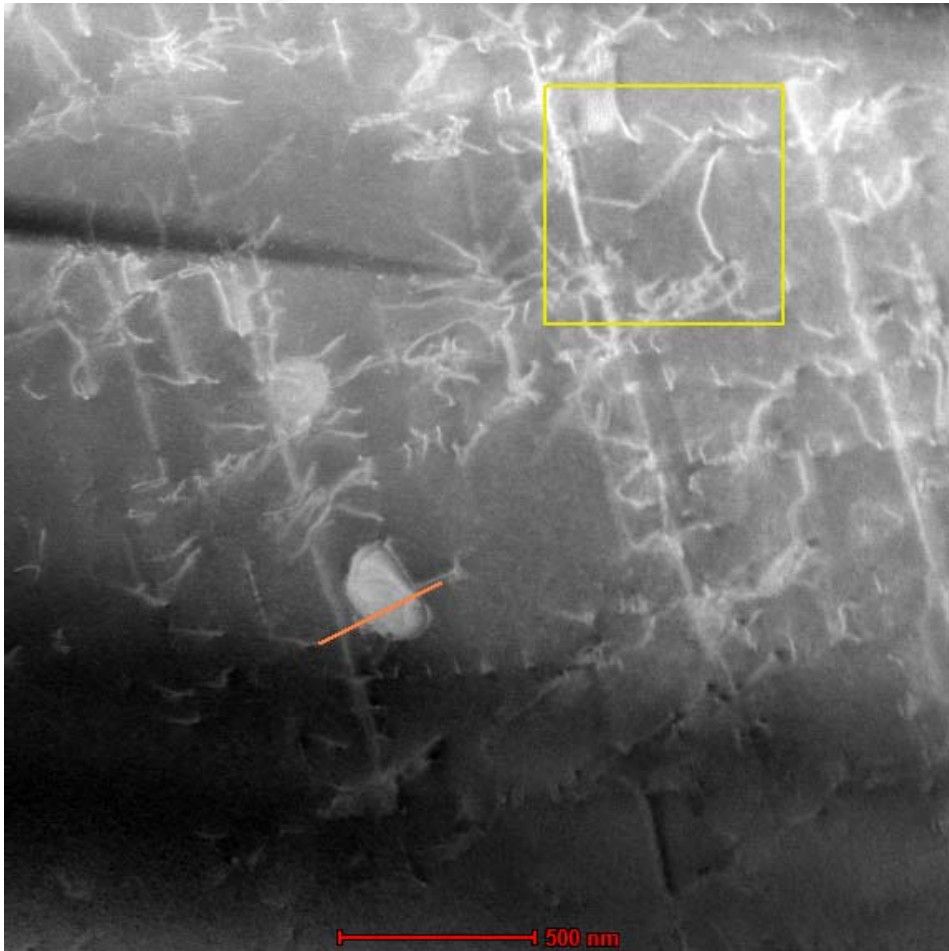
Control + 4 regions



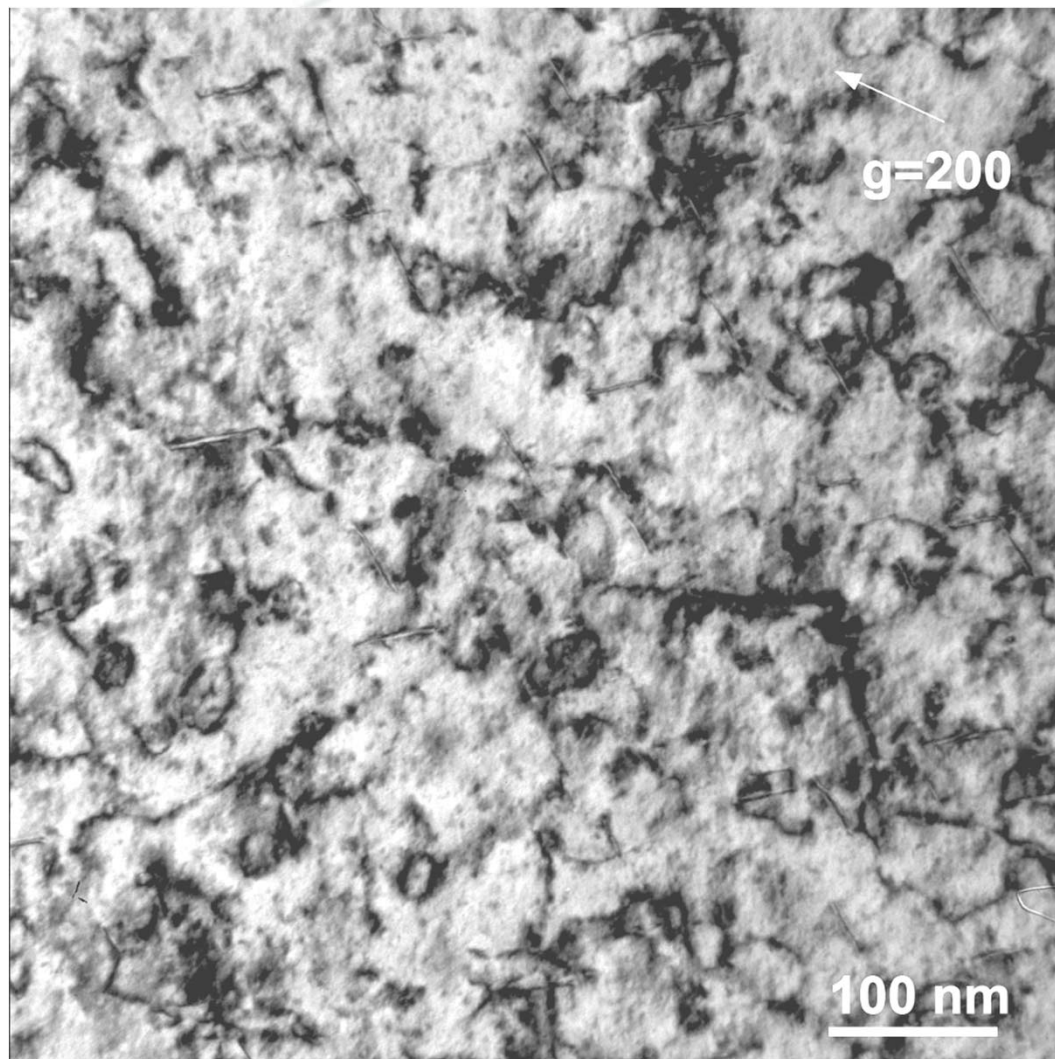


■ Dark and bright field TEM images showing smaller precipitates and dislocations.

# Unirradiated Inconel 718 Co, Cu) rich precipitate



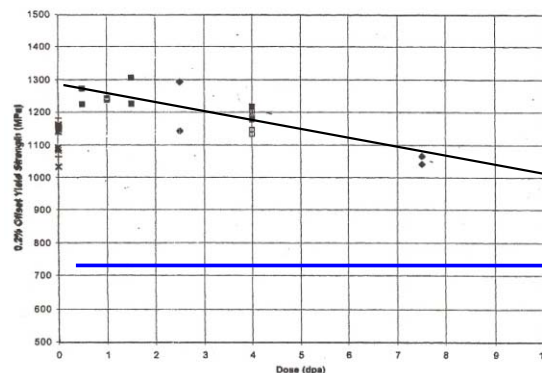
# Inconel 718 #5 ~14 dpa @109°C



# Irradiation Damage and Replacement

- Beam transmission through the window incurs heating causing thermal stress.
- Beam irradiates the window causing mechanical properties to change.
- Beam window design criteria is 20 dpa (displacement per atom). Beam window reached the end of its life.
- Estimate dose rate is 100 R/hr at contact without shielding and highly contaminated.
- We replaced window in March 2010, stored at Area A and shipped to CMR in November 2010.

718 Yield



Fi Dose, dpa

Calculated Von Mises stress under pressure and thermal load at the center of the target is ~ 510 MPa. Window will fail when Von Mises stress > the yield strength

15 18 20

End of run cycle 2009



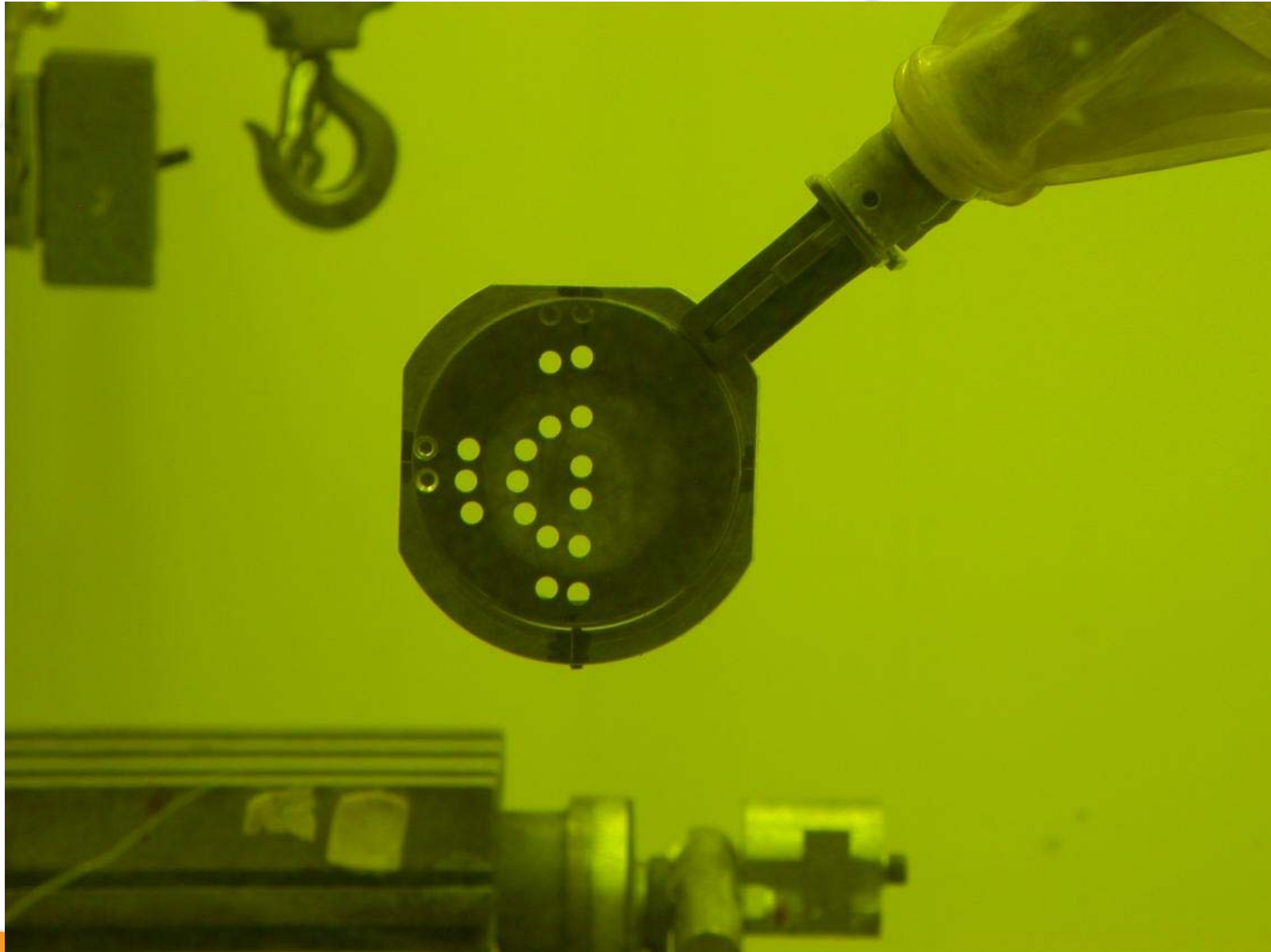
# Image of Beam Window Surface



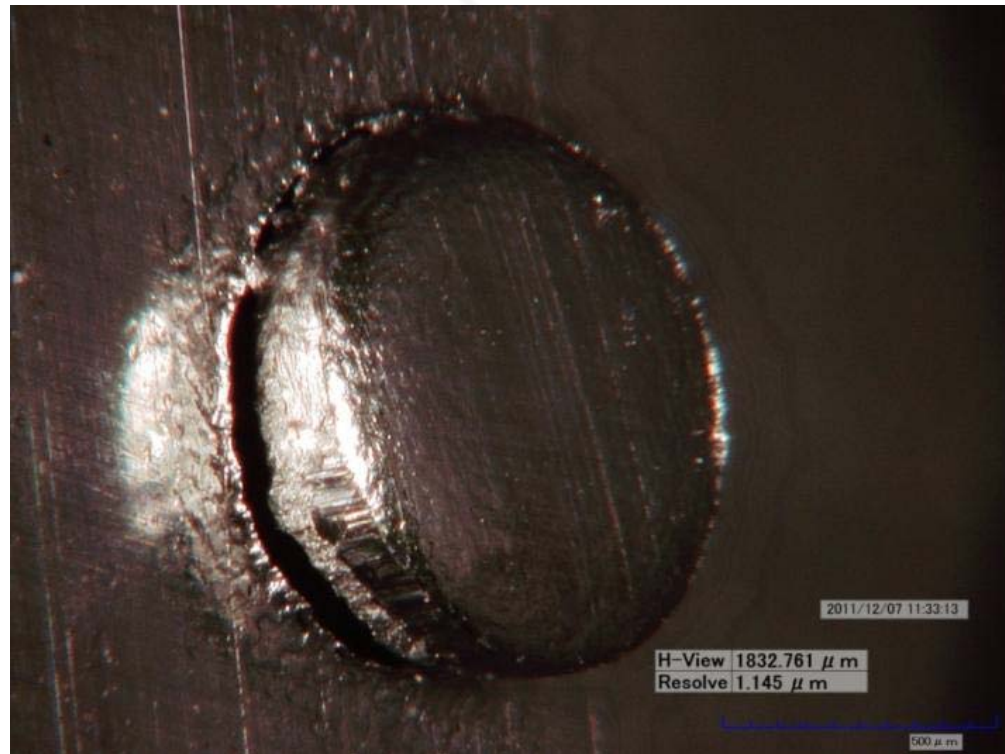
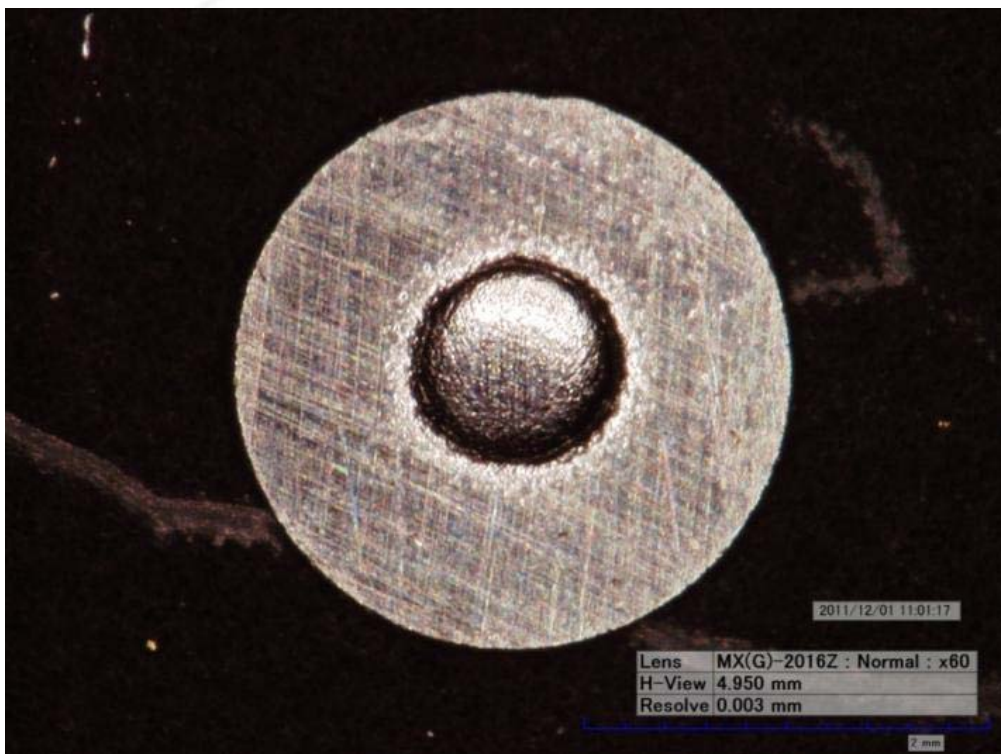
Outline of beam collimator is evident on beam window

Approximate dimensions are 10 cm diameter x 0.5 mm thick.

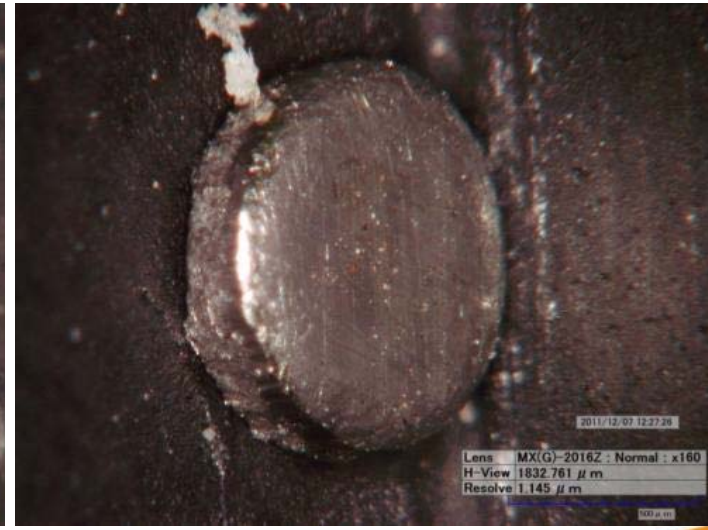
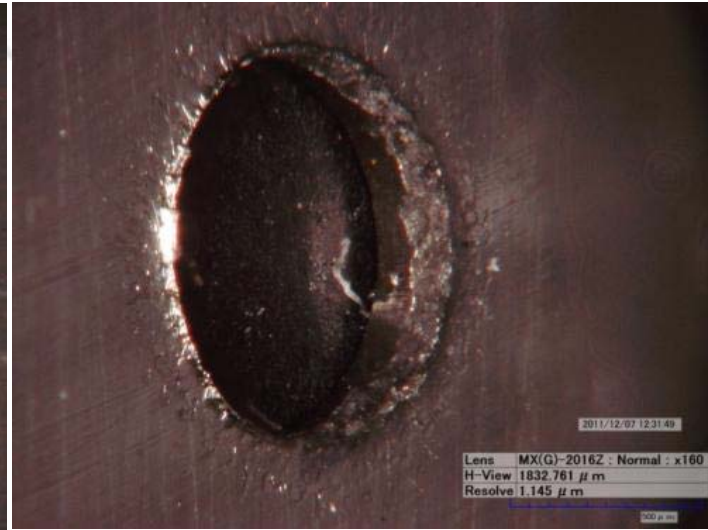
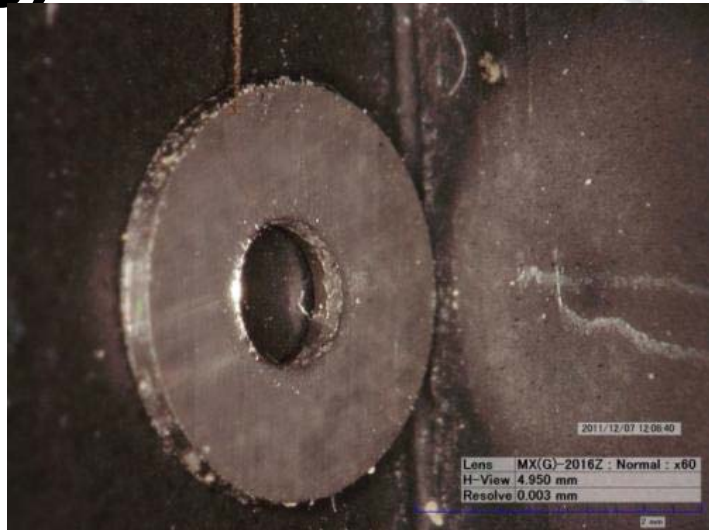
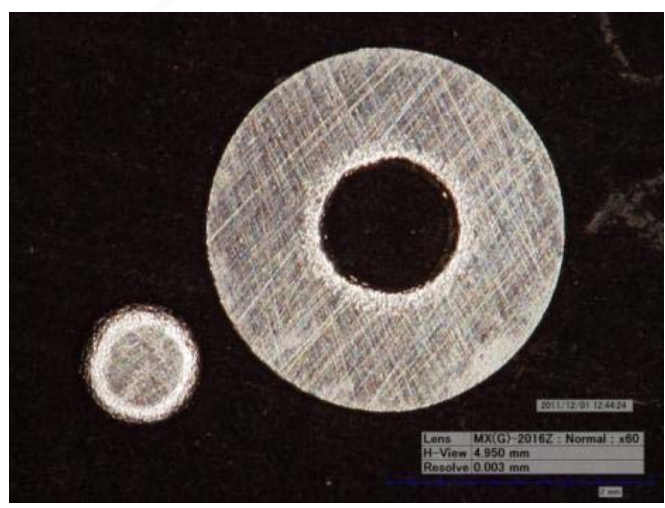
# Machined Window

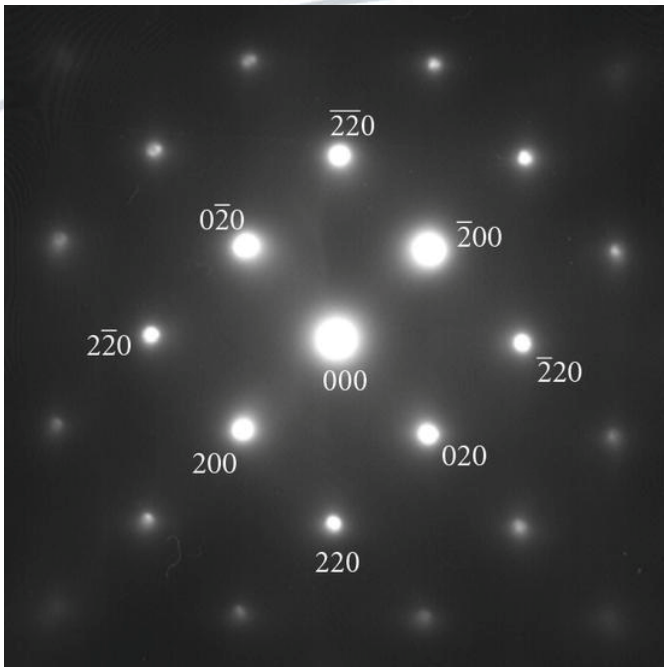


# Optical Images of Control Sample #6 (Unirradiated)

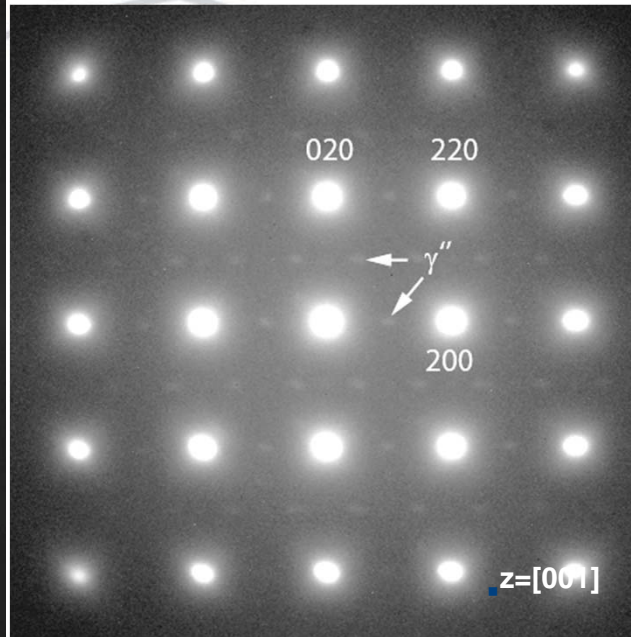


# Optical Images of Sample # 8 (High Radiation Ring)

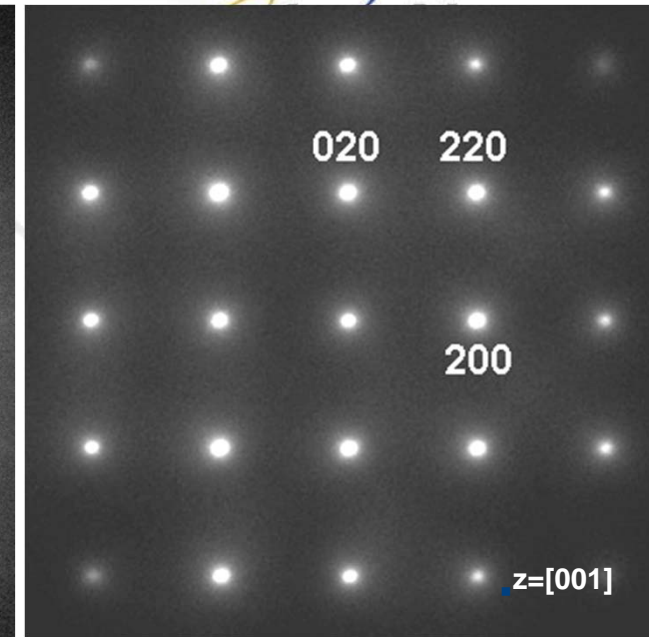




Unirradiated (SA)

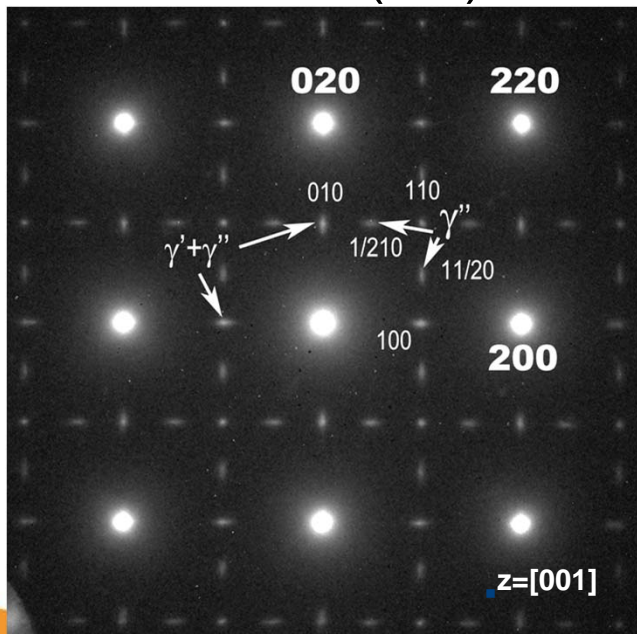


0.5 dpa at 50°C

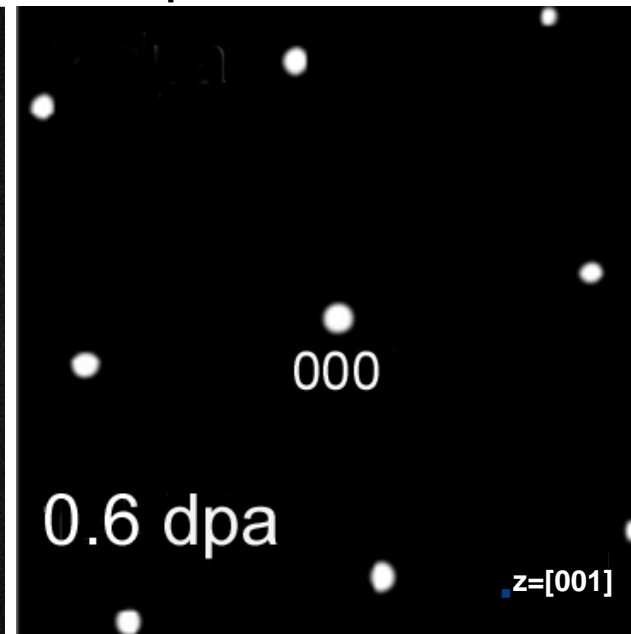


12.5 dpa at 109°C

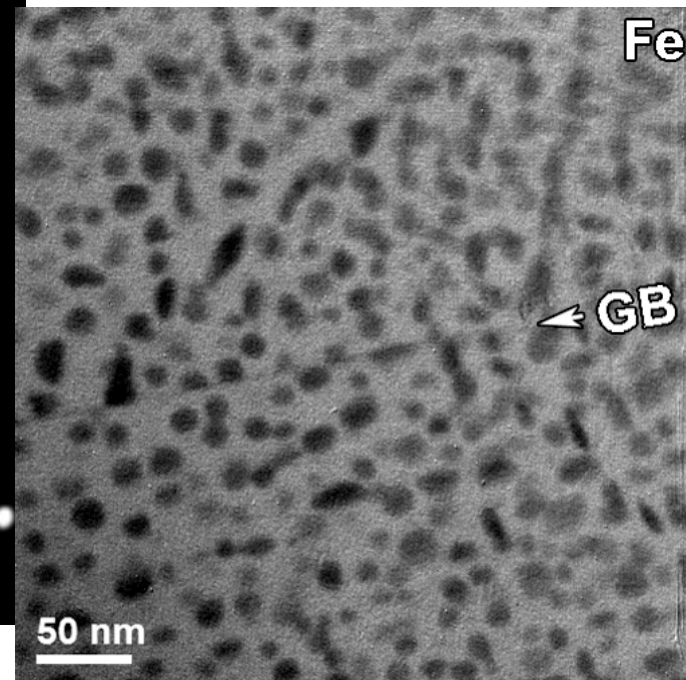
Reference: M. Dehmas et al. Advances in Mat. Sci. and Eng (2011)



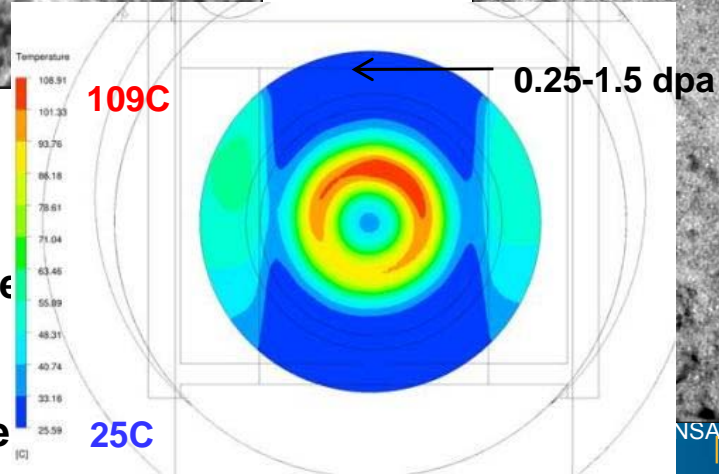
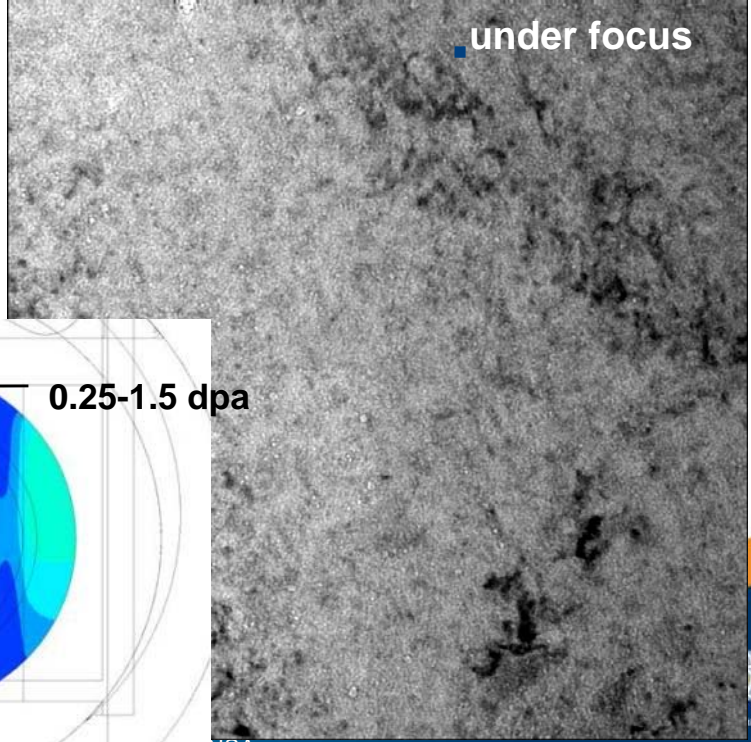
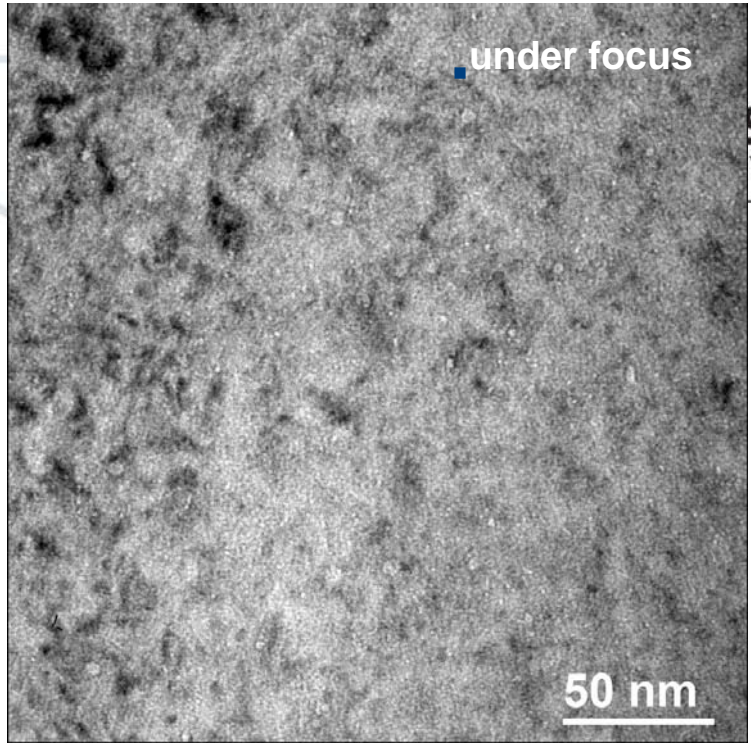
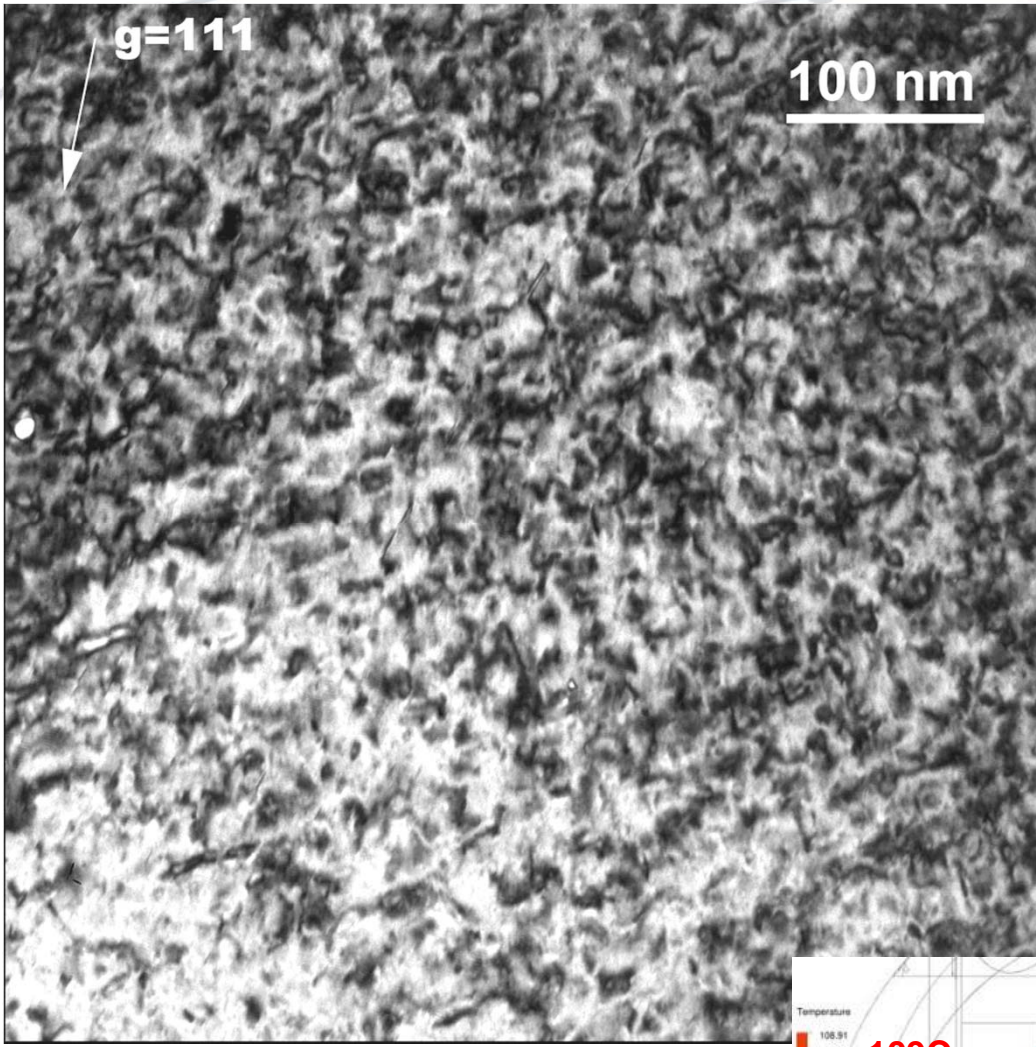
Unirradiated-PH



0.5 dpa at 30-60°C



# Inconel 718 #19 ~ 0.5 dpa @50°C

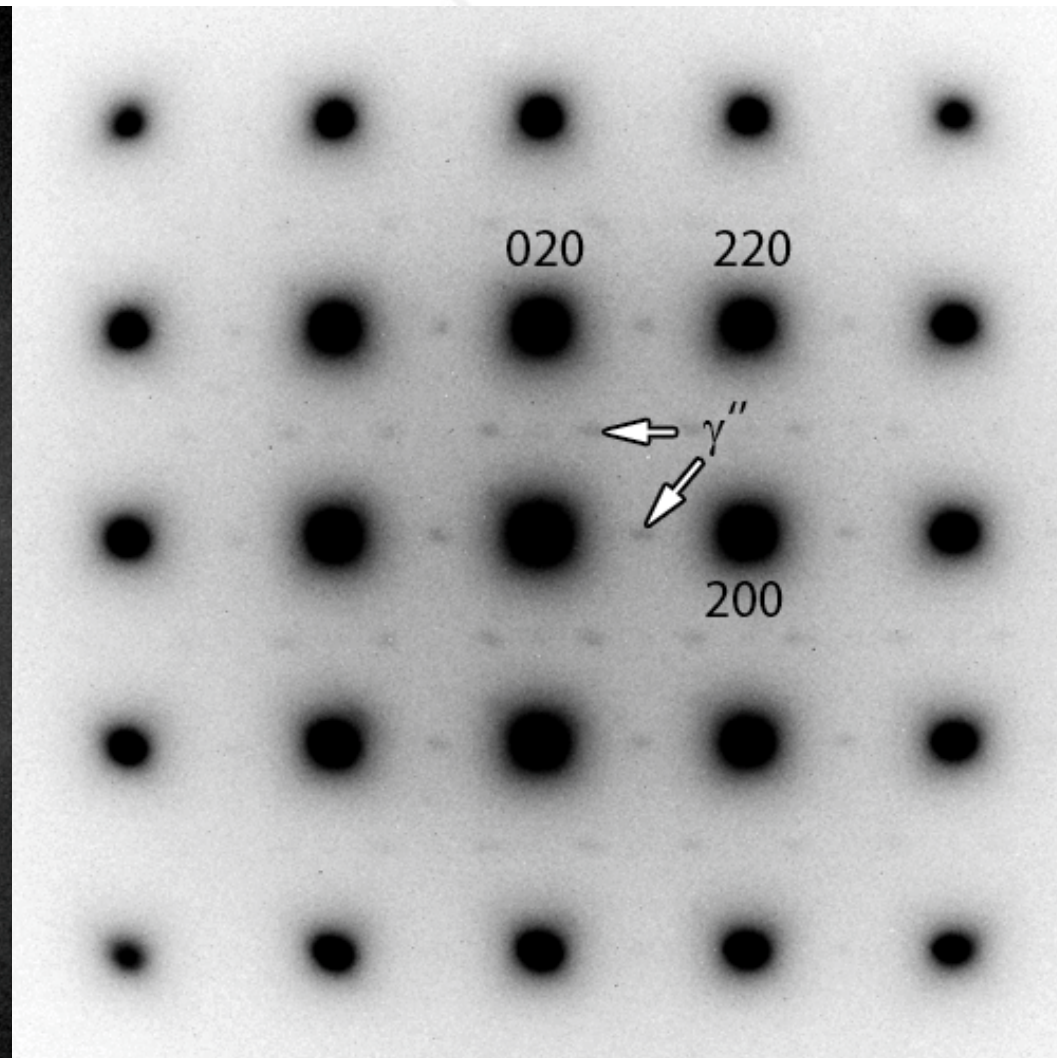
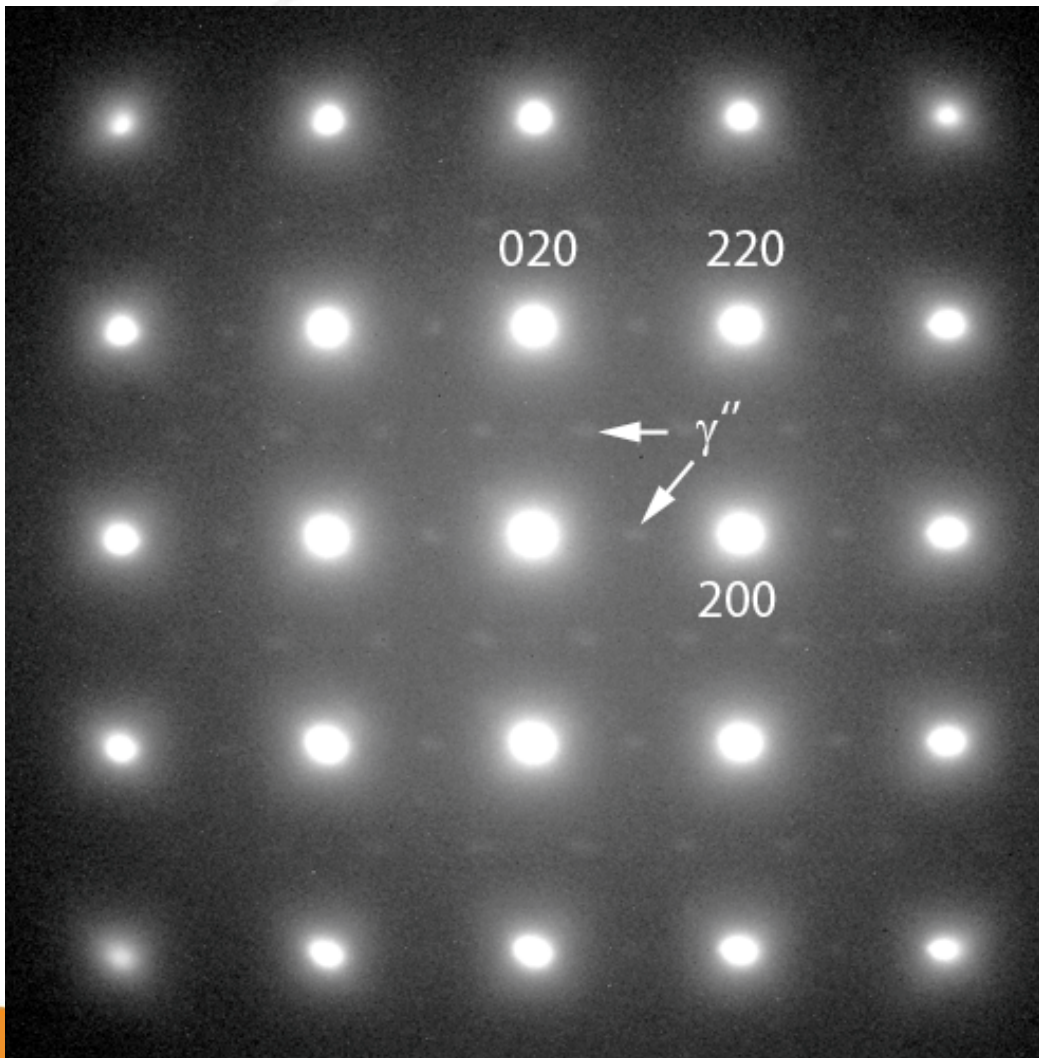


•Bright field TEM images showing dislocation loops.

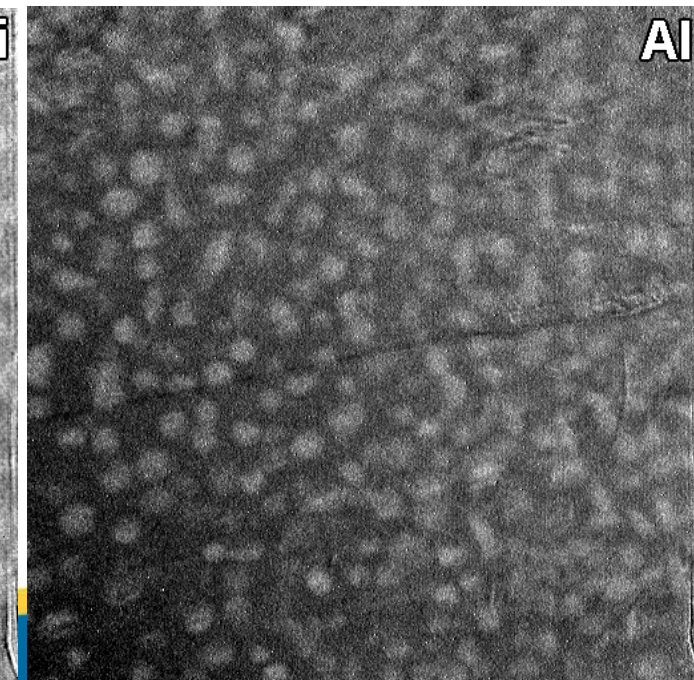
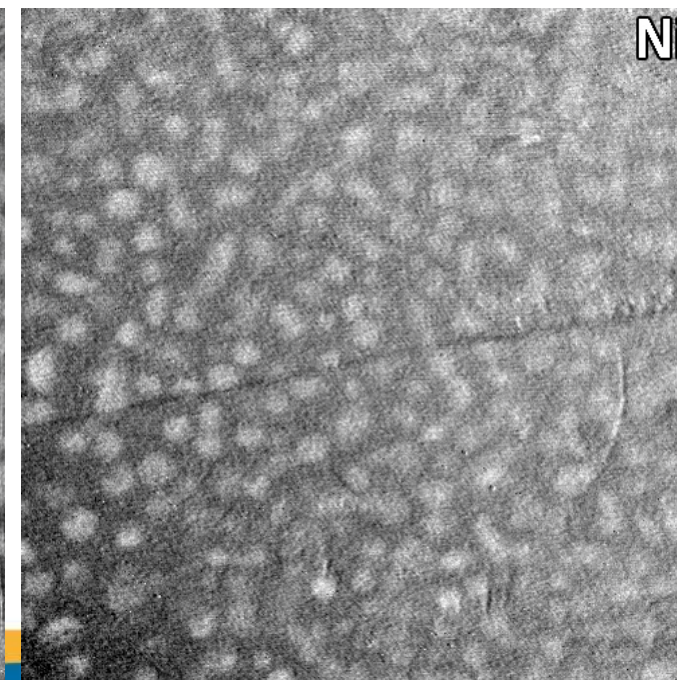
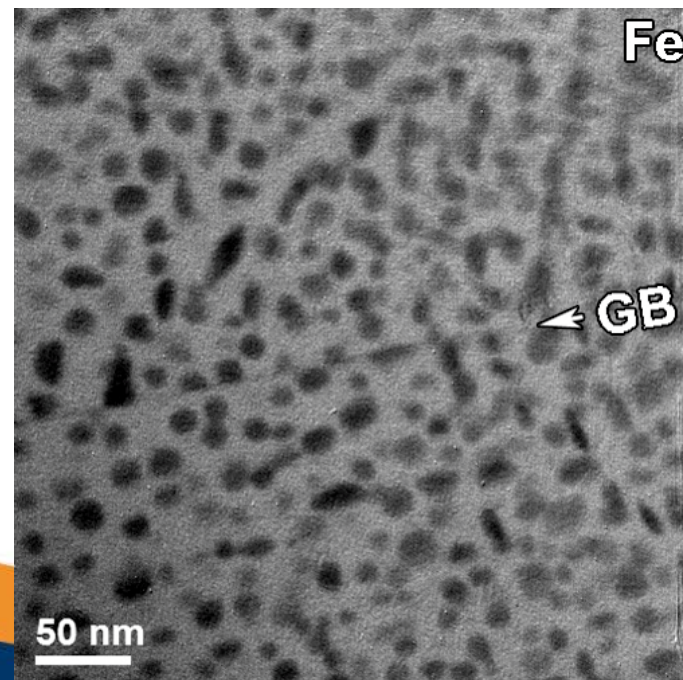
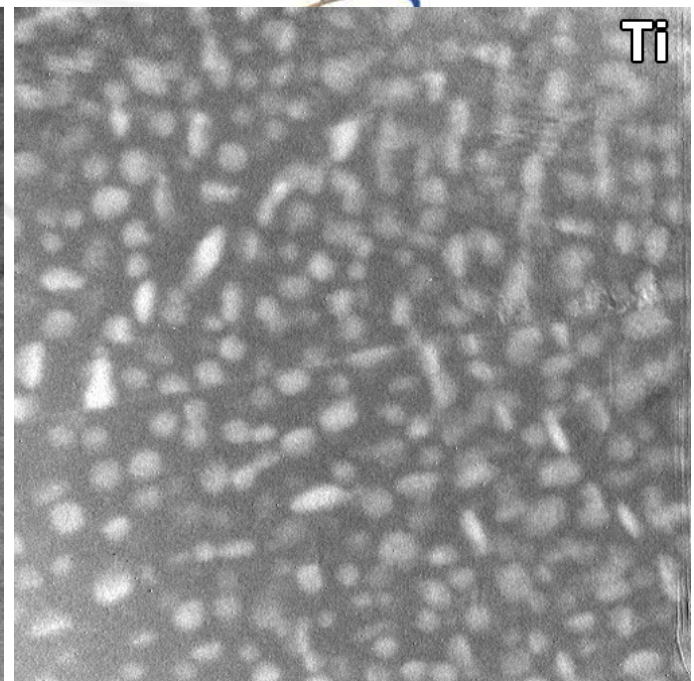
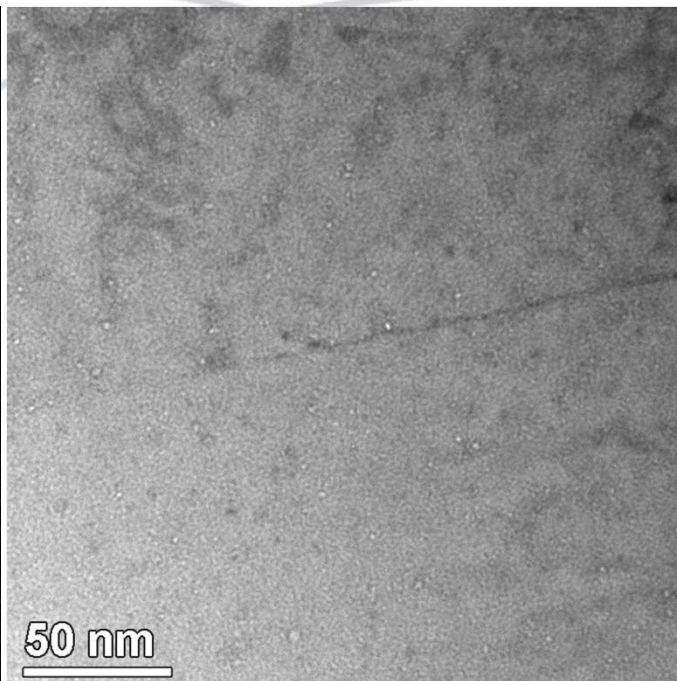
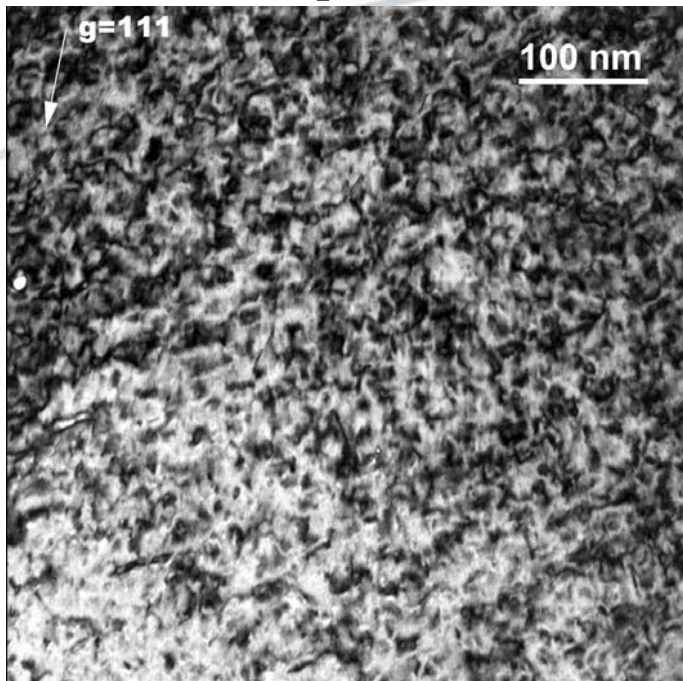
•Under-focus TEM images on the right are showing a high

•Density of bubbles/voids that are on the order of 5-10nm

# Inconel 718 #19 ~ 0.5 dpa @50°C $\gamma''$ precipitates

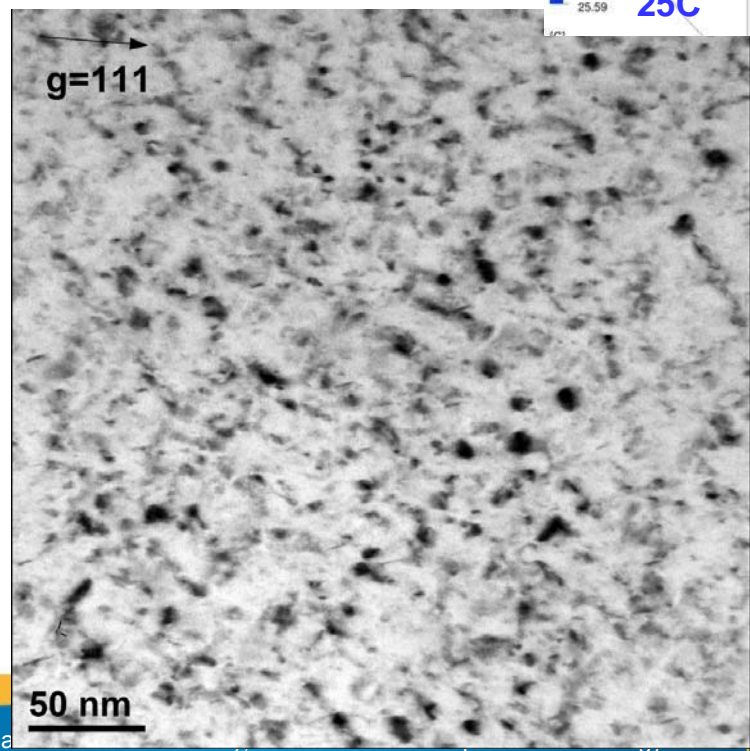
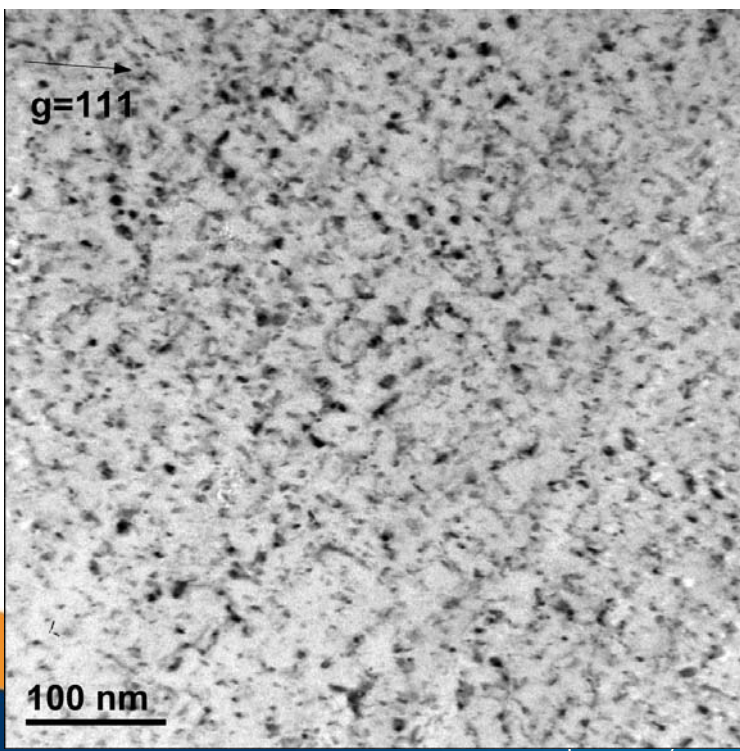
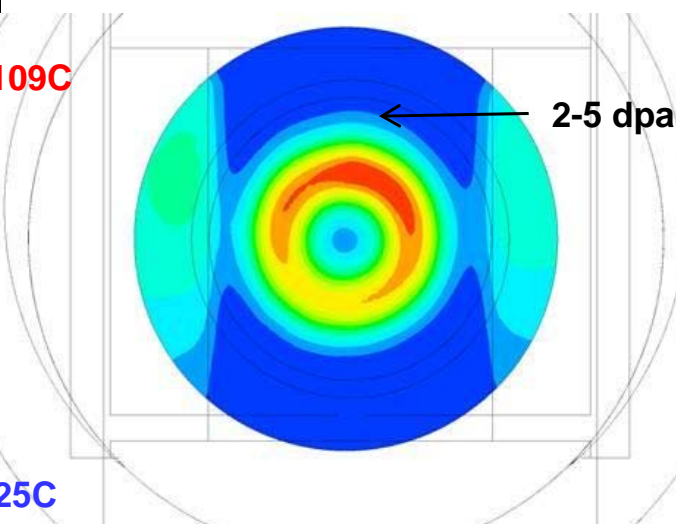
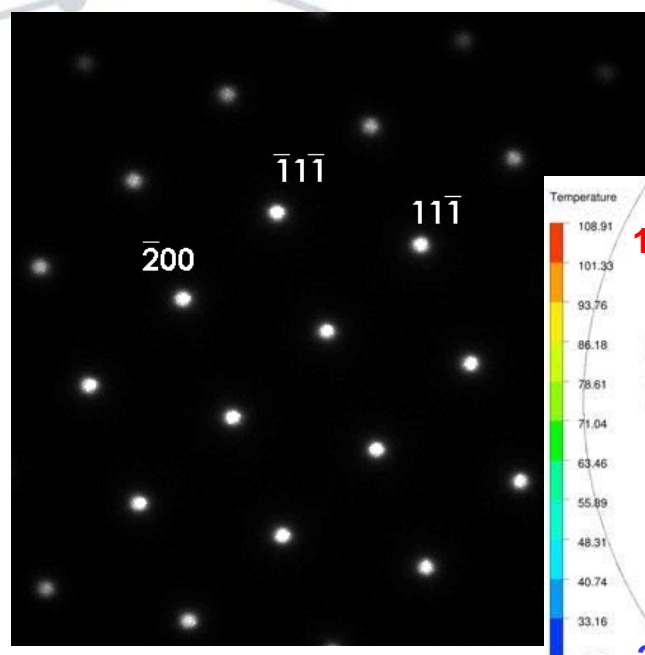
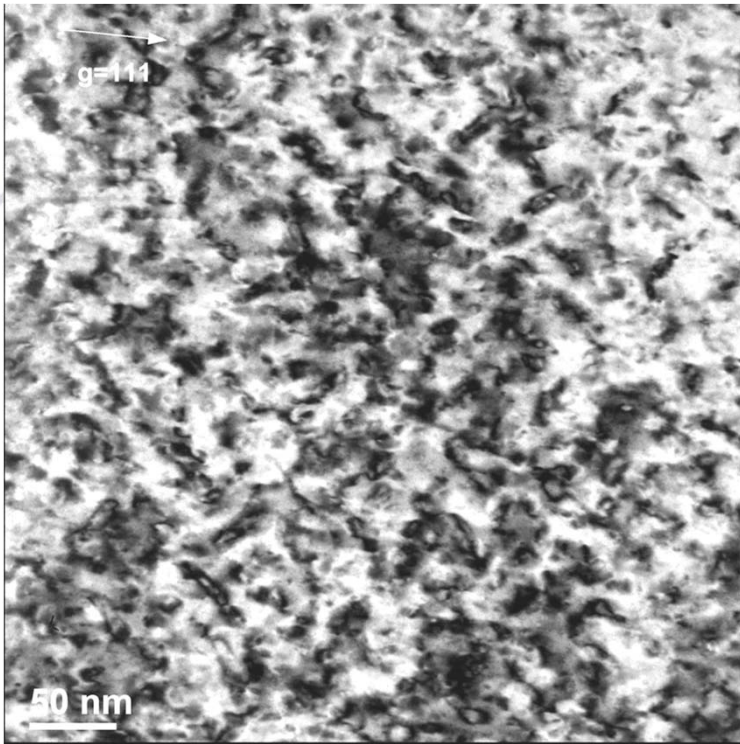
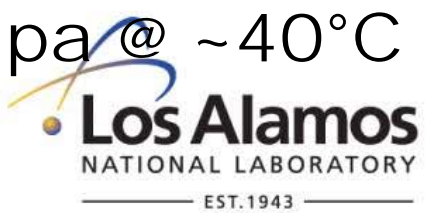


# 0.5 dpa 50°C

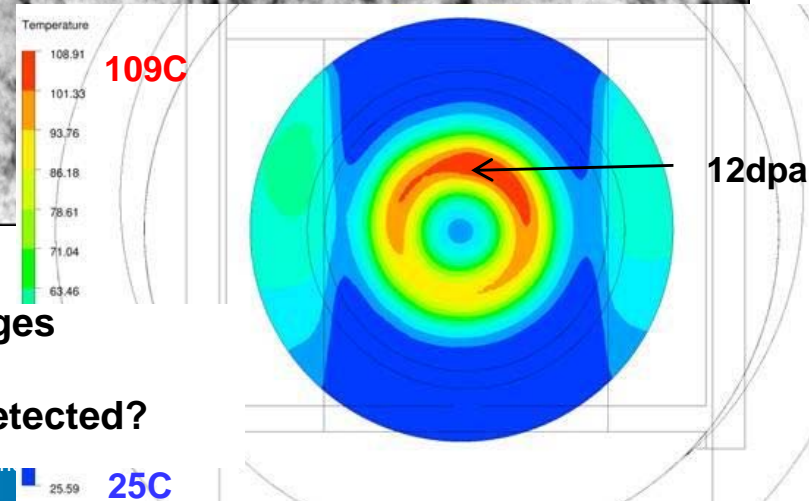
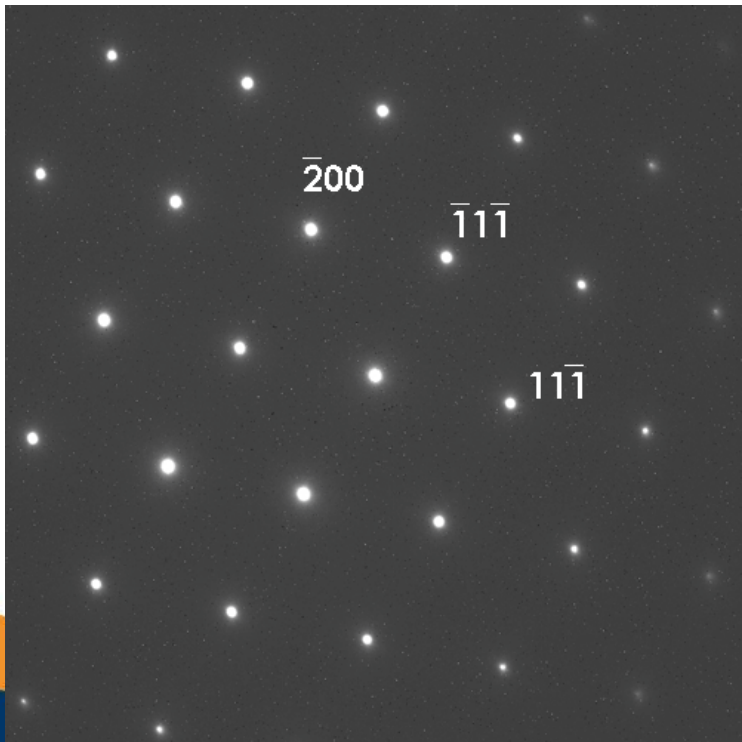
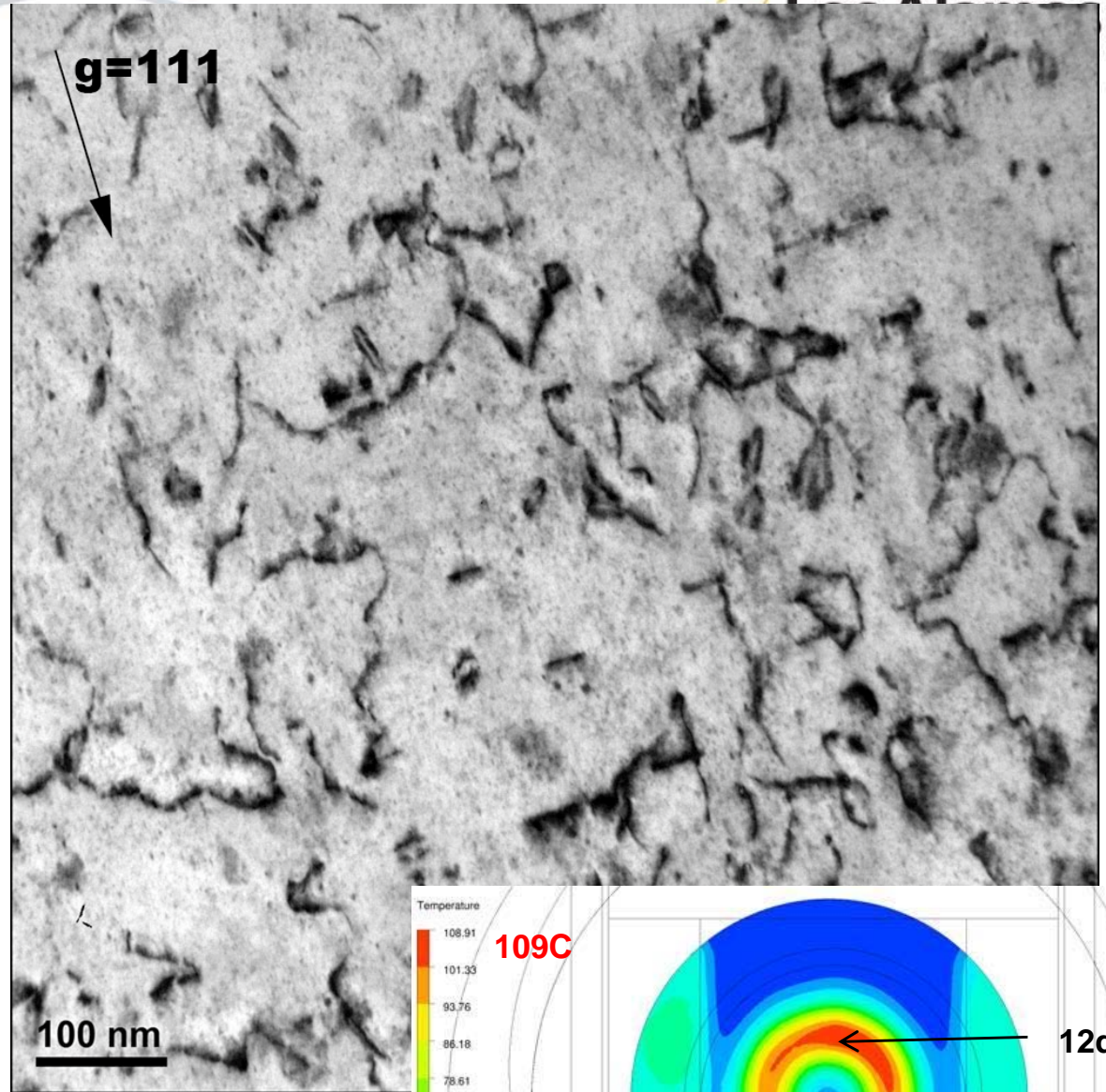
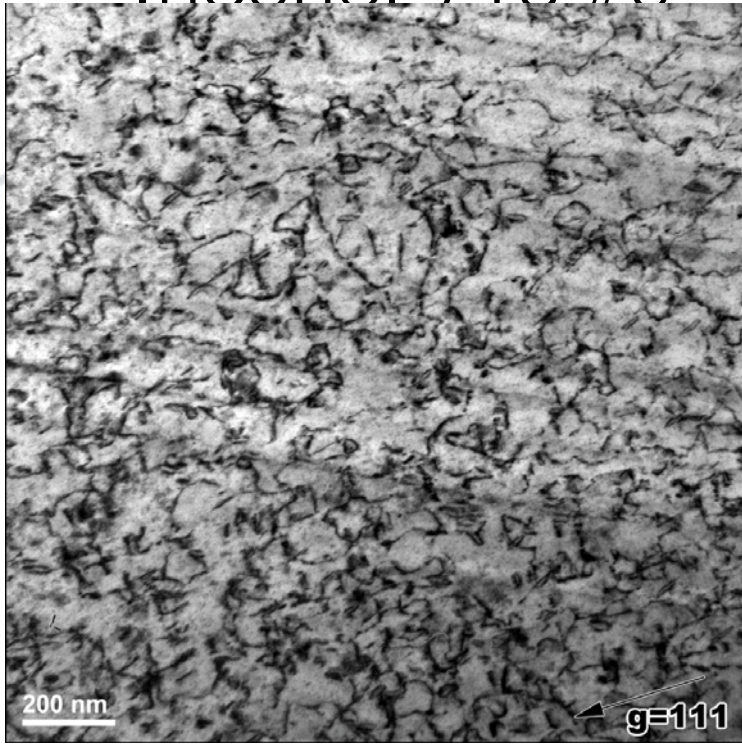




# Inconel 718 #16 ~ 2.5 dpa @ ~40°C

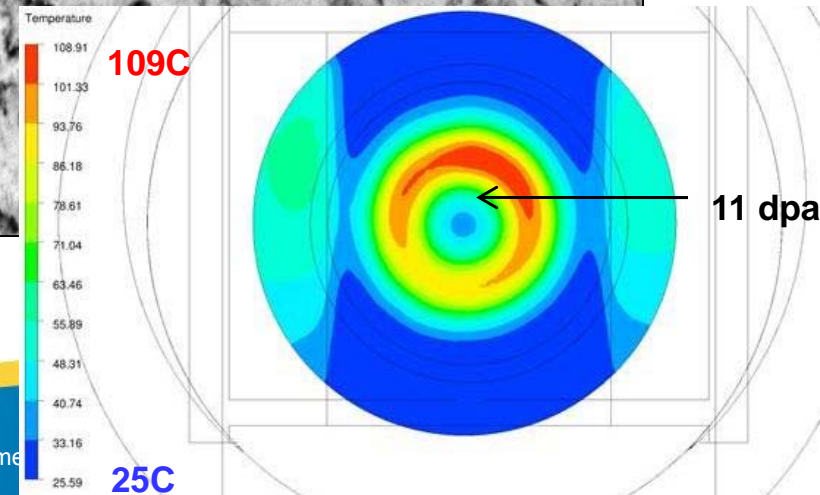
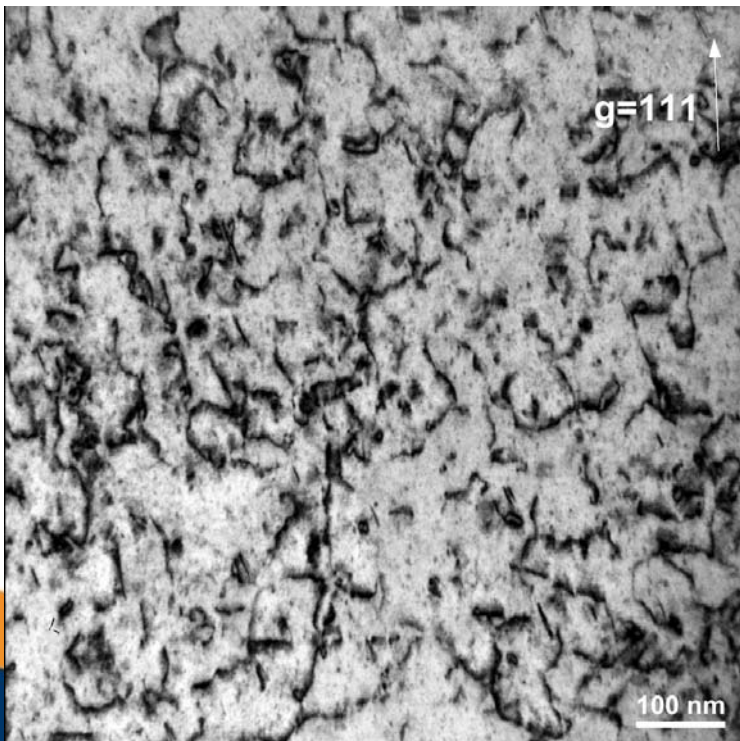
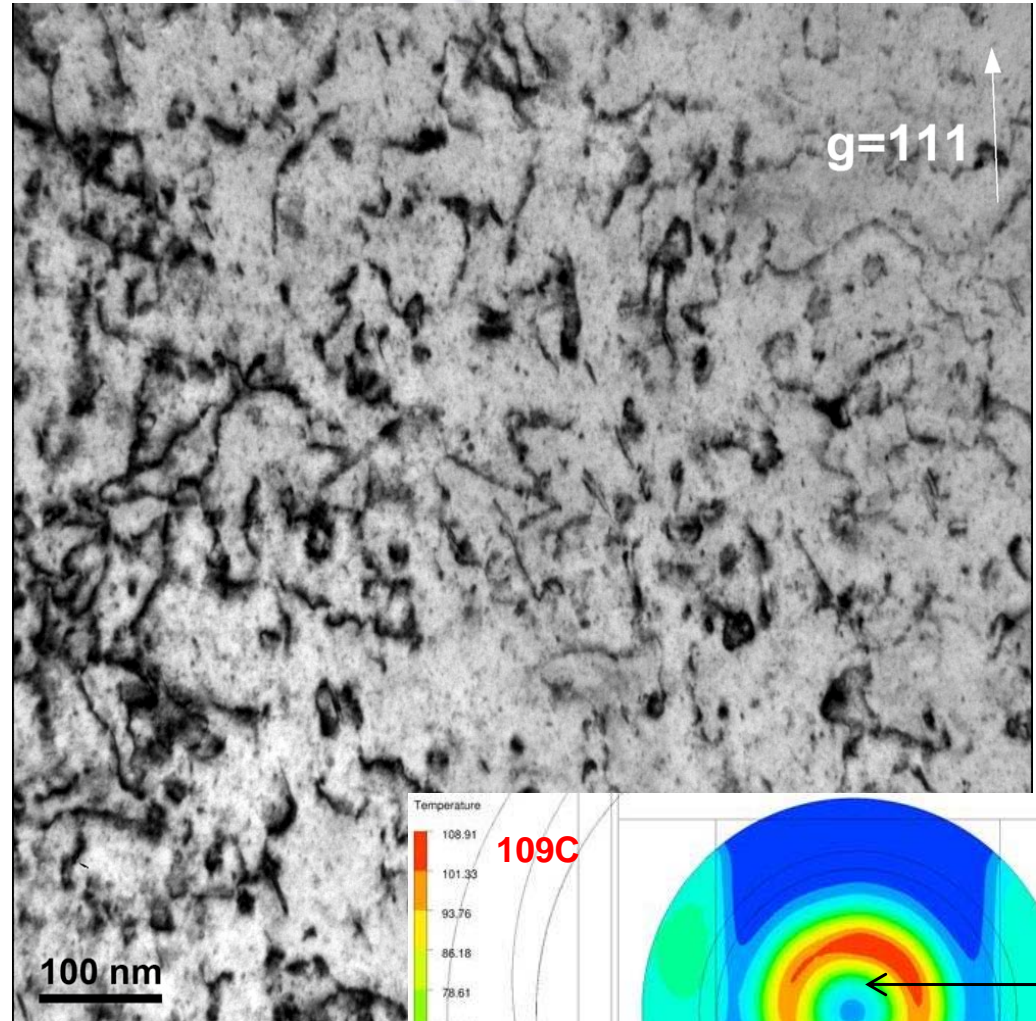
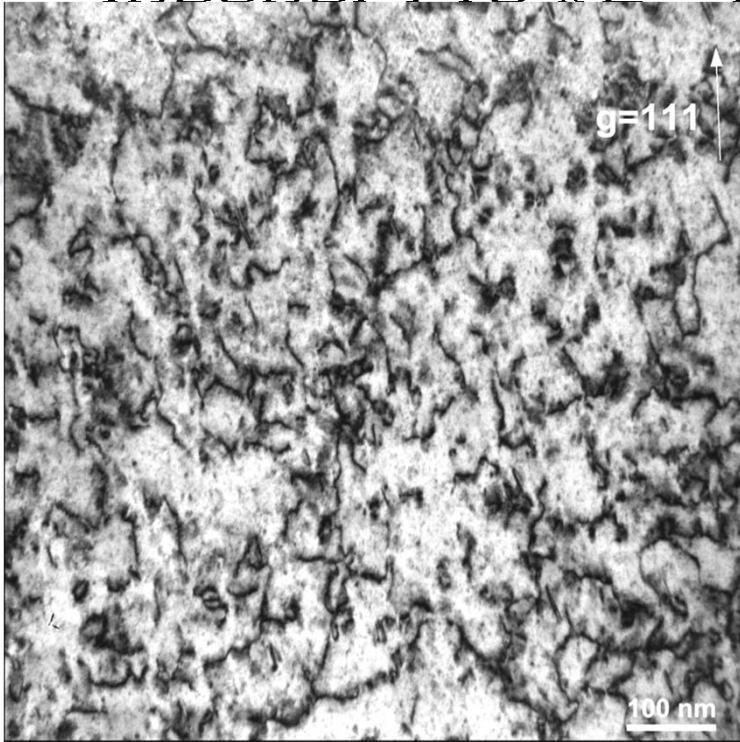


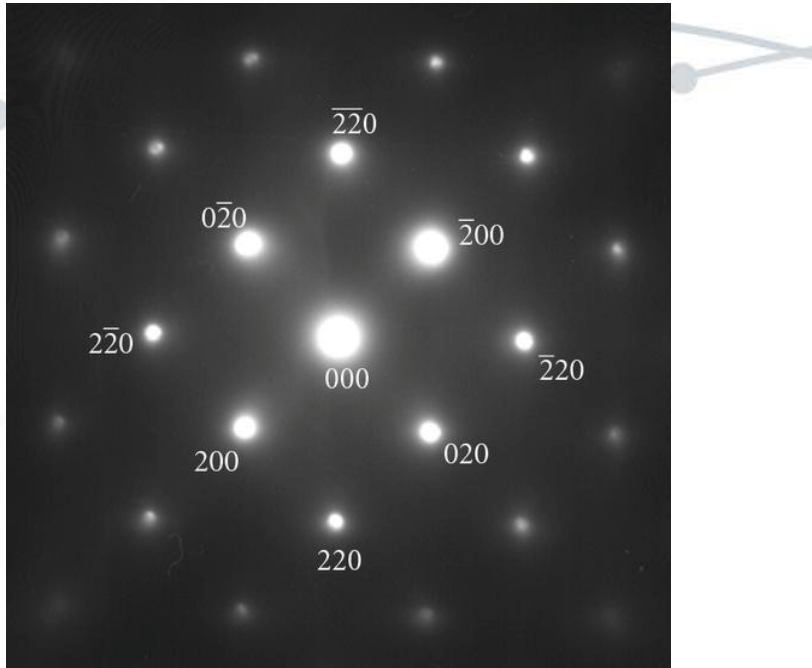
# Inconel 718 #5 ~12 dpa @109°C



Bright field TEM images showing dislocations, precipitates are not detected?

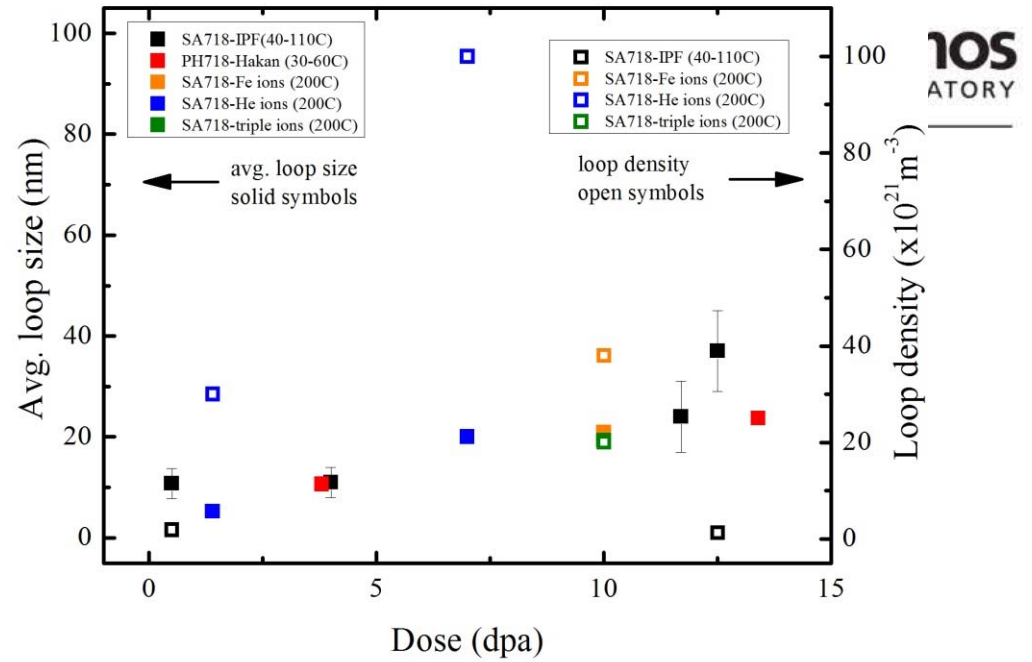
# Inconel 718 #E ~11 dpa @~75°C



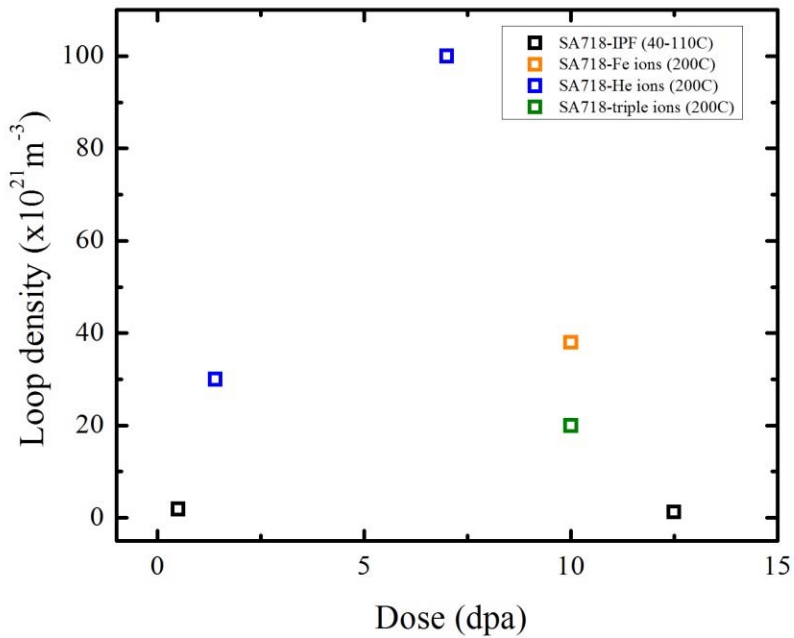


Reference: M. Dehmas et al. *Advances in Mat. Sci. and Eng* (2011)

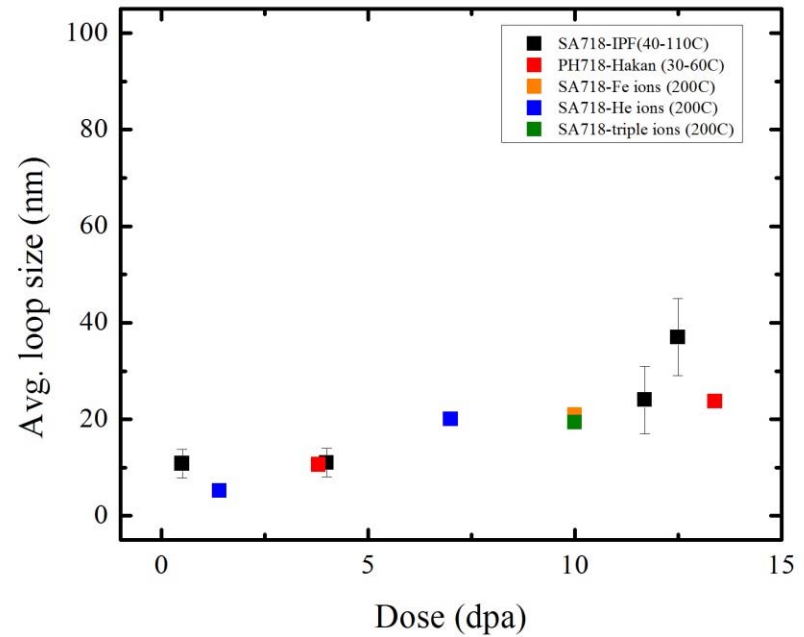
## Loop size and density



105  
ATORY



Loop density



Loop size

