

Progress in advanced sintering of ultrafine and nanocomposite tungsten-based PMI coatings

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Outline

- Motivation for tungsten work at Purdue
- Processing of tungsten: SPS
- Results of multi-modal fine-grained W
- Future work
- Conclusions

Tungsten as PMI material of choice

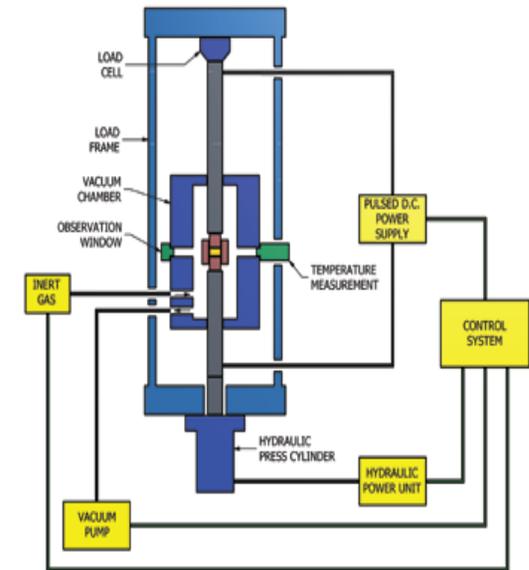
- Tungsten is preferred due to its high temperature properties and low sputter threshold
- Irradiation tolerance against bubble stability and surface morphology evolution (e.g. W fuzz¹) require advances in tungsten-based surfaces
 - Coatings
 - Ultra-fine grain tungsten for radiation tolerance and possible 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. EACP¹, SPD²), 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

¹Q. Wei, Acta Materialia, 54 (2006) 77

²K. Cho et al, ARL-RP-180, June 2007

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

Sample	Temperature (C)	Pressure (MPa)	Time (min)	Density (g/cm ³)	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

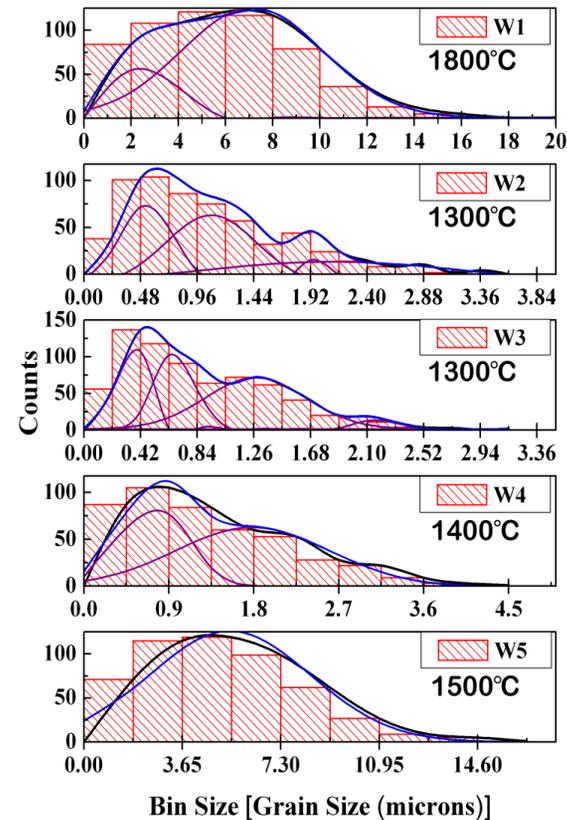
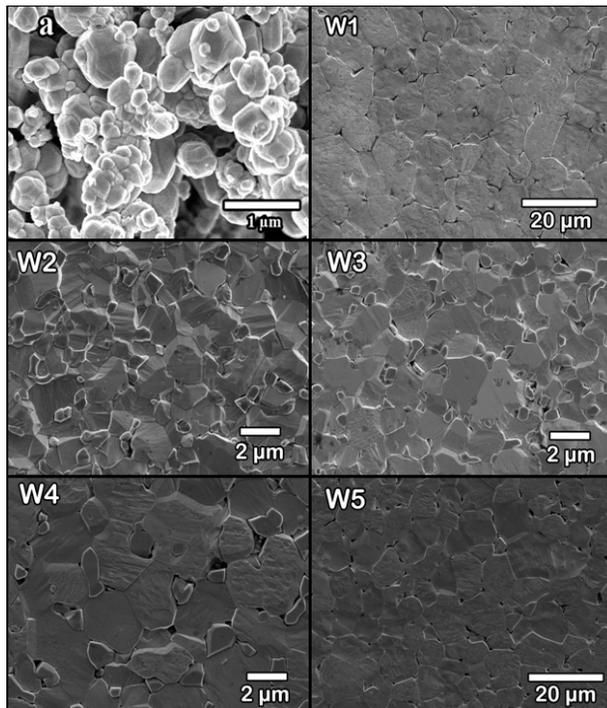


- Plasma activated sintering¹ pioneered by J. Groza is used for short-time high-temperature densification technique for tungsten
- Typical sintering times of hours reduced to 1-5 minutes leading to density values between 90-94% of theoretical density
- Various conditions of time, temperature and pressure used for processing highly-dense sub-micron grain microstructures

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

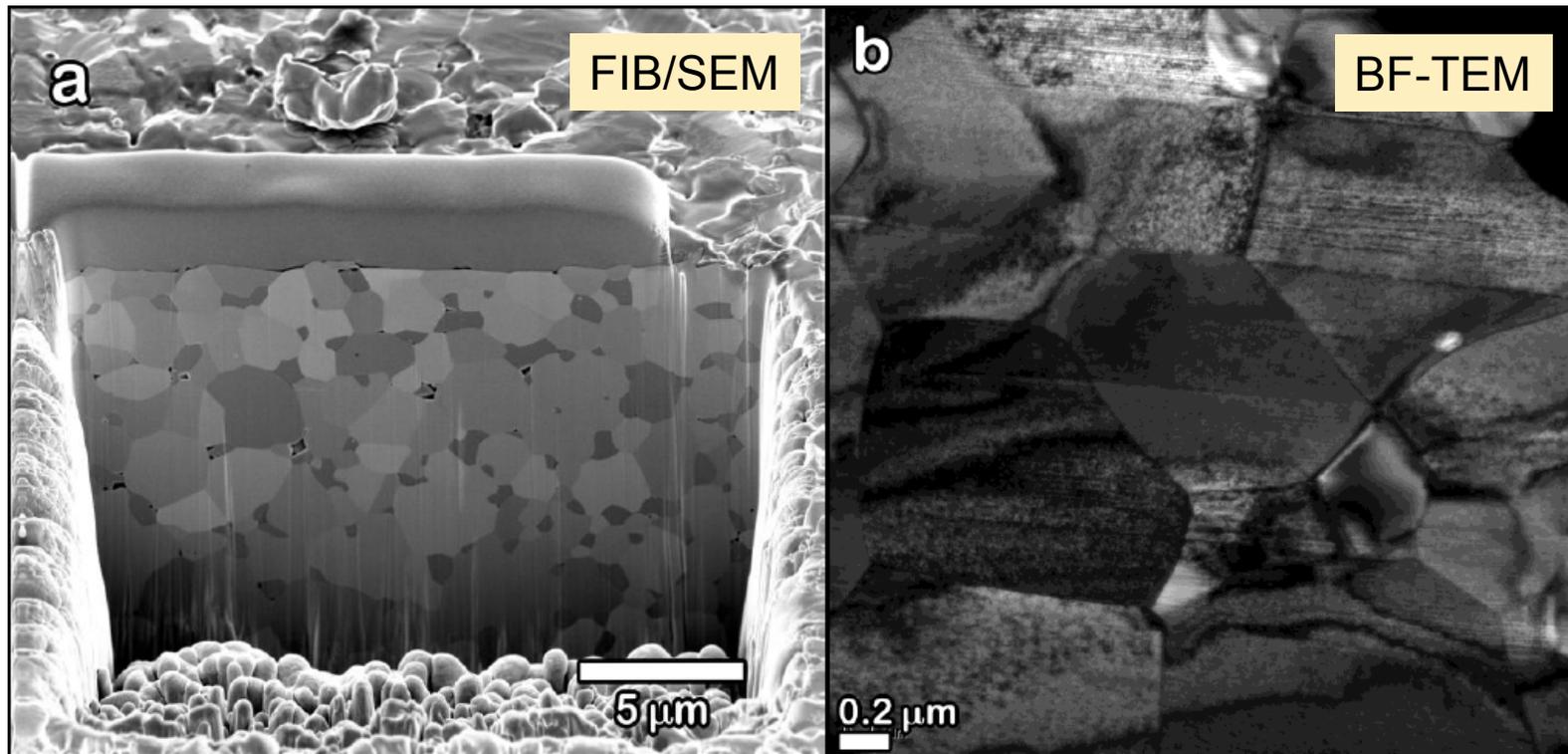
Studying the MMG microstructure of SPS-W



- 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^{1,2}

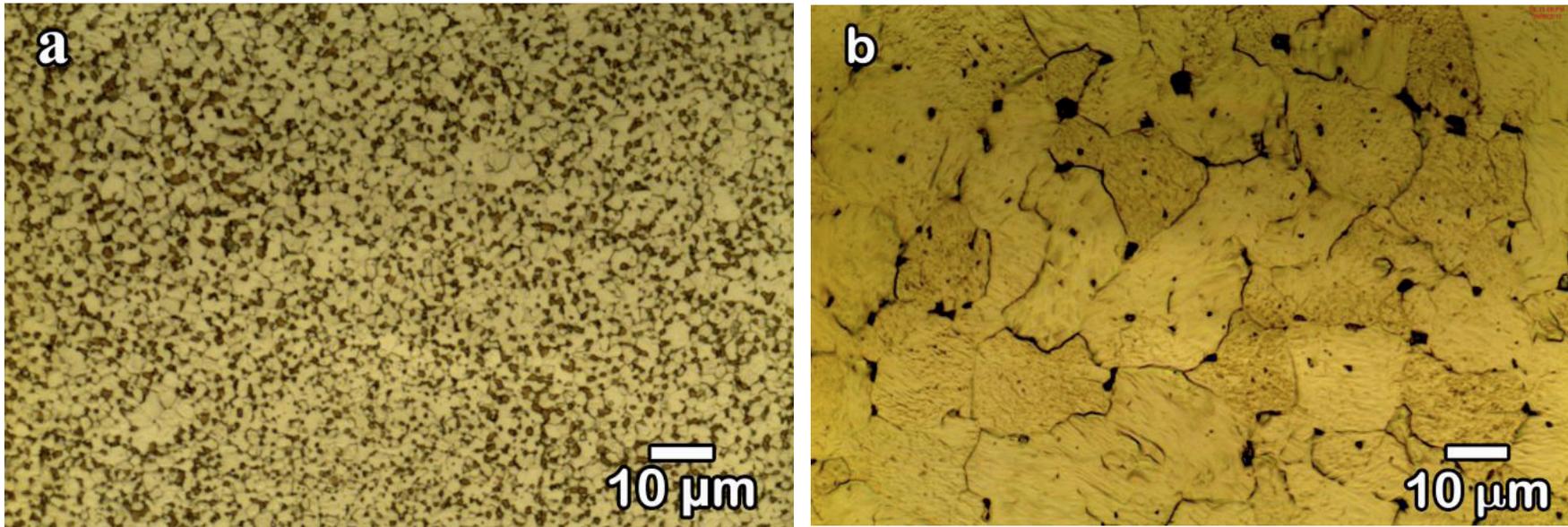
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High-resolution microscopy of MMG-W



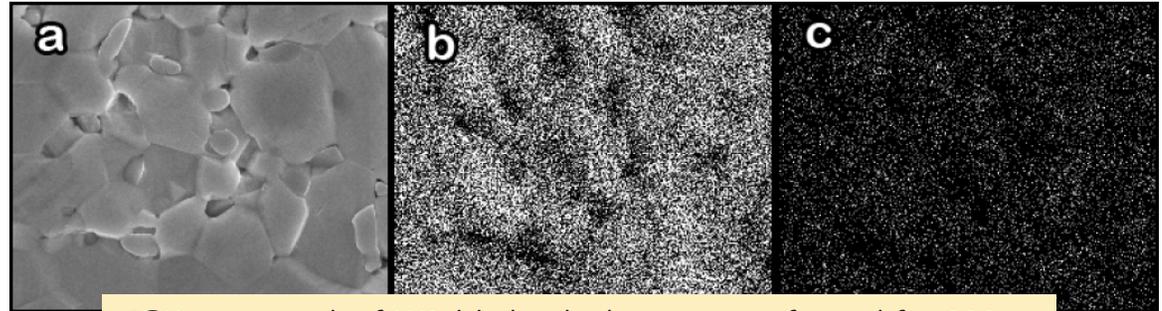
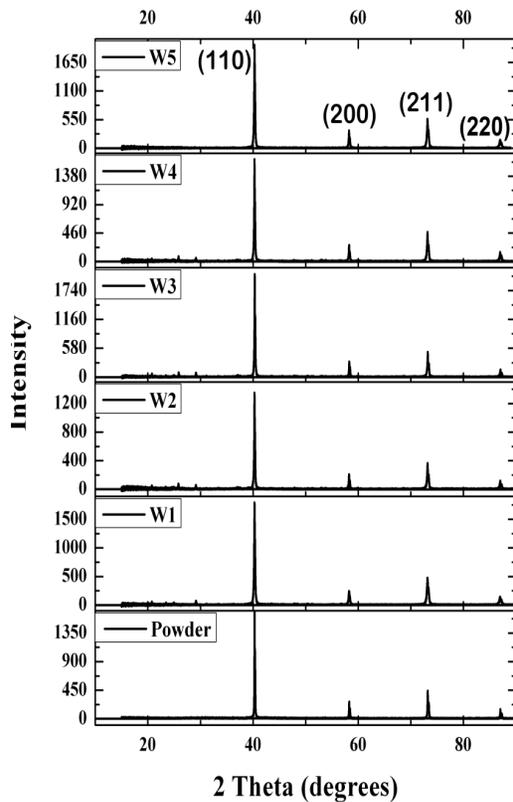
- (a) MMG-W sample (W2) cross section made with FIB/SEM and (b) Bright field TEM image showing small evidence of porosity
- The contrast between the grains (channeling contrast and diffraction contrast in the FIB and TEM images respectively) indicate that the grain boundaries are of high-angle.
- Multi-modal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

Comparison to commercial tungsten

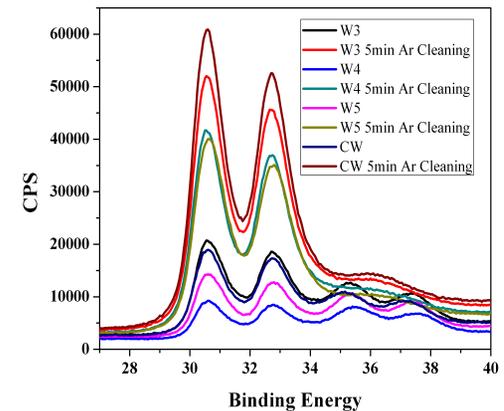
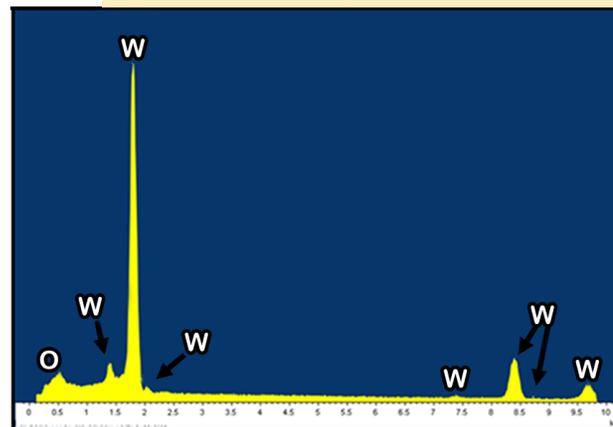


- Optical micrographs of W2 (a) and a commercial tungsten sample (b). W2 was sintered at 1300°C, a pressure of 200 MPa applied, and for 5 minutes.
- Processing tungsten with SPS affects the grain size of tungsten.
- Commercial tungsten microstructure likely influenced by both impurities and slow processing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

Controlling impurities in MMG-W



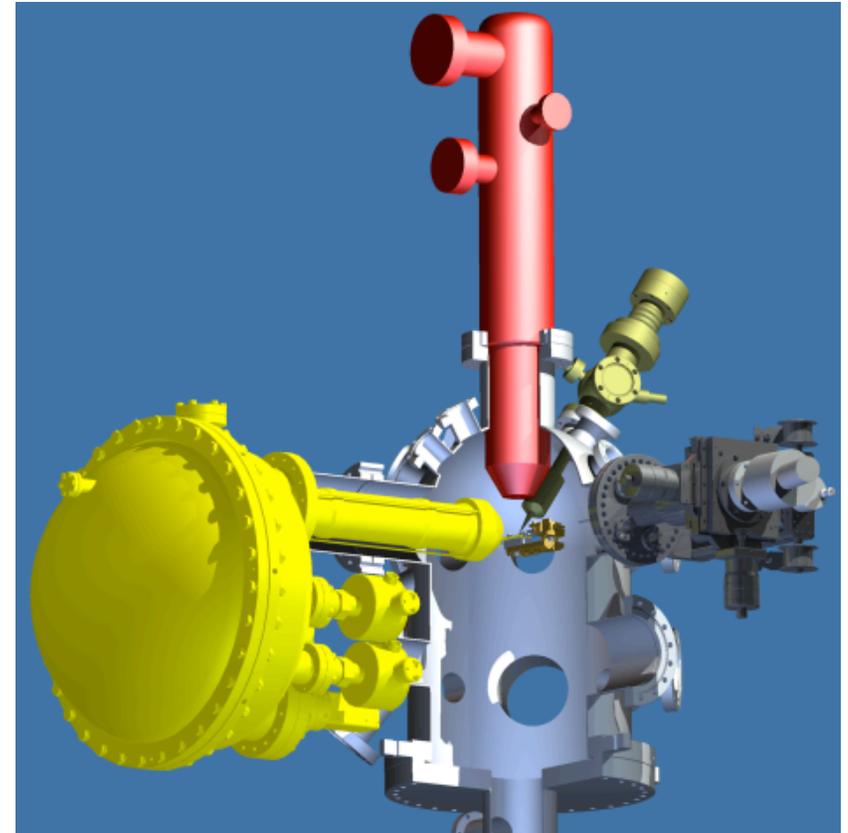
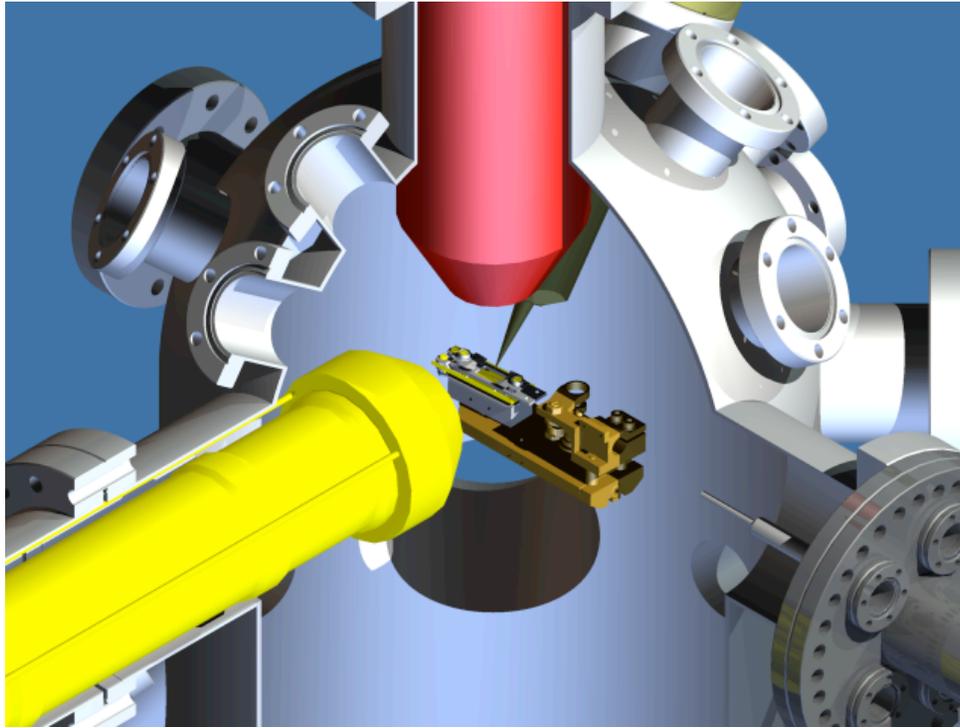
SEM micrograph of W3 (a) that had mapping performed for 900 seconds. The results can be seen in (b) and (c). (b) shows the Tungsten on the surface and the dots in (c) represent oxygen and noise from the system.



- Work by K. Cho et al¹, 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

Future Work: In-situ testing in PRIHSM¹



- ARPES, XPS, UPS, LEISS, DRS, XPD, QCM-DCU, QMS, ellipsometry, SEM/PEEM; all in one table-top facility
- Modification sources: broad ion-beam sources, rf plasma, atomic source, UV laser system, cluster ion-beam source
- Characterize surface physical chemistry and morphology along with the eroded plume **during particle irradiation** (angles between 0-75 deg, 0.05-1 keV, T = 200-900 C, 10^{19} cm⁻²)



Collaboration with Brian Wirth, R. Doerner and D.G. Whyte under new PSI Center

Conclusions

- High external pressure applied during SPS of the powders led to high density consolidated samples at temperatures below the second recrystallization temperature of tungsten (1400 °C) where no grain growth was observed.
- Wang *et al*¹ conjectured that materials with bimodal or multi-modal grain size distribution are responsible for improved ductility and strength.
- For a multi-modal grain size distribution the smaller grains give the high strength while the larger grains enable ductility, thus increasing toughness (fracture toughness tests are underway).
- Microstructure results showed a multimodal size distribution similar to the powder distribution, where ultrafine grains are primarily responsible for the very high Vickers hardness obtained. their high purity – demonstrated from surface and bulk chemical analysis results – also was found as a critical parameter for processing of MMG-W.

¹ O. El-Atwani, J.P. Allain, et al, submitted Acta Materialia, 2010

Final Thoughts: PMI issues in a steady-state burning plasma environment

- How does a material surface behave as erosion levels reach 100's of kg/yr levels; what is the day-to-day effects and performance?
- What is the synergy of 'materials processing' inside a fusion reactor induced by transients and non-transient environments?
- Addressing steady-state plasma-surface interface behavior
 - Erosion-induced morphology
 - Morphology effects on sputtering
 - Morphology effects on particle emission and behavior in magnetic sheath region
- Surface morphology, chemistry and ion-driven self-organization mechanisms
 - Design materials that can self-heal
 - Extend lifetime, however replacement is inevitable
 - Combination with wall conditioning scenarios (low Z coatings, etc...)
- How are *plasma-surface interaction* properties affected by these new material systems and their evolving interface processed by fusion plasma?