SAND2022-7721C

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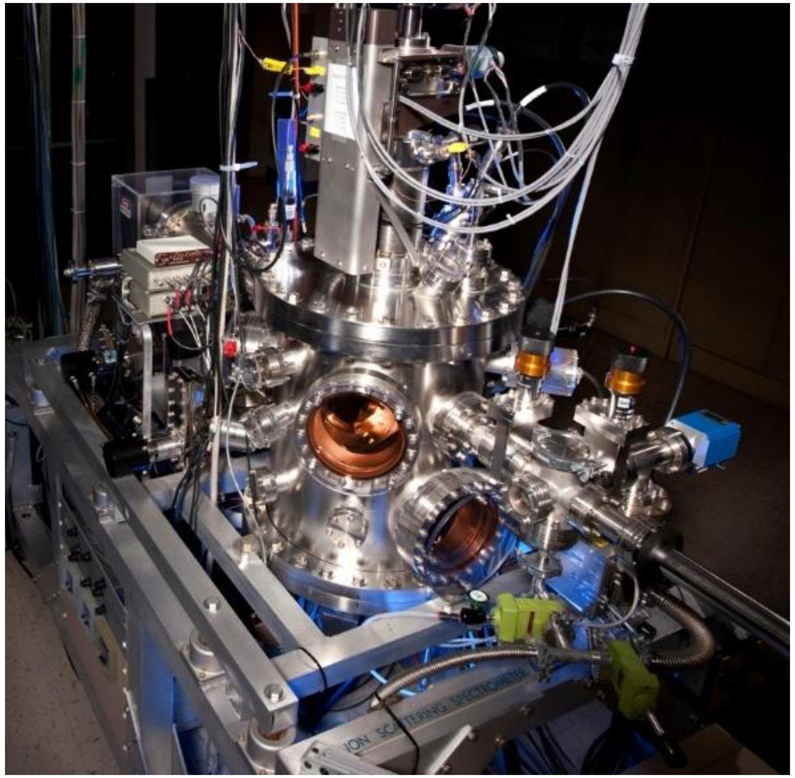
Recrystallization, melting, and erosion of dispersoid-strengthened tungsten materials during exposure to DIII-D plasmas



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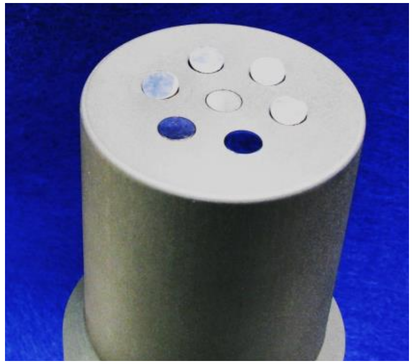

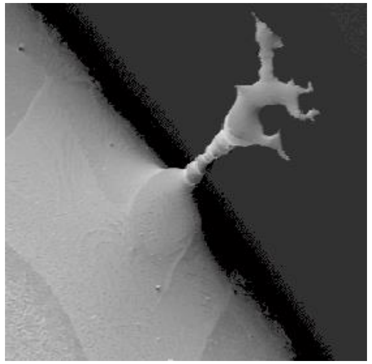

25th International Conference on Plasma-Surface Interactions
13 – 17 June 2022

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Motivation: Understanding W material response to combined high heat flux / particle flux

Dispersoid strengthening can improve mechanical properties, resiliency of W materials against recrystallization / n-damage

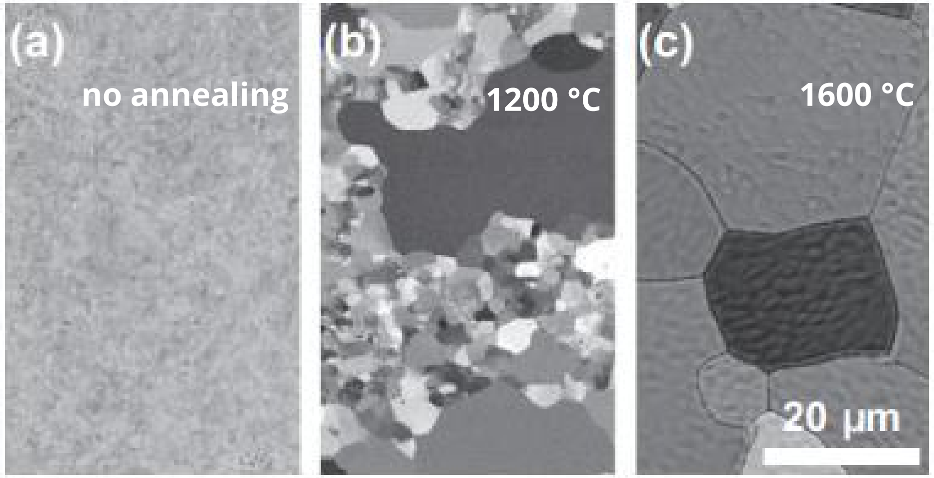
- Initial laboratory testing promising, response to combined effects in a relevant environment needed

Testing of advanced W materials in DIII-D:

- Materials: ITER W, Dispersoid–strengthened W (W-TiO_2, W-Ni)

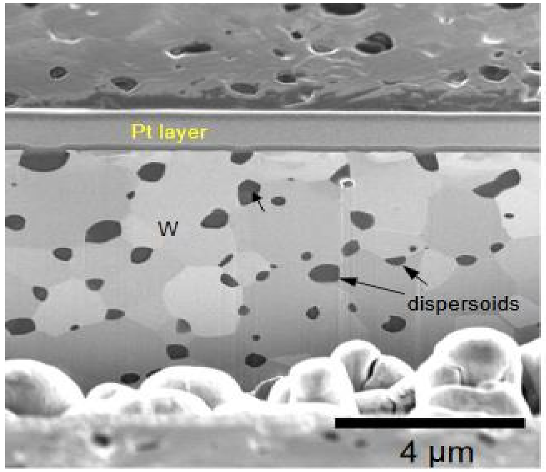
Goals are to quantify:

- Effects of large **thermal gradients** on surface damage / recrystallization.
- Material response to **transients**
- **Preferential sputtering**



grain growth in ITER W

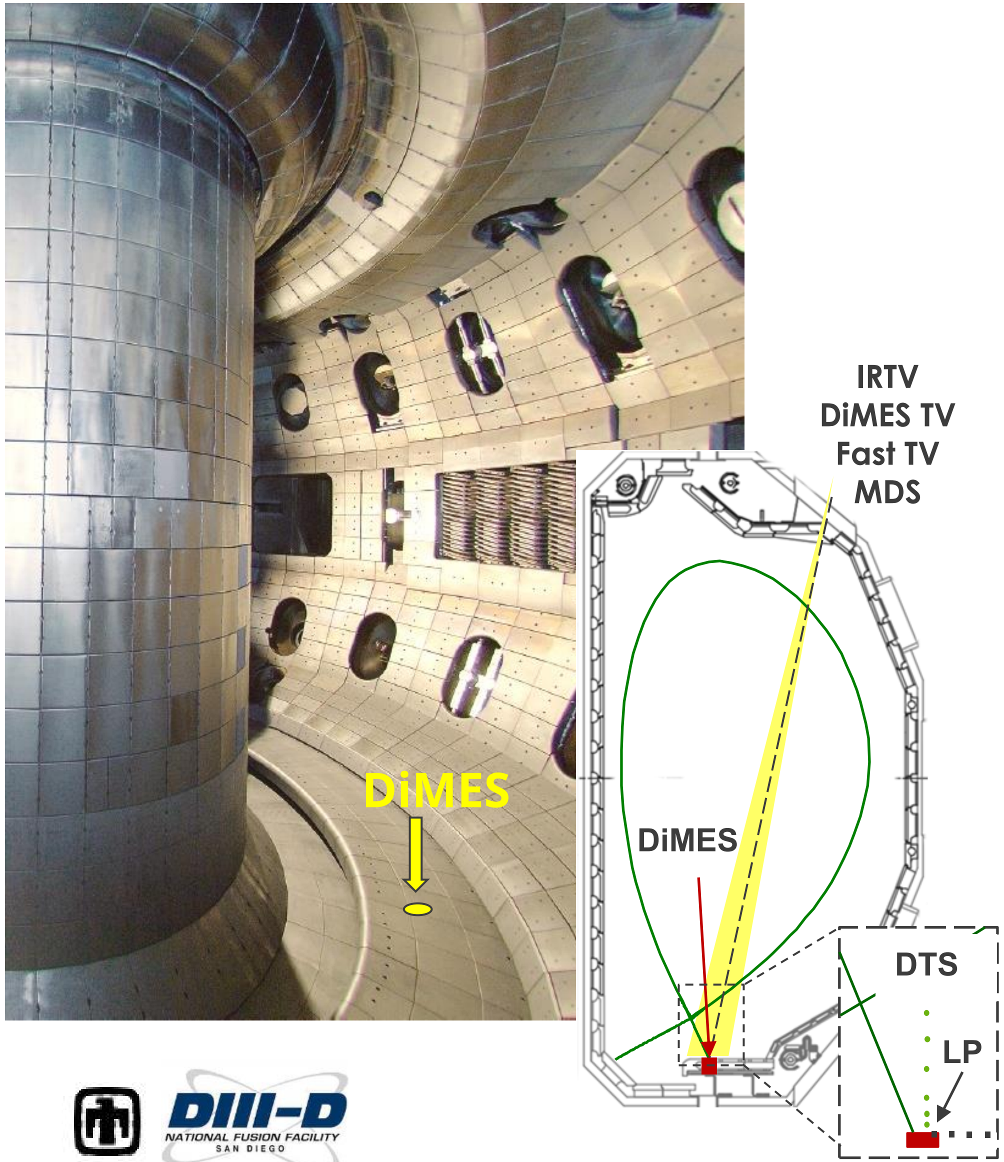
A. Manhard, K. Schmid, et al., *J. Nucl. Mater.* **415** (2011) S632.



dispersoid - strengthened W

R. D. Kolasinski, D. A. Buchenauer,et al., *Int. J. Ref. Met. Hard Mat.* **60** (2016) 28.

Experimental approach: Divertor Material Evaluation System (DiMES) at DIII-D



We used DiMES to expose W samples in the DIII-D divertor

- Located near the Outer Strike Point (OSP) of Lower Single Null (LSN) plasmas
- Well diagnosed with Langmuir probes, Fast Camera, spectroscopy

Approach:

- **Angled, protruding samples** intercept higher heat flux
- Goal: Exceed sample temperature of 1200 °C needed for fast recrystallization
- Thermal analysis to determine sample temperatures

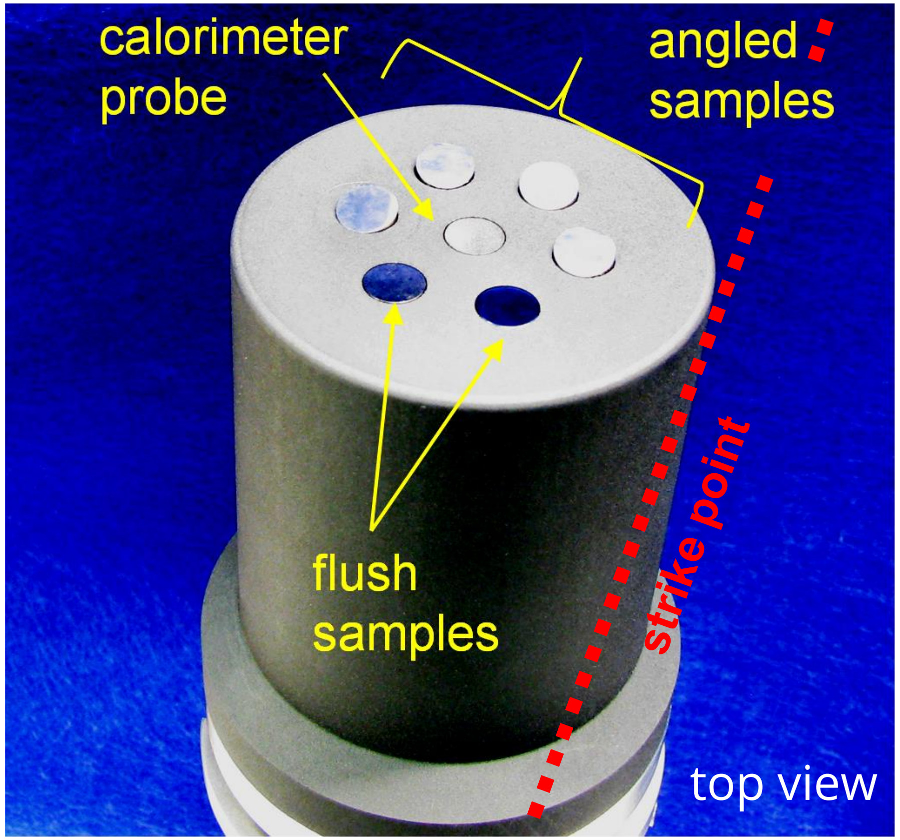
Plasma conditions in the DIII-D divertor

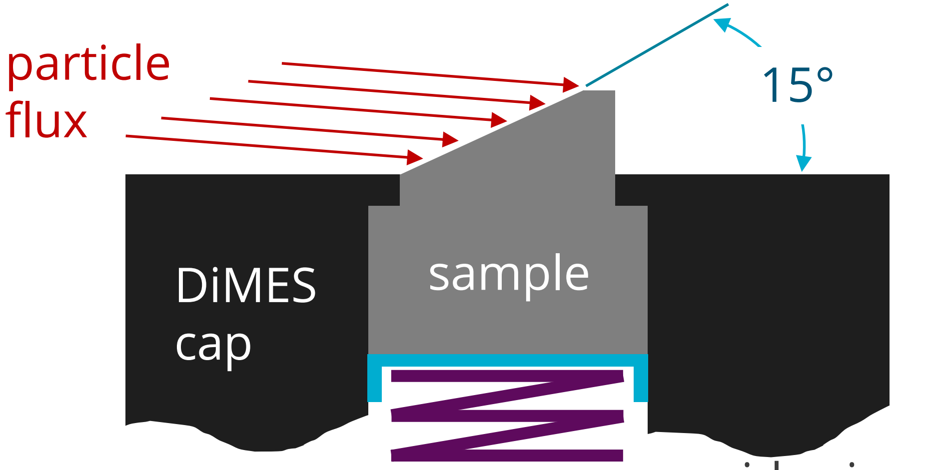
Exposure included 9 H-mode shots in DIII-D

- Samples angled 15° relative to surface
- Steady state heat flux on protruding surfaces:
  - $q_{\perp}$ = 10 – 24 MW/m^2
- 42 Hz ELMs added significant transient heating
- Heat flux, LP, TC meas., and thermal modelling used to determine surface temperatures

Observations:

- Disruption during first shot due to material flaking
- WISE spectrometer detected Ti, W impurities
- Fast camera captured melting of samples closest to strike point (mid-way through exposure)





Top view of DiMES after 9 repeat discharges

ID #	Sample	Config	Observations
1	ITER W	flush	Modest sputtering, C deposits
2	W-TiO_2	flush	Modest sputtering, C deposits
3	ITER W	15° angle	Surface cracking, shallow melting
4	W-TiO_2	15° angle	Flaking, shallow melting, preferential sputtering / evaporation of dispersoid material
5	ITER-W	15° angle	Significant melting / recrystallization
6	W-Ni	15° angle	Significant melting / recrystallization

- Specimens closest to the strike point were more severely damaged, melted material appears to flow along J × B direction
- Extensive microscopy undertaken to compare ITER-grade / dispersoid strengthened W

Significant recrystallization observed in both ITER-grade and DSW materials

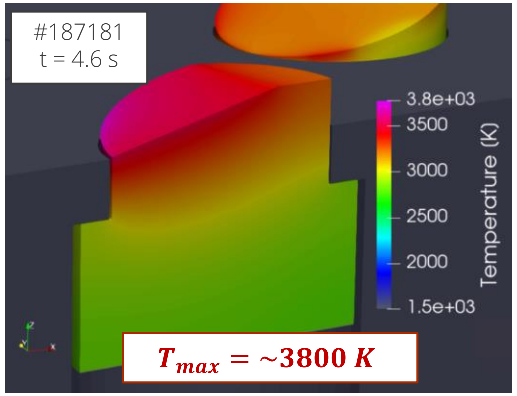
Melted samples:

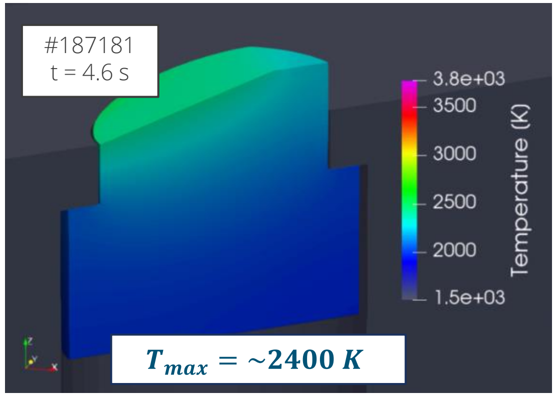
- **Grain size ~ 100 times larger**, entire material recrystallized
- No clear boundary between the melted / un-melted regions
- No obvious differences between ITER-grade / DSW

Un-melted samples:

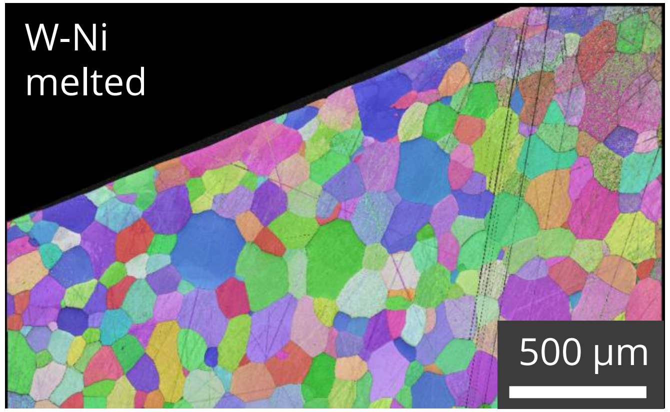
- Changes in microstructure superficial, constrained to within ~5 microns of the surface
- Some deep cracking (up to 100 μm) noted in both ITER and dispersoid strengthened W
- Grain size deeper in material appears comparable to unexposed materials (< 1 μm for W-TiO_2, 5 – 10 μm for ITER W)

Consequences: melted / recrystallized material is much more brittle

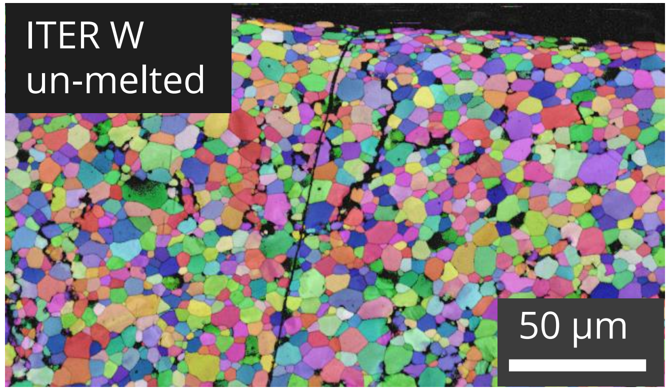


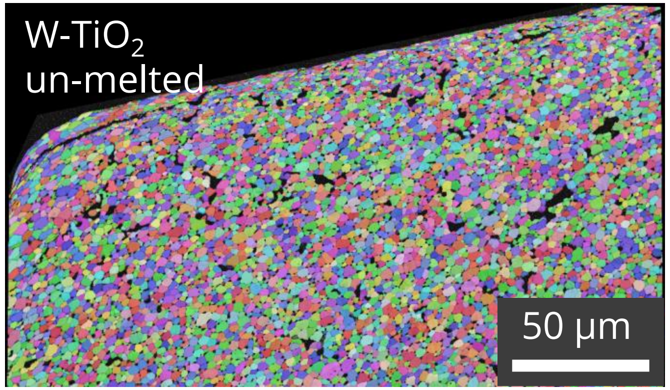


Above: temperature distribution calculated via SIERRA



***note difference in scale



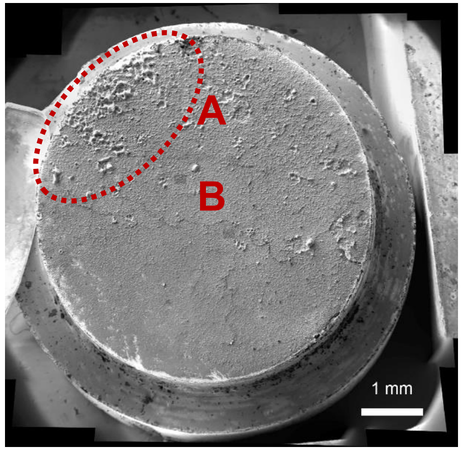


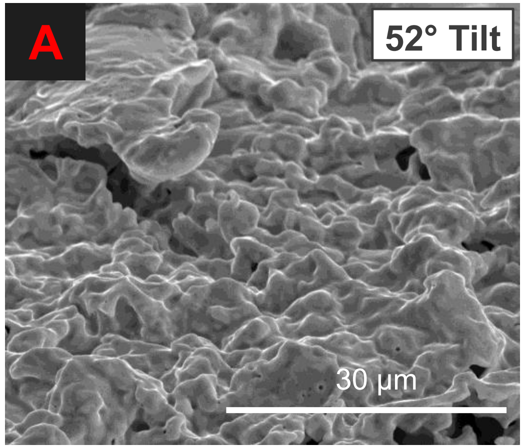
Surface flaking / shallow melting altered surface morphology of samples farthest from strike point

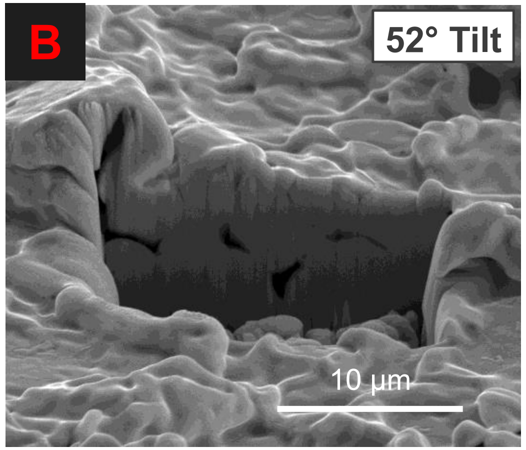
ITER-W

FIB cuts and tilted imaging reveal surface roughening on the order of 10's of μm

- Considerable sub-surface porosity
- Likely that this arises from repeated melting solidification process
- Could have implications for tritium inventory

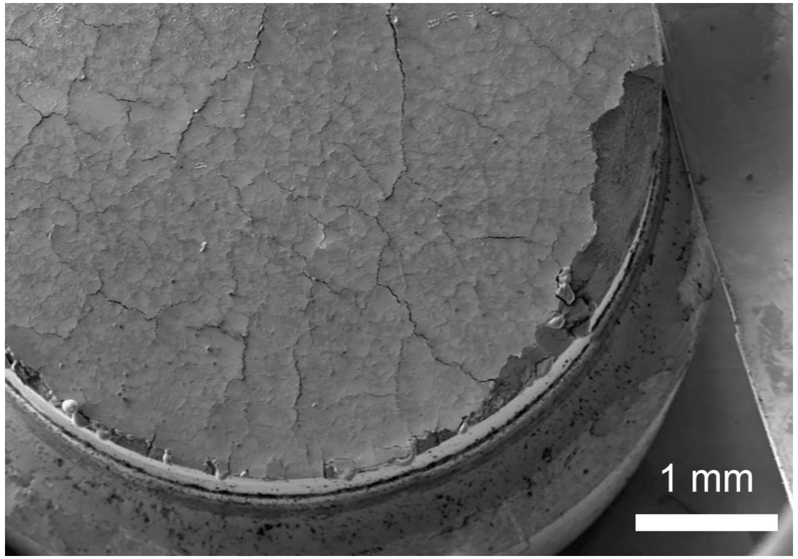


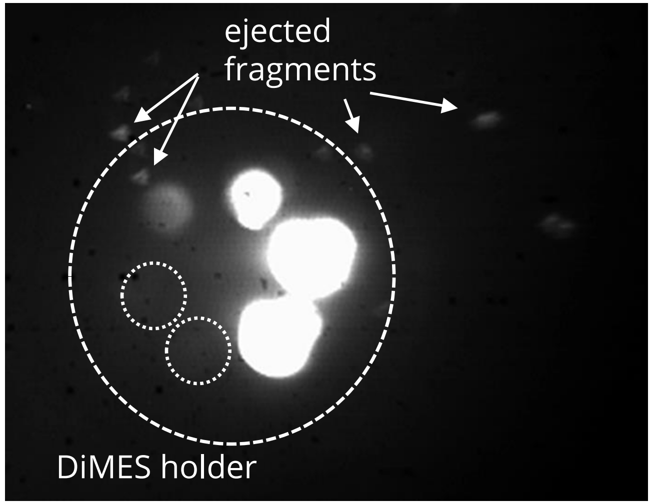




Dispersoid-strengthened W

- Surface melting did not appear as severe, even though the specimen was closer to the strike point
- Noted significant flaking of material at edges of specimen, also observed on Fast camera
- Consequences: dust formation, contributes to plasma impurities



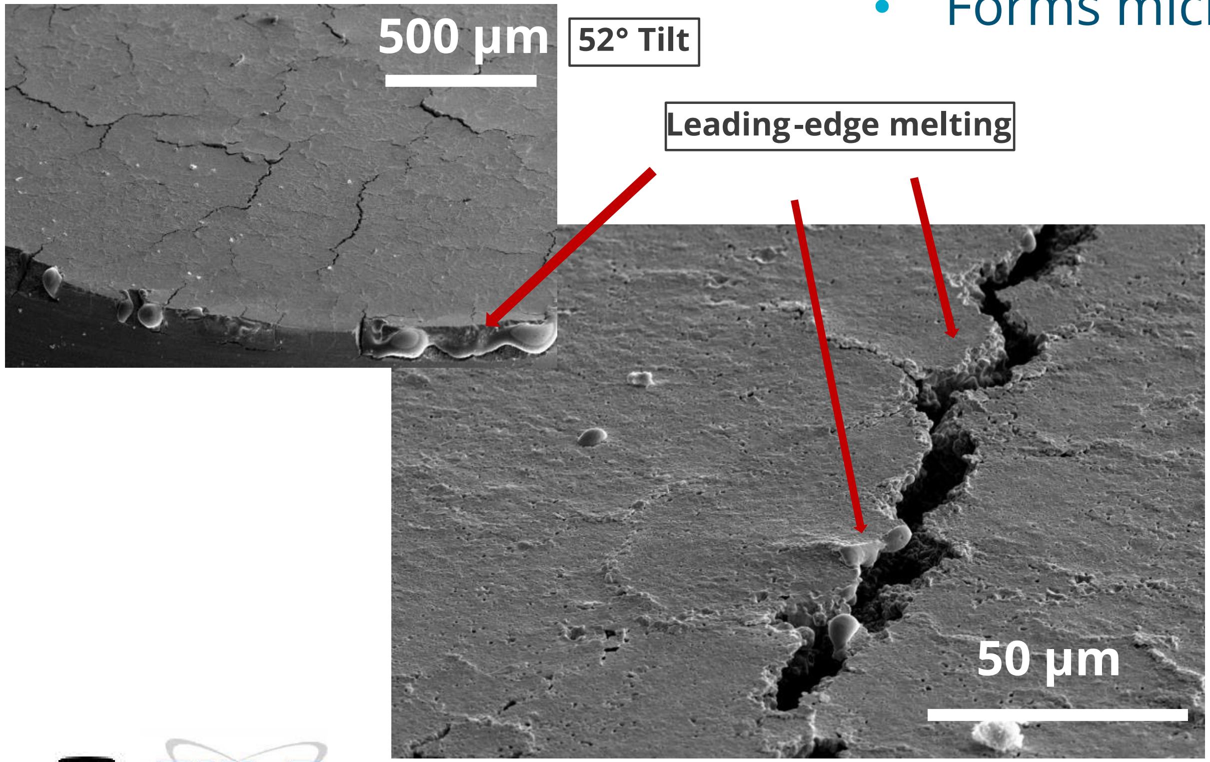


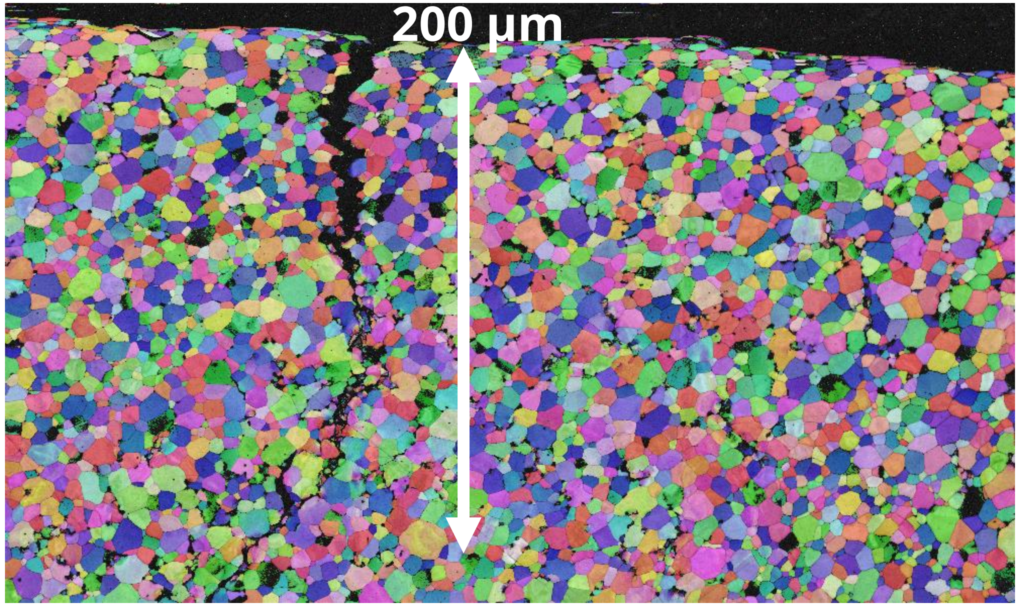
Fast camera view showing flaking of material during plasma exposure

Crack formation observed on all samples but was more severe in the dispersoid-strengthened material

- Cracking was observed in both materials, though was moderately more severe in the DSW.
- Individual cracks were up to several mm long, ~ 5 μm wide, and formed a continuous network across the surface

- Cracks protruded > 200 μm into the material
- Forms microscopic leading edges where melting can initiate

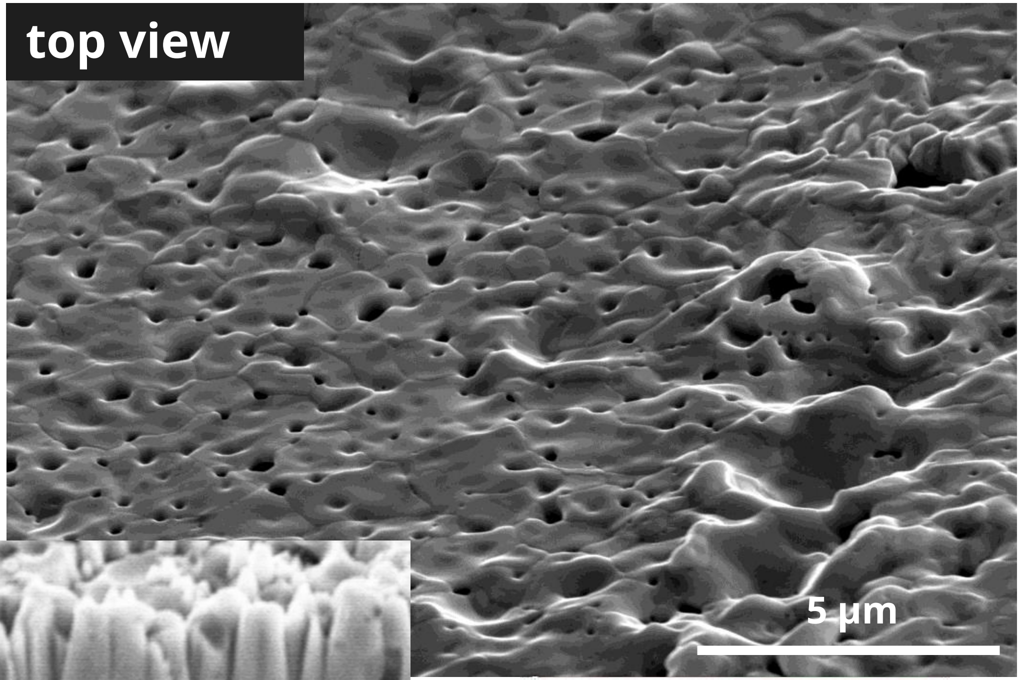




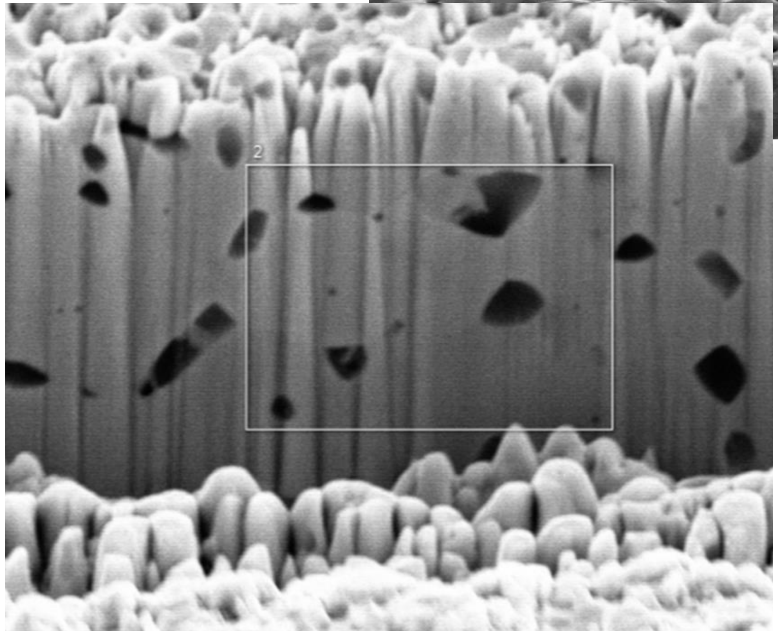
Depletion of dispersoids near the surface alters surface composition / changes sputtered impurities

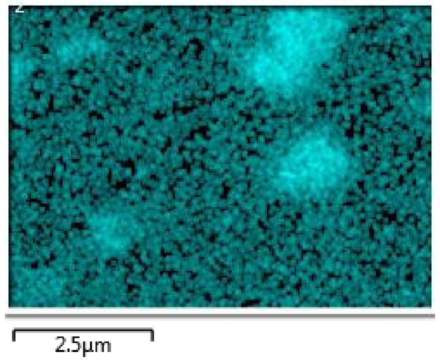
Surface composition analysis:

- Dispersoids appear absent from the surface (only empty pits remain.)
- Consistent with TiO_2 evaporation / sublimated.
- FIB profiling / EDX analysis reveals that sub-surface dispersoids present at > 2-3 μm (effect is superficial)
- **Potential concern for erosion / redeposition?**



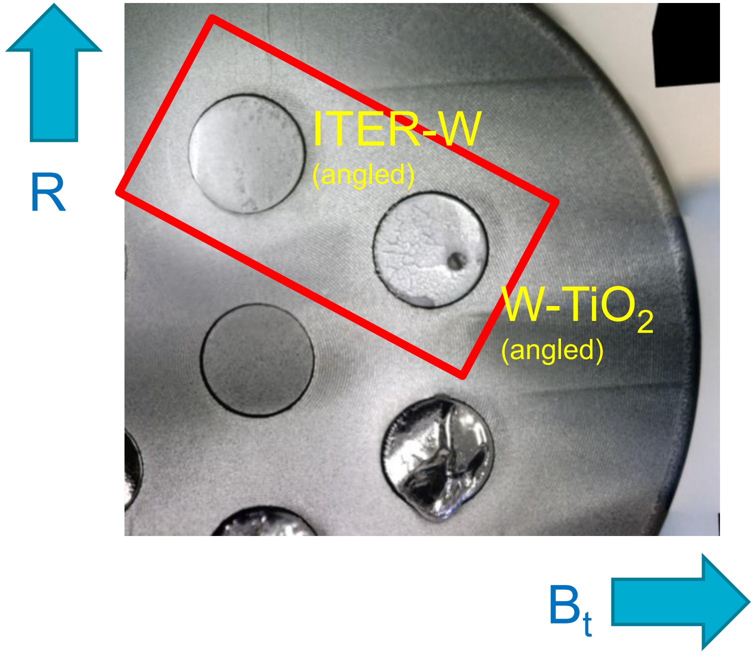
FIB profile

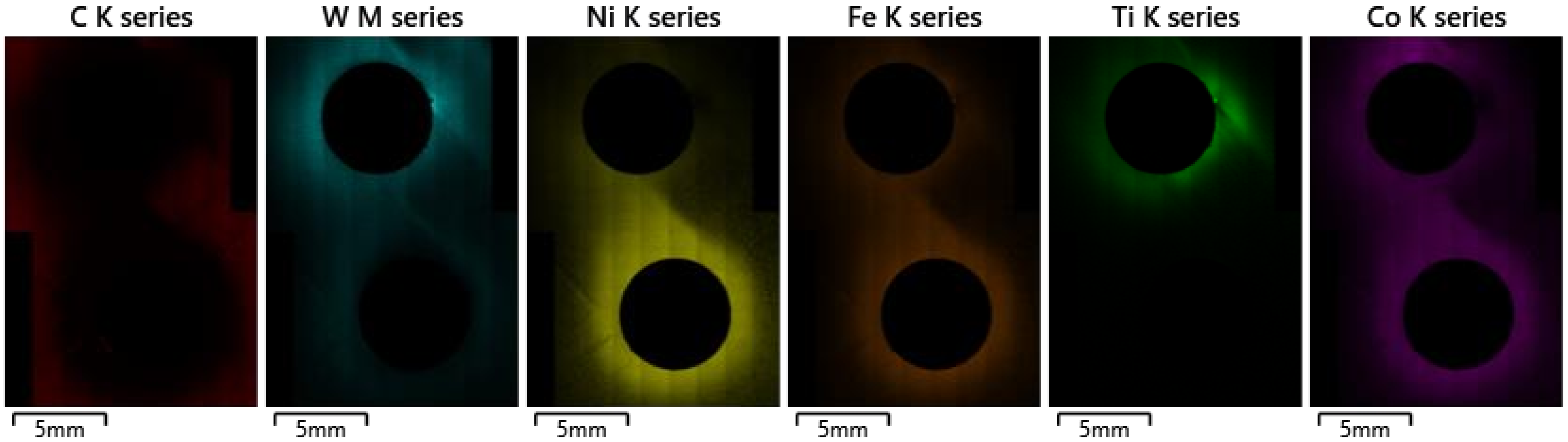




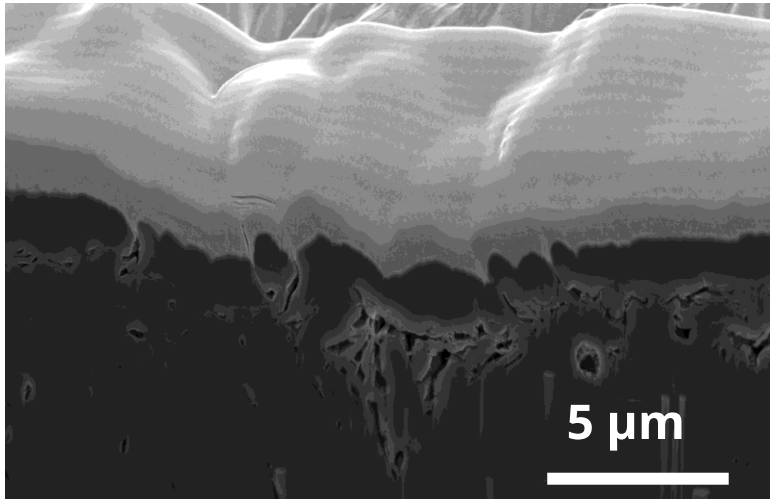
Ti Kα

Dispersoid material has been preferentially sputtered / evaporated





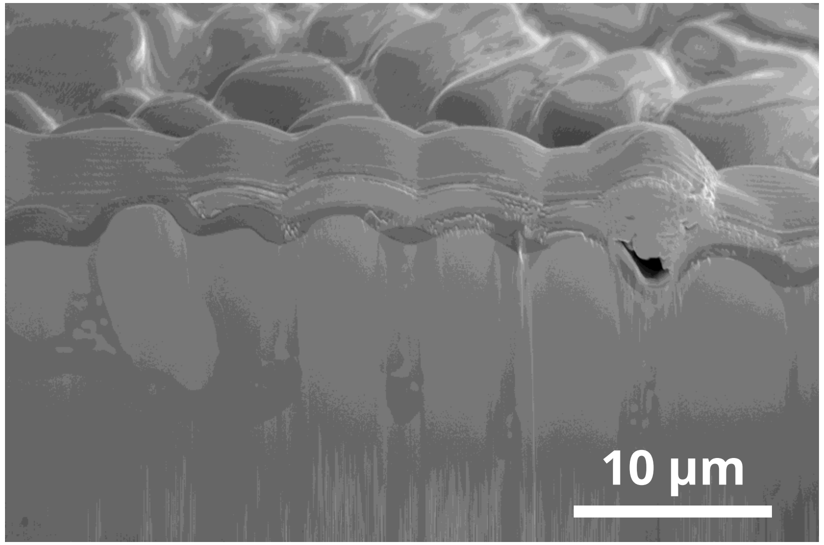
- Redeposited material was approximately 1 µm thick, and consisted mostly of Ti, W, Ni, and stainless steel constituents
- Most material redeposited within a few mm of the sample from where it originated.
- Deposits appear enriched with dispersoid material

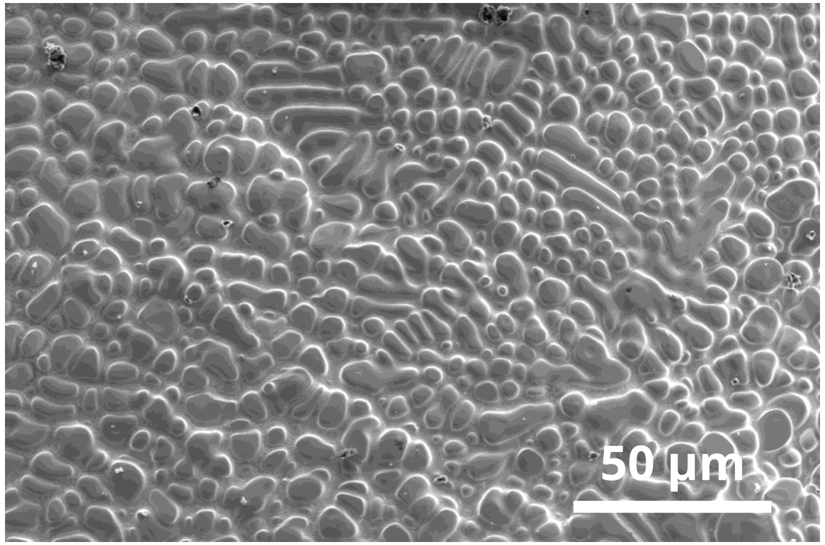


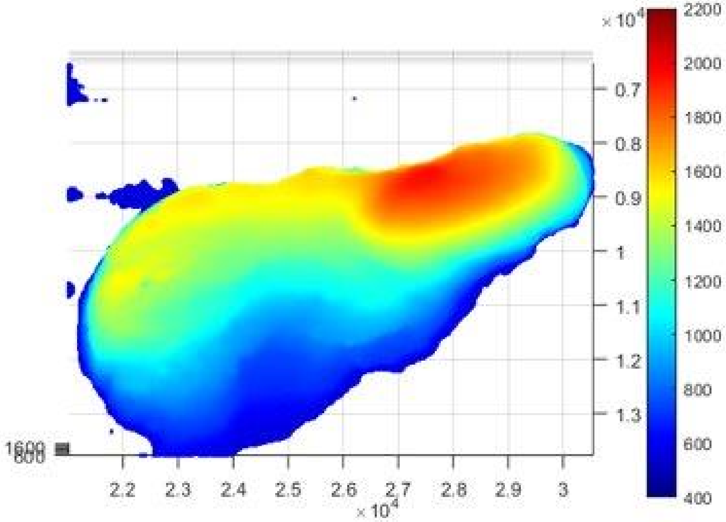
FIB profile of redeposited layer

Features of the melted material

- Unusual surface morphology was observed in the melted regions appears to be due to carbide formation in the near-surface
- Melted material was drawn in the J x B direction, volume of melted material estimated from optical profilometry
- Melt process was captured by Fast Camera, analysis of melt motion is underway.





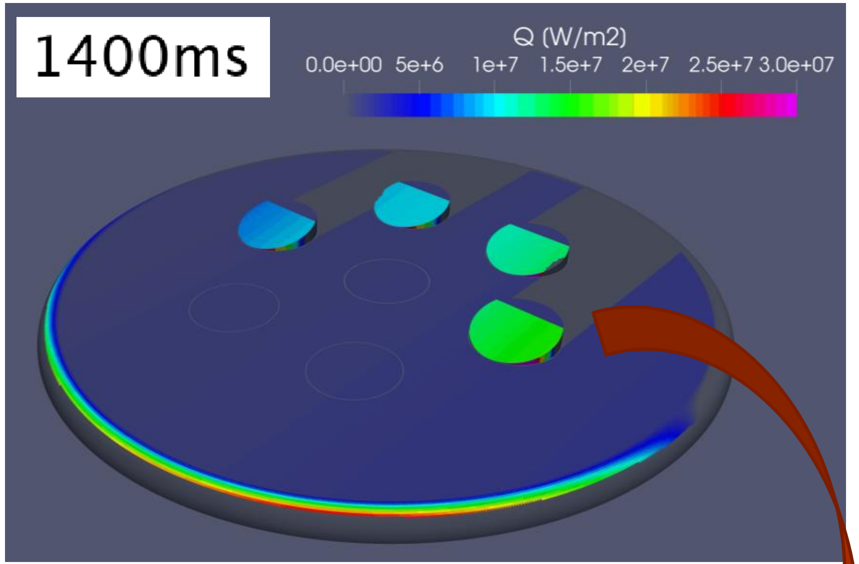


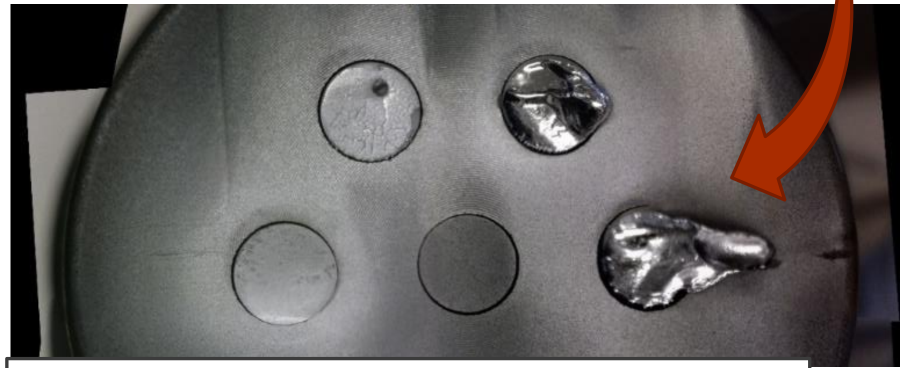
SMITER estimates higher than expected heat fluxes

Thermal analysis observations

- Strong variation in $q_\perp$ across 4 angled samples for each shot
  - **Inter-ELM: 11– 24 MW/m²**
  - **Intra-ELM: up to 115 MW/m2**
  - Samples closest to strike point receive higher $q_\perp$
- Reproduce surface melt temperatures on 2 samples closest to strike point
- Bulk sample temperature **higher than recrystallization temperature for ~3 seconds**
  - Supports microscopy results that show **uniform recrystallization throughout sample depth**, rather than clear transition region

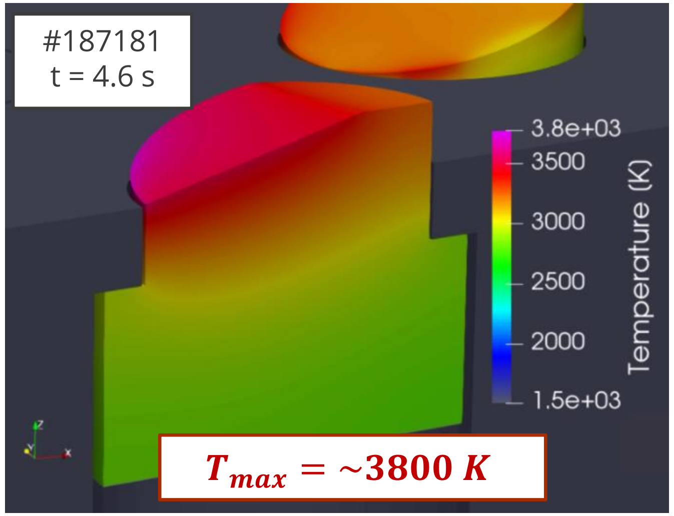
$q_\perp$ during shot #187181

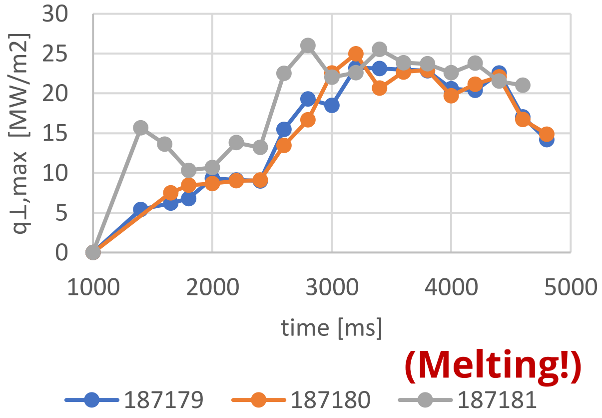




Post-exposure image of angled W samples

SIERRA Thermal Modeling





See poster by J. Coburn **P131(B)**

Concluding remarks

Comparison of ITER-grade and dispersoid strengthened material performance and potential implications:

- Surface roughening of ITER-grade W was significant → erosion & redeposition
- Significant sub-surface porosity was observed → tritium inventory
- For dispersoid-strengthened materials, oxide-based dispersoids near the surface did not appear to survive high temperatures
  - More resilient dispersoid materials (e.g. carbides may provide better performance in this regard)
- Surface roughening / melting on dispersoid-strengthened material was lower, but surface cracking was more severe.

Melted specimens:

- Flow of melted material in J × B direction
- Source of unusual surface topography appears to be carbide formation

Further work: Follow-up experiments planned for current DIII-D run campaign to test improved materials, including tungsten fiber composite / W-TaC, including thermal / melt-motion modelling.

Acknowledgements

We thank the Department of Energy (DOE) Office of Fusion Energy Sciences Materials program for their support of this work. Sandia is a multi-program laboratory managed and operated by Sandia Corporation, a wholly-owned subsidiary of the Honeywell Corporation, for the United States Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. This work was also supported by US DOE under DE-FC02- 04ER54698, DE-FG02-07ER54917, DE-SC0019256, DE-AC05-00OR22725, and DE-AC52-07NA27344.

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