Processing and irradiation of multi-modal grain tungsten

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Outline

- Motivation for tungsten work at Purdue
- Processing of tungsten: SPS
- Irradiation of multimodal fine grain tungsten (powder metallurgy and severe plastic deformation techniques)
- Conclusions and future work

Tungsten as PMI material of choice

- Tungsten is preferred due to its high temperature properties and low sputter threshold
- Irradiation tolerance against bubble stability and surface morphology evolution (e.g. W fuzz[1]) require advances in tungsten-based surfaces
  - Coatings
  - Ultra-fine grain tungsten for radiation tolerance and possible enhanced ductility
  - Alloys
  - Nano-composites (dispersoids in microstructure)
- Scalability to real component dimensions needed in a fusion device places a burden on materials development (processing)
  - Nanocrystalline W possible with extrusion methods (e.g. EACP[1], SPD[2]), however scaling to large component dimensions is a challenge
  - Sintering becomes a possible pathway forward, however challenges exist in processing a large-scale, ultra-fine grained system with radiation tolerant properties

[1] Q. Wei, Acta Materialia, 54 (2006) 77
[2] K. Cho et al, ARL-RP-180, June 2007

Tungsten as PMI material of choice

- *Can SPS lead to nanocrystalline tungsten of high densities without dispersoids?*
- *How much are fine, ultrafine or nanocrystalline tungsten radiation tolerant?*
- *Is a smaller grain size preferred*?

SPS: processing of thin (~several-mm) PMI surfaces using W

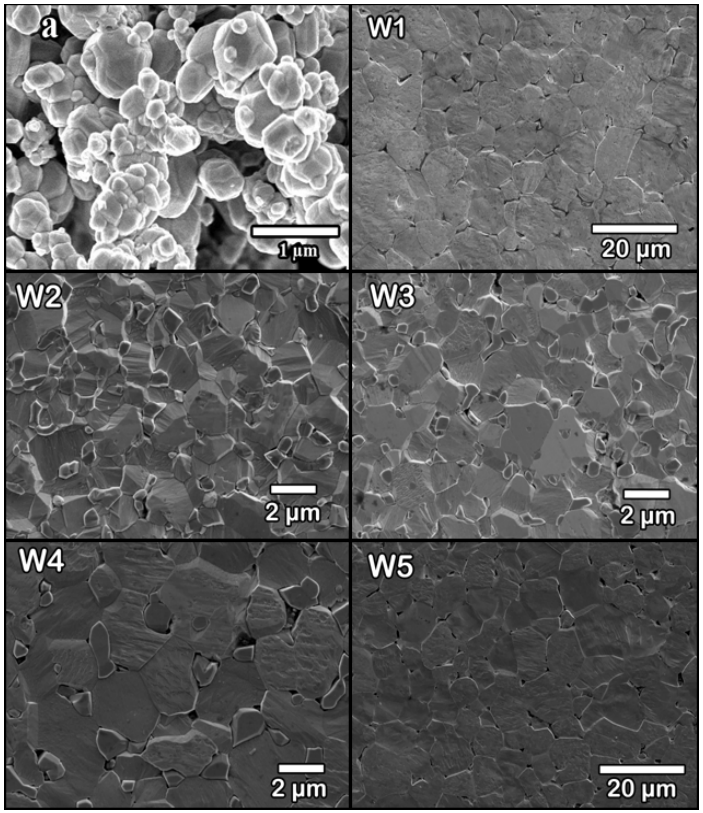
Sample	Temperature (C)	Pressure (MPa)	Time (min)	Relative density (%)	Vickers Hardness (kg/mm^2) ± Standard Deviation
W1	1800	90	1	90.80	231 ± 9.53
W2	1300	200	5	93.76	513 ± 13.1
W3	1300	266	5	93.16	523 ± 23.3
W4	1400	200	5	93.94	518 ± 14.2
W5	1500	200	5	94.30	341 ± 13.5
Bulk W					423 ± 17.6

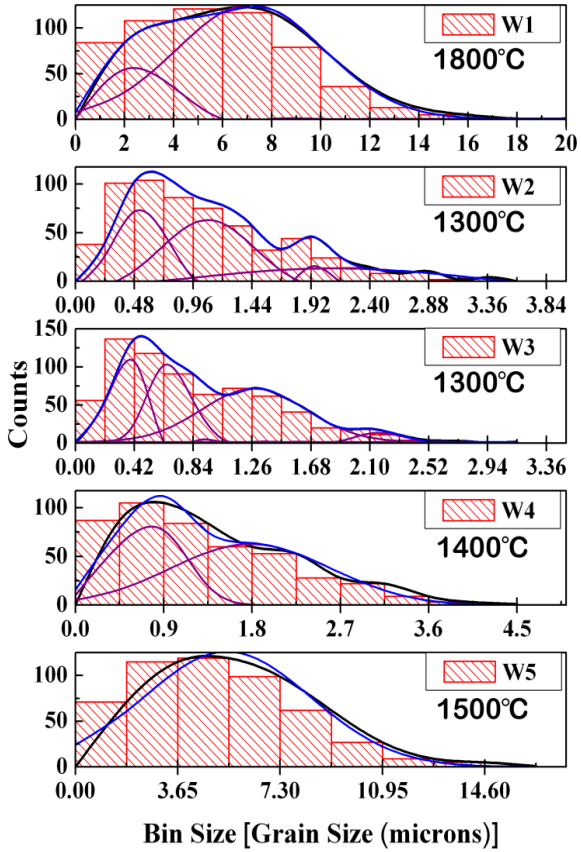
Average Particle Size Distribution	
Micron Range	Weight Percent
0-1	54.9
1-2	35.4
2-3	6.9
3-4	2.8

- Plasma activated sintering[1] pioneered by J. Groza is used for short-time high-temperature densification technique for tungsten
- Typical sintering times of hours reduced to 1-5 minutes leading to density values between 90-94% of theoretical density
- Various conditions of time, temperature and pressure used for processing dense sub-micron grain microstructures of high hardness

[1] Gwen Jones, Joanna Groza et al, Matl Manuf Proc, 9 (1994) 1105
[2] J. Groza, J.F. Shackelford, E.J. Laverna, M.T. Powers, Materials Processing Handbook, CRC Press 2007
3 O El-Atwani et al, Materials Science and Engineering A, under review

Studying the MMG microstructure of SPS-W





Average Particle Size Distribution	
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0-1	54.9
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3-4	2.8

Based on the SEM images, the morphology exhibits bimodal and multi-modal grain size distributions.

W3 at 1300 C is similar to initial powder distribution

As the sintering temperature is increased, the number of modes in the distribution decreases and the peaks shift to the right due to grain growth

- Hardness values in this study are up to 50% higher than those quoted in the literature for consolidated tungsten sintered by PAS[1],PPC[2] or SPS[3]

[1] Gwen Jones, Joanna Groza et al, Matl Manuf Proc, 9 (1994) 1105
[2] Yoo SH et al, Powder Metall.42(2), 181 (1999).
[3] Shearwood C, Ng HB. Proc. of SPIE Vol. 6798 67981B-1.

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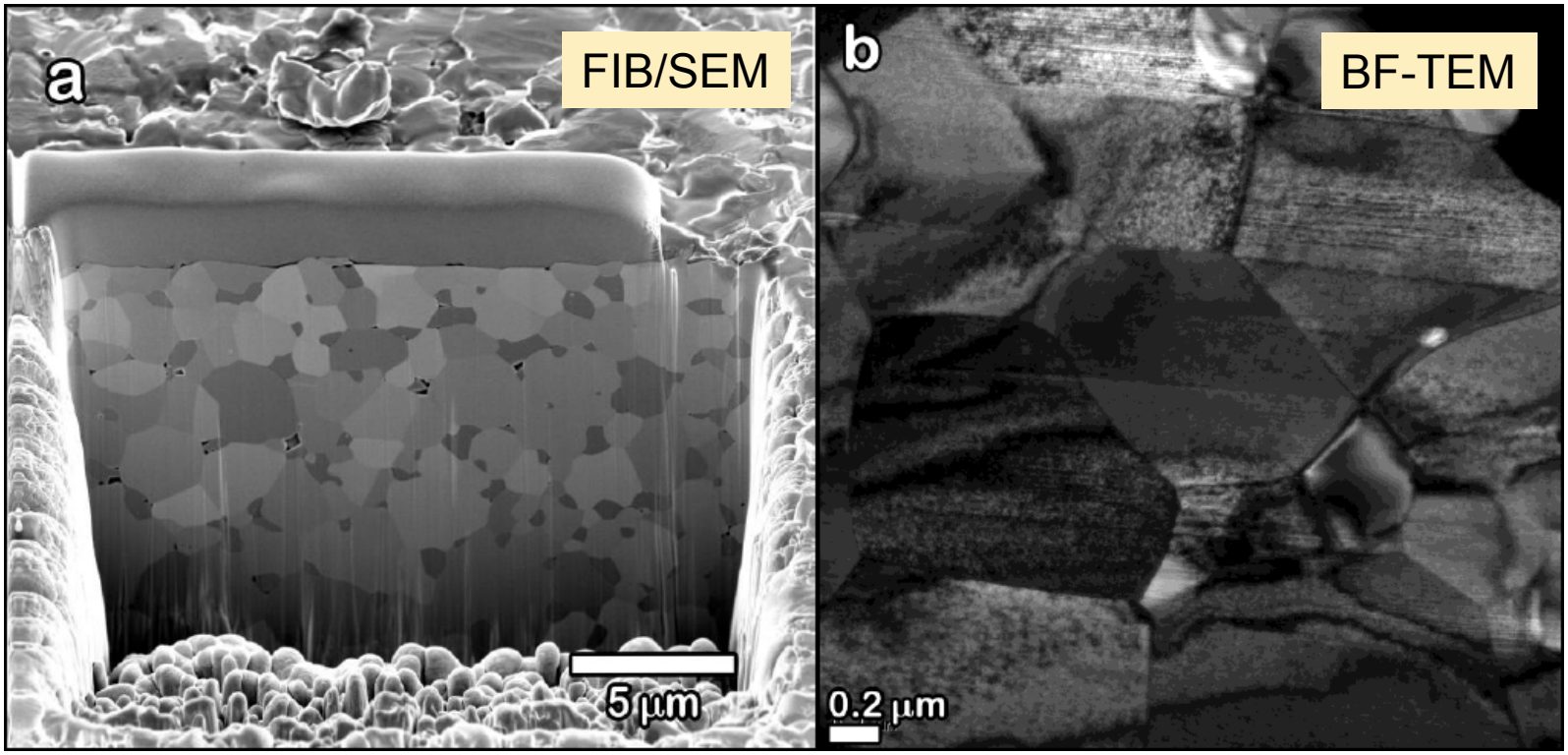
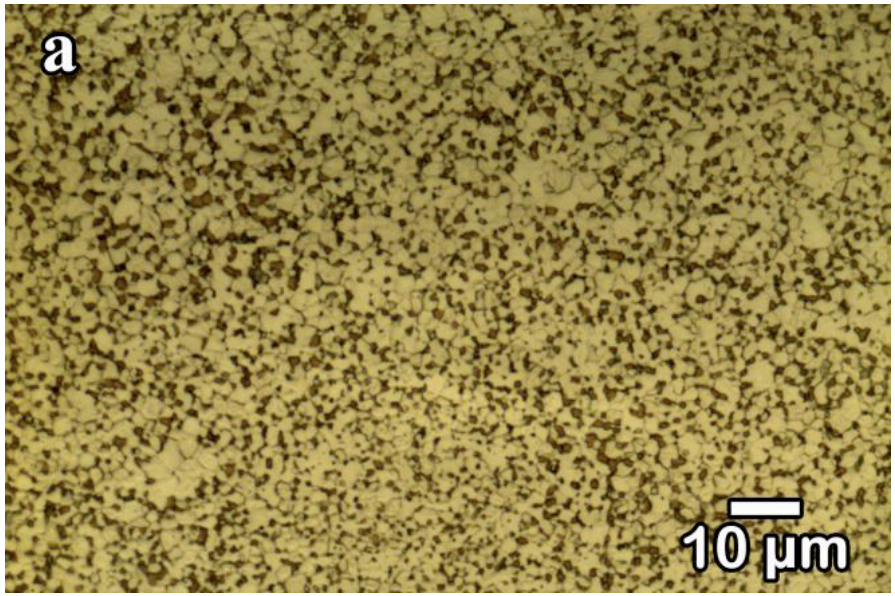
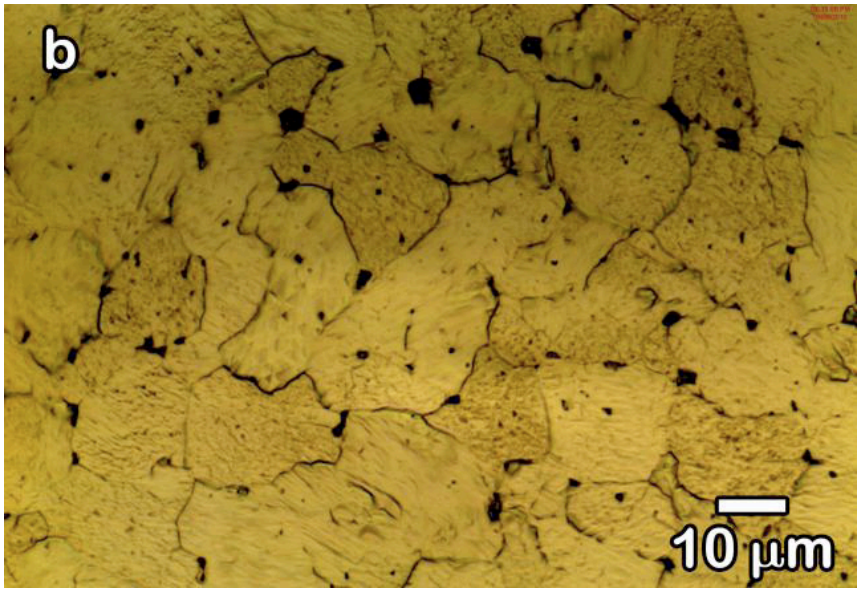
High-resolution microscopy of MMG-W



- (a) MMG-W sample (W2) cross section made with FIB/SEM and (b) Bright field TEM image showing small evidence of porosity
- The contrast between the grains (diffraction contrast TEM images) indicate that the grain boundaries are of high-angle.
- Multi-modal distribution observed is conjectured to be a result of sintering without significant grain growth

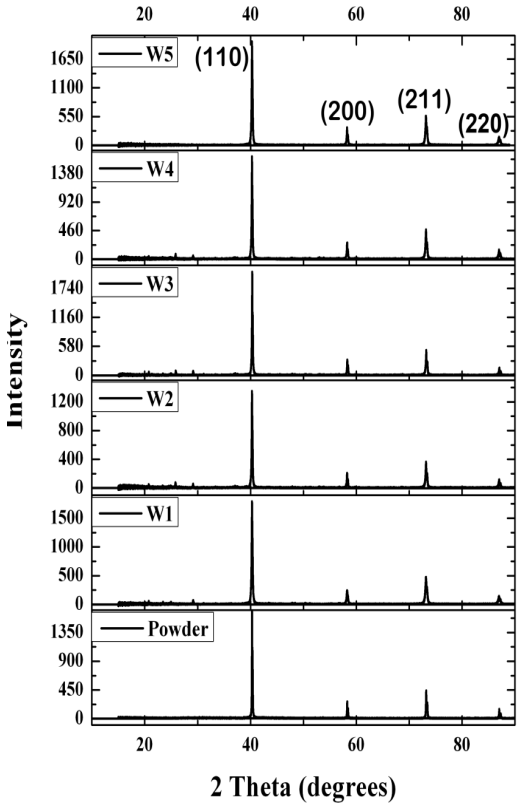
Comparison to commercial tungsten

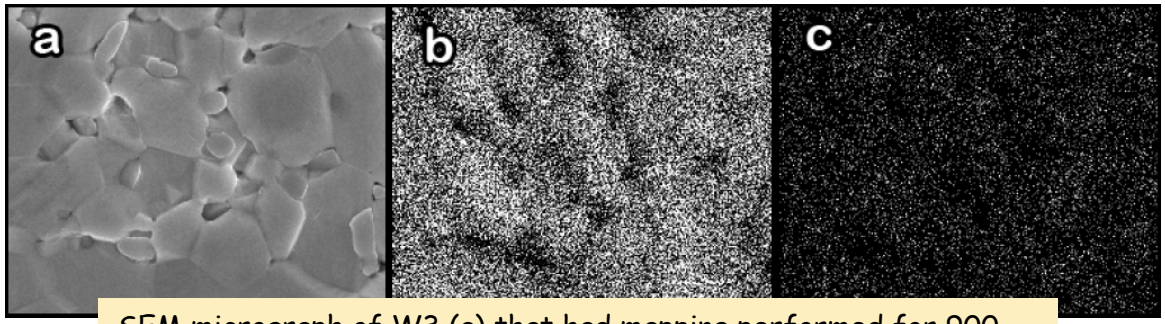




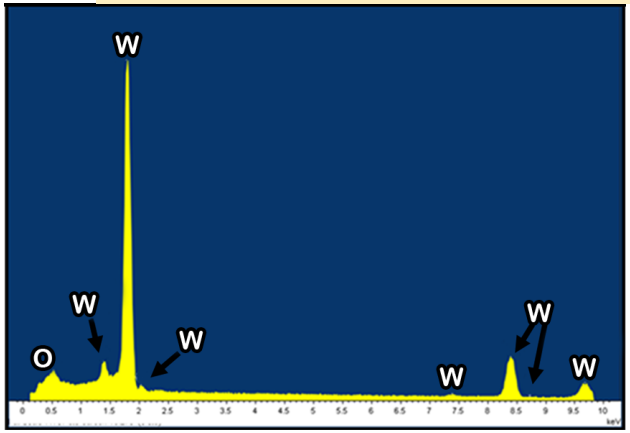
- Optical micrographs of W2 (a) and a commercial tungsten sample (b). W2 was sintered at 1300°C, a pressure of 200 MPa applied, and for 5 minutes.
- Processing tungsten with SPS affects the grain size of tungsten.
- Commercial tungsten microstructure likely influenced by both impurities and slow processing time using conventional sintering techniques

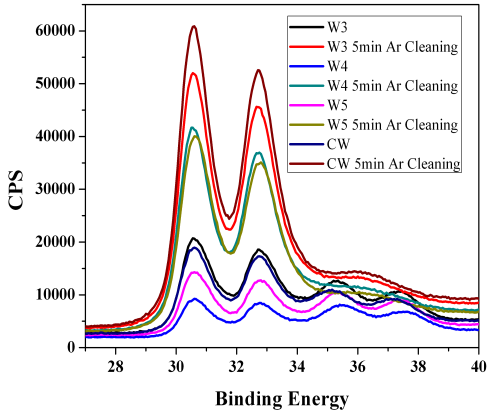
Controlling impurities in MMG-W





SEM micrograph of W3 (a) that had mapping performed for 900 seconds. The results can be seen in (b) and (c). (b) shows the Tungsten on the surface and the dots in (c) represent oxygen and noise from the system.





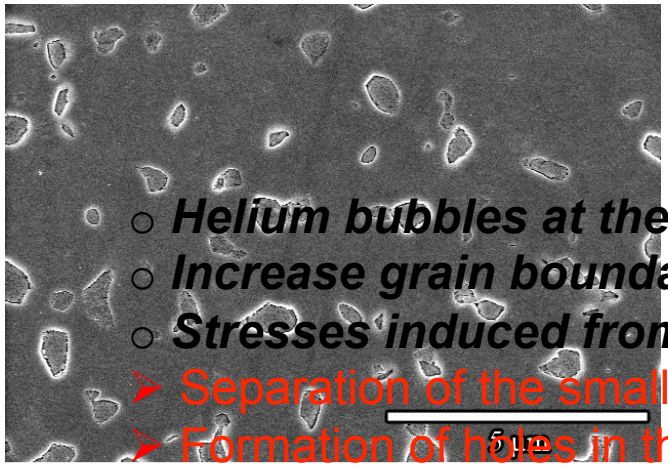
- Work by K. Cho et al[1], conjectures that grain refinement, depletion, redistribution of pre-existing impurities segregated along the GBs are the critical aspects for inducing flow softening while improving the material ductility.
- XRD, EDX, XPS show MMG-W indicate low amounts of oxide or other metal impurities

[1] K. Cho et al, ARL-RP-180, June 2007

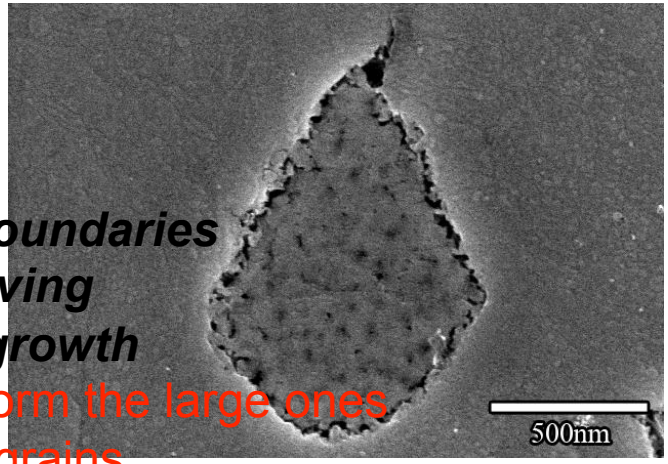
Irradiation of multimodal fine and ultrafine tungsten 1

Irradiation with helium ($1X10^{22}$ ion.m^{-2})
Helium energy = 200eV (no displacement damage)
Temperature = 600 C (no thermal vacancy migration)

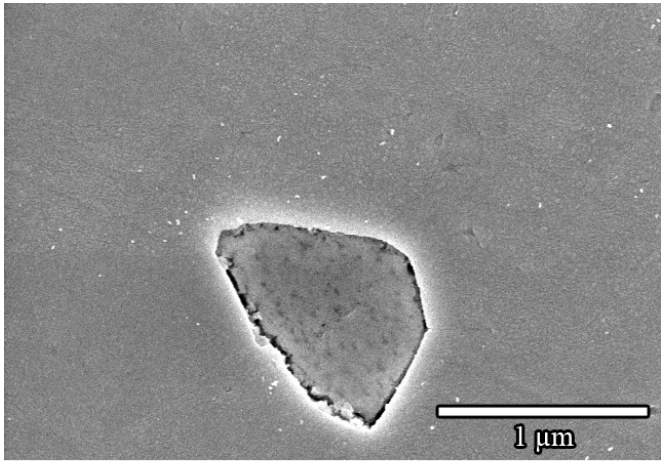
W3



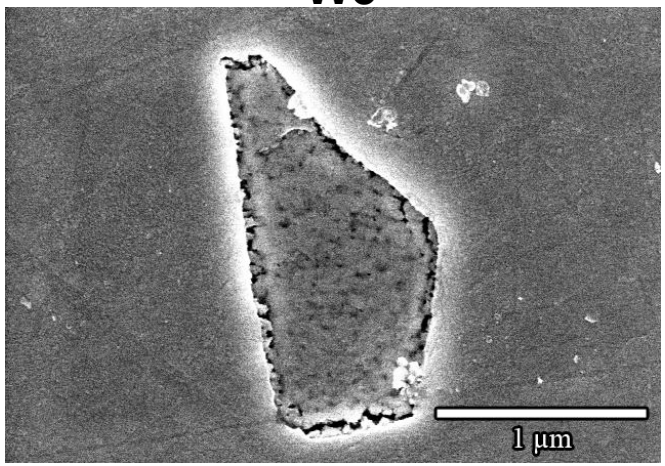
W4



- ***Helium bubbles at the grain boundaries***
- ***Increase grain boundary grooving***
- ***Stresses induced from grain growth***
  - Separation of the small grains form the large ones
  - Formation of holes in the small grains

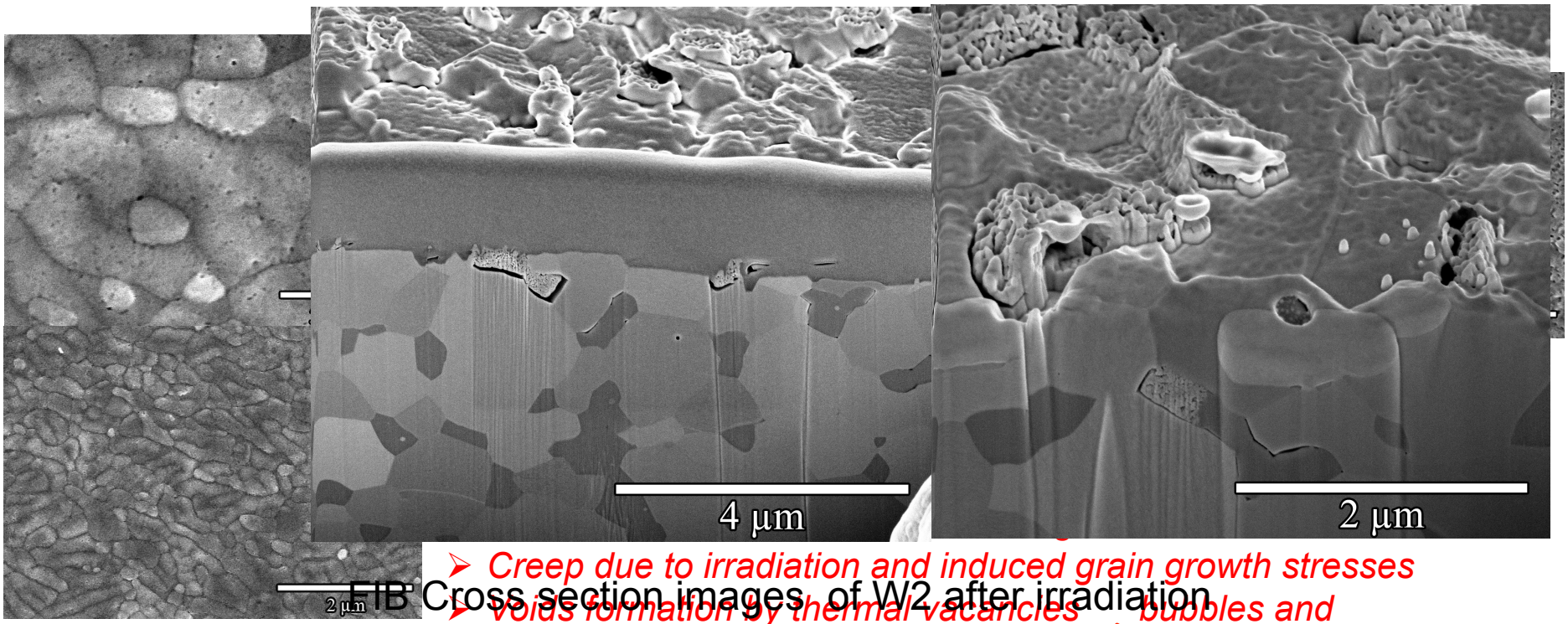


W5



Irradiation of multimodal fine and ultrafine tungsten 2

Irradiation with helium ($1X10^{22}$ ion.m^{-2})
Helium energy = 200eV (no displacement damage)
Temperature = 950 C (both thermal vacancy interstitial migration are possible)



FIB Cross section images of W2 after irradiation

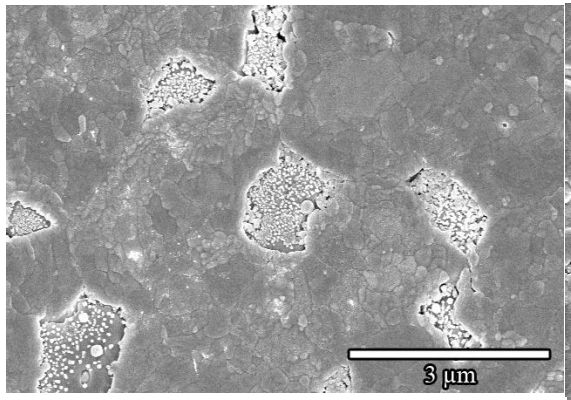
Commercial tungsten

- *Creep due to irradiation and induced grain growth stresses*
- *Voids formation by thermal vacancies → bubbles and blistering*
- *Grain boundaries should lead to high radiation tolerance [1] ,thus, separation of small grains (no grain boundaries) lead to less radiation tolerance (more bubbles and nanostructure formation and low fluencies*

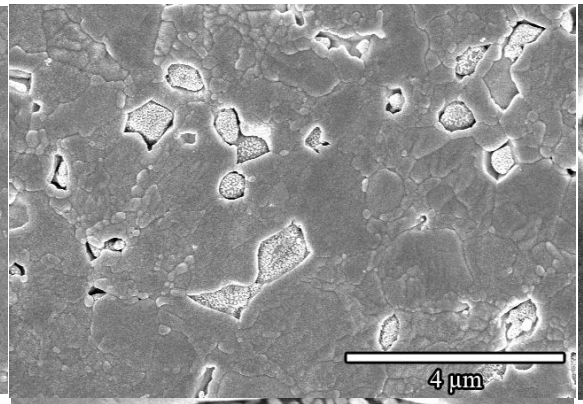
Xian-Ming Bai et al, SCIENCE VOL 327 26 MARCH 2010

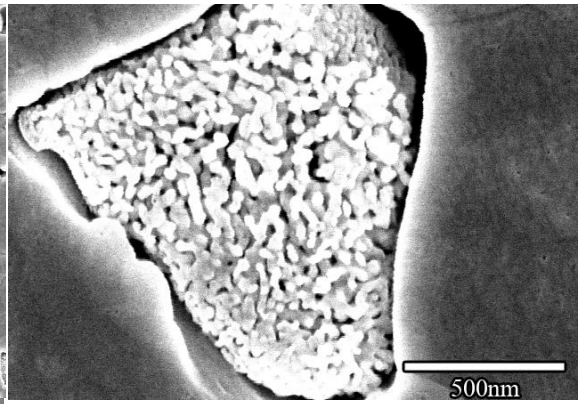
Irradiation of multimodal fine and ultrafine tungsten 3

Irradiation with helium ($1X10^{21}$ ion.m^{-2})
Helium energy = 200eV (no displacement damage)
Temperature = 950 C (both thermal vacancy interstitial migration are possible)

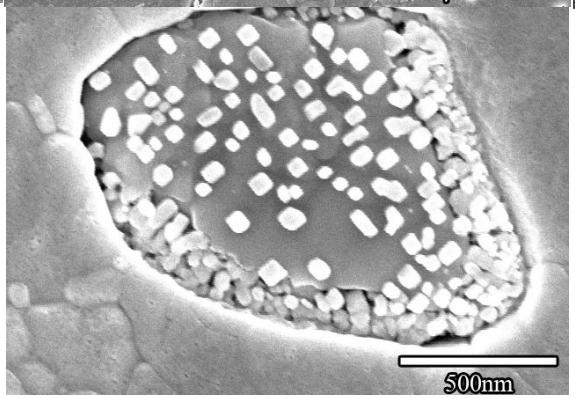


W3





W3



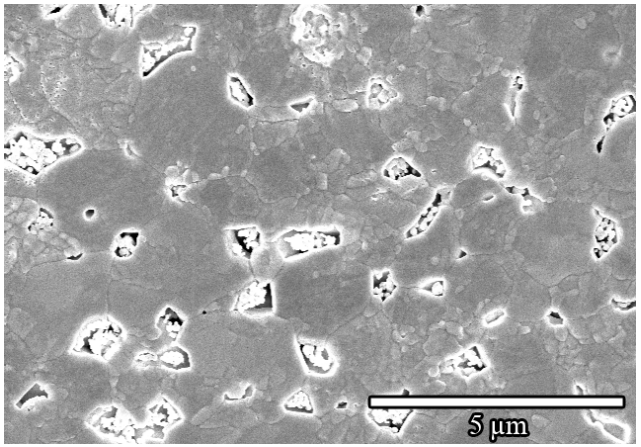
W3

- **Separation of grains in the first step (low fluence)**
- **Structure nucleation on the small grains due to bubble formation [1]**
- **Low blistering**

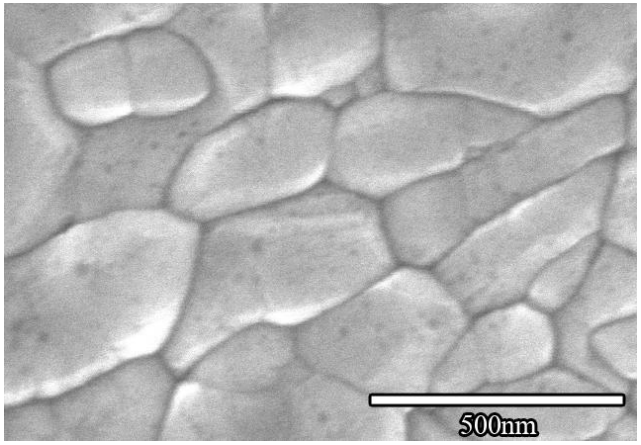
Kajita et al, Nucl. Fusion 49 (2009)

Irradiation of multimodal fine and ultrafine tungsten 4

Irradiation with helium ($5X10^{21}$ ion.m^{-2})
Helium energy = 200eV (no displacement damage)
Temperature = 950 C (both thermal vacancy interstitial migration are possible)



W3



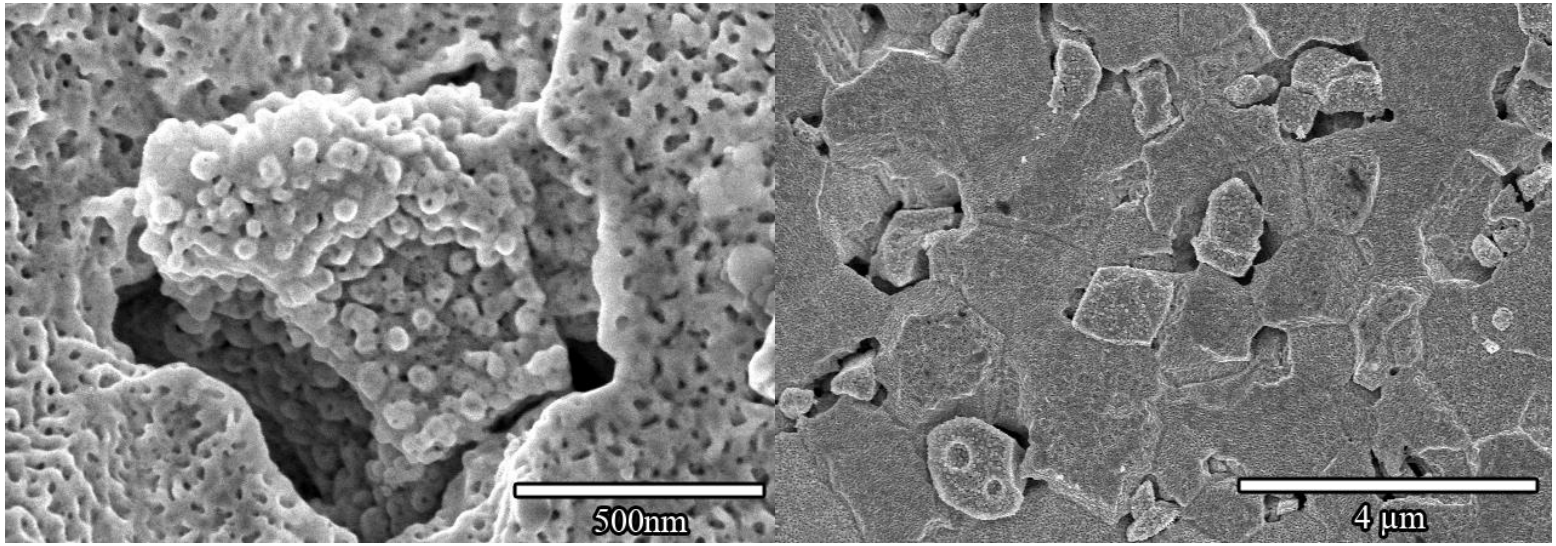
W4

- **Damage to the small grains**
- **Large blistering, bubbles coalescence (oriented in some cases)**

Same results were obtained at 50eV

Irradiation of multimodal fine and ultrafine tungsten 5

Irradiation with helium ($1X10^{22}$ ion.m^{-2})
Helium energy = 200eV (no displacement damage)
Temperature = 950 C (both thermal vacancy interstitial migration are possible)



W3 etched then irradiated

- **Structures on the small grains**
- **more pores on the surface**
- **increased gb grooving**

Sequenced process for TEM samples

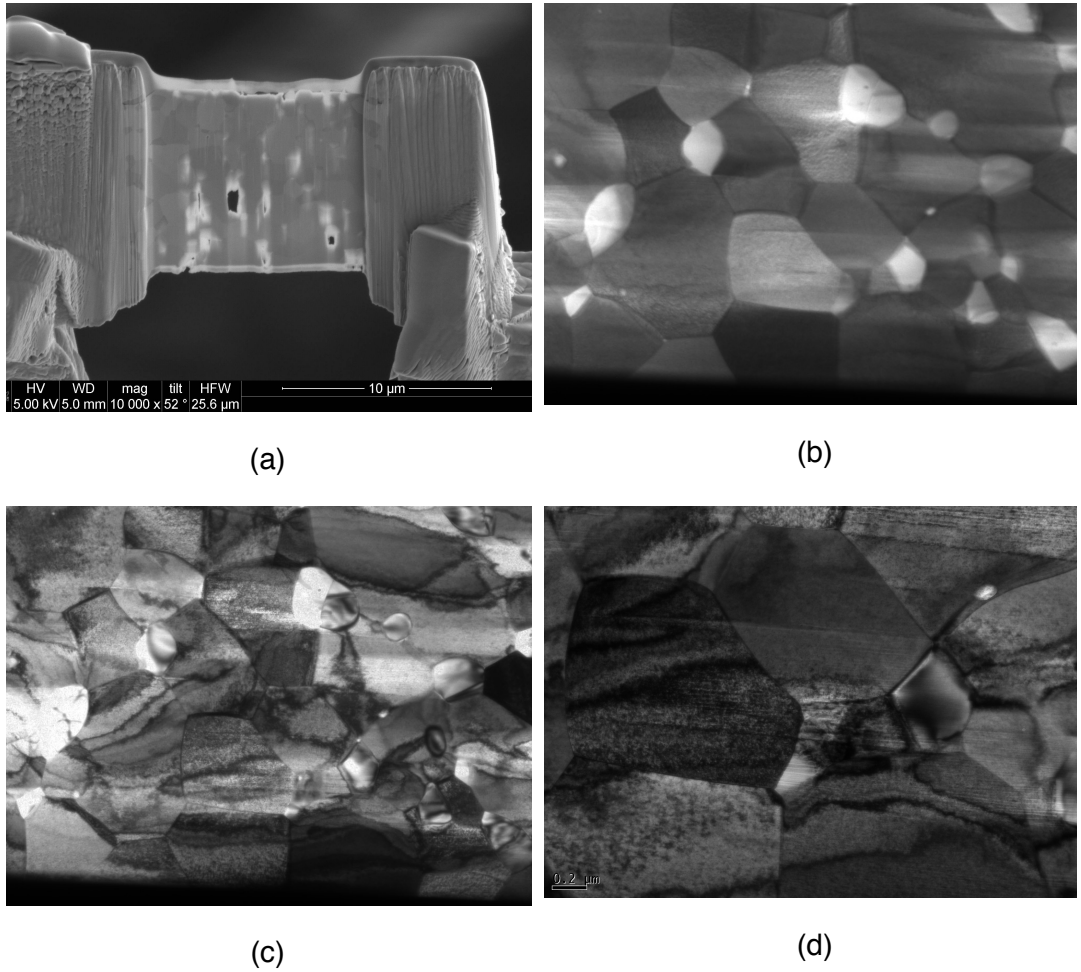


Fig. 2.12. Sequenced process of preparing TEM tungsten samples for nanoscale damage analysis and processing characterization. (a) The sample preparation of tungsten is tedious and must be reduced to a very thin (~ 100 nm) sample anchored on 10-um tungsten structures.

TEM and hardness results

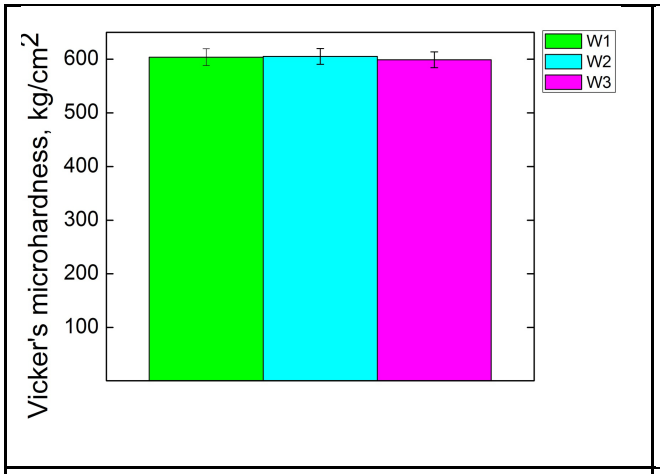


Fig. 3.4 (a). Vickers microhardness data of three tungsten SPS samples synthesized as described in above section.

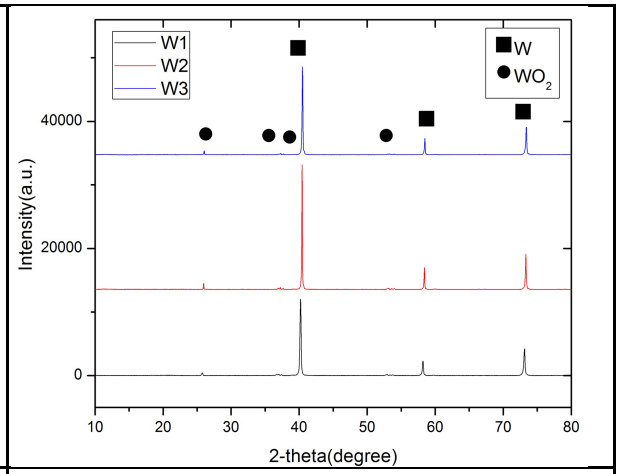


Fig. 3.4 (b). X-ray diffraction data of three tungsten SPS samples synthesized as described in above section.

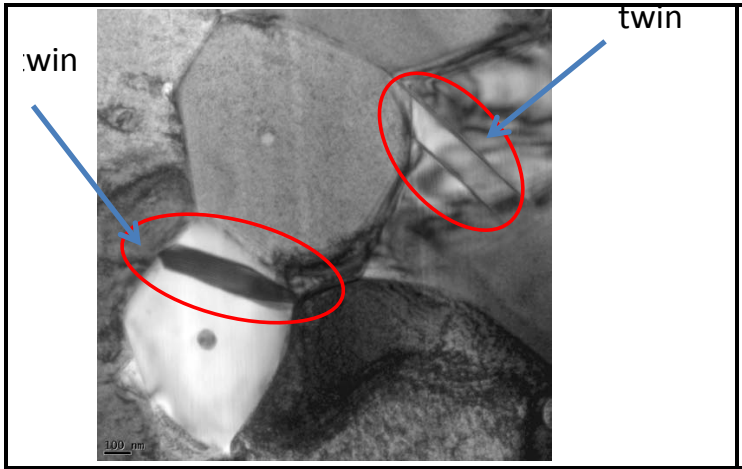


Fig. 3.8 (a). TEM micrograph near the small grains showing these grains are twinned.

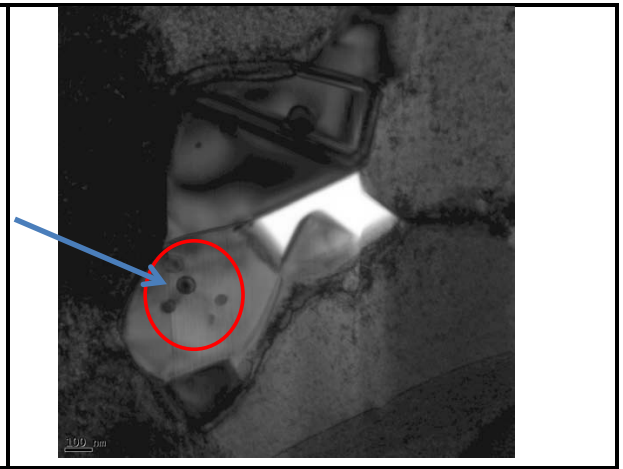
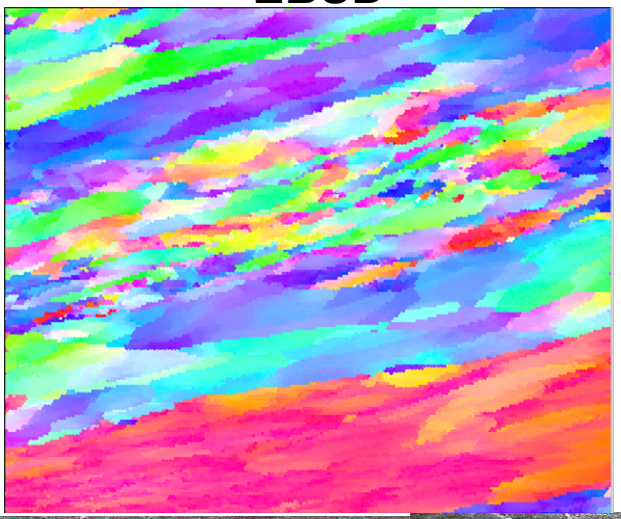


Fig. 3.8 (b). TEM image with signs of twinned grains.

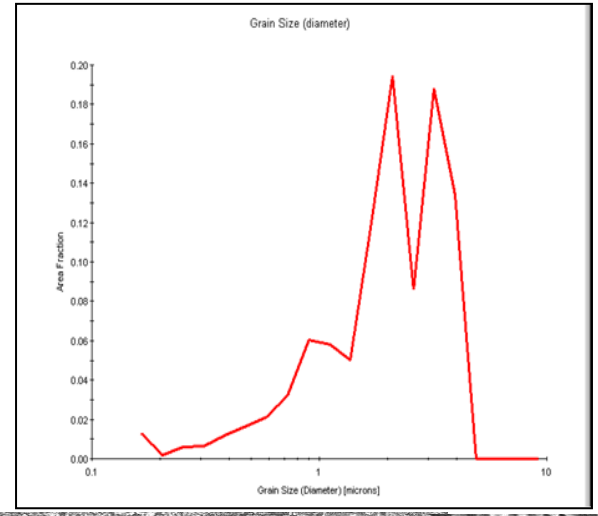
Irradiation of SPD tungsten sample

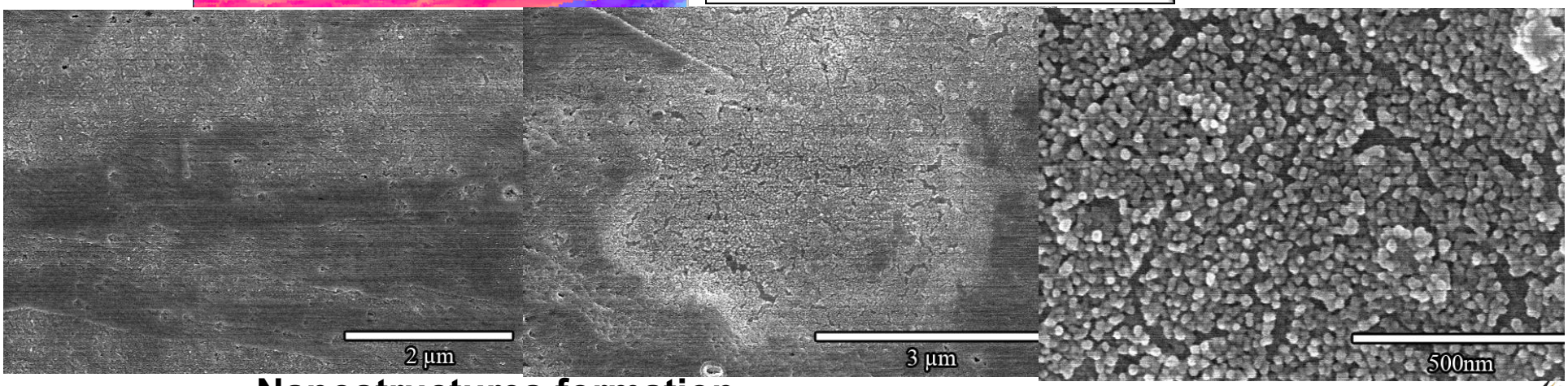
Irradiation with helium ($2X10^{22}$ ion.m^{-2})
Helium energy = 200eV (no displacement damage)
Temperature = 950 C (both thermal vacancy interstitial migration are possible)

EBSD



Size profile





- **Nanostructures formation**
- **Role of dislocations**

EBSD: electron backscatter diffraction

Summary

- Multimodal grain size distribution leads to high hardness
- Modification and irradiation-induced structure was found to dominate on the small grains than on large grains on multimodal distribution tungsten
- As the grain size decreases, blistering decreases
- Dislocations may play a role on structure formation on SPD samples
- Erosion *in-situ* studies are planned to correlate with nano and micro-structure behavior
- More investigation on radiation tolerance as a function of grain size (In-Situ TEM) is planned

Thanks for your attention
Questions??