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Periodic Surface Morphology characteristics of Tungsten Under Simultaneous Helium and Deuterium Bombardment at 30 keV

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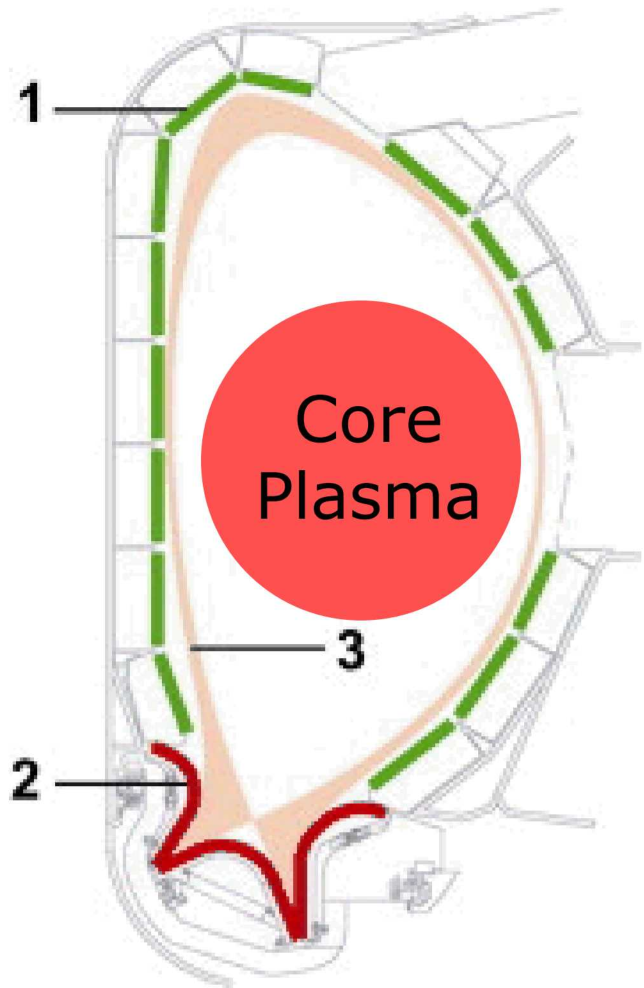
Overview

- Introduction
- The DAISIE Experiment
- Implantation Results and Discussion
  - Erosion
  - Morphology
  - Retention
- Conclusions

Plasma-Material Interactions in Burning Fusion Plasmas

- Particle fluxes of $\sim 10^{18}$–10^{20} cm^{-2} s^{-1}
- Accumulated fluence of $\sim 10^{26}$ cm^{-2}
  - Ions and neutrals
  - H isotopes with ~5-10% He
- Heat fluxes at the divertor of ~10–20 MW m^{-2}
- Neutron flux of $\sim 10^{14}$ cm^{-2} s^{-1}
  - 14 MeV for D-T fusion
  - 2.45 MeV for D-D fusion



Key: 1) First wall, 2) Divertor 3) Scrape-off layer

Quoted parameters from Ueda et al., *Nuclear Fusion*, 2017
Image adapted from: Reith et al., *Journal of Nuclear Materials*, 2013

Advantages of Tungsten as a Plasma-Facing Component

- 2013: ITER Council moves from carbon fiber-based divertor armor to full-tungsten divertor (150 m^2)
- High melting temperature (3422 °C)
- High thermal conductivity (173 W/m-°C)
- Low H isotope retention (1 kg inventory limit in vessel and pumping systems)
- Low sputtering yield (plasma impurities)

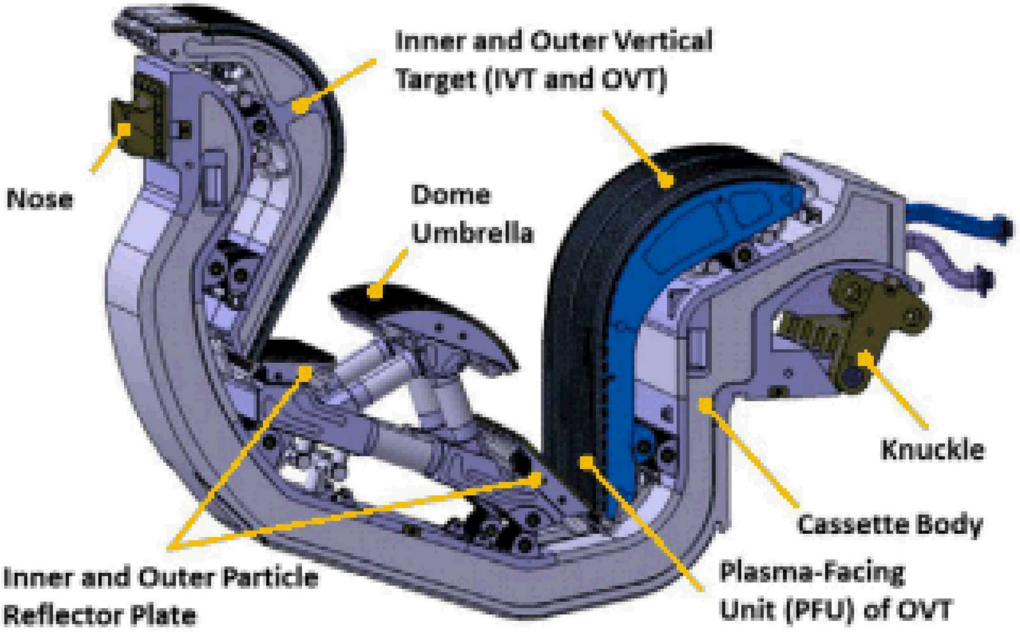
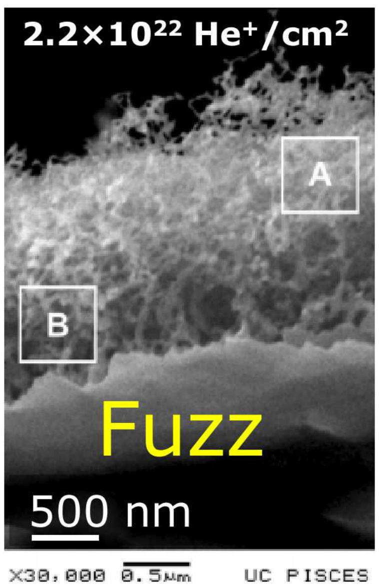


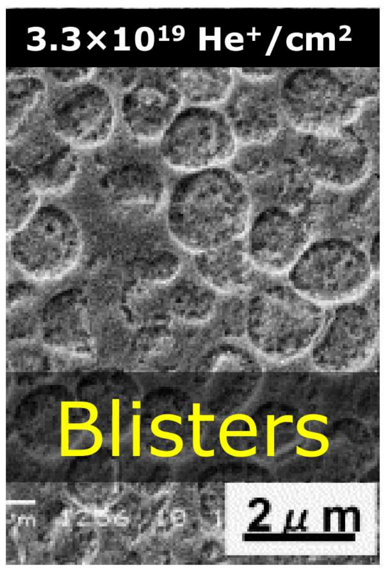
Image from: Hirai, et al., *Physica Scripta*, 2014

Previously-Observed He-Induced Morphologies on W Surfaces Under Various Exposure Conditions

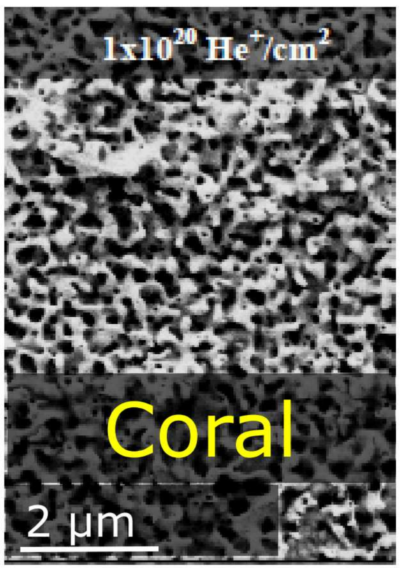
- Driven by diffusion of He and sputtering (at sufficient ion energies)
- D blistering observed at lower temperatures than those studied in this thesis



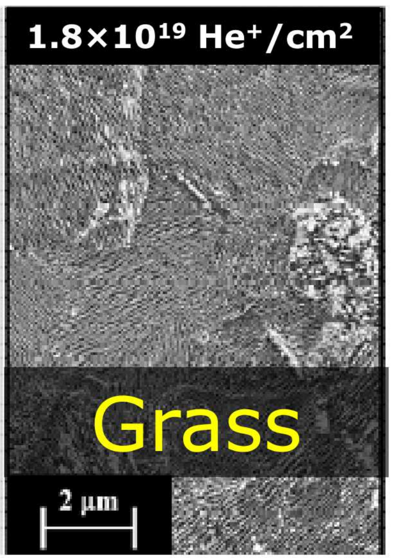
PISCES-B[1]
(60 eV, 850 °C)



PBEF[2]
(19 keV, 800 °C)



HOMER[3]
(30 keV, 1150 °C)



MITE-E[4]
(30 keV, 900 °C)

Images from: 1) Baldwin and Doerner, *Nuclear Fusion*, 2008; 2) Tokunaga, et al., *Journal of Nuclear Materials*, 2004
3) Radel and Kulcinski, *Journal of Nuclear Materials*, 2007; 4) SJ Zenobia et al., *Journal of Nuclear Materials,* 2012

W Grass Evolution Dependent on Crystal Orientation

- Forms for T > 700 °C
- Fiducial markers to image same zone
- In MITE-E:
  - Grains within 4°-8° of {100} least eroded
  - Grains closer to {100} more eroded, needle structure
  - Grains near {110}, {111} more eroded, grass structure

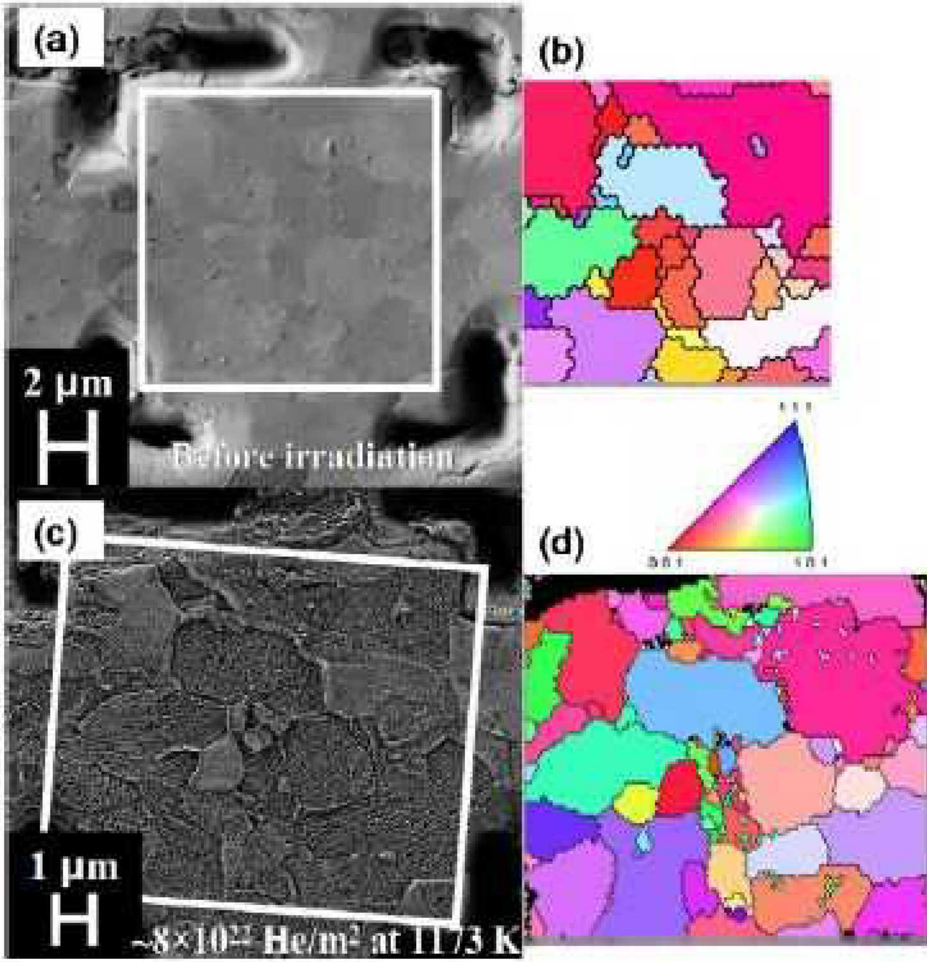
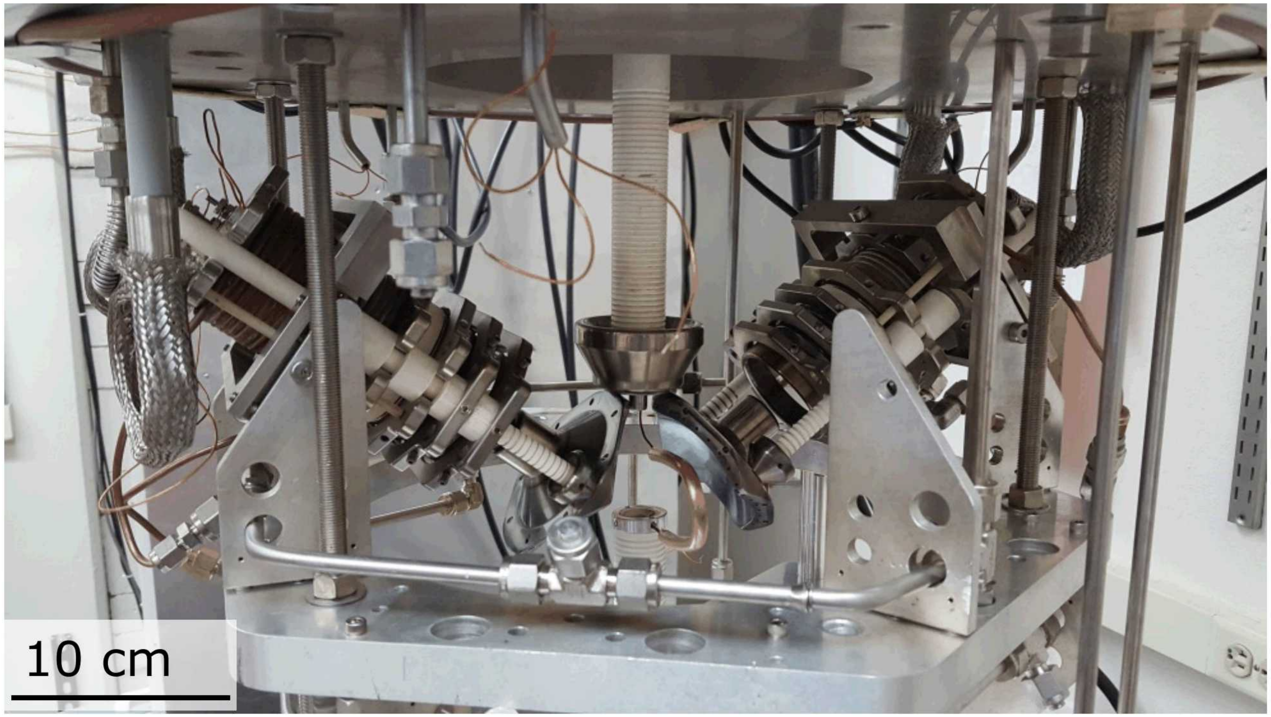
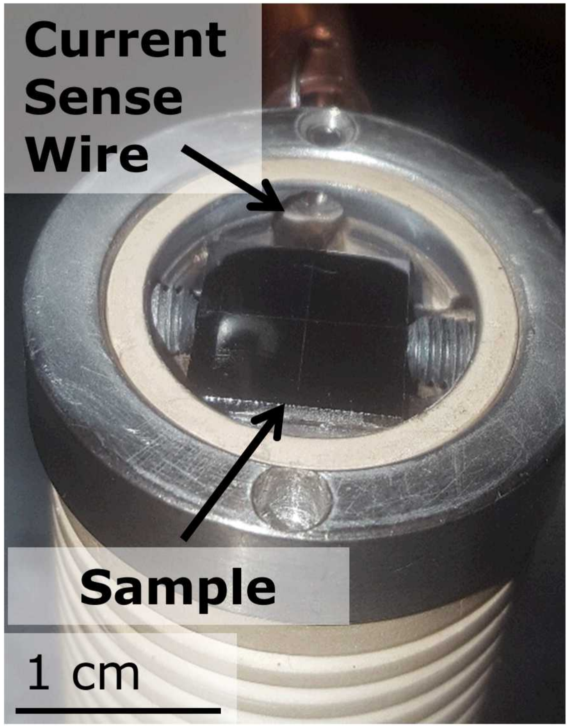


Image from: LM Garrison, and GL Kulcinski, *Physica Scripta,* 2014

The DAISIE Experiment

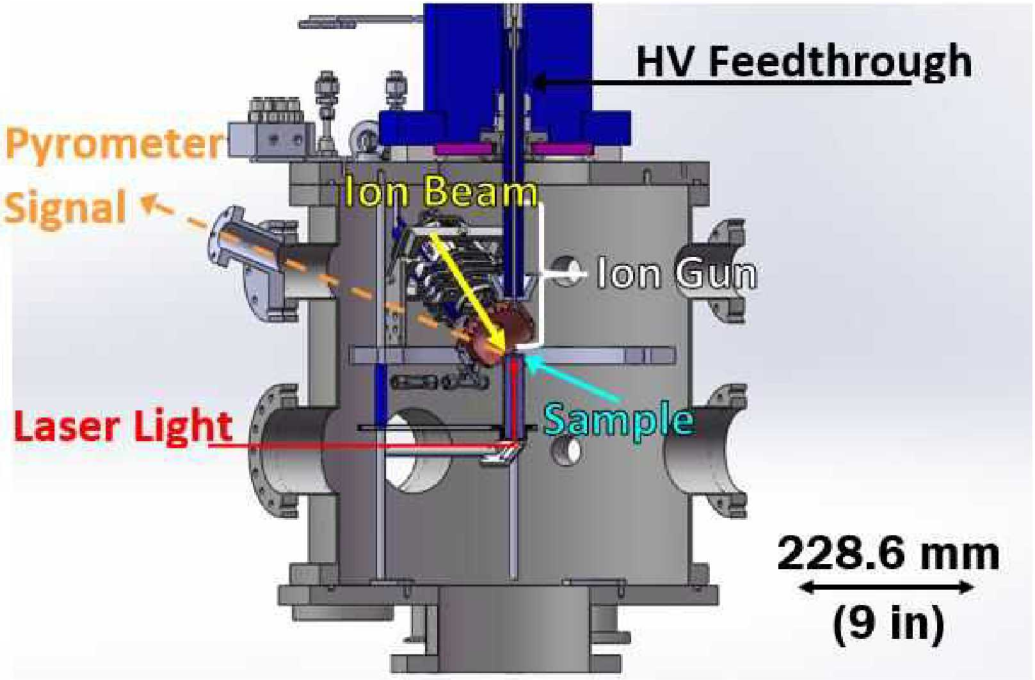
Objective: Develop a dual-ion beam experimental facility capable of simultaneously implanting multiple ion species (specifically He and D) into candidate PFC surfaces

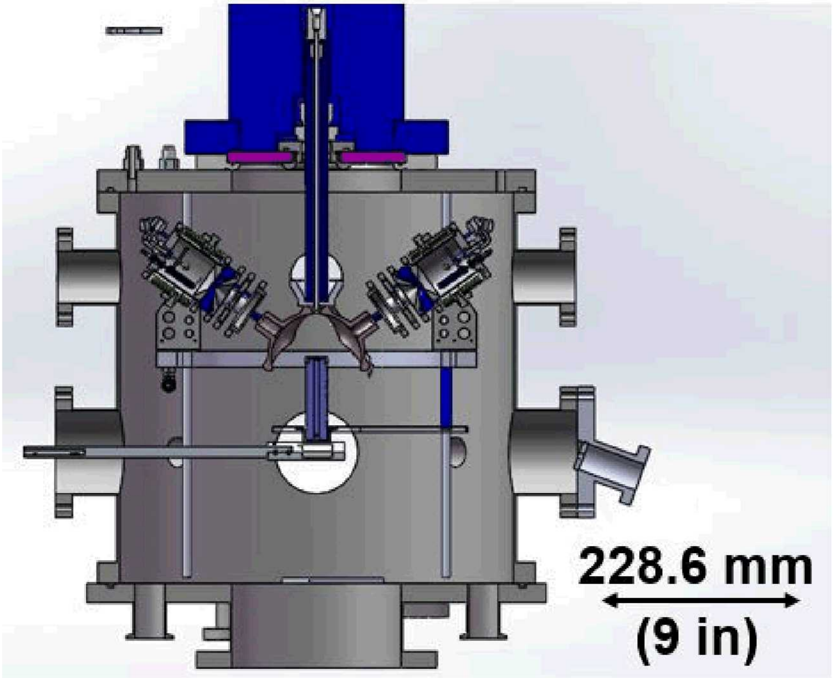




The DAISIE Experiment

Dual-ion beam experimental facility capable of simultaneously implanting multiple ion species (specifically He and D) into candidate PFC surfaces with independent control of each beam





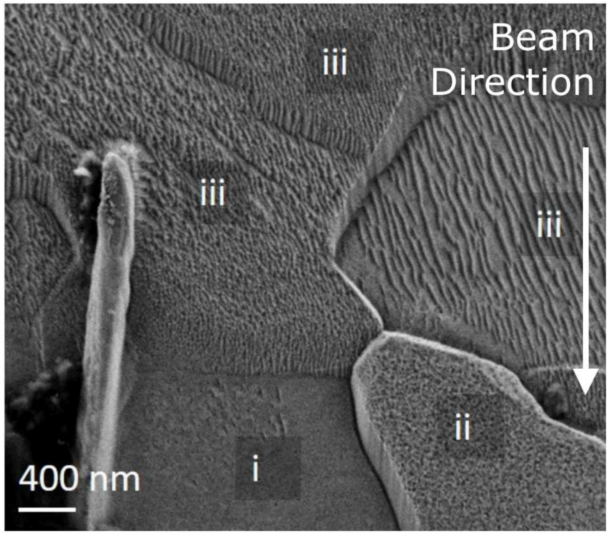
Both laser and beam heating options available to reach target temperature of 900 °C for this thesis, depending on ion flux

Implantation Parameters

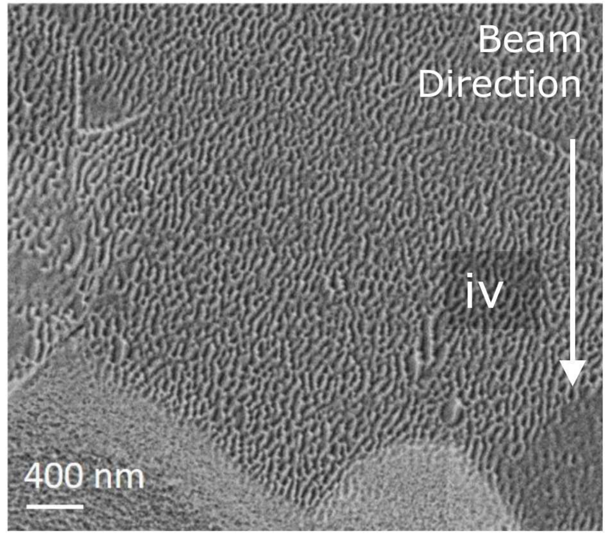
Sample ID	Ion Energy [keV]	Temperature [°C]	He Flux [He $cm^{-2}s^{-1}$]	D Flux [D $cm^{-2}s^{-1}$]	He Fluence [He cm^{-2}]	D Fluence [D cm^{-2}]
DW06	30	900	1×10^{15}	–	3×10^{18}	–
DW05	30	900	1×10^{15}	–	6×10^{18}	–
DW07	30	900	1×10^{15}	–	6×10^{18} (2 beam)	–
DW09	30	900	9×10^{14}	8×10^{15}	3×10^{18}	2.7×10^{19}
DW08	30	1100	1×10^{15}	9×10^{15}	3×10^{18}	2.7×10^{19}
DW03	30	1100	1×10^{15}	9×10^{15}	6×10^{18}	5.4×10^{19}

- Individual fluences (fluxes) selected to reflect fluence (flux) ratio of 90 at% D, 10 at% He
- Dual-beam, He-only implantations split fluence (flux) equally between each beam
- Beam Compositions:
  - He composition: 100% He^{+}, 0% He^{++}
  - D_i^{+} composition: 10% D^{+}, 32% D_2^{+}, 58% D_3^{+}
- No significant morphological differences observed between 900 °C and 1100 °C mixed-species samples

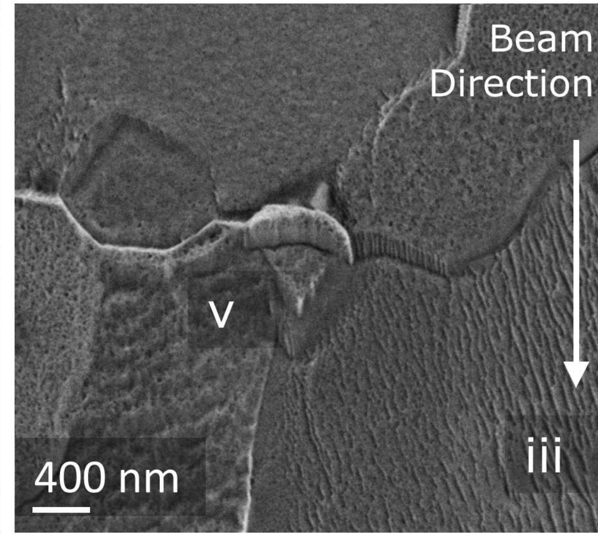
Low-Fluence He Morphologies: Smooth Grains, Pores, Ripples, and Broken Ridges



3.4×10^{17} He/cm^2



1.2×10^{18} He/cm^2

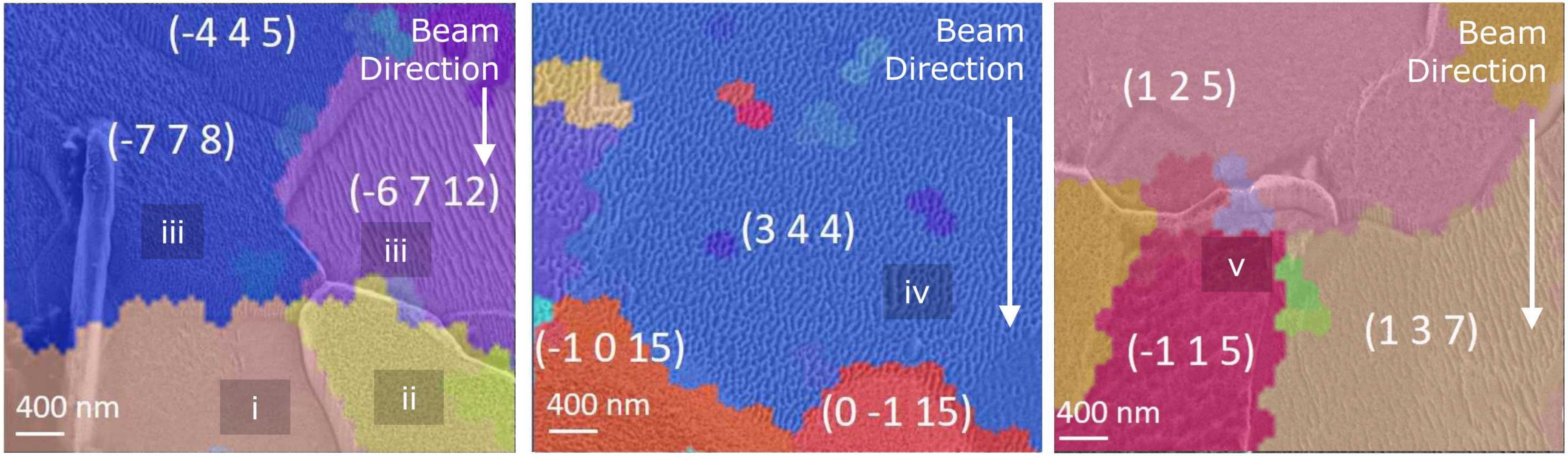


3.4×10^{17} He/cm^2

- **Low He fluence (< 2×10^{18} He/cm^2)**
- Morphologies vary grain-to-grain
- Grains eroded nearly uniformly
- Shape and orientation are functions of sputtering and thermal diffusion mechanisms

Key:
(i) Smooth surface
(ii) Pores
(iii) Parallel ripples
(iv) Broken ridges
(v) Perpendicular ripples

Low-Fluence He Morphologies: Grain Orientation Patterns Indicate Morphology Dependence on Crystallography

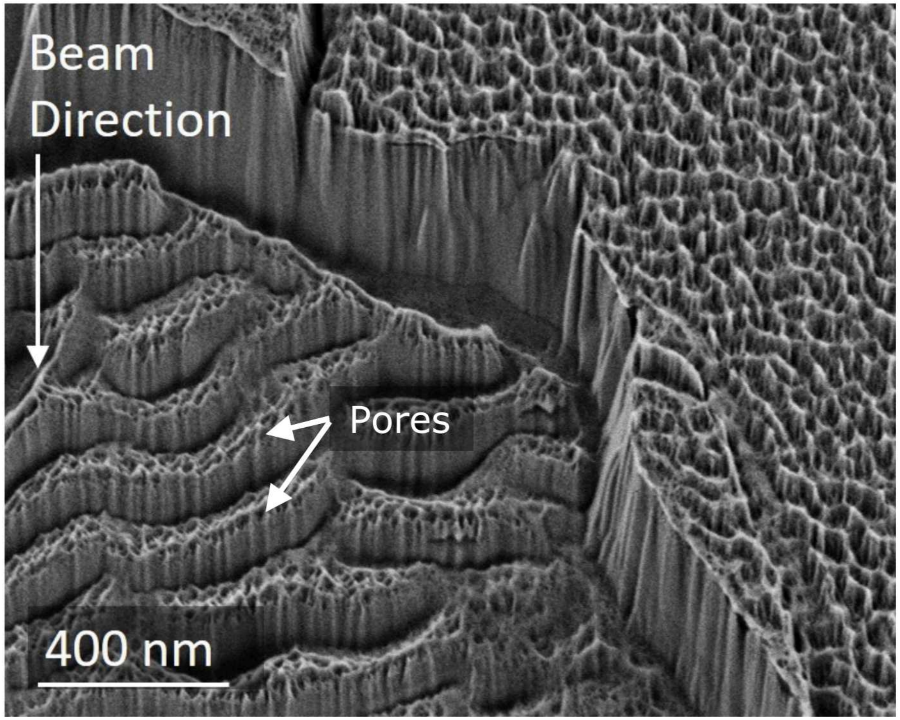


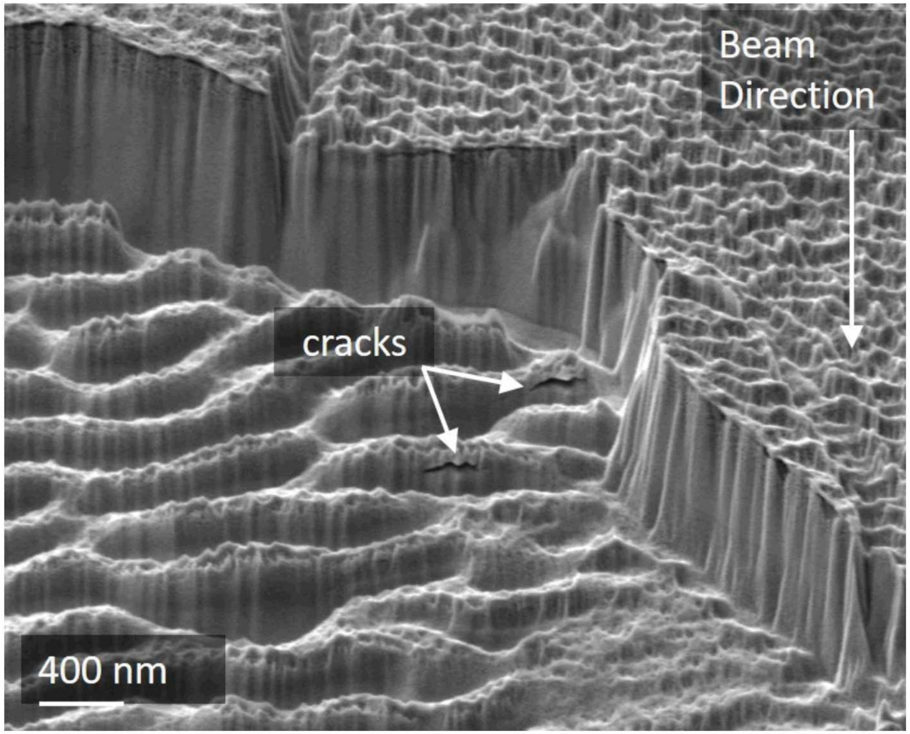
3.4×10^{17} He/cm^2 — 1.2×10^{18} He/cm^2 — 3.4×10^{17} He/cm^2

- **Low He fluence (< 2×10^{18} He/cm^2):**
- Pores, perpendicular ridges favored at orientations closer to {100}
- Parallel ripples/ grass favored orientations closer to {111}, as under 0° incidence
- No grains nearest {101} available

Key:
(i) Smooth surface
(ii) Pores
(iii) Parallel ripples
(iv) Broken ridges
(v) Perpendicular ripples

Pores and Cracks in Ridges Suggest Macroscopic Erosion Mechanisms



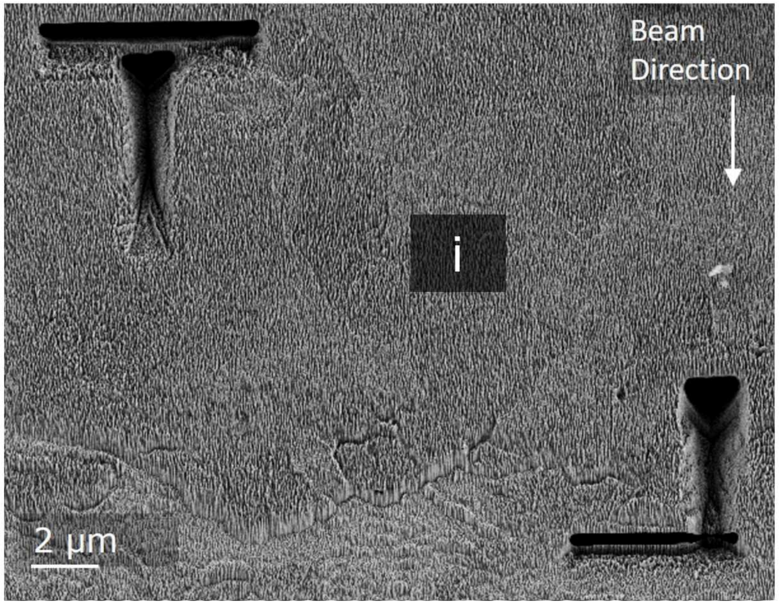


5.7×10^{18} He/cm^2

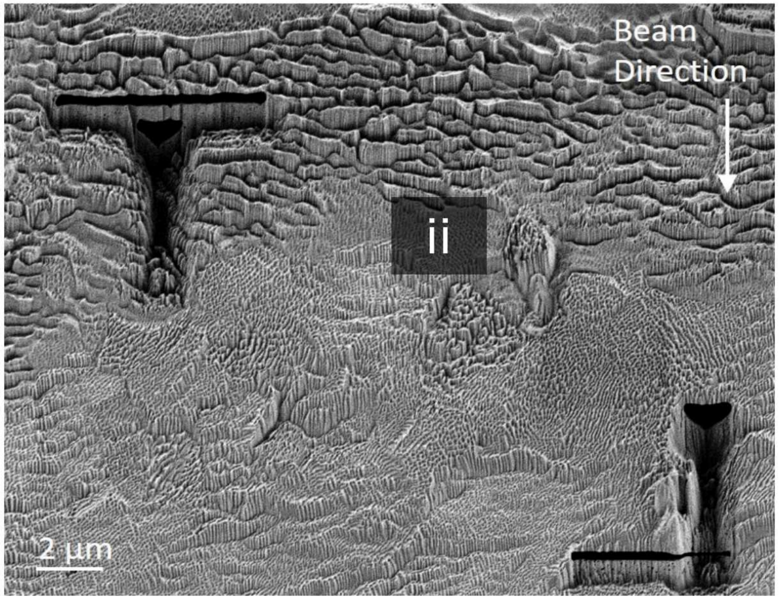
SEM Tilt: 55°

- Difference in grain height ~900 nm
- Bubbles and cracks as deep as 200 nm beneath ridge peak

High-Fluence He Morphologies: Grass, Perpendicular Ridges, Needles, and Webbed Ridges



6.3×10^{18} He/cm^2

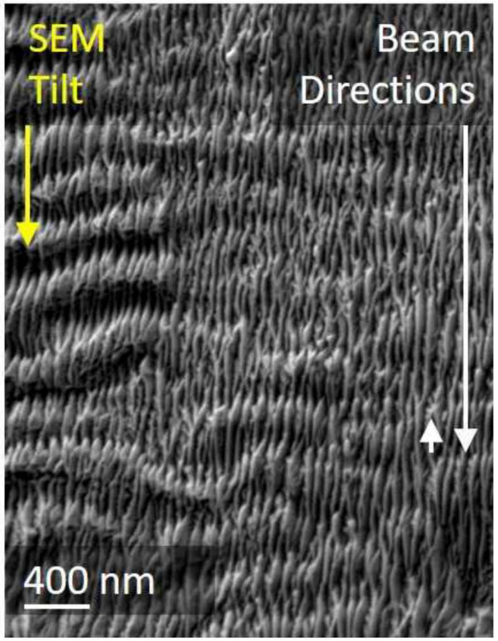


1.4×10^{19} He/cm^2

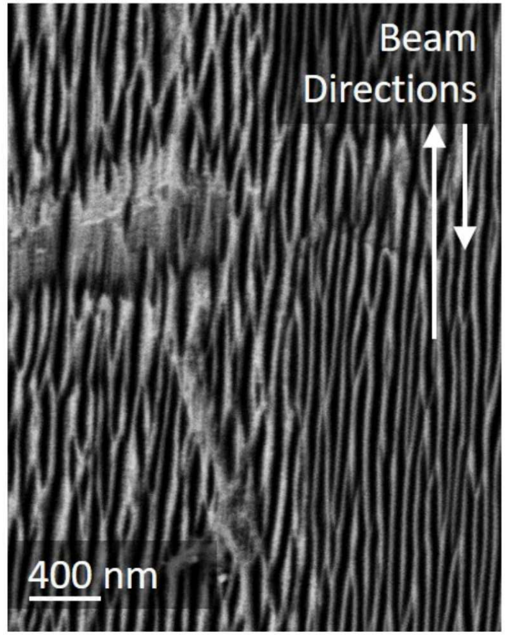
- **High He fluence (6×10^{18} – 2×10^{19} He/cm^2)**
- Common morphologies extend across grain boundaries
- Grass wavelength ~100 nm
- Perpendicular ridge wavelengths ~500 nm
- No meaningful grain orientation patterns obtained from EBSD data at higher fluences

Key:
(i) Perpendicular ridges
(ii) Grass (Parallel)

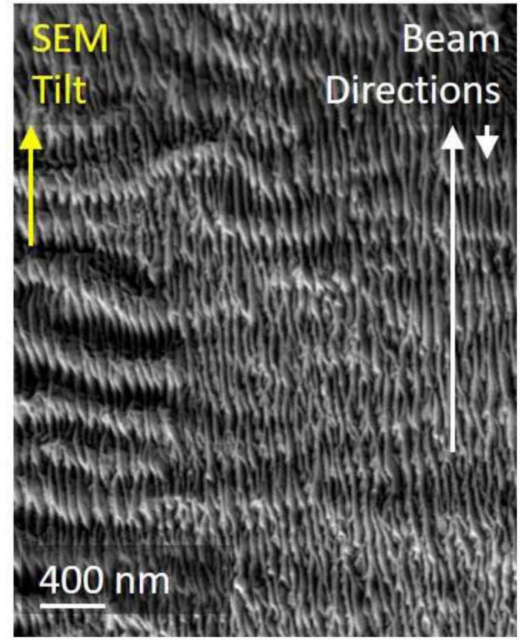
Morphologies on Dual-Beam, He-Only Sample Display Strong Dependence on the Incident Beam Direction at 900 °C



5.6×10^{18} He/cm^2



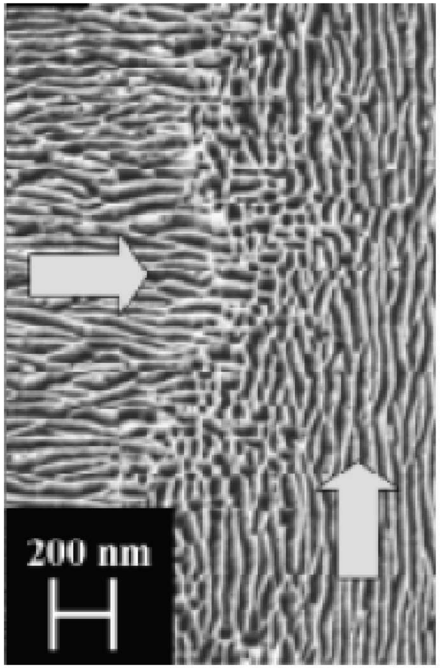
8.1×10^{18} He/cm^2



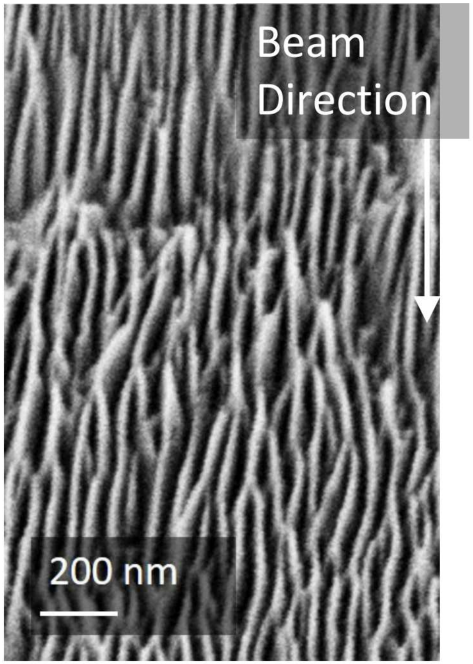
7.5×10^{18} He/cm^2

- Regions dominated by single beam direction develop features *in the direction of the dominant beam*
  - SEM tilted at 55° such that electron beam aligned with dominant ion beam direction
- Grass appears in regions receiving similar fluences from each direction
  - Grass aligned parallel to beam, no topology preference for either direction

“Grass” from He Implantation at Angled Incidence Strongly Resembles Grass at Normal Incidence



1.8×10^{19} He/cm^2
0° ion incidence*

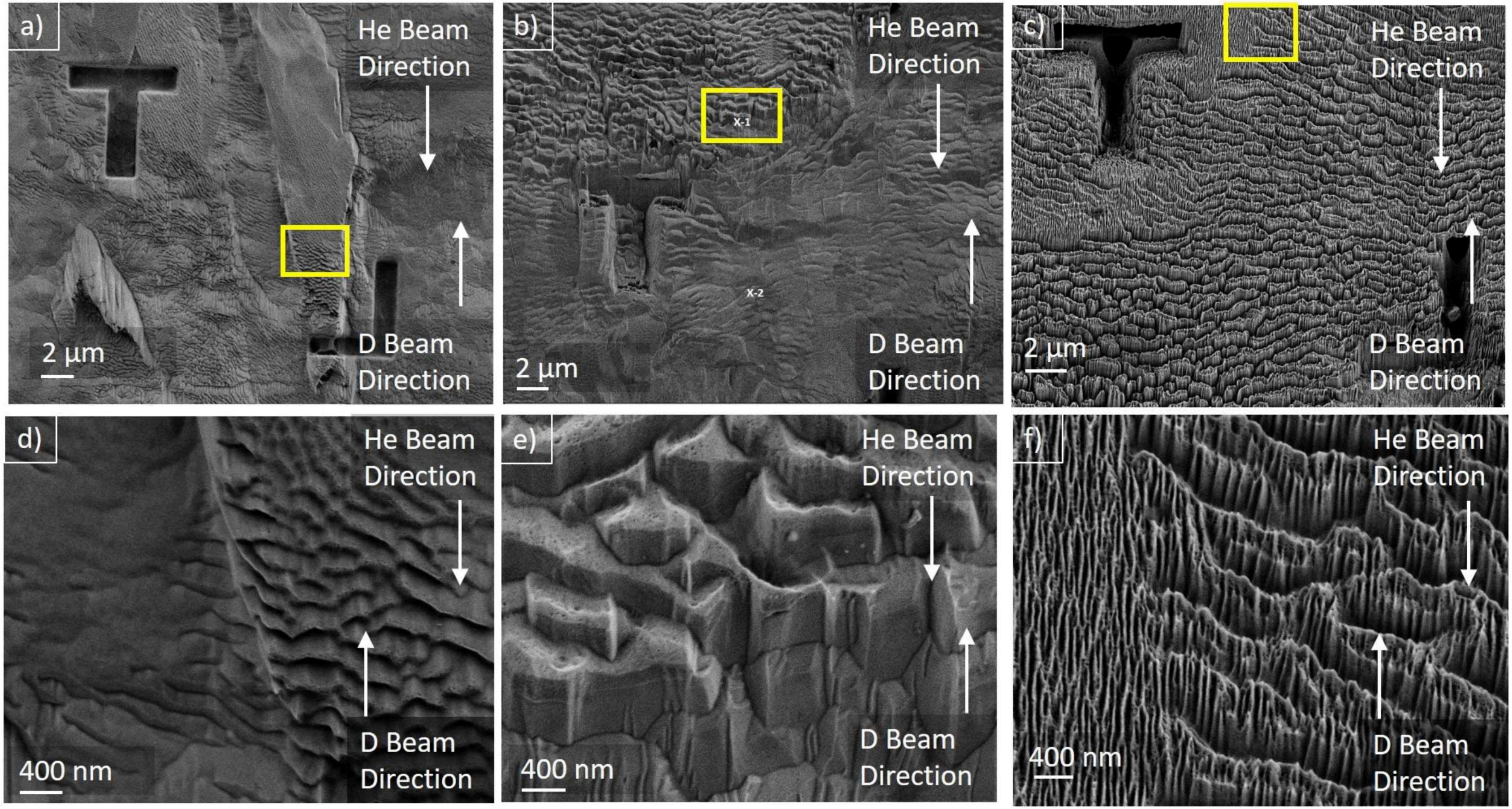


6.3×10^{18} He/cm^2
55° ion incidence

- Normal incidence: orientation of grass varies with grain orientation (diffusion-dominated)
- Angled incidence: orientation of grass aligned with beam across grain boundaries (sputtering-dominated)
- Both sets of grass have similar feature widths, wavelengths

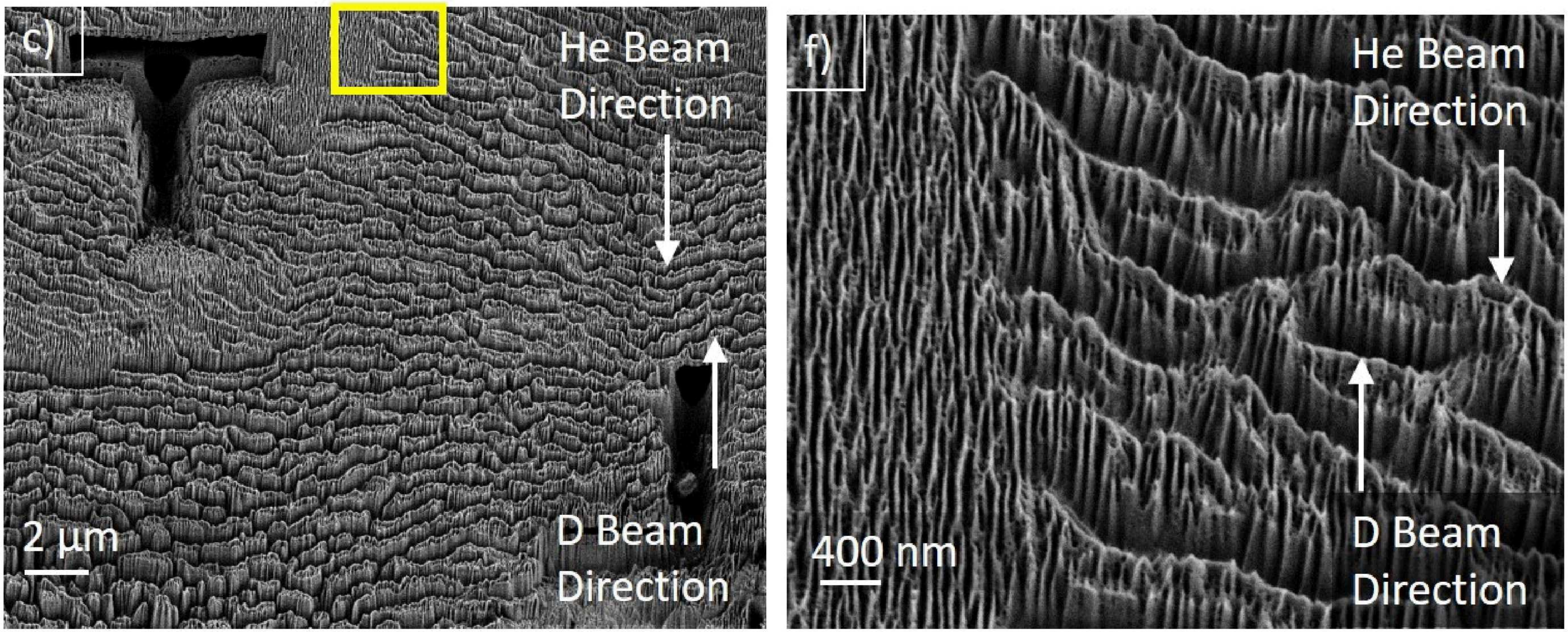
*Image from: SJ Zenobia, LM Garrison, and GL Kulcinski, *Journal of Nuclear Materials,* 2012

Mismatch in Beam Spots Created Range of He-D Compositions on Simultaneous He-D Samples



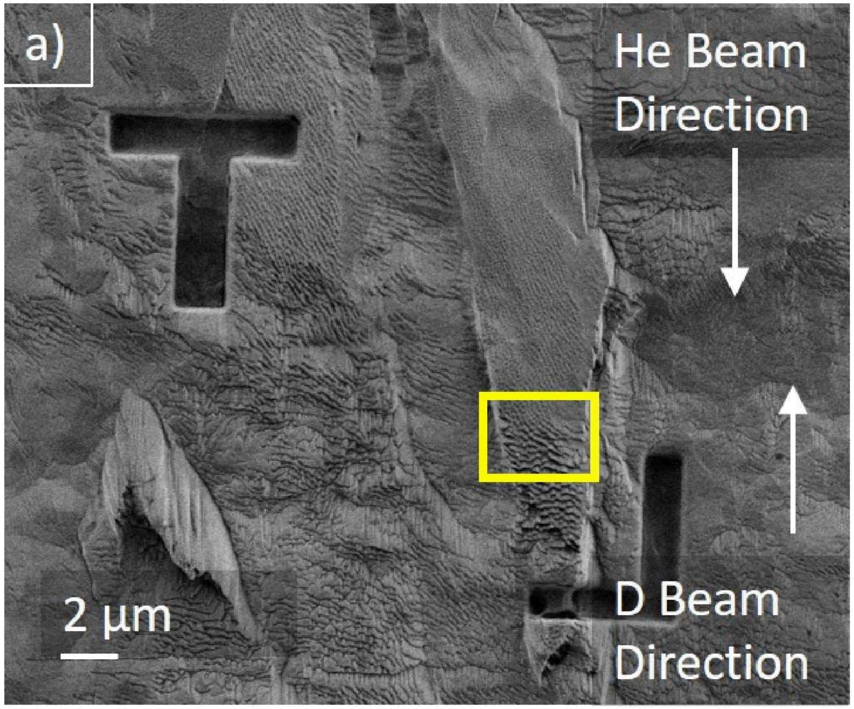
5.2×10^{17} He/cm^2	5.5×10^{18} He/cm^2	7.0×10^{18} He/cm^2
3.2×10^{19} D/cm^2	1.1×10^{19} D/cm^2	4.5×10^{18} D/cm^2
(1.6% He)	(33% He)	(61% He)

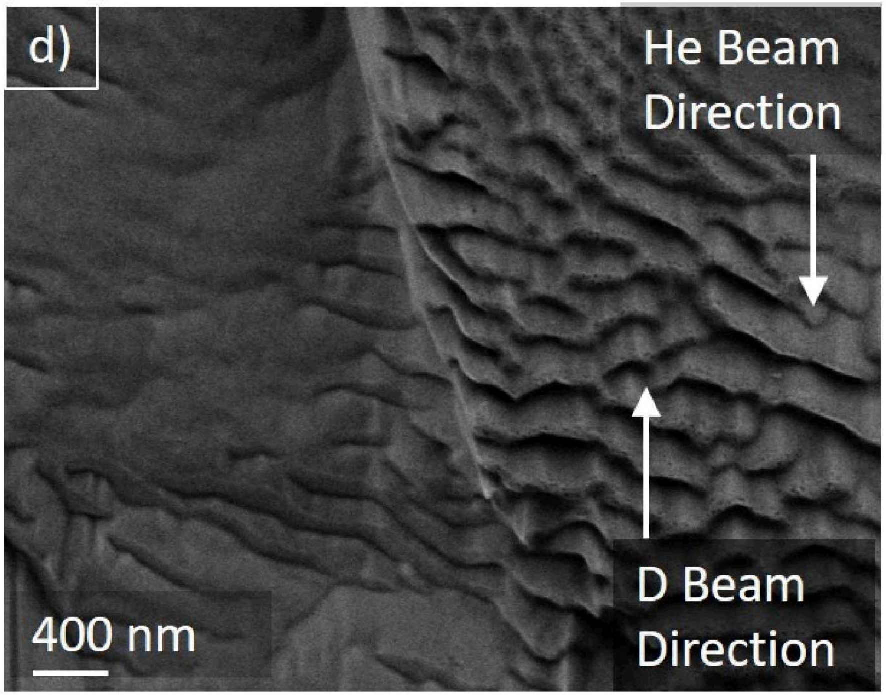
Simultaneous He-D Samples Dominated by He Strongly Resemble He-Only Samples



- Sample fluence: 7.0×10^{18} He/cm^2, 4.5×10^{18} D/cm^2 (61% He)
- Parallel and perpendicular ridge structures develop
- Perpendicular ridge structures develop in the direction of He beam

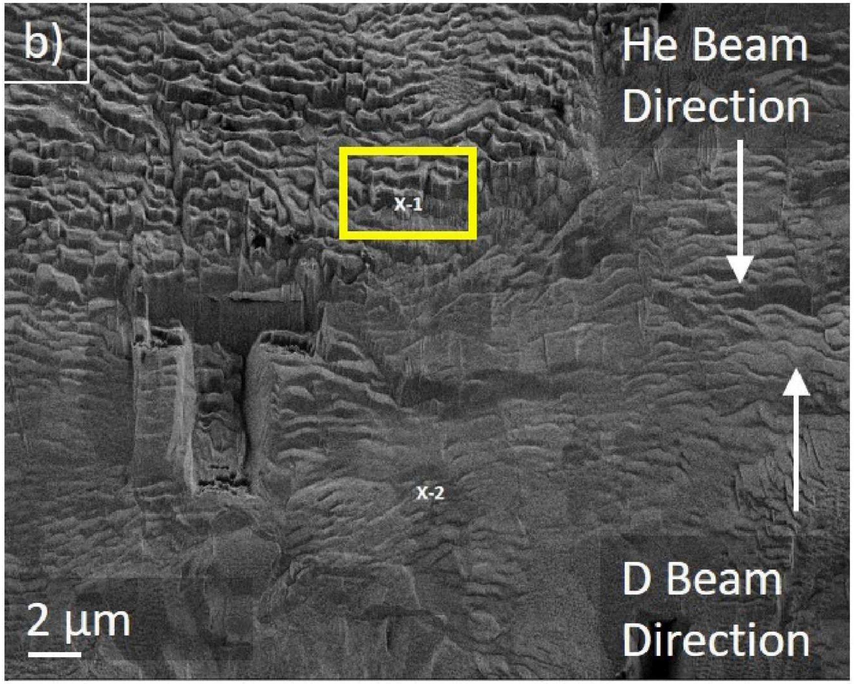
Simultaneous He-D Samples Dominated by D Strongly Exhibit Faceting Not Present on He-Only Samples

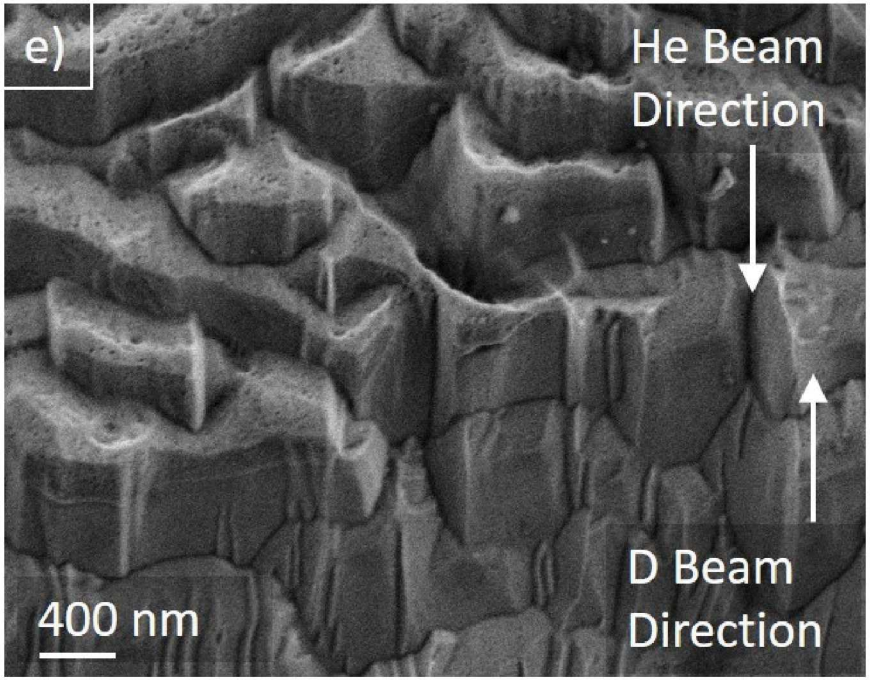




- Sample fluence: 5.2×10^{17} He/cm^2, 3.2×10^{19} D/cm^2 (1.6% He)
- Perpendicular ridge structures develop in the direction of D beam
- Pores visible on ridges
- Facets believed to be intergranular fractures caused by H embrittlement
- He samples *mostly laser heating*; He-D samples *entirely beam heating* to reach 900 °C

New Morphologies Observed Under Simultaneous He-D Implantation Due to Beam Geometry and Embrittlement





- Sample fluence: 5.5×10^{18} He/cm^2, 1.1×10^{19} D/cm^2 (33% He)
- Implantation geometry—ridged structure displays less preference for either beam direction
- Embrittlement fracturing affects development of sputtering-driven morphologies, such as grass
- Similar sizes and wavelengths to single-species implantations—similar underlying mechanisms

Conclusions

- Surface morphologies and orientations under 55° incidence implantation were highly sputtering-dominated, compared to more diffusion-dominated implantation under normal incidence.
- Morphologies exhibit strong directional dependence ion incident beam(s).
- Morphologies from simultaneous He-D implantation driven by similar mechanisms and also highly beam geometry-dependent.
- Addition of high-power D implantation resulted in surface fractures.
- A strong dependence of morphologies on grain orientation was observed at low-moderate He fluences, consistent with previous observations at 30 keV, 900 °C and normal incidence.

Acknowledgments

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Thank You

Additional slides

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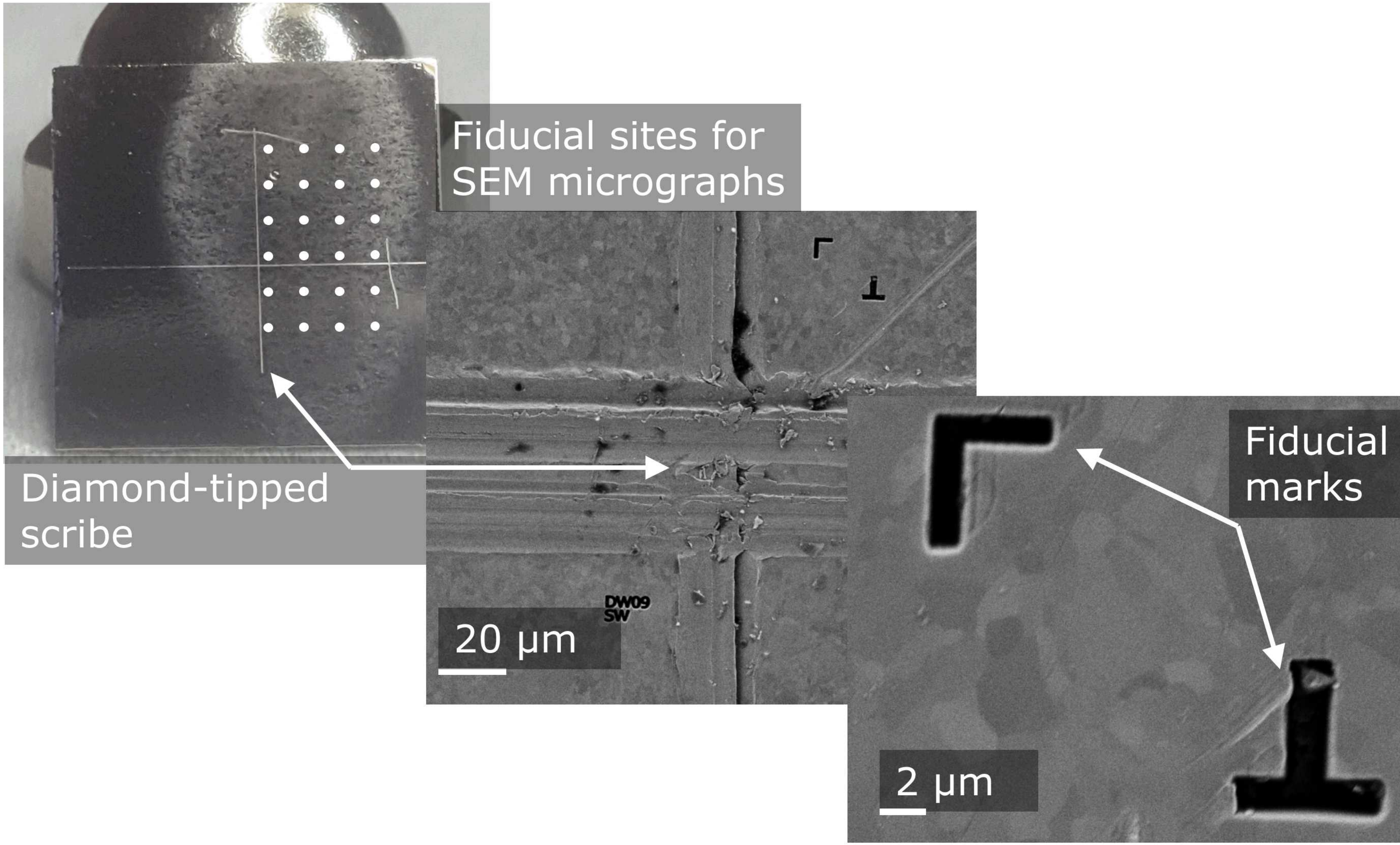
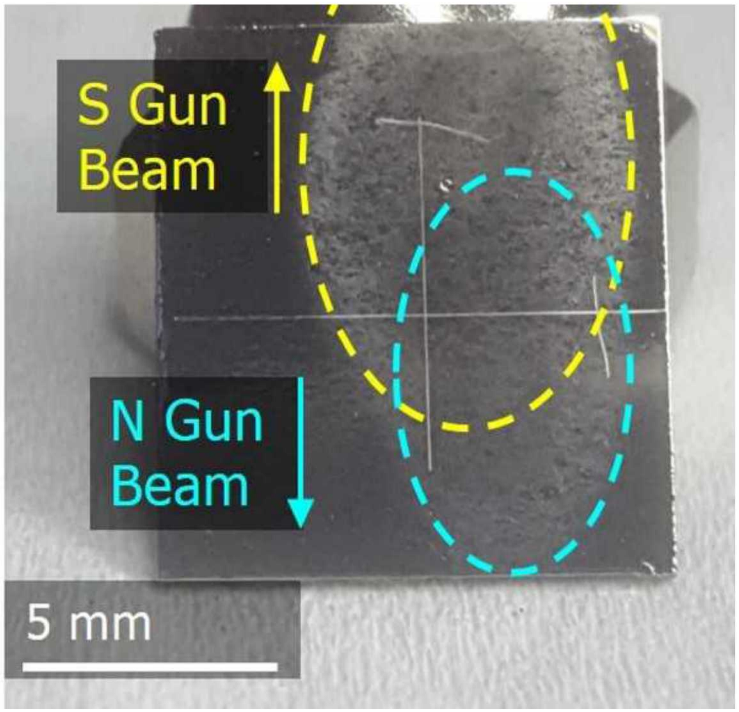
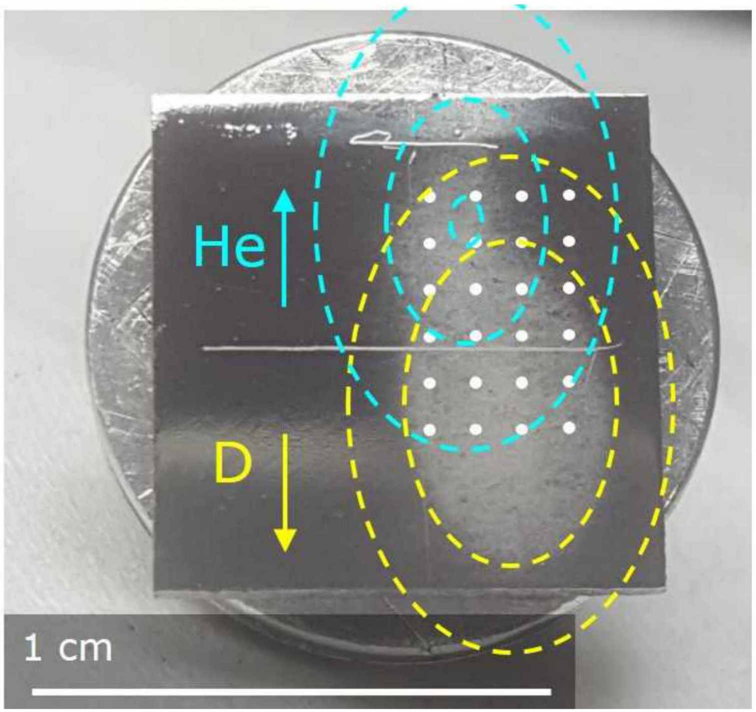
SEM Siting Methodology to Determine Local Fluence



Effect of Beam Overlap on Local Fluence



3×10^{18} He/cm^2 + 3×10^{18} He/cm^2



3×10^{18} He/cm^2 + 2.7×10^{19} D/cm^2

- Result is range of beam fluence ratios

H-He Charge Exchange Cross-Section

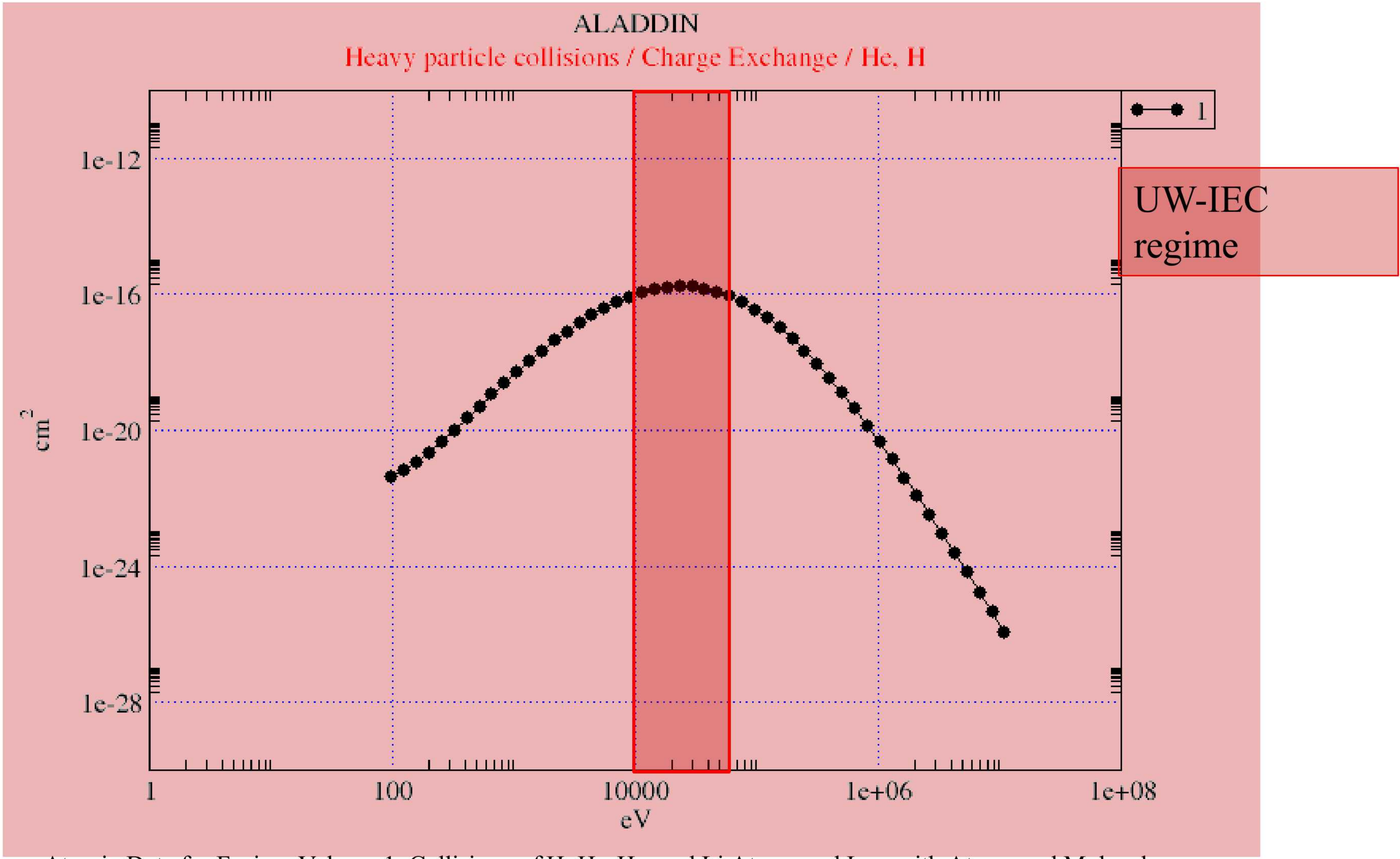
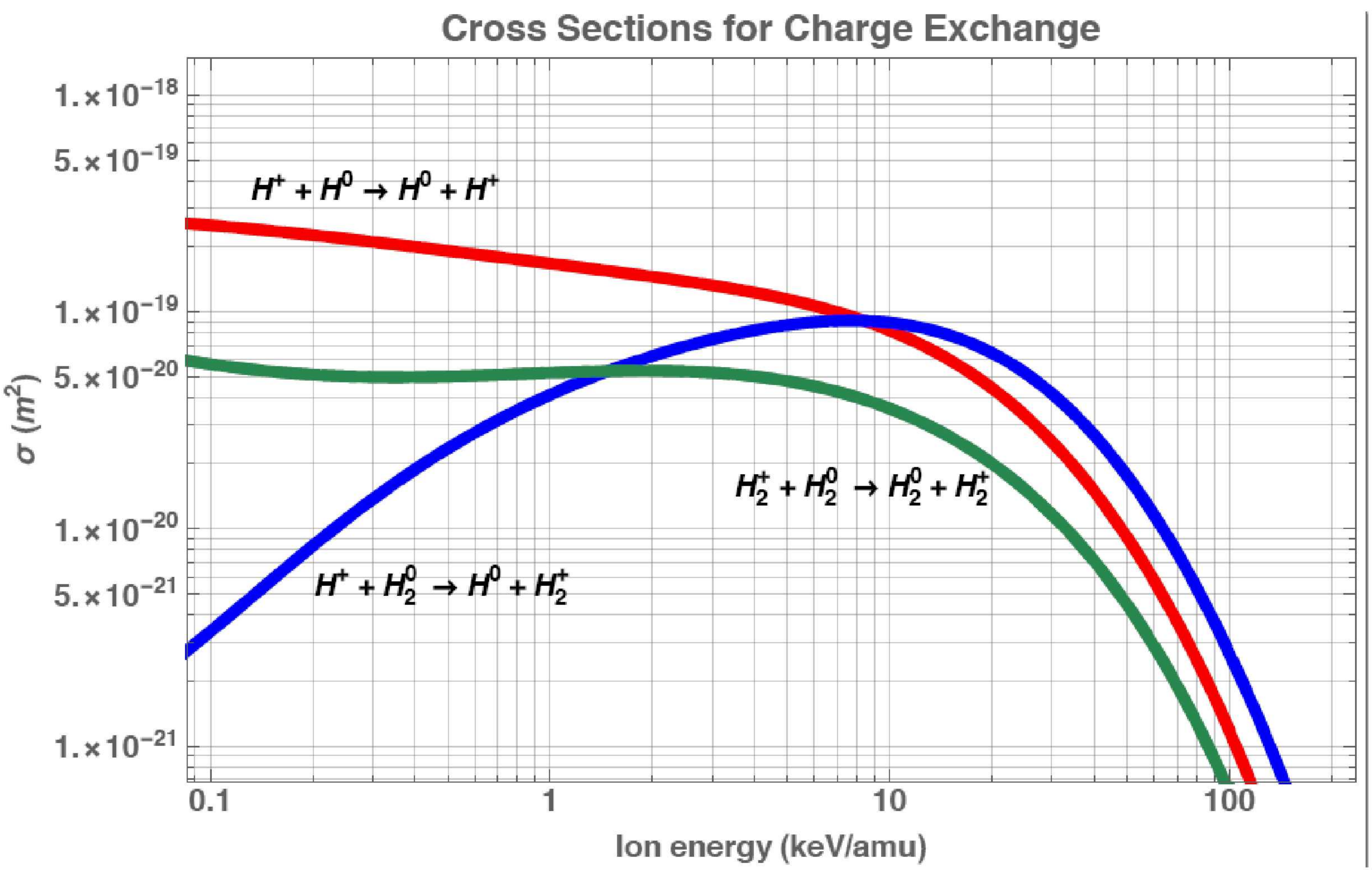


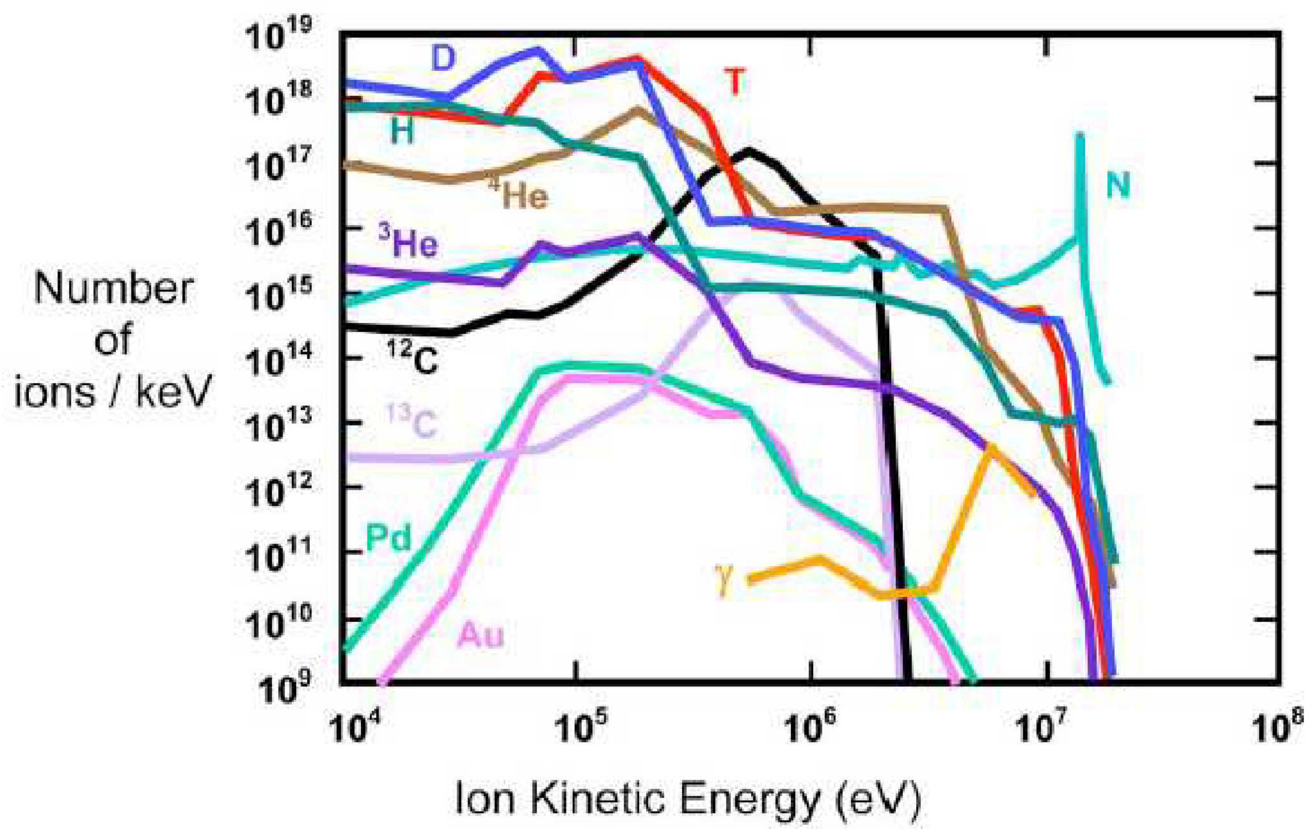
Image from: Atomic Data for Fusion. Volume 1: Collisions of H, H_2, He, and Li Atoms and Ions with Atoms and Molecules (C. F. Barnett ed.), ORNL-6086 (1990)

H-H Charge Exchange Cross-Sections



Data from: International Atomic Energy Agency *Atomic Molecular Data Services ALADDIN Database,* June 26, 2018

Ion Energy Spectrum of IFE Wall Flux includes Significant ^{4}He and D Flux at 30 keV



- Taken from HAPL IFE study

Image from Sethian, et al., *IEEE Transactions on Plasma Science, 2010*

DAISIE: Ion Gun (B. Egle Design)

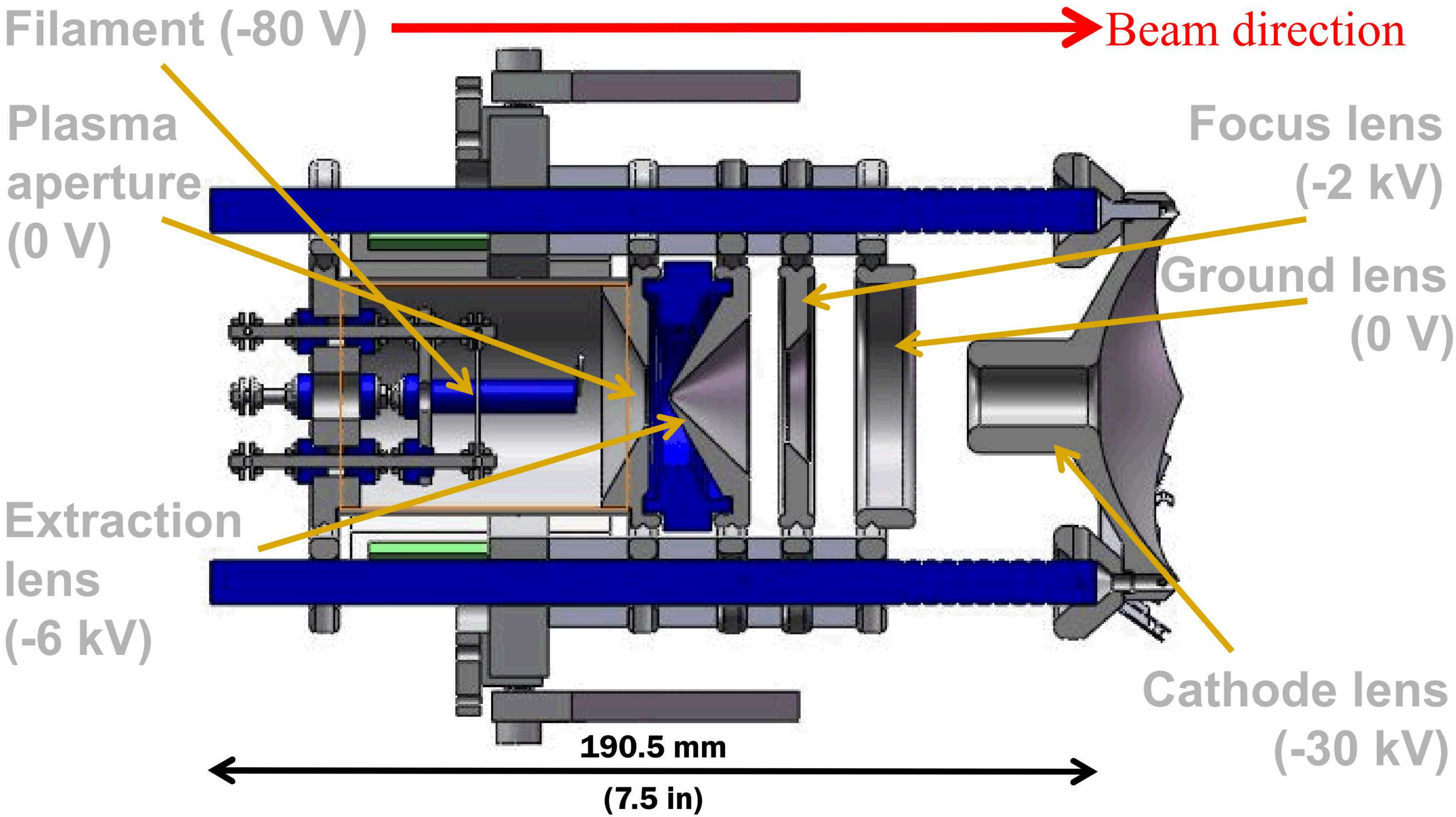
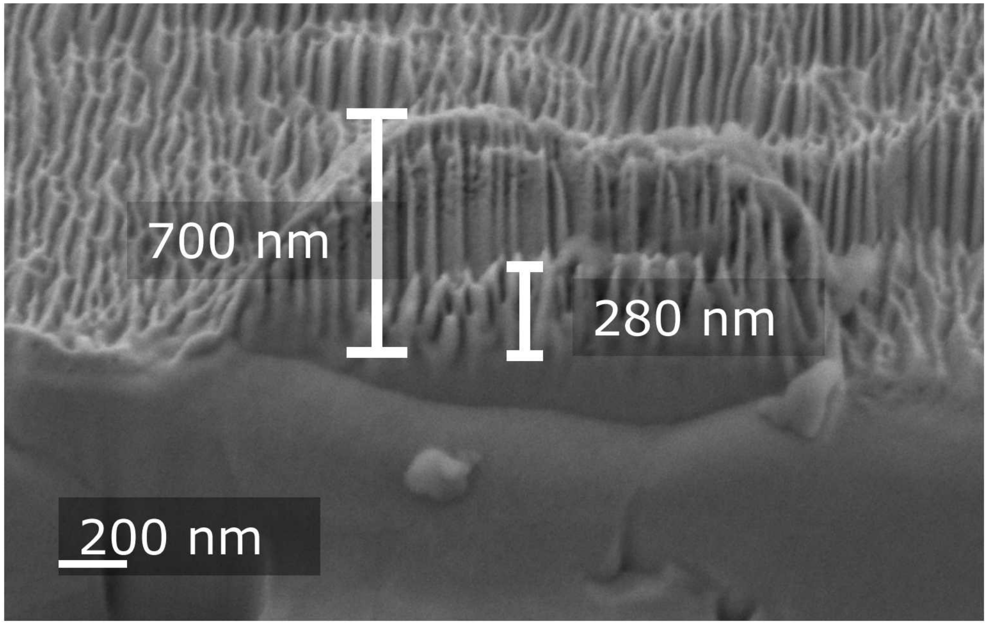
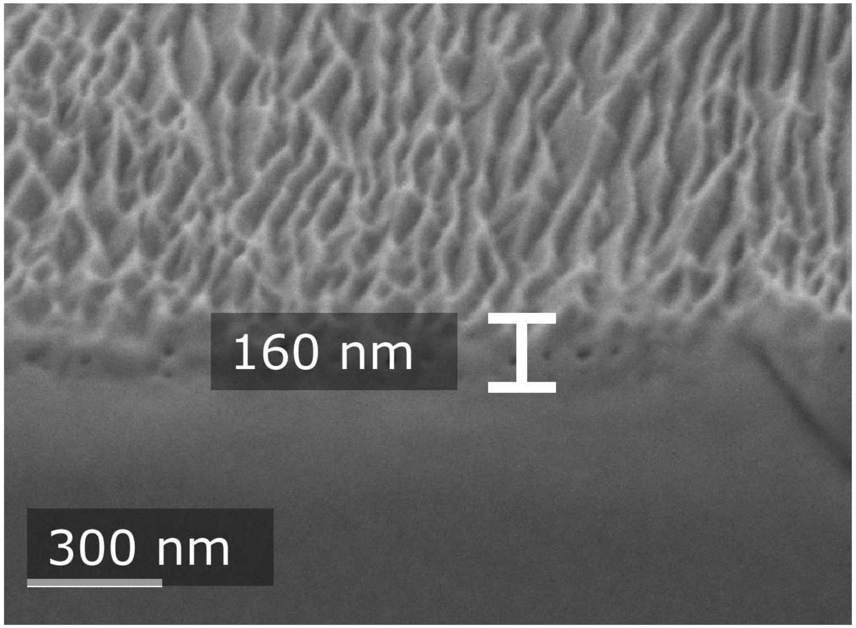


Image from: B. Egle, *Nuclear Fusion of Advanced Fuels Using Converging Focused Ion Beams,* PhD Thesis, 2010

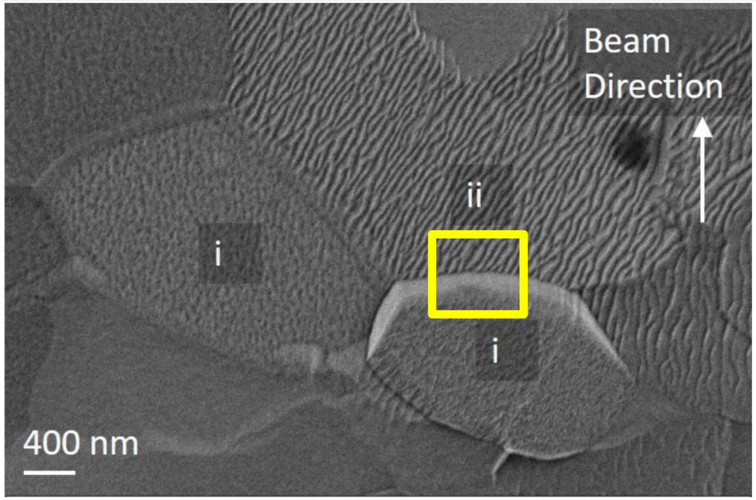
Depth of Microstructure Growth from He^+ Exceeds Calculated Implantation Depth



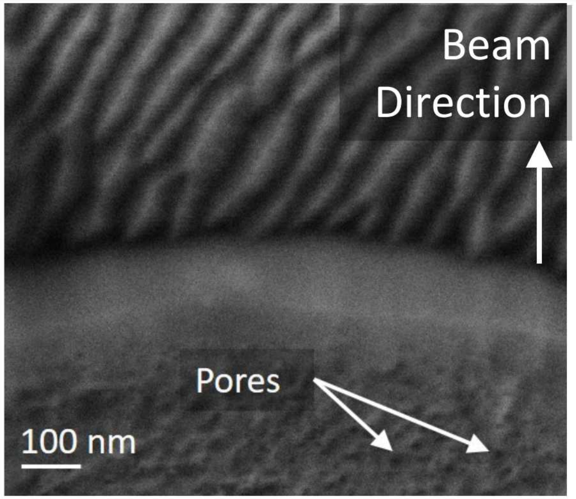


- Local fluence 7×10^{22} He/m^2, 30 keV He ions, 900 °C
- Surface feature heights vary from 160-700 nm, with grain
- Pore sizes on the order of ~30 nm
- Predicted He implantation depth only 64 nm

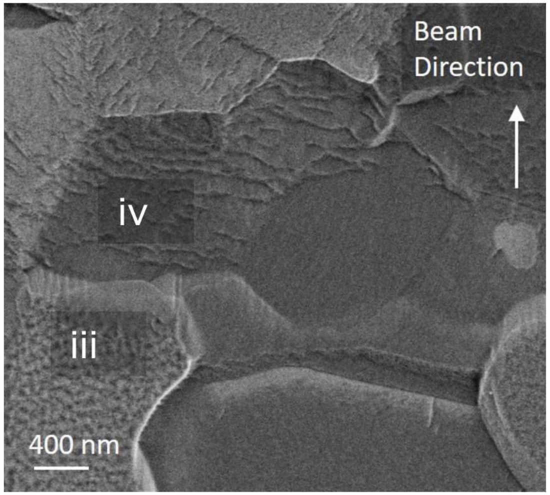
Low D-Fluence Morphologies are Similar to Those That Developed Under He Implantation



3.5×10^{18} D/cm^2



3.5×10^{18} D/cm^2
(High mag)

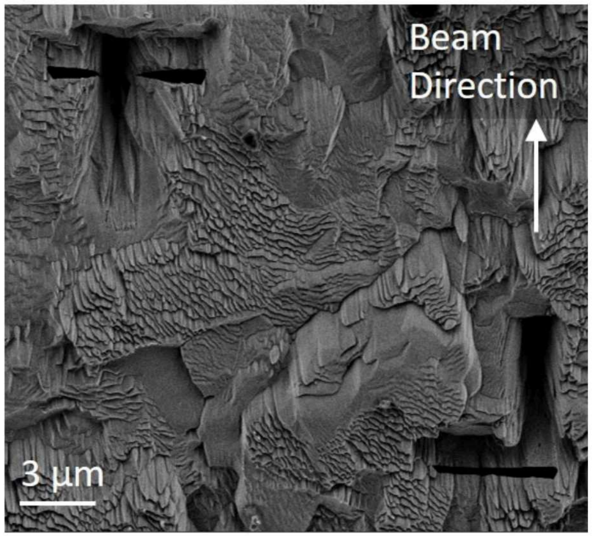


3.3×10^{18} D/cm^2

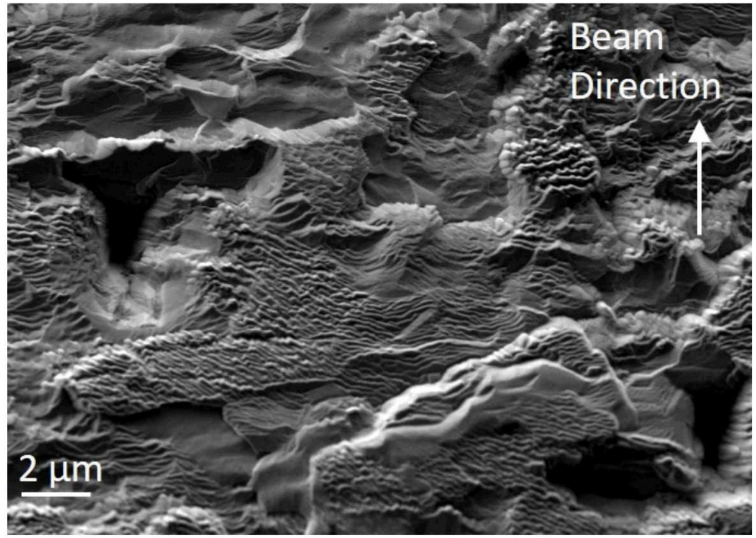
- **Low D fluence (< 1×10^{19} D/cm^2)**
- Diagonal ripples more common under D implantation. Morphology may extend beyond single grain boundary
- Sizes and wavelengths of features similar to those observed under low He fluence

Key:
(i) Pores
(ii) Diagonal ripples
(iii) Broken ridges
(iv) Perpendicular ripples

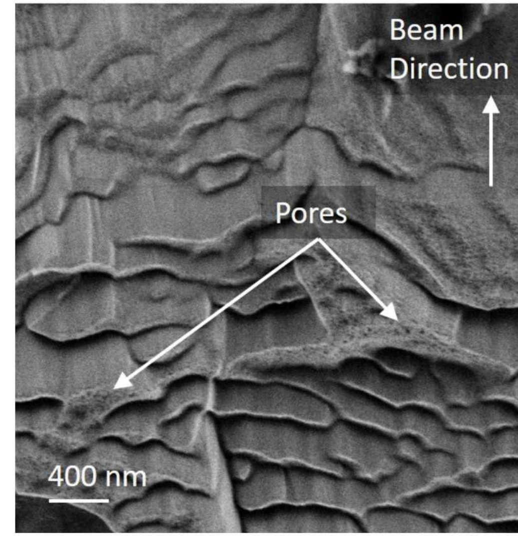
High D Morphologies Exhibit Faceted Surfaces not Present Under He Implantation



2.5×10^{19} D/cm^2



2.5×10^{18} D/cm^2, 55° SEM tilt

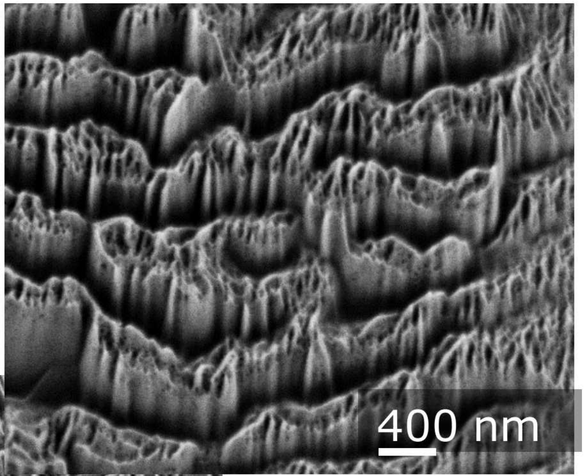


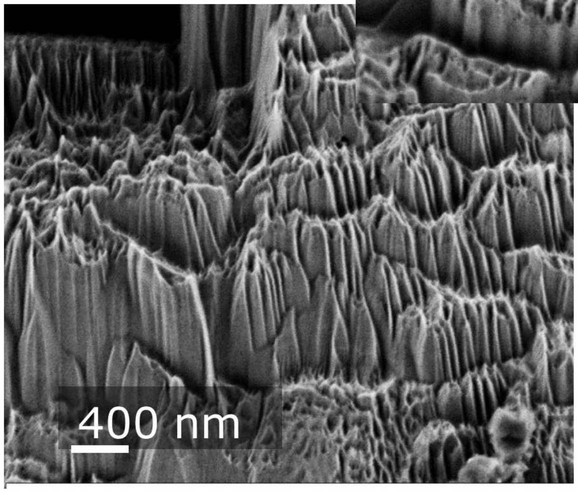
1.7×10^{19} D/cm^2

- **High D fluence (1×10^{19} – 1×10^{20} D/cm^2)**
- Facets believed to be intergranular fractures caused by H embrittlement
- Embrittlement fractures require: (i) D concentrations (ii) surface stresses
- He samples *mostly laser heating*; D samples *entirely beam heating* to reach 900 °C
- Faceting vs ridges not fluence-dependent, possibly crystallography-dependent
- Facets may host ridges, ripples, or pores
- Perpendicular ridges develop in direction of incident beam

Highly Similar Morphologies Between 0.9D-0.1He Samples Implanted at 900 °C vs 1100 ° C

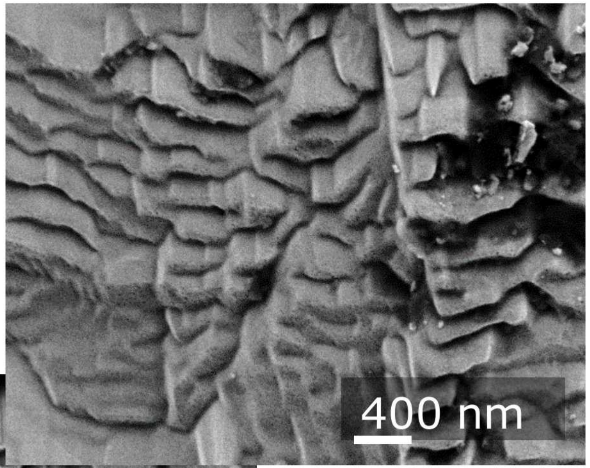
1100 °C
4×10^{22} He/m^2
2×10^{21} D/m^2

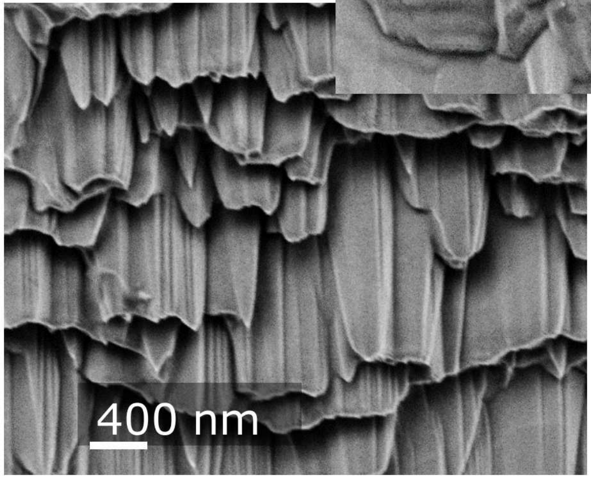




900 °C
4×10^{22} He/m^2
1×10^{22} D/m^2

1100 °C
2×10^{20} He/m^2
3×10^{23} D/m^2

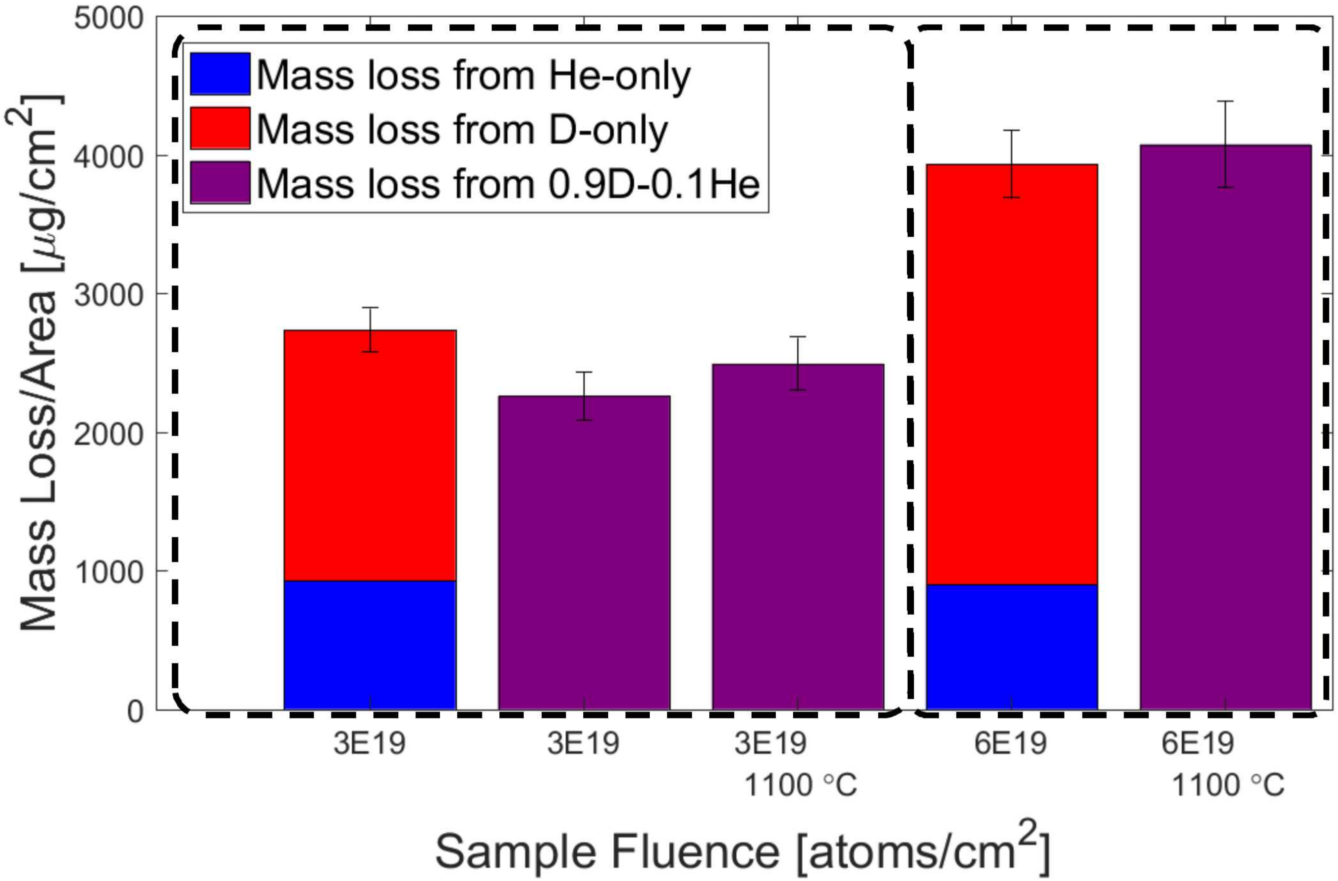




900 °C
2×10^{21} He/m^2
3×10^{23} D/m^2

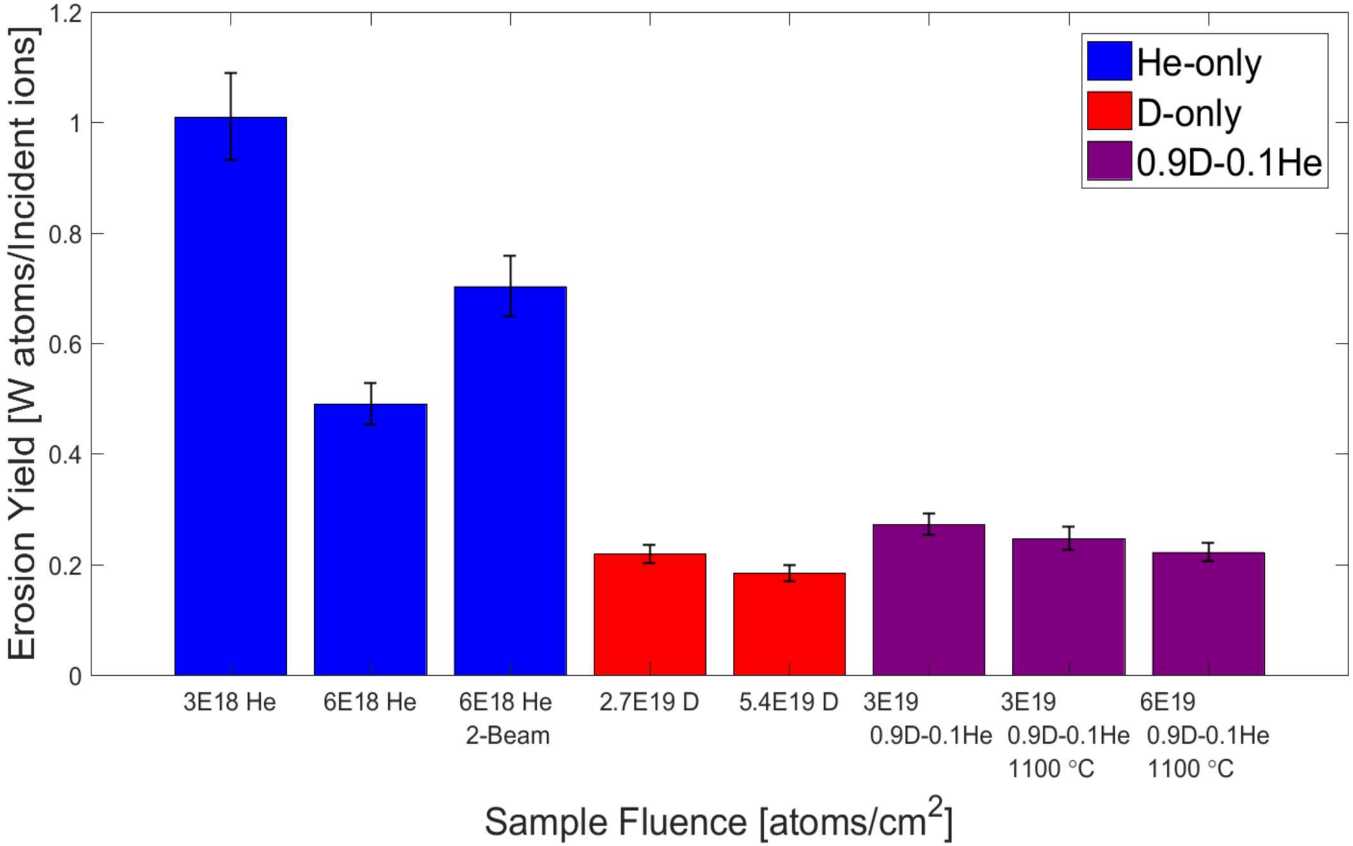
- No significant difference in mass loss observed
- Is this related to these temperatures falling in similar desorption regimes for H (peak from 100-500 °C) and He (peaks below 700 °C, above 1100 °C)?

No Significant Mass Loss Differences Between Mixed-Species and Sum of Single-Species Implantations



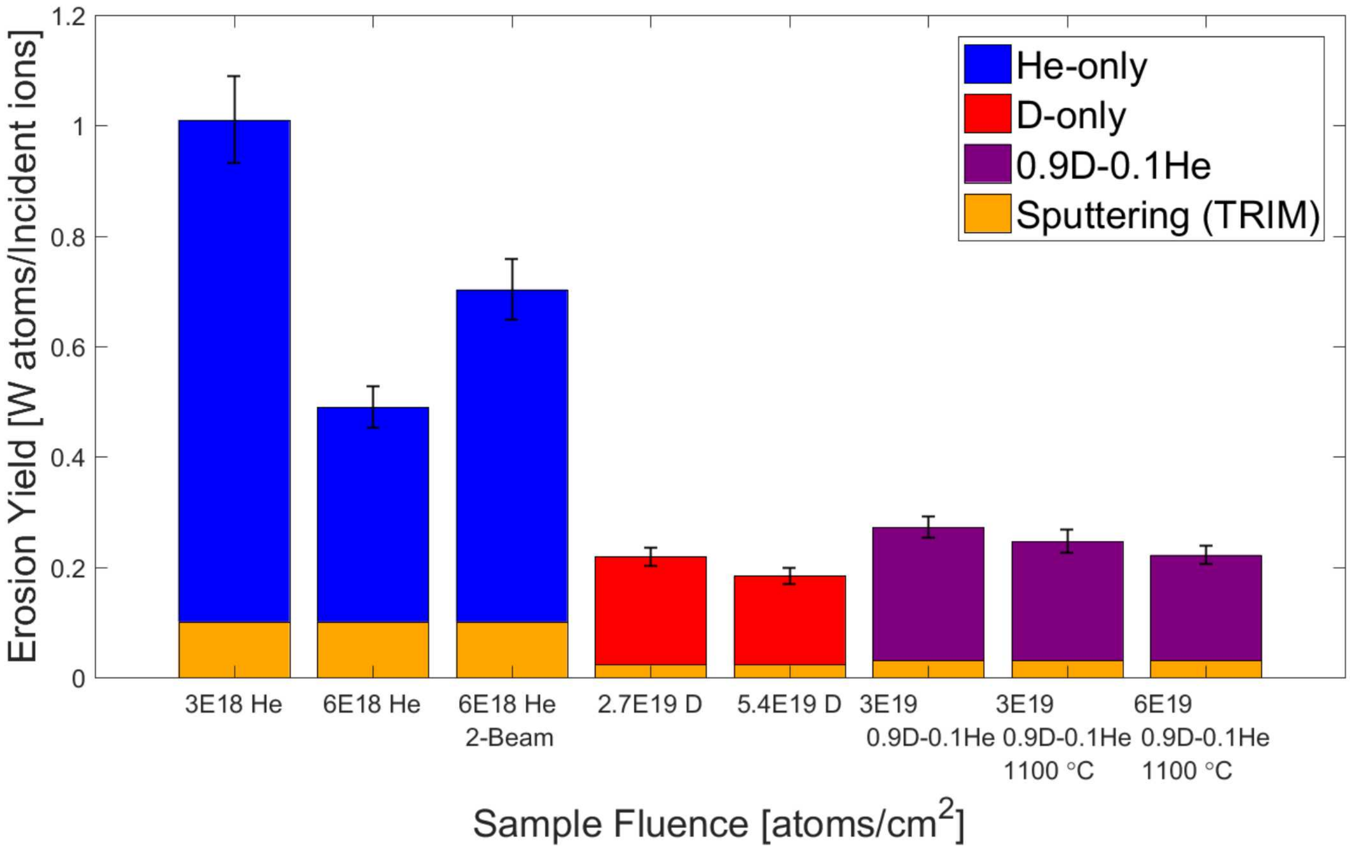
- All ion energies 30 keV, all sample temperatures 900 °C, except where indicated
- 2000 μg/cm^2 corresponds to ~ 0.1 μm W eroded (averaged over beam spot)

Erosion Yield can Decrease as a Function of Fluence



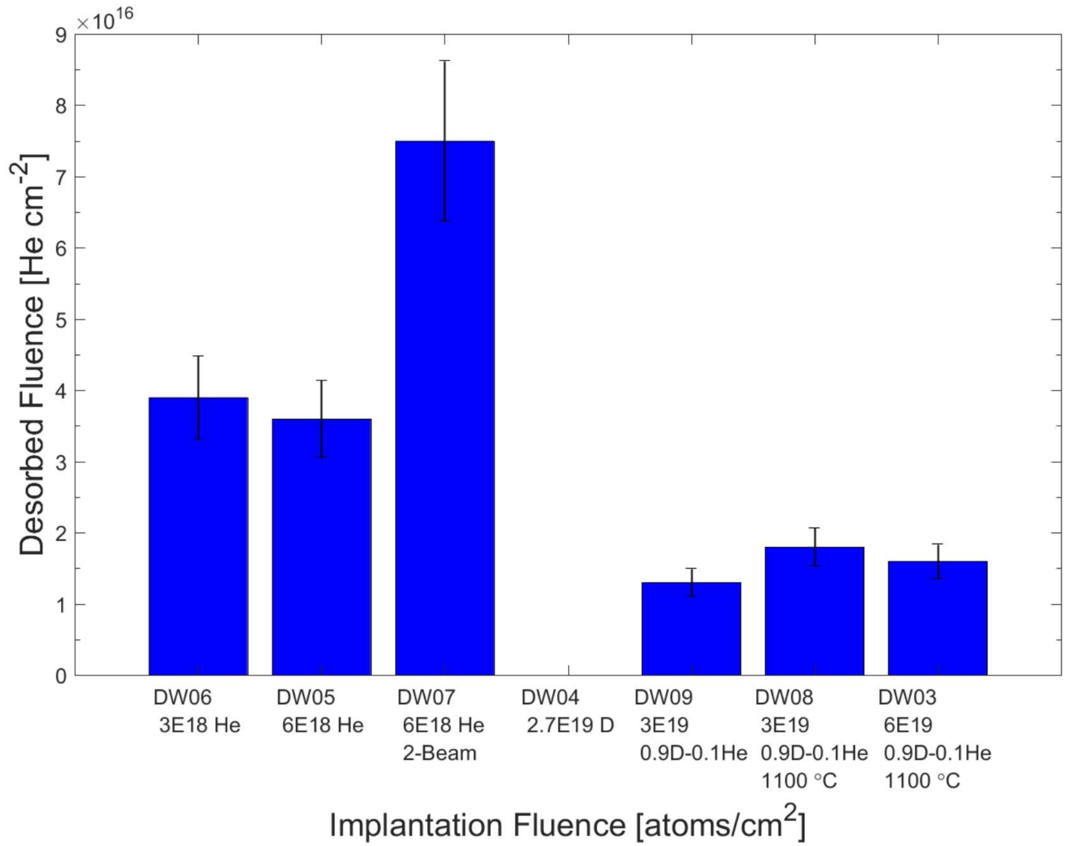
- Yields derived from mass loss, measured in [W atoms lost/incident ion]
- Largest discrepancy in erosion vs fluence for He samples, less obvious trend for D or mixed He-D samples. More samples necessary to determine a trend
- Surface temperatures of 900 °C vs. 1100 °C not a significant factor

Measured Erosion Yields Exceed Sputtering Calculations by Factor of 6-10



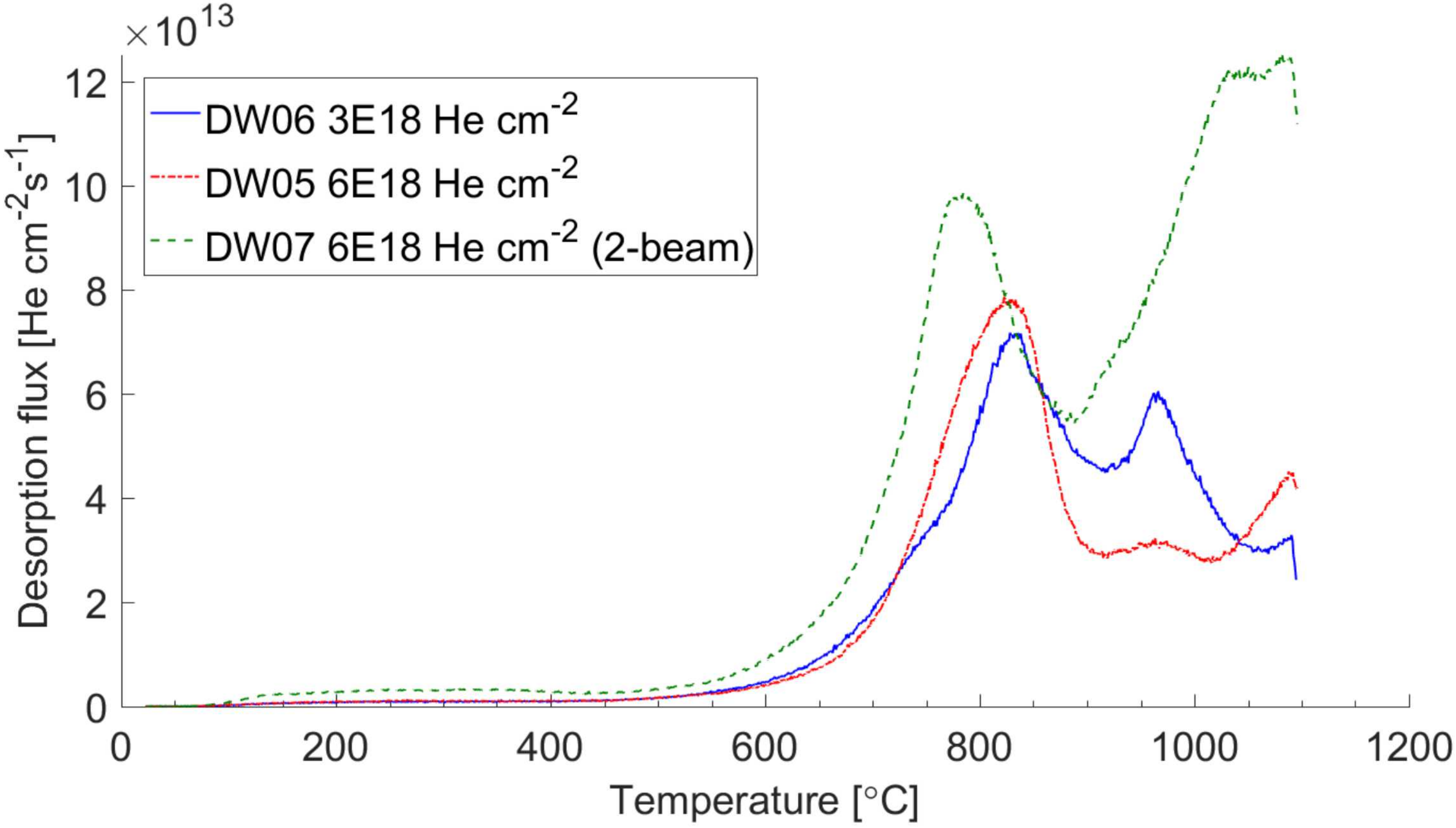
- Microstructure development leads to macroscopic mass loss vs atomic sputtering
- High temperature may be a factor, although no clear trend with temperature suggested by available data

Total He Desorption Reduced by Factors of 2-3 Under Simultaneous He-D Implantation



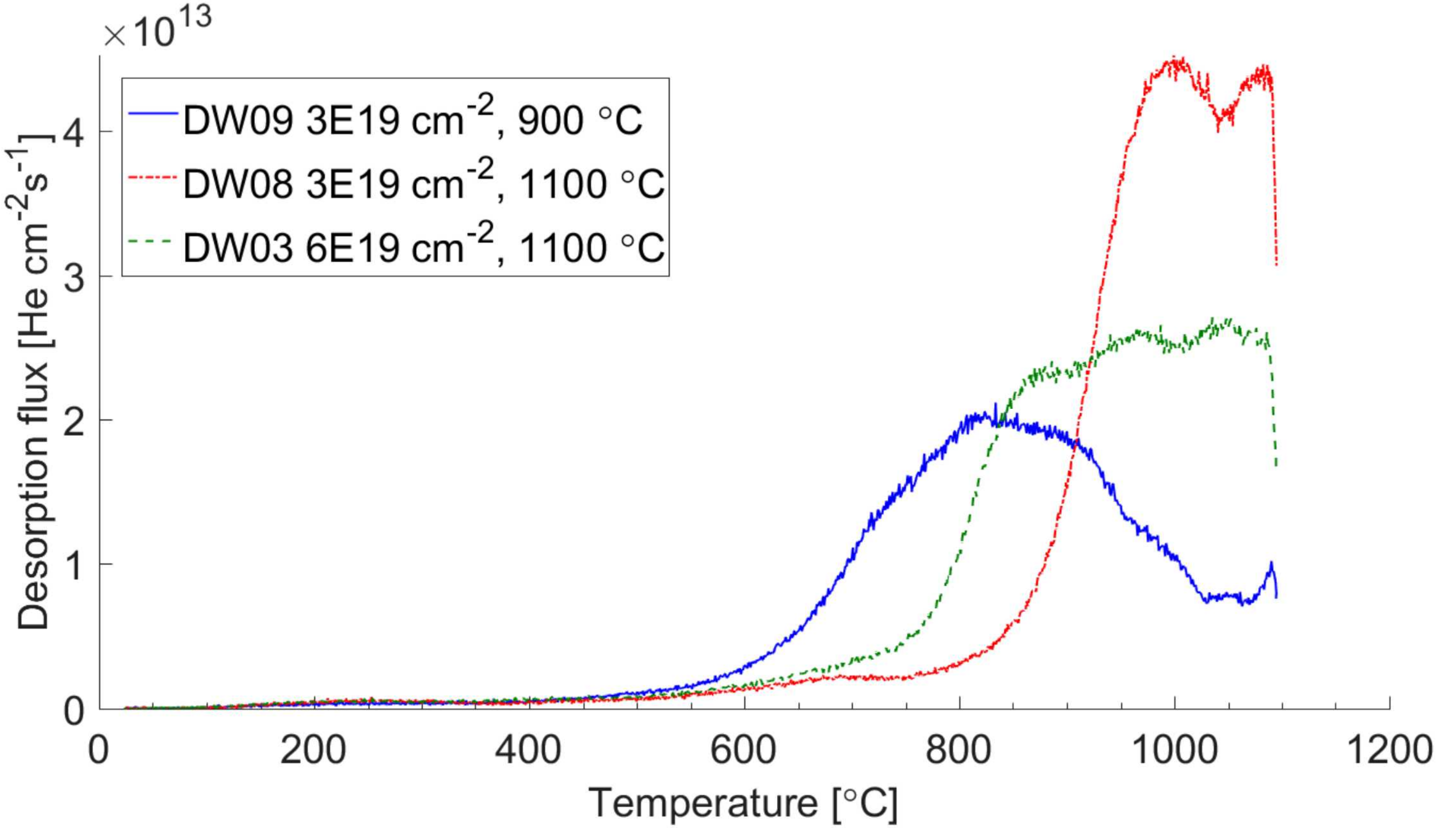
- Time between implantation and desorption not significant past ~100 hours
- Is the difference between single and dual-beam, He-only related to morphology developments?

He Desorption in He-Only Samples



- Shift in desorption peaks with increased fluence
- Altered trapping behaviors introduced by dual-beam, He-only morphologies?

He-D Samples Exhibit Different Desorption Behaviors than He-only Samples



- Some similar desorption peaks to He-only: 800 °C, 1000 °C, 1100 °C
- More complex 800 °C desorption regime
- Effect of implantation temperature uncertain