4.2 Evaluation of Mechanical Properties of Tungsten After Neutron Irradiation — L. L. Snead, L. M. Garrison, T. S. Byun, N. A. P. K. Kumar, and W. D. Lewis (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this work is to evaluate the effect of neutron irradiation on the mechanical properties of tungsten with selected microstructures to aid in developing plasma-facing materials for fusion reactors.

SUMMARY

A total of 440 tungsten samples were irradiated in HFIR at temperatures from 70 to 900°C and fast neutron fluences of 0.01 to 20 $\times 10^{25}$ n/m^2 at E>0.1 MeV over the last year. Types of tungsten included were [110] single crystal tungsten, [100] single crystal tungsten, wrought tungsten foils, annealed tungsten foils, and tungsten-copper laminate. Samples that were irradiated at 2×10^{25} n/m^2 and below have been tensile and hardness tested at 300°C and below. The defect structure of selected low dose samples has been investigated in the TEM. The analysis of the lower dose samples is ongoing, and analysis of the higher dose samples will follow.

PROGRESS AND STATUS

Introduction

Tungsten will be the primary divertor material in the ITER tokamak and is a leading candidate for plasma facing components in other advanced fusion reactors. Tungsten has many beneficial characteristics such as a low sputtering yield, high melting temperature, and low activation properties. However, tungsten has a ductile to brittle transition temperature between ~400-900°C [1] (the temperature depends strongly on the microstructure and test method) and can be difficult to manufacture. Additionally, there is limited data available on the behavior of tungsten under neutron irradiation. It is known that microstructure can greatly change the mechanical properties of a material. Several different microstructures of tungsten were selected for neutron irradiation and post irradiation analysis in order to form a data set on neutron-irradiated tungsten properties.

Experimental Procedure

The full list of irradiated specimens was published in the previous semi-annual report [2]. There were a total of 440 samples irradiated in HFIR at selected temperatures between 70 and 900°C and fast neutron fluences of 0.01 to 20 $\times 10^{25}$ n/m^2 at E>0.1 MeV over the last year. Samples of each different tungsten material--single crystal tungsten [110], single crystal tungsten [100], wrought tungsten foil, annealed tungsten foil, and tungsten-copper laminate--that received doses up to 2×10^{25} n/m^2 have been tensile tested and hardness tested. Additional tensile testing at temperatures above 300°C for the low dose samples is planned. Selected samples from each different microstructure type have been analyzed in the TEM. The higher dose samples have finished irradiation but have not been analyzed yet. Figure 1 illustrates the irradiation parameters used in the study.

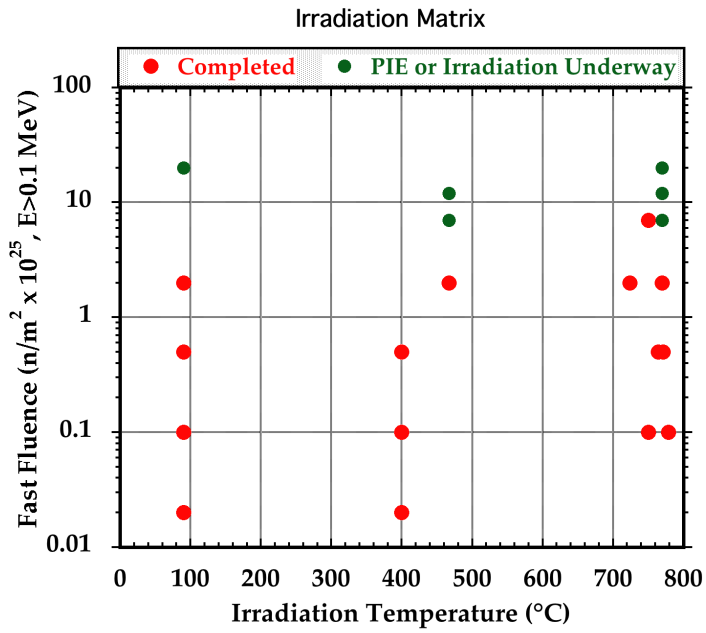

Figure 1. Irradiation matrix for the tungsten samples irradiated in HFIR in 2013.

Bar samples 0.630 X 0.157 inches (16 X 4 mm) and tensile test specimens with the same outer dimensions were used for the irradiations. The foil samples were 0.004 inches thick (0.1 mm), while the single crystal and laminate samples were 0.020 inches thick (0.5 mm). Inside the rabbits, CVD SiC pieces were used to measure the irradiation temperature. Figure 2 shows the dimensions of a tensile test sample.

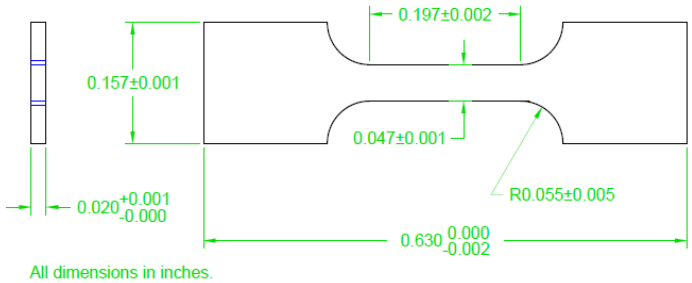

Figure 2. Tensile test specimen dimensions.

RESULTS

Microhardness

Single crystal tungsten samples in [100] and [110] orientations were hardness tested (Figure 3). For both orientations, hardness increased with dose, but there was not a clear trend of hardness with increasing irradiation temperature. The indentation of the hardness test induced triaxial deformation in the samples, so there was little difference between the different orientations for the hardness values.

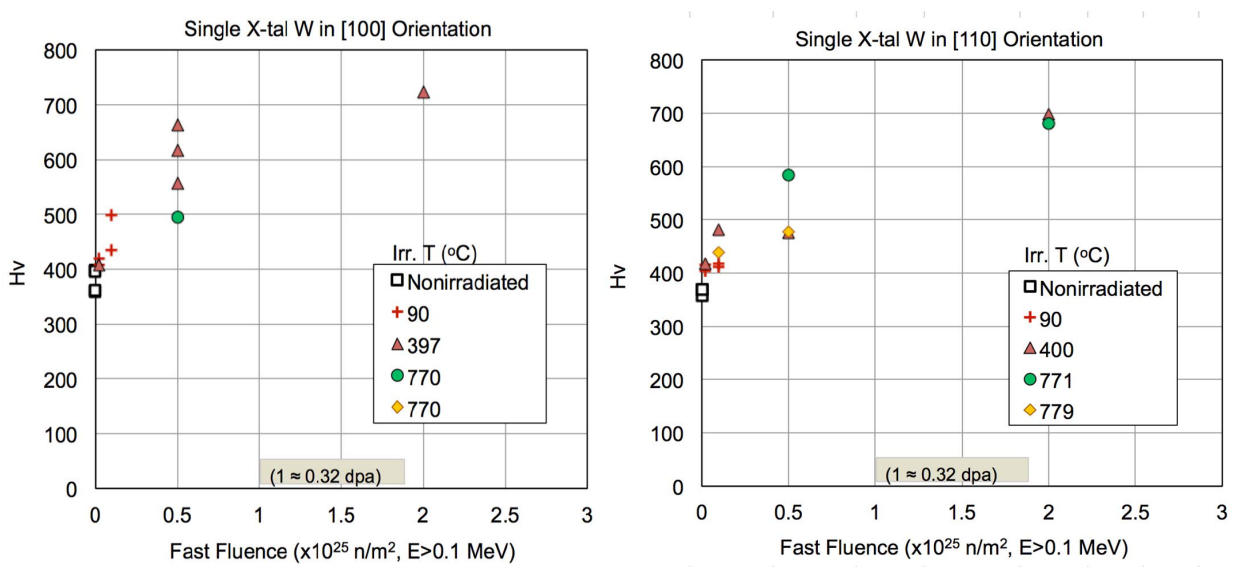

Figure 3. Hardness testing was completed for single crystal tungsten samples in orientations [110] and [100] after neutron irradiation over a range of fluences and temperatures.

Tensile Test Results

Single crystal tungsten samples in [110] and [100] orientations were tensile tested in an unirradiated state at temperatures from room temperature up to 650°C. The results from the [110] samples are presented in Figure 4. In the unirradiated condition, both the [100] and [110] samples showed ductility starting at 300°C. Thus far, samples of [110] tungsten irradiated to two different doses have been tensile tested at room temperature, 90 and 300°C. At both doses, the [110] samples are also ductile at 300°C (Figure 4). Yield as well as fracture stress increased with irradiation dose. Additionally, total ductility started to decrease from 0.03 dpa. Samples of [100] single crystal tungsten that was irradiated to 0.02×10^{25} n/m^2 were also tensile tested at room temperature, 90, and 300°C. For the [100] orientation samples, some ductility is observed on the sample tested at 300°C. However, the overall elongation of the [100] sample at 300°C is much less than the [110] samples tested at the same temperature. Higher temperature tensile tests will be performed on irradiated specimens in 2014.

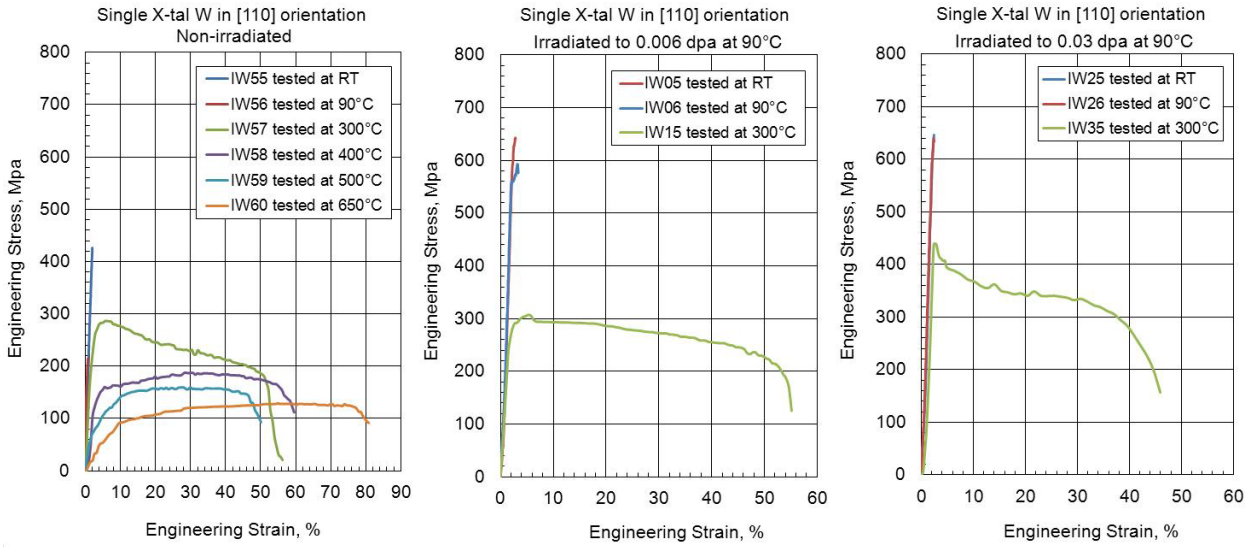

Figure 4. Tensile test results of [110] single crystal tungsten unirradiated, after 0.006 dpa, and after 0.03 dpa.

Wrought Tungsten-Copper Laminate Material

The tungsten-copper laminate material was produced in Germany at KIT. The material consists of repeated layers of 0.1 mm tungsten and 0.1 mm copper braised together into a laminate with a total thickness of 0.5 mm. Tungsten foil of thickness 0.1 mm was found by Rieser et al. to be ductile at room temperature [3]. The motivation for the tungsten-copper laminate is to determine if a bulk material can be made that retains the ductility of the thin tungsten foils.

In the unirradiated condition, samples of wrought tungsten-copper laminate material were tensile tested at temperatures from room temperature up to 650°C (Figure 5). Both strength and ductility decreased with increasing test temperature. Embrittlement was shown at 90°C, which is consistent with the single crystal specimens.

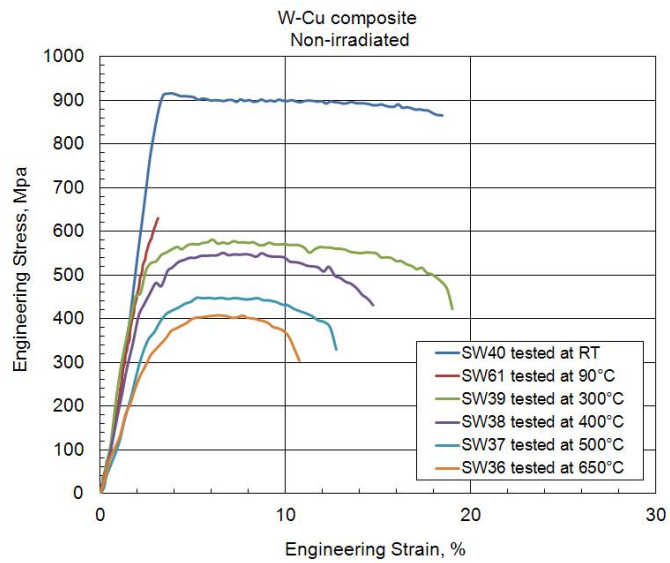

Figure 5. Tensile test results of the tungsten-copper laminate material before irradiation.

The W-Cu laminate material was irradiated at 400, 750, and 770°C and fast fluences between 0.01 and 10×10^{25} n/m^2, E>0.1 MeV. These samples were then tensile tested at room temperature (Figure 6). There is not a clear relationship between the strength of the material and the irradiated dose. As the W layer (0.1 mm thick) was heavily deformed before irradiation, additional hardening by irradiation should be limited. Therefore, the strength dependence was subjected to the statistical behavior of crack initiation and propagation in the composite. This led to the high variation in strength at different conditions. Significant ductility was retained below a fast fluence of $0.1\text{x}10^{25}$ n/m^2.

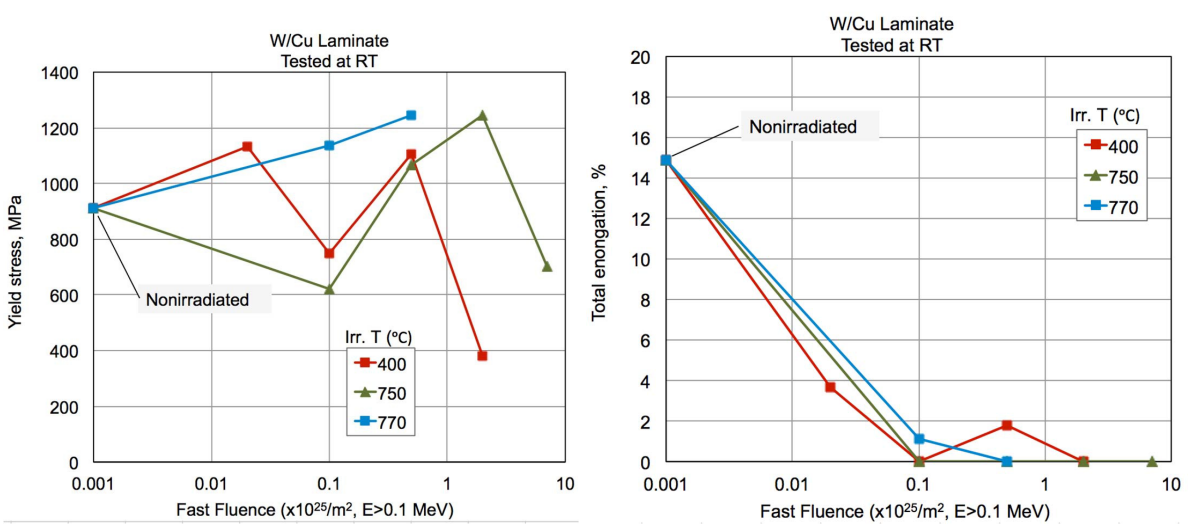

Figure 6. Yield strength and elongation of the tungsten-copper laminate material after irradiation at 400, 750, and 770°C.

TEM Analysis of Tungsten

Thus far, TEM analysis has been completed on selected single crystal, wrought foil, and annealed foil tungsten samples. Samples were analyzed in the unirradiated state and then after fluences of 0.1×10^{25} n/m^2, 0.5×10^{25} n/m^2, and 2.0×10^{25} n/m^2. A focused ion beam (FIB) system with Ga^+ ion beam was used to prepare TEM specimens. In order to minimize the unwanted damage caused by Ga^+ ions, very low energy and current (2 kV and 27 pA) was used on the TEM specimen during final thinning process. A summary of the irradiation defects is presented in Table 1, with descriptions of the different materials following. Samples of annealed foil and deformed foil samples that were irradiated at higher temperatures are currently being prepared for TEM analysis for comparison with the single crystal tungsten samples.

Table 1. Summary of dislocation loops observed in TEM analysis of single crystal, wrought foil, and annealed foil tungsten samples.

Material	Fluence in n/m^2 & (dpa)	Irr. Temp. in °C	Dislocation loop density (x 10^{22} m^{-3})	Average loop diameter in nm
Single Crystal -W	0.1 x 10^{25}n/m^2 (0.032)	80-100	2.97	4.85
Single Crystal-W	2 x 10^{25}n/m^2 (0.639)	80-100	4.55	4.98
Single Crystal-W	2 x 10^{25}n/m^2 (0.639)	724	1.41	6.37
Single Crystal-W	0.5 x 10^{25}n/m^2 (0.160)	764	Low	6.56
Annealed foil-W	0.1 x 10^{25}n/m^2 (0.032)	80-100	3.01	4.38
Annealed foil-W	2 x 10^{25}n/m^2 (0.639)	80-100	4.11	4.45
Deformed foil-W	0.1 x 10^{25}n/m^2 (0.032)	80-100	2.59	5.07
Deformed foil-W	2 x 10^{25}n/m^2 (0.639)	80-100	3.55	8.08

Deformed tungsten foil

Samples of deformed tungsten foil were analyzed in the TEM in the unirradiated state and then after irradiation at 80-100°C to fluences of 0.1×10^{25} n/m^2 and 2.0×10^{25} n/m^2. There was a noticeable increase in damage with increased neutron dose. A high density of dislocation loops was observed for both irradiated samples. With the increase in irradiation dose, the defects appeared to coalesce, which led to a coarsening of dislocation of loops. The average loop diameter of the sample exposed to 0.1×10^{25} n/m^2 was ≈ 5.074 nm (average of 50 loops), while the average loop diameter of the sample exposed to 2×10^{25} n/m^2 was larger, ≈ 8.076 nm (average of 50 loops). The pre-existing dislocation network in the deformed specimens could be the reason for the increase in loop diameter. This observation is in agreement with previous

studies, which showed that the presence of a pre-existing, low network dislocation density would promote the decrease in loop density with simultaneous increase in loop diameter.

Annealed tungsten foil

Samples of annealed tungsten foil were analyzed in the TEM before and after irradiation at 80-100°C to fluences of 0.1×10^{25} n/m^2 and 2.0×10^{25} n/m^2. While the deformed tungsten foil showed larger dislocation loops at higher fluences, the annealed foil only had a small change in dislocation loop size for the different fluences. The average loop diameter of the sample exposed to 0.1×10^{25} n/m^2 was ≈ 4.38 nm (average of 50 loops), and the average loop diameter of the sample exposed to 2×10^{25} n/m^2 was ≈ 4.45 nm (average of 50 loops). The average diameter of dislocation loops was less than the average diameter of loops found in deformed tungsten sample. An increase in defect density was observed with the increase in irradiation dose.

Single Crystal [110] tungsten

Four samples of neutron irradiated single crystal tungsten have been analyzed in the TEM thus far as well as a control sample in the unirradiated state. The two samples were irradiated at 80-100°C to fluences of 0.1×10^{25} n/m^2 and 2.0×10^{25} n/m^2. Two samples were irradiated at higher temperatures before TEM analysis: one sample was irradiated at 724°C to 2.0×10^{25} n/m^2, and another sample was irradiated at 764°C to 0.5×10^{25} n/m^2. Dislocations were observed in all of the irradiated samples with the samples irradiated at higher temperatures having a lower dislocation density but larger dislocation loops on average than the samples irradiated at the lower temperatures. The two samples irradiated at 80-100°C did not develop voids (Figure 7), but both samples irradiated at higher temperatures did (Figure 8).

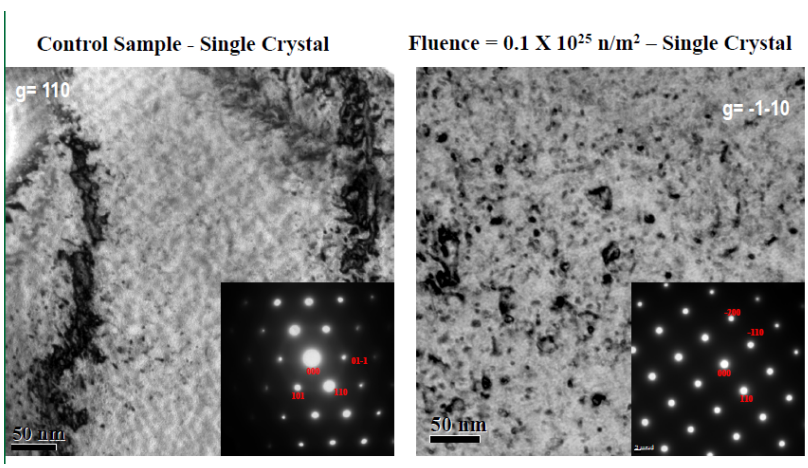

Figure 7. The single crystal tungsten unirradiated sample is shown for comparison. After irradiation to 0.1×10^{25} n/m^2 at 80-100°C dislocation loops are visible.

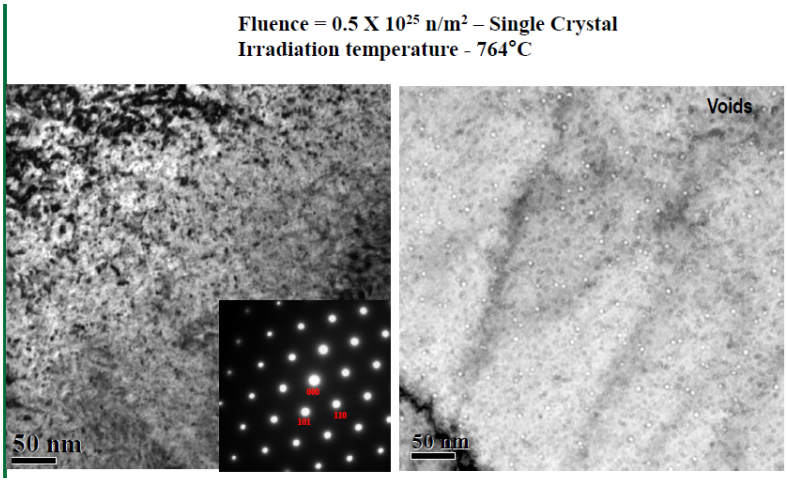

Figure 8. When irradiated at a higher temperature of 764°C, the single crystal tungsten sample developed voids in addition to dislocation loops.

REFERENCES

[1] M. Rieth and A. Hoffmann, International *J. Refractory Met. Hard Mater.* **28** (2010) 679-686.
[2] Y. Katoh, and J. L. McDuffee, Fusion Materials Semiannual Progress Report for Period Ending June 30, 2013, DOE/ER-0313/54, 229-232.
[3] J. Reiser, M. Rieth, B. Dafferner, and A. Hoffmann, *J. Nucl. Mater.* **423** (2012) 1-8.