In-situ electron microscope observations and analysis of radiation damage in tungsten

Xiaoou Yi^{1, 2}, Michael L Jenkins³, Marquis A Kirk⁴ and Steven G Roberts^{1, 2}

^{1.} Department of Materials, University of Oxford, Parks Road, OX1 3PH, Oxford, U.K.

^{2.} CCFE, Culham Science Centre, OX14 3DB, Abingdon, U.K.

^{3.} Trinity College, University of Oxford, Broad Street, OX1 3BH, Oxford, U.K.

⁴ Nuclear Engineering Division, Argonne National Laboratory, IL 60439, Argonne, U.S.A.

Tungsten is a prime candidate for building divertor components in fusion reactors. During its service life, these components may undergo up to 30-40 dpa of displacement damage per year, originated from the collision cascades of fusion neutrons. In this work, we investigated the production and evolution of radiation damage in tungsten with in-situ observations and analysis of 150 keV W⁺ ion irradiations, so as to mimic the effects of the average primary recoil energy of 14 MeV fusion neutrons [1].

TEM foils of pure tungsten (typically > 99.996 wt%, Plansee) were annealed (1673K, 20 h) and prepared from jet-electropolishing in a 0.5 wt.% NaOH aqueous solution. The in-situ irradiations were performed on the IVEM-Tandem facility at Argonne National Laboratory, with a focused beam of W⁺ ions directed to the specimen surface, at a high incident angle of ~75°. The irradiation conditions covered a wide temperature range from 30 K to 1073 K and a dose range from 10^{16} to 10^{18} W⁺/m² (0.01 ~ 1.0 dpa) at a constant rate of ~ 6.25×10^{14} W⁺/m²s. We recorded the defect dynamics at 15 frames/s, and performed defect characterizations and analyses (population; size distribution; geometry; nature) following the methods described in Jenkins *et al* [2].

This comprehensive study of damage production and evolution in tungsten has led to several new findings. We have discovered that at doses ≤ 0.01 dpa, the first observable defects nucleated in tungsten were vacancy loops, predominantly of $\mathbf{b} = \frac{1}{2} <111$, and were formed within individual collision cascades. With the increase of temperature from stage I (30 K) to stage IV (1073 K), loops with $\mathbf{b} = \frac{1}{2}$ <111> gained increasing predominance over those with **b** = <100> and the analysis of defect size versus the frequency of occurrence suggested the engagement of strong elastic interactions among all radiationinduced defects in the cascade (more details in Mason et al) [3]. At doses beyond the overlap of cascades (> 0.01 dpa), radiation damage in tungsten evolved through the 1D migration of defect clusters, the elastic interactions and (typically) non-conservative reaction among these defect clusters, rendered by irradiation temperature and dose. Notably, a transition from random distribution to spatial ordering of loops has been observed in the $z = \langle 001 \rangle$ grains at doses $\rangle 0.4$ dpa and T ≥ 773 K, as partially illustrated in Figure 1. We've also noticed that non $z = \langle 001 \rangle$ orientations may considerably lower the threshold of this ordering phenomenon. The highlights of damage analysis in tungsten are summarized in Figure 2. The rates of defect accumulation in Figure 2a indicated a rapid saturation for T < 773 K. whereas for $T \ge 773$ K, the first signs of saturation did not occur until > 0.4 dpa. Post-irradiation analysis of defect size distributions (Figure 2b) and defect geometry (Figure 2c) at 1.0 dpa suggested that 773 K (stage III, migration of monovacancies) is a characteristic temperature in the radiation damage evolution of tungsten. Together with evidence found in defect dynamic behavior and the results of defect nature determination, we have found that temperature and dose tend to drive the damage microstructure in tungsten towards an increased proportion of interstitial $\frac{1}{2} < 111 > 100$ boys, an increased degree of spatial ordering among them and facilitate their size increase through coalescence reactions.

References:

[1] A. E. Sand, S. L. Dudarev and K. Nordlund, EPL. 103 (2013), 46003.

[2] M. L. Jenkins in "Characterization of Radiation Damage by Transmission Electron Microscopy", 1st ed., (IOP Publishing, Bristol & Philadelphia) 27-69, 74-89, 110-128.

[3] D. R. Mason, X. Yi and M. A. Kirk, J. Phys: Condens. Matter. 26 (2014), 375701.

[4] The authors acknowledge funding from the EPSRC, UK (Grant No. EP/H018921/1) and the DOE Office of Nuclear Energy, USA (Contract No. DE-AC02-06CH11357, UChicago Argonne, LLC) for the in-situ electron microscopy work accomplished at ANL (IVEM-Tandem Facility). XY thanks Mr. Pete Baldo and Mr. Edward Ryan for their generous help with the irradiations, and CCFE for supporting a Junior Research Fellowship via St Edmund Hall, University of Oxford.



Figure 1. Temperature and dose dependence of damage microstructures in W irradiated with 150 keV W^+ at close to z = [001] orientations. Micrographs were recorded under weak-beam dark-field conditions: g = 200, 3-4g.



Figure 2. Highlights of radiation damage analysis in W: a) the evolution of defect population over a dose range of $0.1 \sim 1.0$ dpa as a function of temperature; b) defect size distributions and c) defect geometries at a dose of 1.0 dpa as a function of temperature.