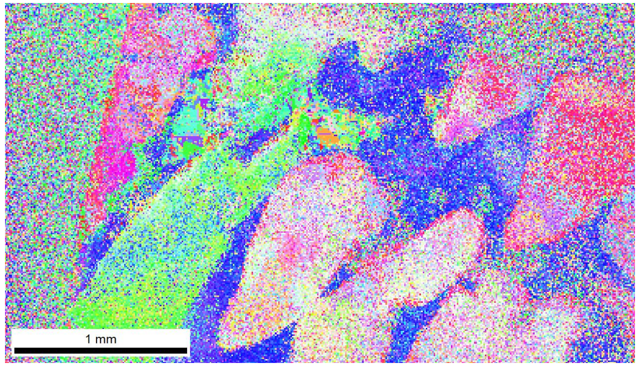
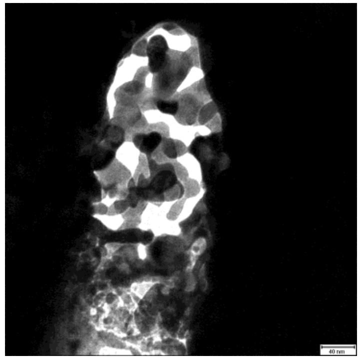
SAND2022-1975C

Sandia National Laboratories

In-situ TEM of the Microstructure and He Behavior of AM W Alloys





AIME–TMS 150th Celebration TMS2022 Annual Meeting & Exhibition SINCE 1871

PRESENTED BY

Eric Lang, Streit Cunningham, Ian McCue, Jason Trelewicz, Khalid Hattar

U.S. Department of Energy NNSA

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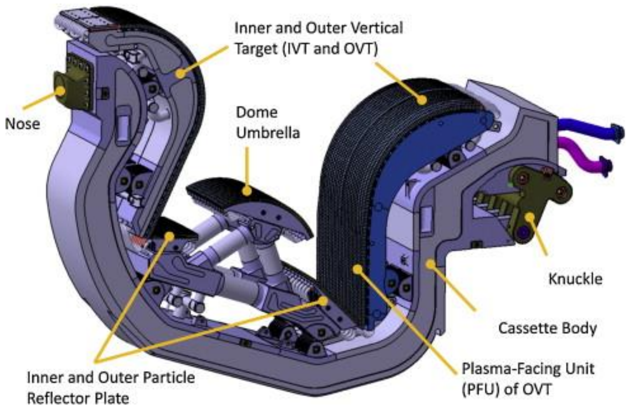
Acknowledgements

- **Sandia National Laboratories**: Khalid Hattar
- **Northwestern University**: Ian McCue
- **Stonybrook University**: Streit Cunningham, Jason Trelewicz

Tungsten proposed for use in divertor region in fusion reactors



T. Hirai, Fusion Eng. & Design, 2013.

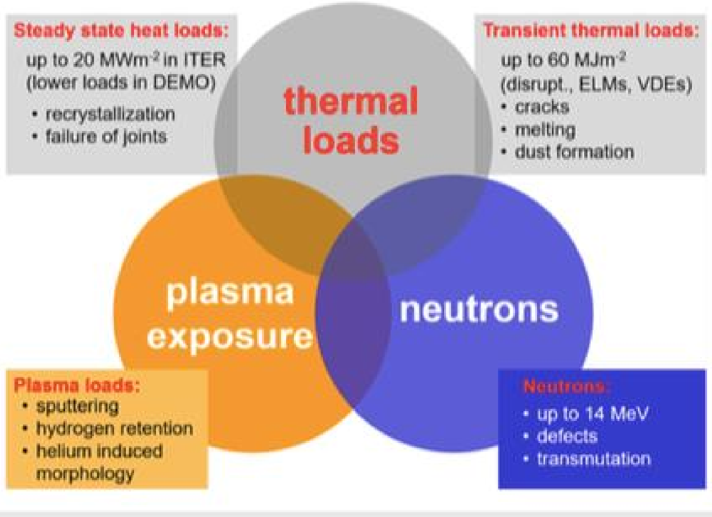
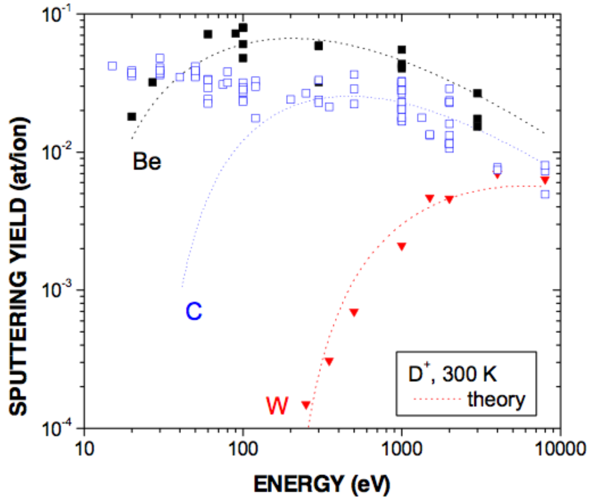
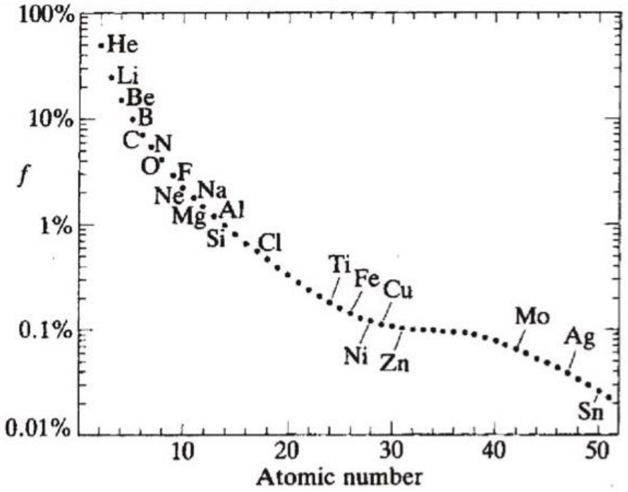


FIG. 1. Synergistic wall loads in D-T-burning magnetic confinement experiments.

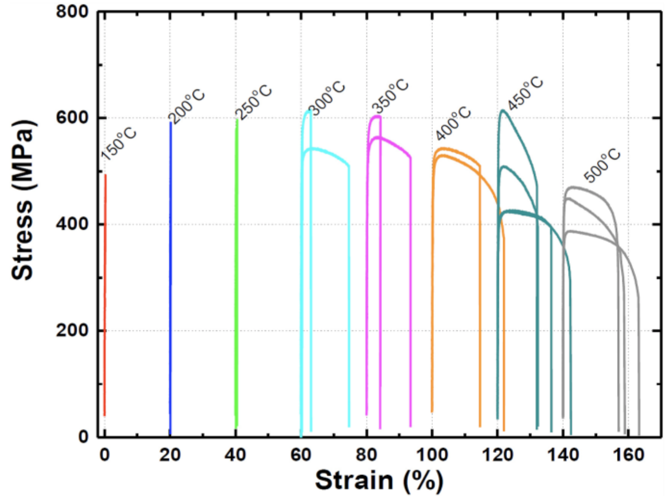
J. Linke. *Matter Radiat. Extremes* 2019.

Low sputtering of W, but small amounts tolerable





High DBTT, and embrittled under irradiation



<u>Ways to improve W:</u>

Impurity inclusions

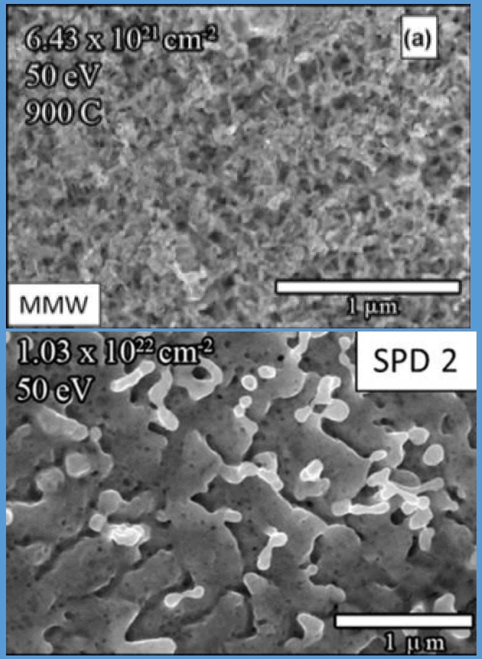
Grain refinement

Alloying

X T. Shen. *JNM* 2016.
T. Miyazawa. *Nuclear Materials and Energy* (2018).
G. Federici. Nuclear Fusion (2001)

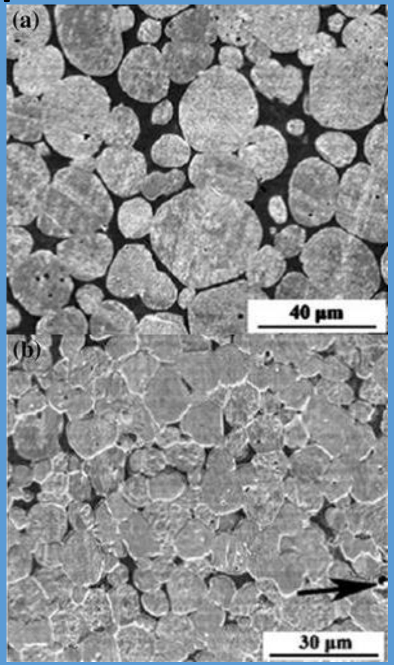
Tungsten alloys can reduce the grain size, increase ductility

Reducing the grain size can improve the radiation tolerance, but unstable at high temperatures



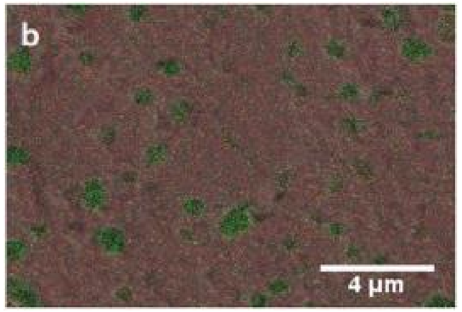
O. El-Atwani. *JNM* 2015.

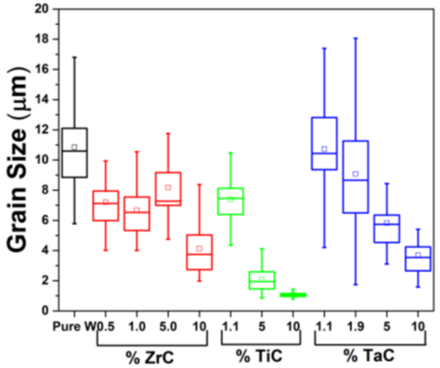
Tungsten heavy alloys used as kinetic penetrators, NiFe acts as a binder phase



K. Hu, *Metall Mater Trans A* 2013.

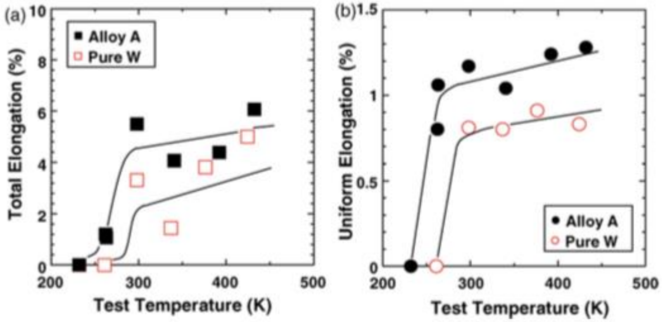
Dispersion strengthening: introducing second phase carbide or oxide to pin grains



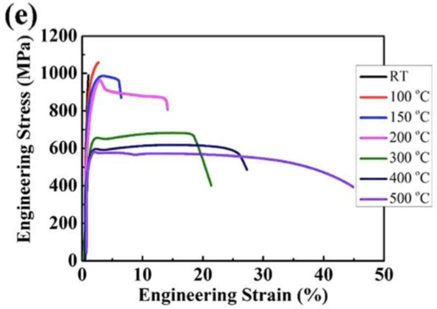


E. Lang, et al. *IJRMHM 2018*

Dispersion strengthening reduces grain size, increases interface concentrations



Y. Ishijima. *MSE A* 2008

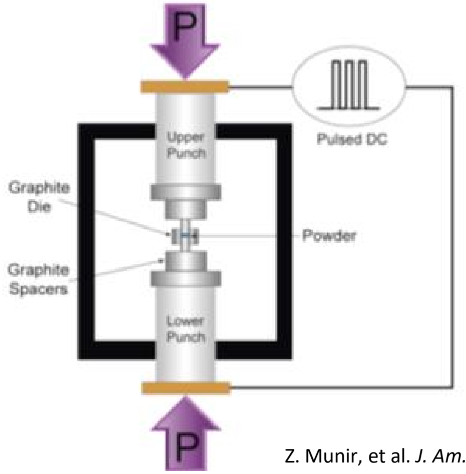


Z.M. Xie. *Sci. Rep.* 2016

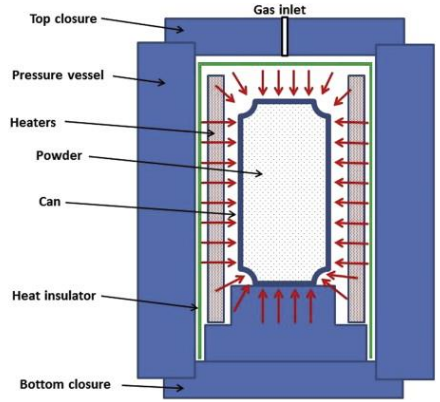
Alloying W with TiC (left) and ZrC (right) can improve bulk mechanical properties

Tungsten alloys typically pressed or sintered

Arc melting processing of W produces large-grained microstructures



Z. Munir, et al. *J. Am. Ceram. Soc.*, **94** [1] 1–19 (2011).

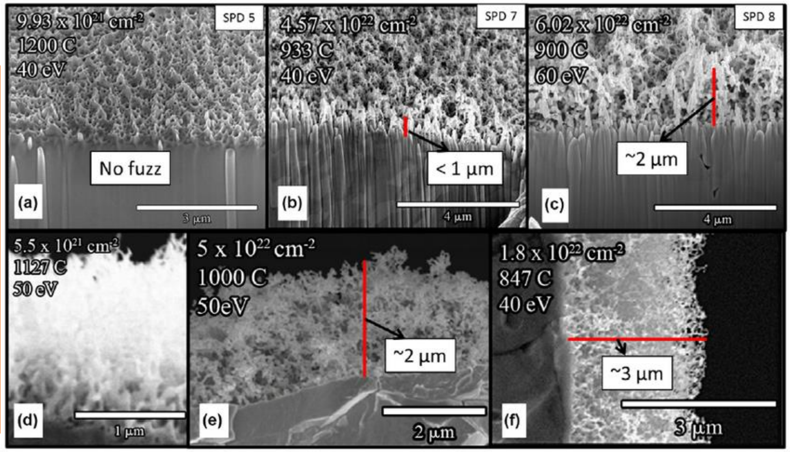


J.M. Torralba, Comprehensive Materials Processing, 2014

Need net shaping of components for fusion environment

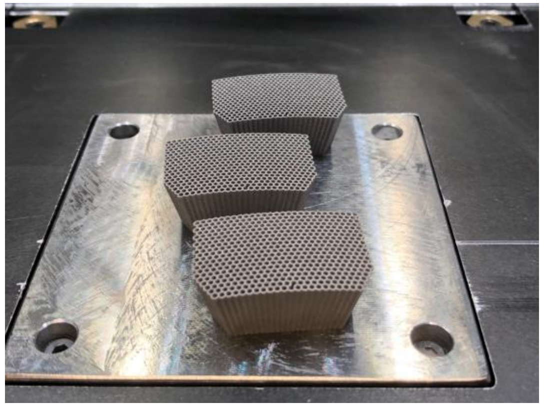
For fusion: W-Cu gradients, flow channels, curved geometries

W microstructure impacts its mechanical properties and response to ion irradiation

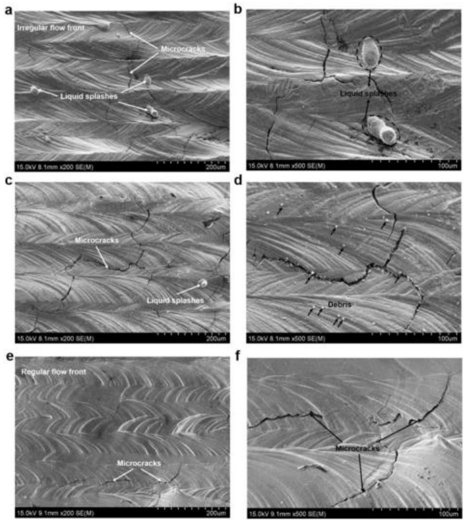


O. El-Atwani, et al. Nuclear Fusion, 2014

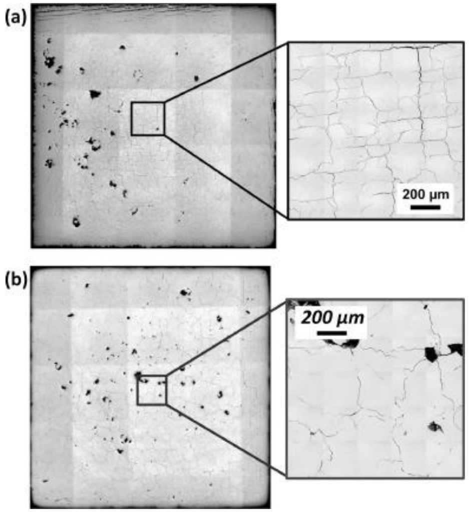
Additive Manufacturing techniques for complex geometries, rapid builds, composition/property gradients…



Additive manufacturing of W has challenges: cracking, anisotropic microstructures

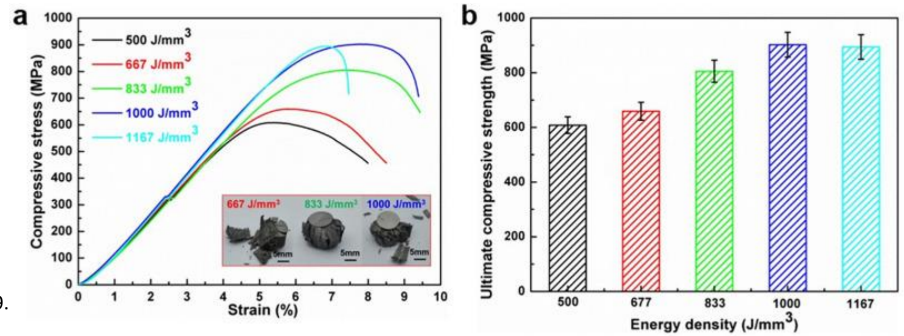


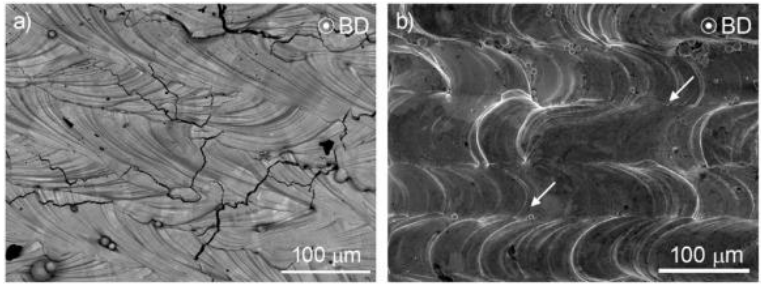
M. Guo, *International Journal of Refractory Metals and Hard Materials*, 2019.



A.v. Muller, *Nuclear Materials and Energy*, 2019.

Build parameters affect microstructure and properties

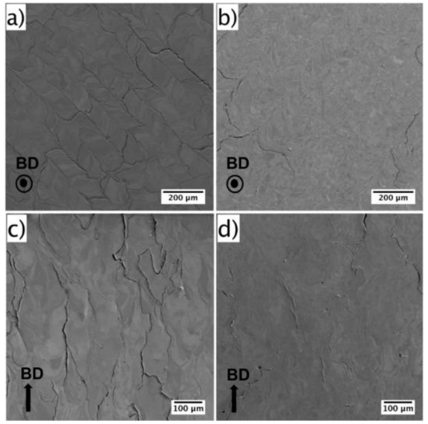




A. Ivekovic, et al. IJRMHM, 2018.

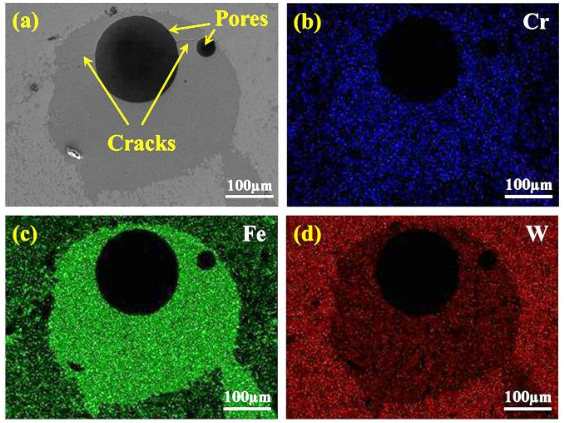
<u>Ways to improve W:</u>
Impurity inclusions
Grain refinement
Alloying

Alloying with ZrC has reduced cracks



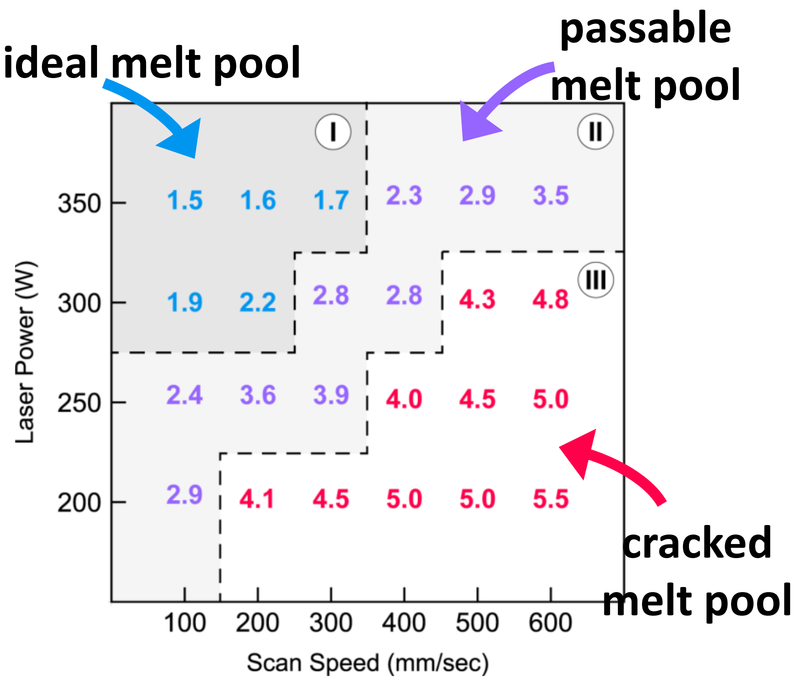
K. Li, et al. IJRMHM, 2019.

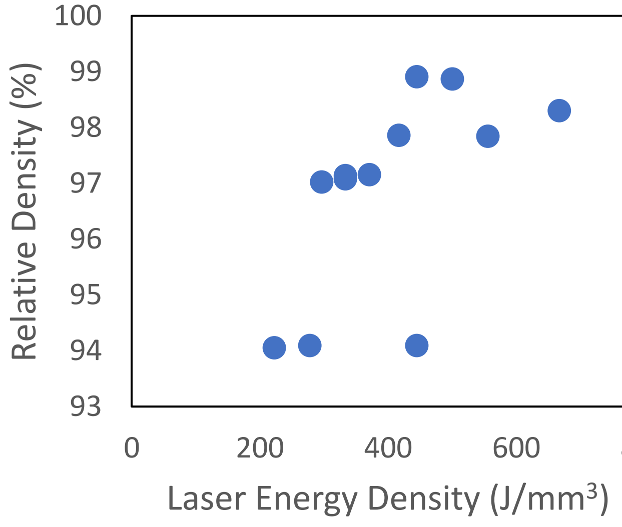
Alloying with Cr, Fe can mimic a WHA microstructure

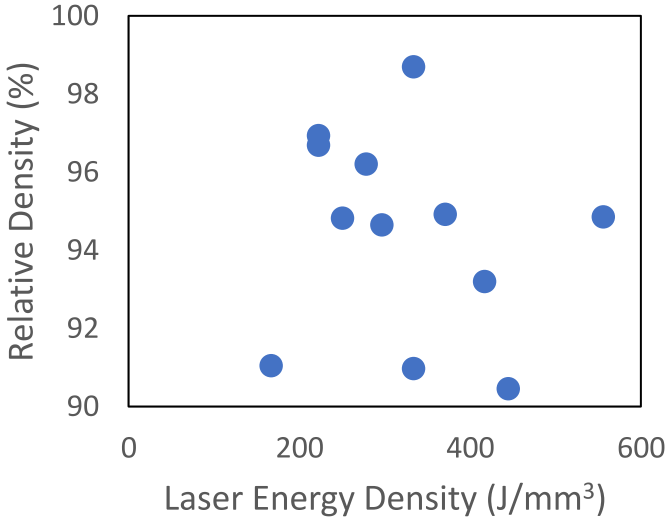


J. Xie, *Surface Coatings and Technology*, 2021.

Directed Energy Deposition used to fabricate W and W alloys

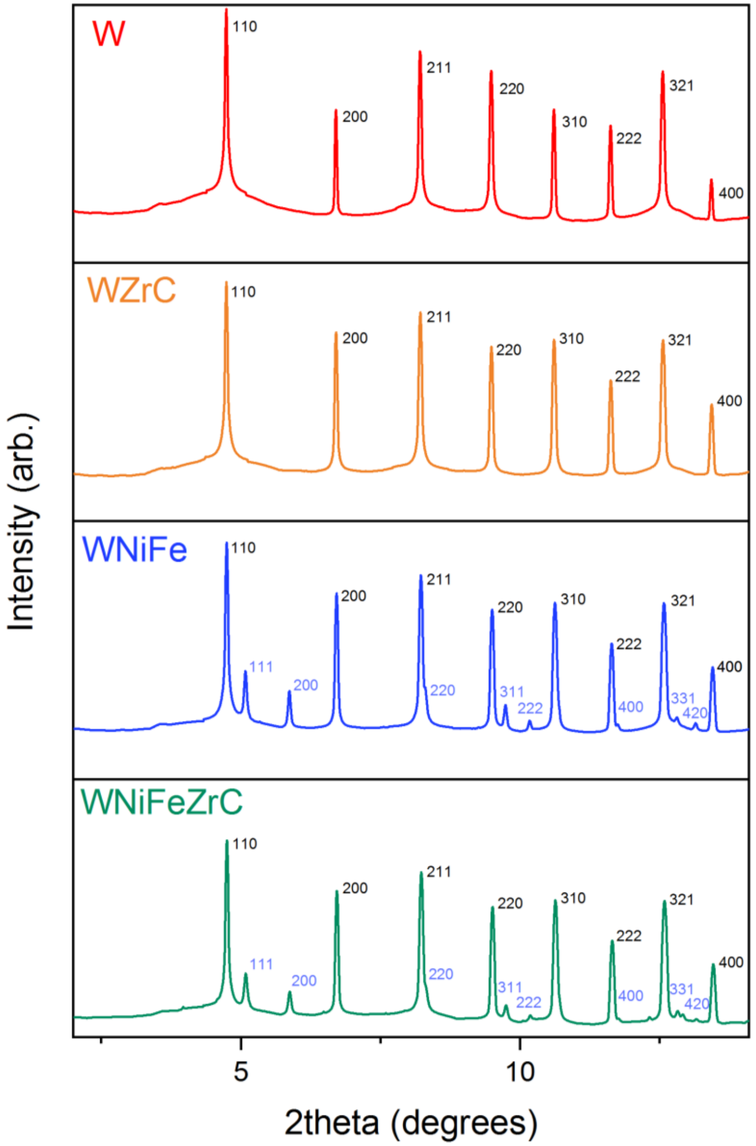






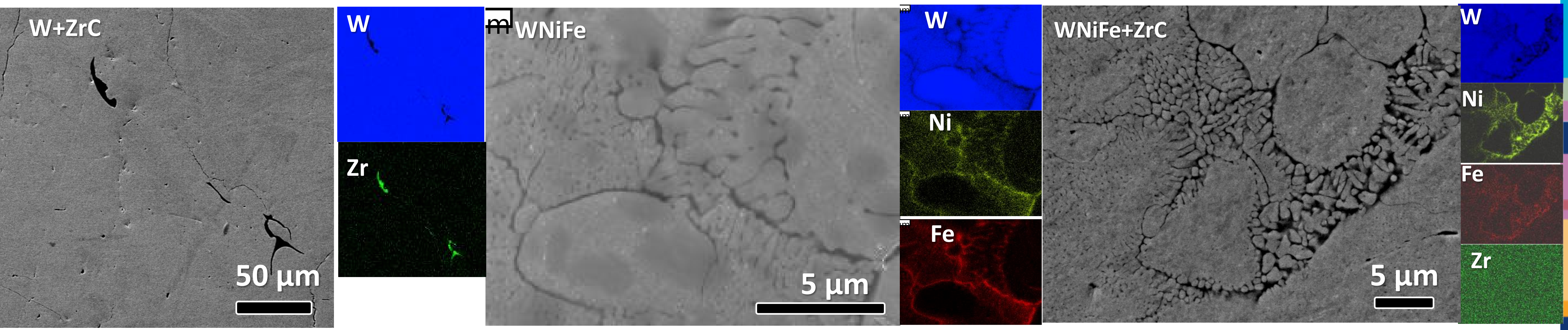
- **Density trend in pure W has a clear maximum between 400 and 500 Jmm^{-3}**
- **Density trend in W-NiFe is less obvious due to Ni and Fe evaporationc**

BCC W fabricated with FCC secondary phases



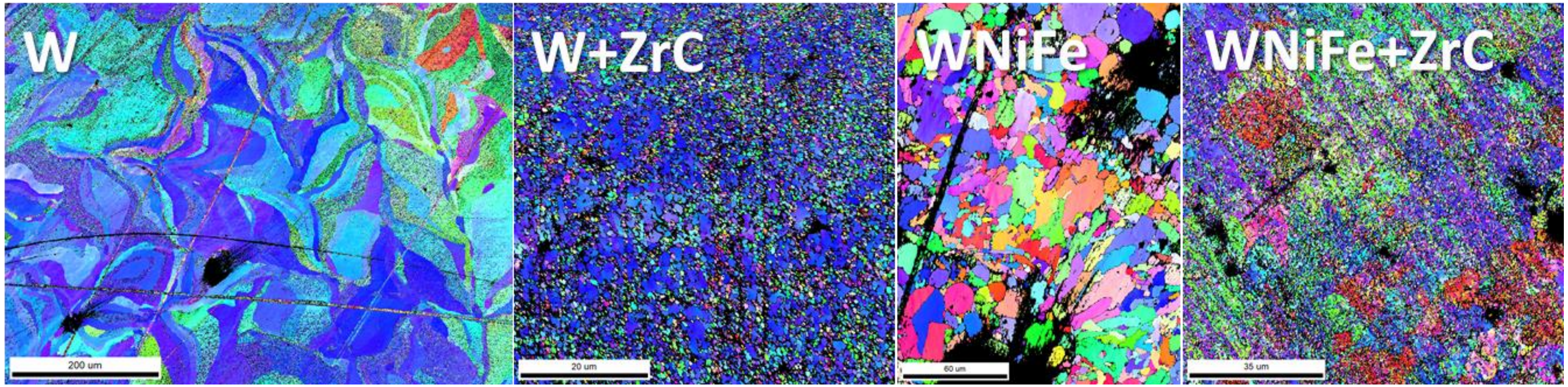
	Nominal Composition	XRD Composition
W	100% W	100% W
W+ZrC	99.95% W 0.05% ZrC	99.95% W 0.01% Cubic Zr 0.03% ZrC
WNiFe	95% W 3.5% Ni 1.5% Fe	96.23% W 3.71% Ni 0.06%Fe
WNiFe+ZrC	94.5% W 3.5% Ni 1.5% Fe 0.05% ZrC	99.00% W 0.92% Ni 0.001% Fe 0.009% Cubic Zr 0.08% ZrC

Grain refiners and lower melting point alloying additions were the most effective in reducing crack density

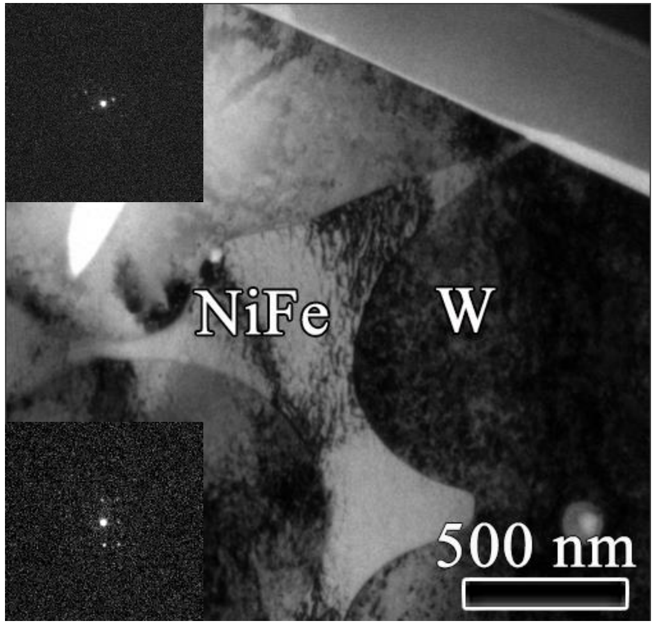


ZrC forms micron-sized particles, refines grains

NiFe segregation to interfaces, refine grain size

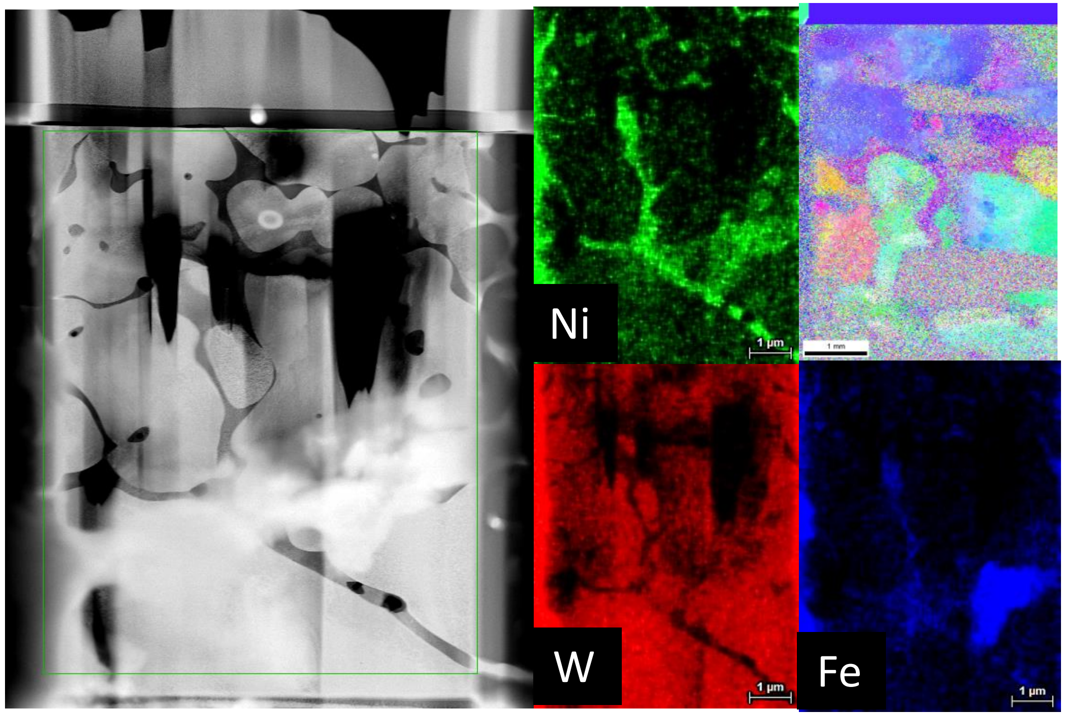


FCC NiFe-rich phases within BCC-W matrix

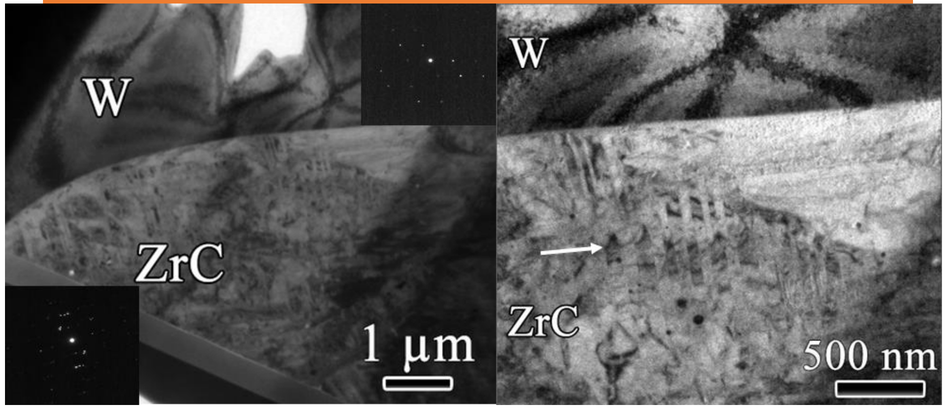


FCC precipitates within BCC W matrix

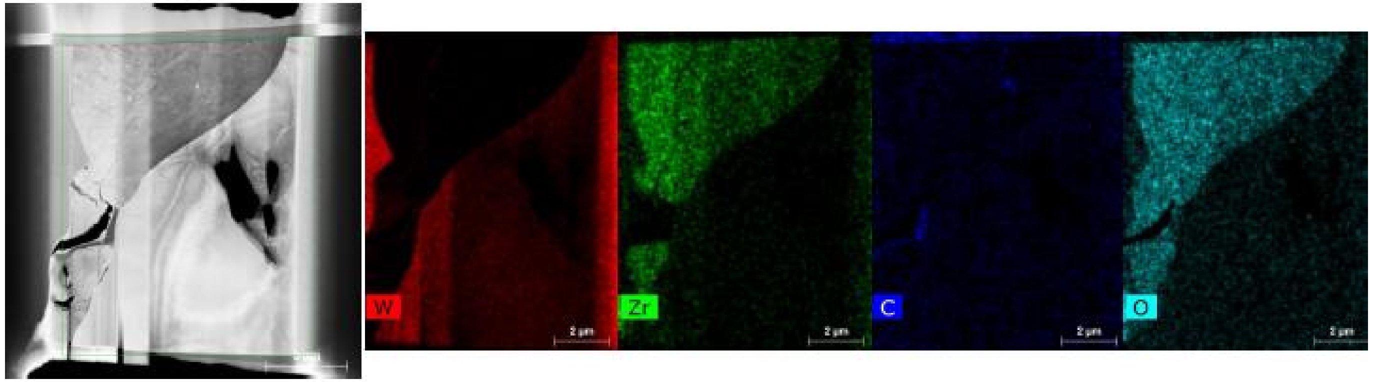
Ni and Fe segregate to boundaries, precipitates



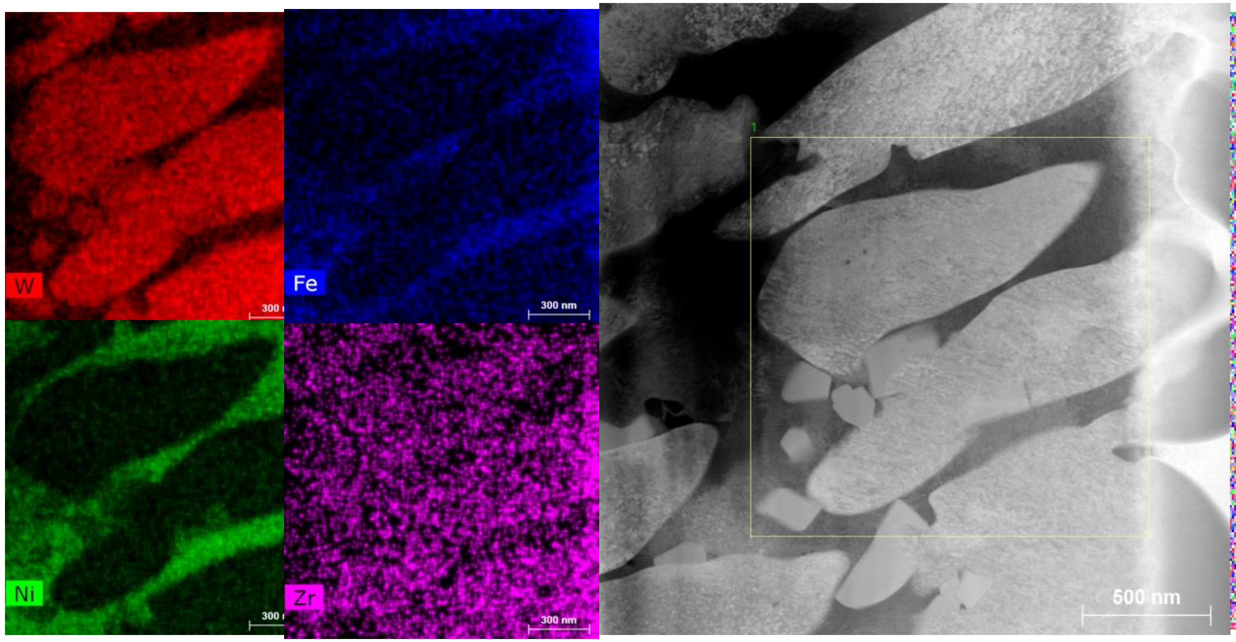
W+ZrC shows zirconia formation, twining`

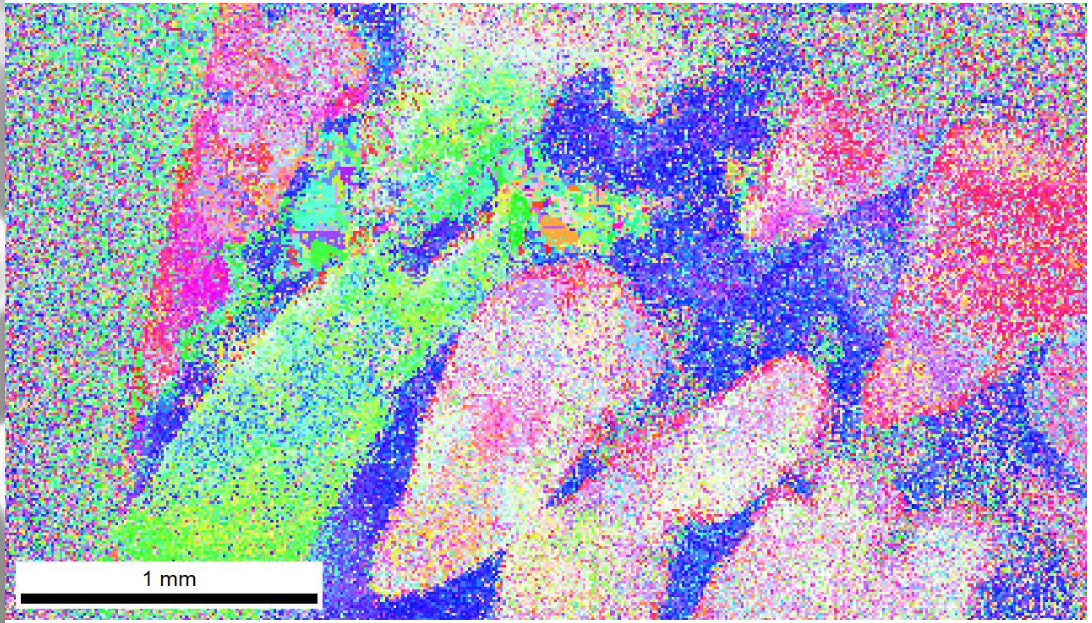


Zr segregation into micron-sized particle



Kitchen sink sample shows elongated W within NiFe sea, Zr homogenously distributed





Elongated W rich precipitates within NiFe matrix

ACOM map: NiFe in primarily 111 orientation, W randomly oriented

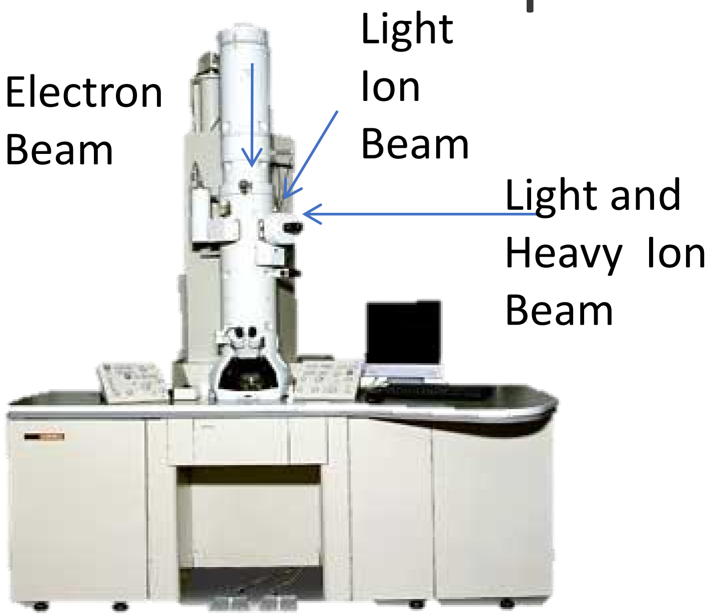
In-situ TEM irradiation and heating allows for viewing of transient processes

<u>Proposed Capabilities</u>

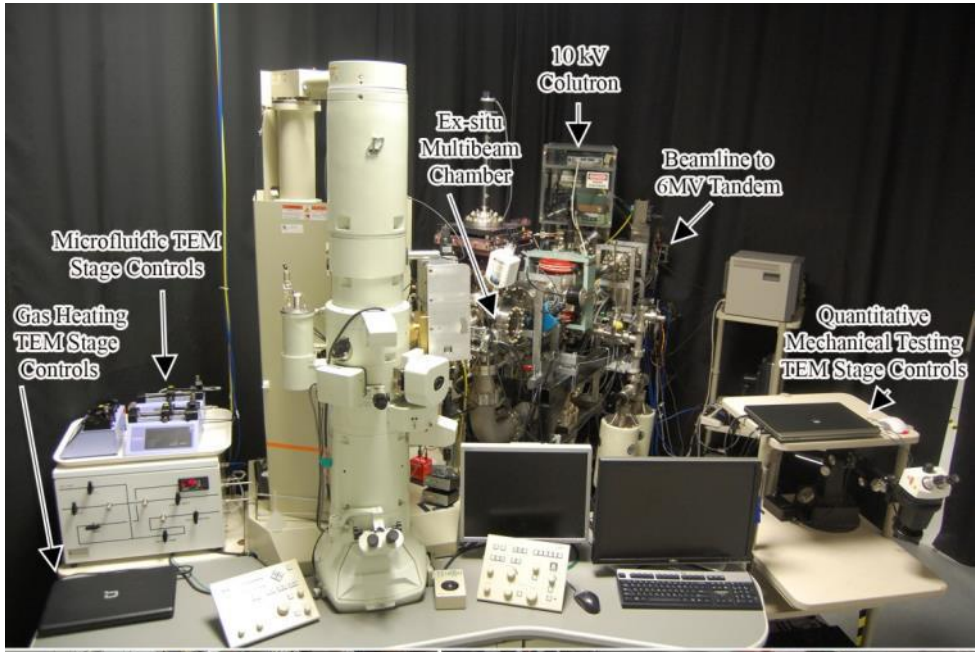
- 200 kV LaB_6 TEM
- Ion beams considered:
  - Range of Sputtered Ions
  - 10 keV D^{2+}
  - **10 keV He^+**
- All beams hit same location

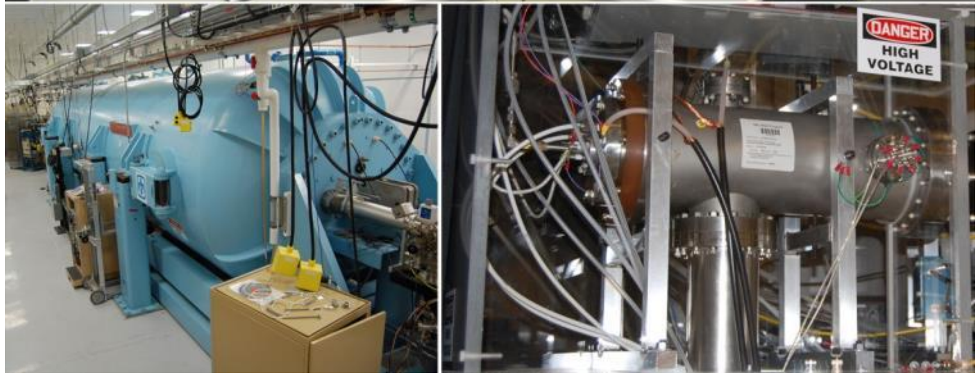
- Nanosecond time resolution (DTEM)
- Procession scanning (EBSD in TEM)
- *In situ* PL, CL, and IBIL

- *In situ* vapor phase stage
- *In situ* liquid mixing stage
- ***In situ* heating**
- Tomography stage (2x)
- *In situ* cooling stage
- *In situ* electrical bias stage
- *In situ* straining stage (3x)



In-situ annealing and He irradiation to simulate PFC conditions





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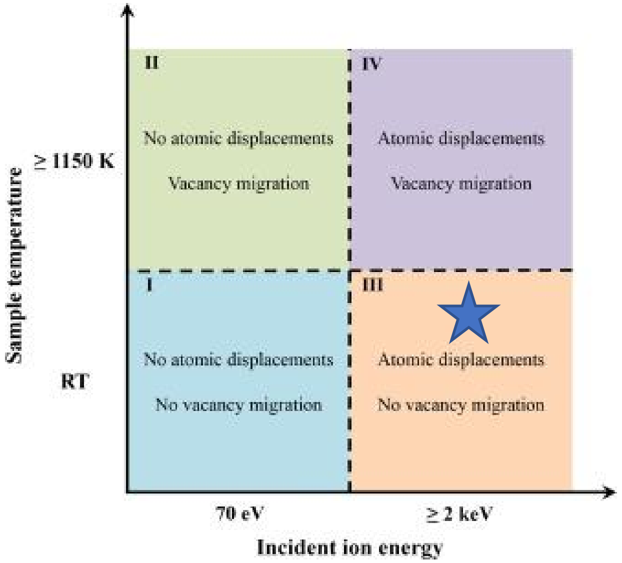

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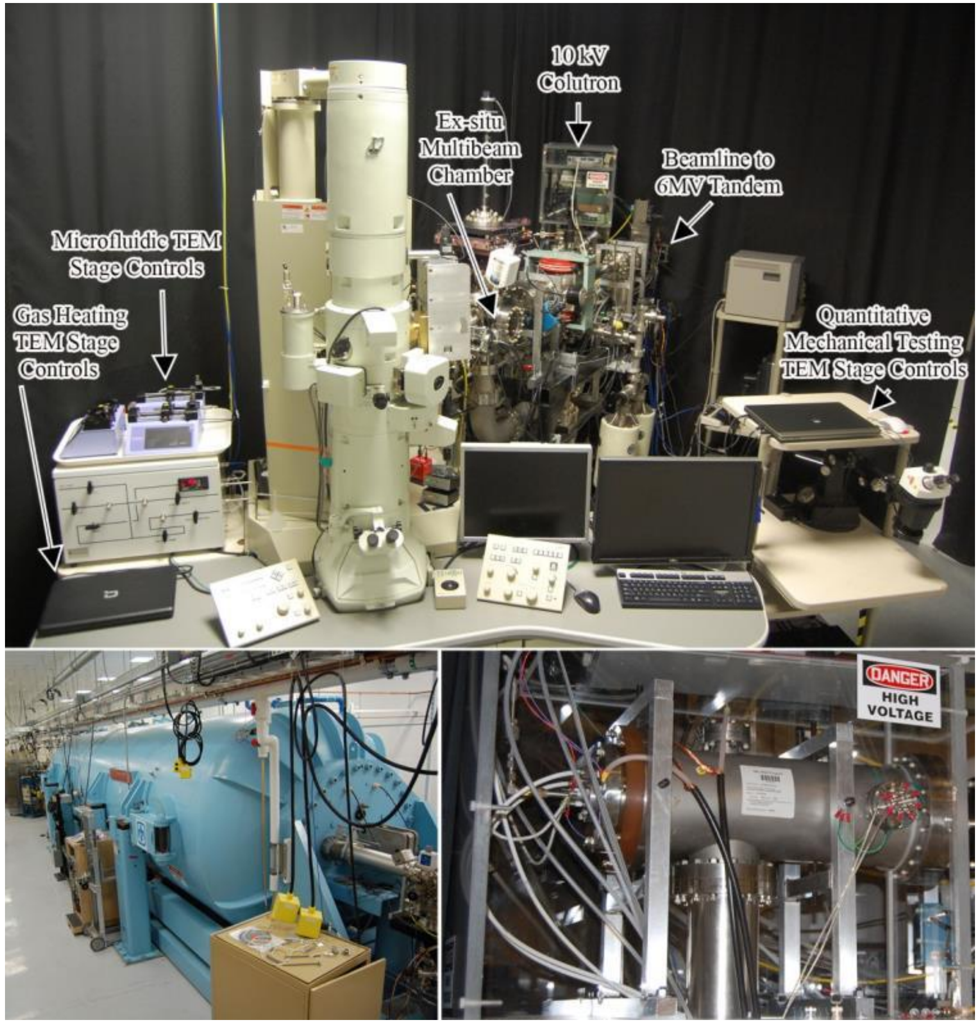
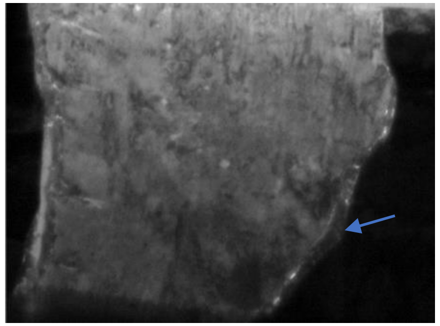
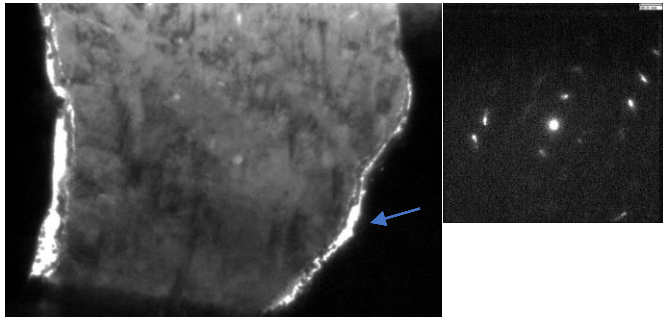


O. El-Atwani, JNM 2015

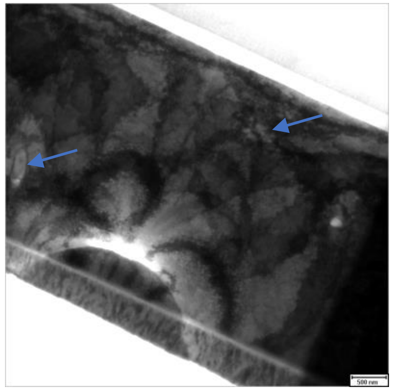
In-situ annealing up to 900 C shows dispersoid evolution at ~700 C

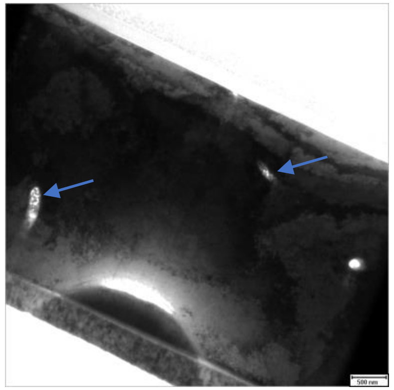
- ZrO_2-W interface delaminates at ~700 C
  - Maintains crystalline ZrO_2



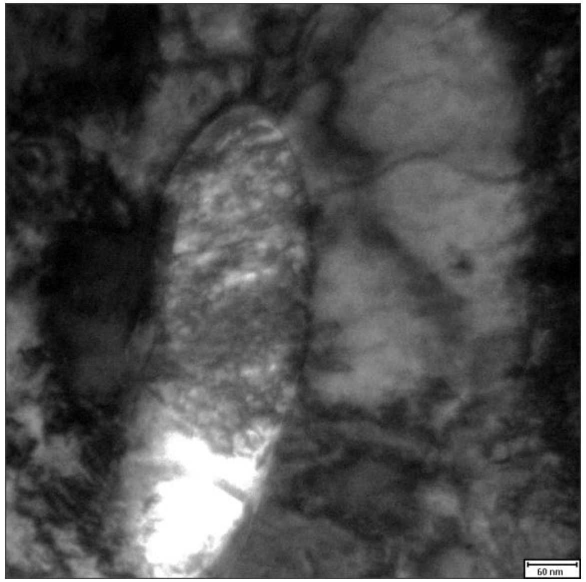


- NiFe precipitate melts at ~800 C

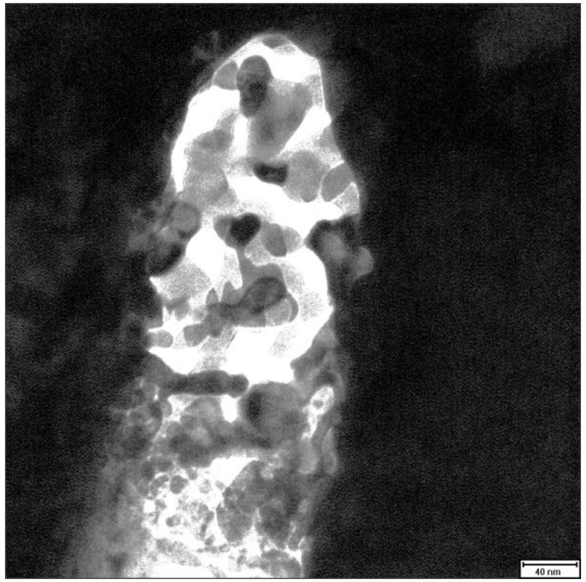




600 C



900 C



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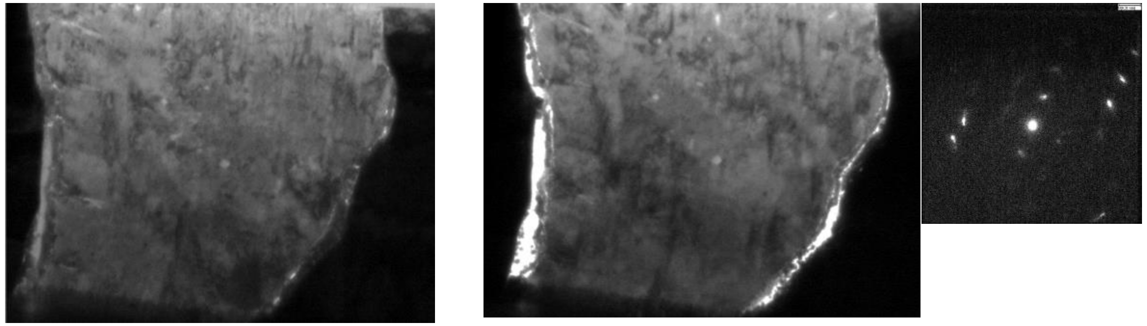

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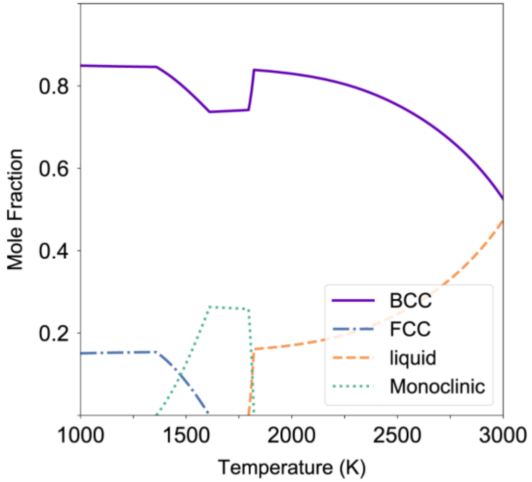
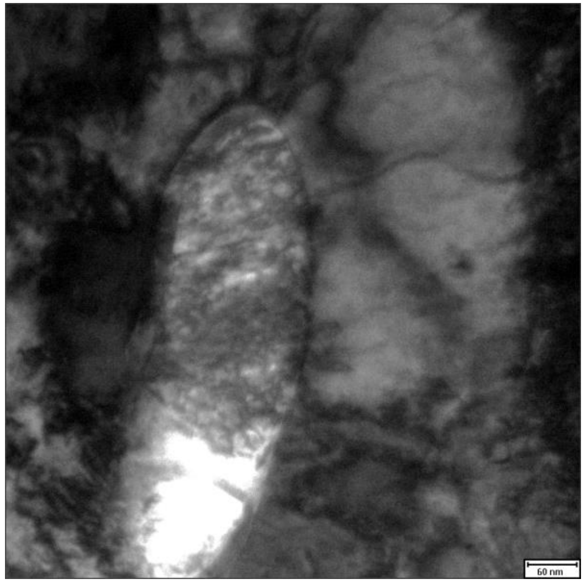
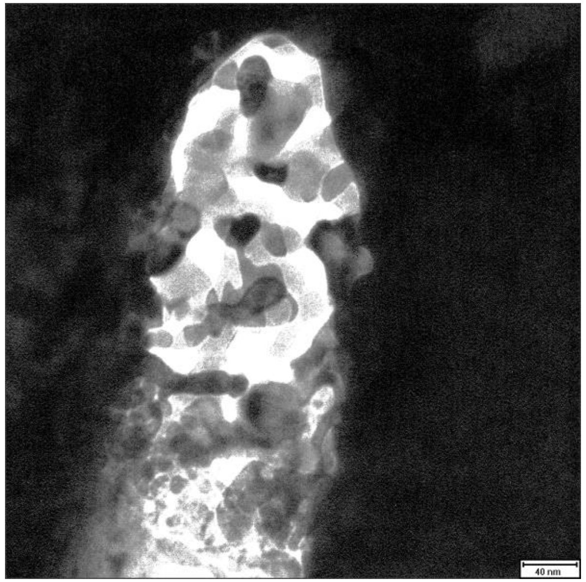


CALPHAD predicts a phase transformation at 1300K

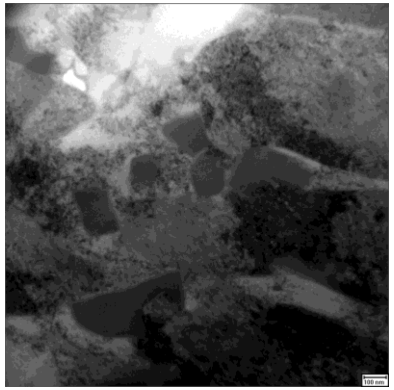
600 C



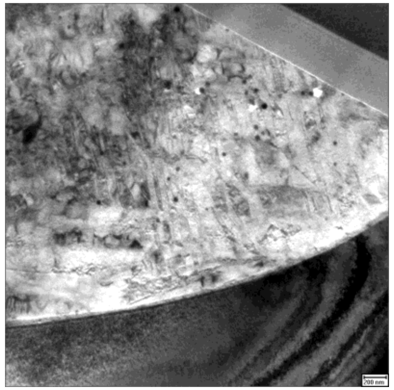
900 C



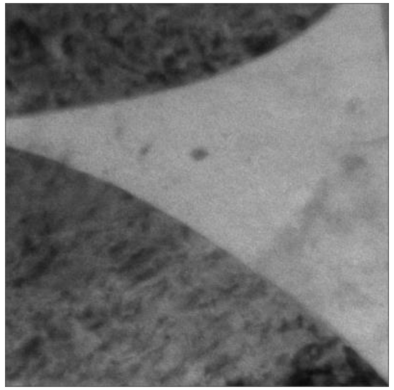
In-situ He irradiation at 650 C shows no accumulation of He bubbles



WNiFeZrC at 650 C Pre-Irradiation

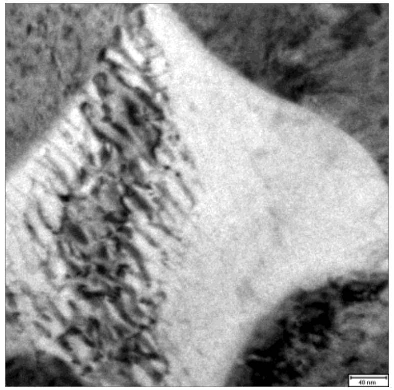


WZrC at 650 C Pre-Irradiation

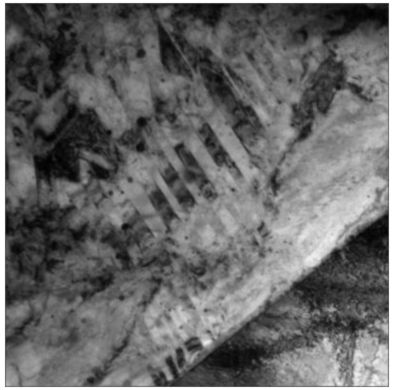


WNiFe at 650 C Pre-Irradiation

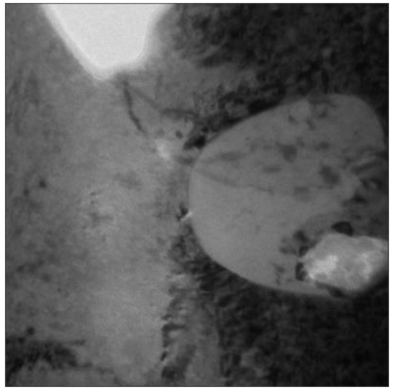
Post-Irradiation



Post-Irradiation



Post-Irradiation



Temperature limited to 650C to ensure clean interfaces – limited vacancy migration

10 keV He – do have displacement damage

No evidence of He bubbles following He irradiation

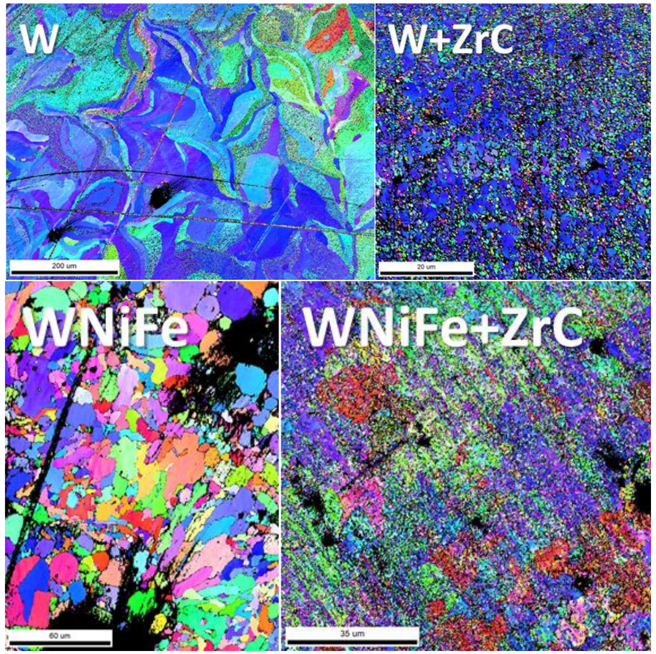
No evidence of He bubbles following 3 weeks of aging either

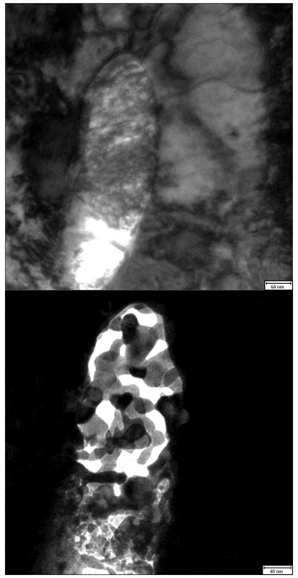
Conclusions and impact

- Alloyed tungsten may improve the irradiation performance in a fusion reactor
- Additive manufacturing represents a new design space for interrogating refractory alloy synthesis
- Dense W alloys with Ni, Fe, and ZrC fabricated via DED
- Thermal stability a current issue
- Design space preliminarily investigated, these materials show some promise

Future work

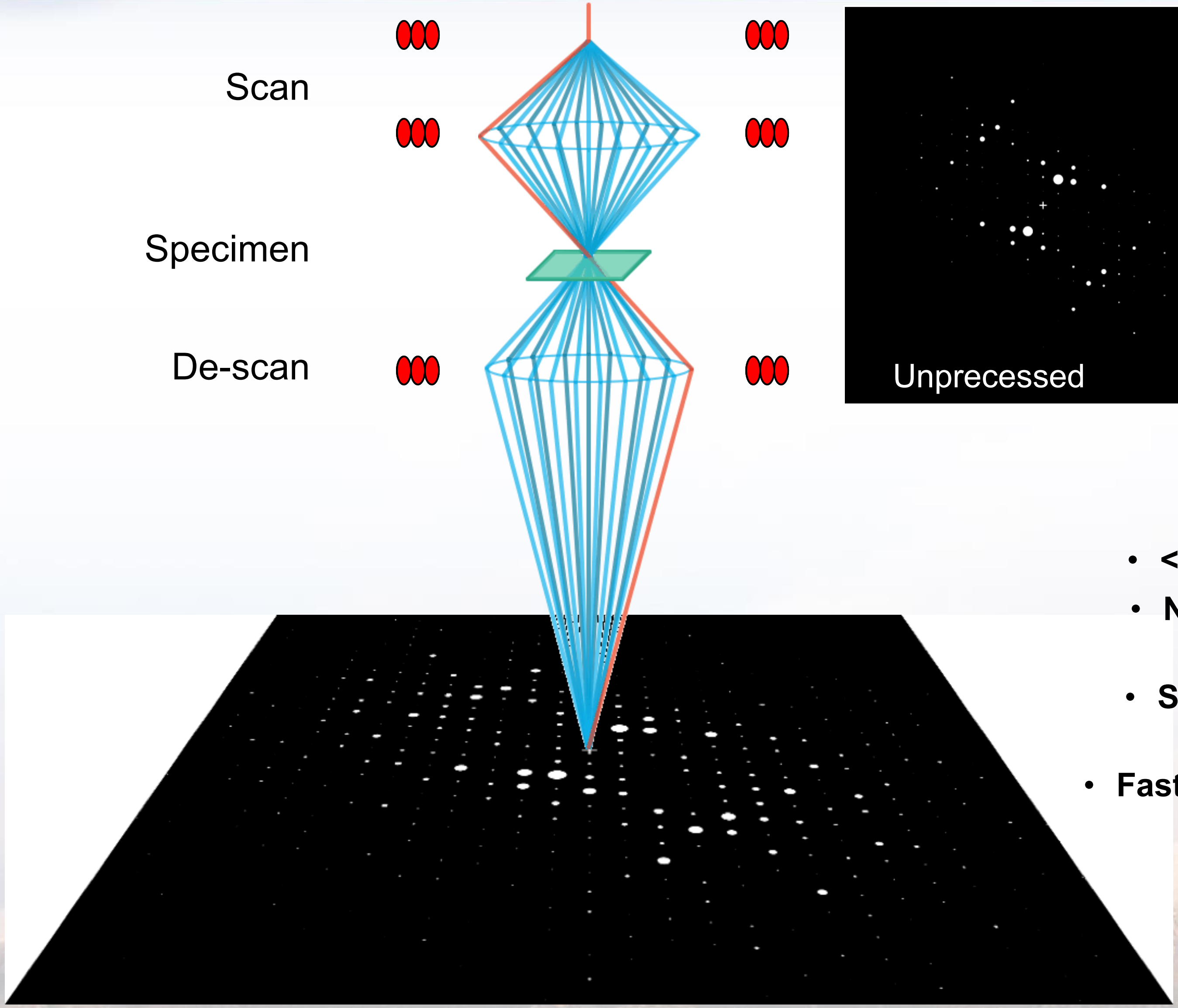
- Ex-situ irradiations and characterization to implant He at high fluence reactor-relevant conditions
- Thermal treatments
  - DSC characterization for impact of alloying on thermal conductivity
  - Recrystallization inhibition of alloyed materials

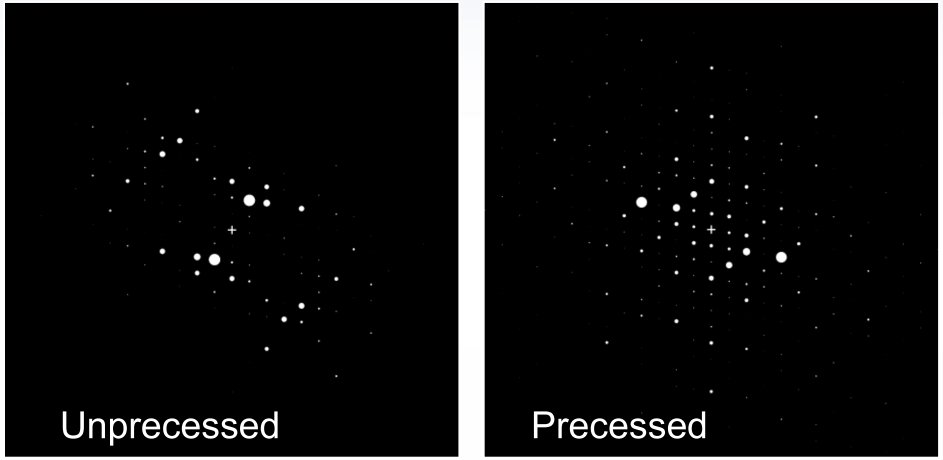




Precession Electron Diffraction (PED) Microscopy

Collaborators: K.J. Ganesh, S. Rajasekhara, & P.J. Ferreira





<u>Advantages</u>

- **< 10 nm spatial resolution**
- **Near kinematical electron diffraction**
- **Symmetry ambiguities are resolved**
- **Fast and automated acquisition**
  - **~200 grains in 15 min.**

Chris Own, PhD Dissertation, Northwestern University, 2004

Slide courtesy: K. J. Ganesh, K. Hattar