4.4 ANALYSIS OF NEUTRON IRRADIATED TUNGSTEN MICROSTRUCTURES

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OBJECTIVE

The objective of this project is to analyze the neutron irradiated microstructures of tungsten for evaluating its potential use in plasma facing components in fusion reactors.

SUMMARY

There are two components to neutron irradiation response that change the structure of tungsten: neutron created lattice displacement defects such as interstitials and vacancies, and transmutation products, especially rhenium and osmium. This study is examining the effects of both of these on the tungsten microstructure. Tungsten irradiated to four different doses from 0.7 to 4.5 dpa was examined by transmission electron microscopy, scanning transmission electron microscope, and electron dispersive spectrometry. The tungsten types included single crystal tungsten and as-rolled tungsten foils. The Re and Os transmutation products form needle-shaped precipitates preferentially along the <110> direction. The average precipitate and void sizes increased with increasing dose.

PROGRESS AND STATUS

In our earlier reports microstructure evaluation in tungsten at lower neutron doses were described. The dominate features were dislocation loops at lower temperatures and voids at higher temperatures. This report present the microstructural changes in both polycrystalline and single crystal tungsten specimens exposed to higher neutron doses. The irradiation conditions of examined high dose tungsten specimens are presented in Table 1. A focused ion beam (FIB) system with Ga^+ ion beam was used to prepare transmission electron microscopy (TEM) specimens. In order to minimize the unwanted damage caused by Ga^+ ions, very low voltage and current (2 kV and 27 pA) were used on TEM specimens during the final thinning process.

Table 1. High Temperature-High Dose Tungsten specimens examined under TEM

Sample ID	Material	Design Temp (°C)	Actual irradiation temperature (°C)	Fluence ($x10^{25}$ n/m^2)	dpa
1W17	Single Crystal	500	705	2.2	0.7
1W19	Single Crystal	500	770	9.0	2.9
OW125	Polycrystalline	500	Not available	14.1	4.5
OW158	Polycrystalline	650	Not available	7.4	2.4

Figure 1 provides the summary of microstructural changes in single crystal and polycrystalline tungsten as a function of irradiation dose and temperature. In order to avoid confusion, henceforth only design temperatures instead of actual exposed temperatures will be shown on all the micrographs and tables.

The TEM micrographs were recorded under two-beam condition with diffraction vectors g = 110 and $1\bar{1}0$. Line dislocations and loops were seen in 500°C, 0.7 dpa specimens. With increasing dose, considerably fewer loops were seen and the transmuted second phase dominates the microstructure. Both polycrystalline specimens exposed to higher irradiation doses showed heavy precipitation in the microstructures. A dense platelet type structure was observed in all high dose irradiated specimens (see Figure 1). In polycrystalline tungsten, apart from the platelet type structures within the grains, huge pancake type precipitates were also seen at the grain boundaries. The irradiation induced defect sinks such as dislocation loops, voids, which are formed in the early stage of irradiation may have acted as nucleation sites for the second phase precipitates. Therefore a drop in void number density was observed

at higher irradiation doses (Table 2). The size of the precipitates also increased with the irradiation dose and temperature. However the number density of precipitates remained in the same range of about 10^{22} m^{-3}, and did not change with the change in irradiation temperature and dose (Table 2).

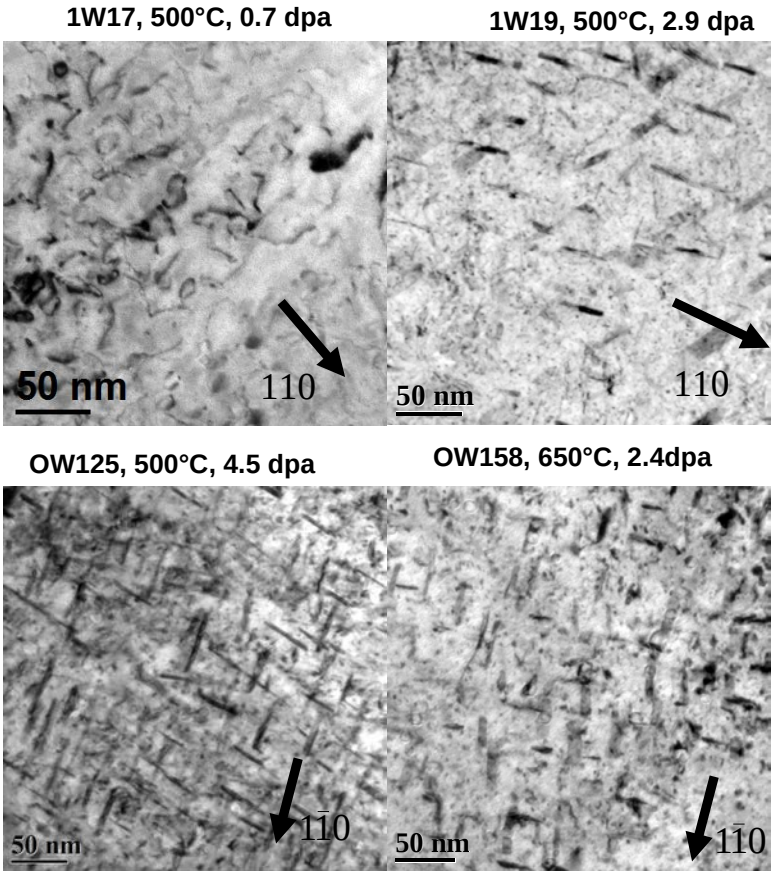

Figure 1. Micrographs illustrating the damage microstructure in tungsten. All images were recorded at B= [001].

Table 2. Summary of microstructural observation in pure tungsten after neutron irradiation

Material	Irradiation Temp (°C)	Fluence ($x10^{25}$ n/m^2)	Mean dislocation loop diameter (nm)	Mean void size (nm)	Void density (m^{-3})	Precipitate length (nm)	Precipitate Density (m^{-3})
Single Crystal	500	2.2	6.37	3.03	$1.22X10^{22}$	-	-
Single Crystal	500	9.0	-	5.28	$7.23X10^{20}$	20	$4.77X10^{22}$
Polycrystalline	500	14.1	-	7.40	$4.11X10^{21}$	33.5	$3.04X10^{22}$
Polycrystalline	650	7.4	-	6.40	$5.45X10^{21}$	36	$1.24X10^{22}$

Transmutation

It is widely known that tungsten produces transmutation elements such as rhenium (Re) and osmium (Os) upon neutron irradiation. The compositional change in pure tungsten after exposing to a neutron dose of

1 dpa in HFIR is estimated to be W-9.22%Re-5.02%OS (M.Fukuda et al, Journal of Nuclear Materials, 455, (2014), 460-463). In general, the solubility of Re and Os in pure tungsten is approximately 27 at.% and 5 at.% respectively at room temperature (B. Ralph, D. Brandon, Philosophical magazine, 8, (1963), 919-934 and A. Taylor, B. Kagle, N. Doyle, Journal of Less Common Metals, 3, (1961), 333-347). However during irradiation, Re and Os form precipitates at compositions well below their solubility limits. Figure 2 shows the scanning transmission electron microscope (STEM) images of polycrystalline tungsten irradiated to 2.4 dpa at 650°C with platelet and pancake shaped precipitates within the grains and at the grain boundaries respectively. STEM embedded with Electron Dispersive Spectrometry (EDS) was used to measure the composition of the precipitates. EDS confirms the presence of high concentrations of Re and Os in the platelets, but due to limited width of the platelet type structures the elemental ratio is still unclear. However, the pancake type structures at the grain boundaries showed an average composition of W- 40%Os-24%Re. Figure 3 shows the atomic profile of pancake type precipitates. Further study on these precipitates is ongoing.

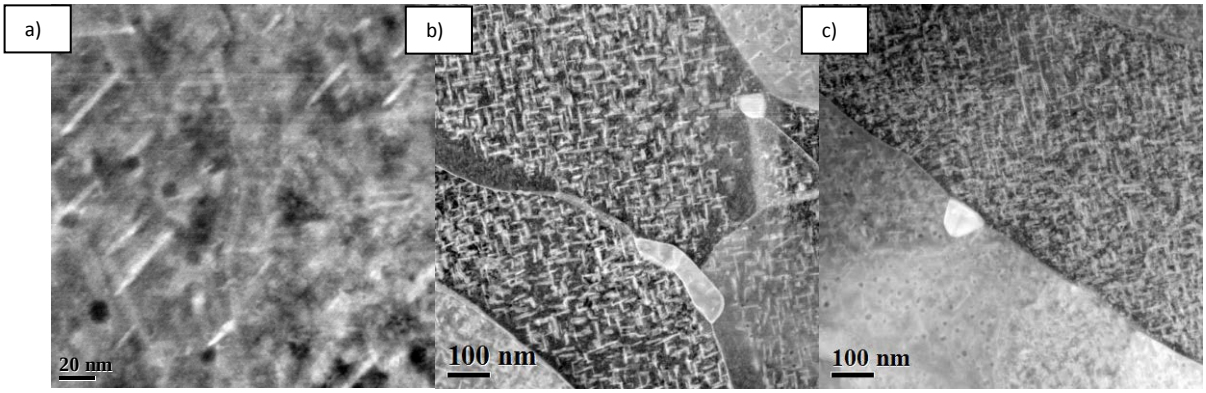

Figure 2. STEM micrographs showing the transmuted phase within grains and at the grain boundaries in OW158, 650°C- 2.4 dpa, polycrystalline tungsten a) platelet type precipitates within the grains, b) c) grain boundary precipitates.

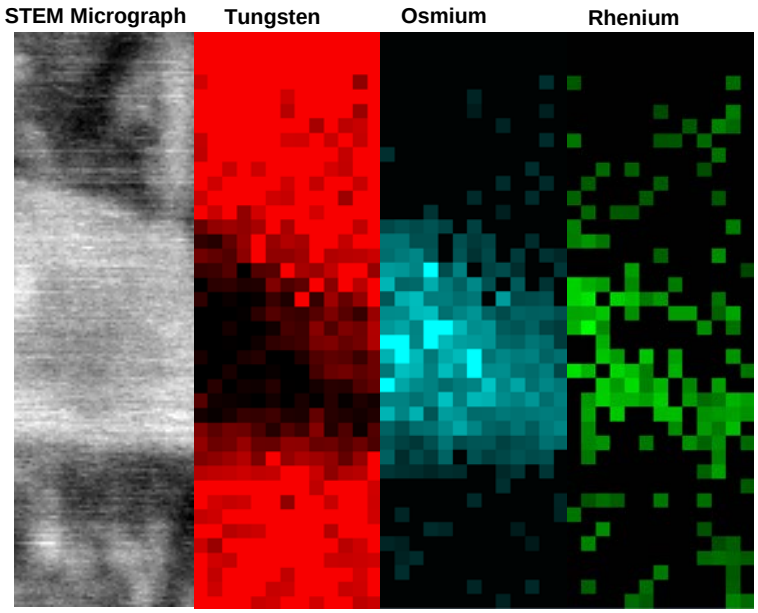

Figure 3. Atomic profile of the pancake type grain boundary precipitate.

Voids

Microstructural examination of the irradiated specimens revealed the presence of voids for all four irradiation conditions, Figure 4. The mean size of voids increased with the increasing irradiation dose and temperature (Table 2). Alignment of voids, usually referred to as void lattice formation, in irradiated tungsten is usually observed at doses above 10 dpa (K. Krishan, Radiation Effects, 66, (1982), 121-155). Recently Tanno et al., have report void lattice formation even at lower dpa in JOYO test reactors (T. Tanno et al, Materials Transactions, 48, (2007), 2399-2402). However, such void lattice formation was not observed in current irradiated specimens. Voids were rather randomly distributed in both polycrystalline and single crystal specimens. Void size distribution as a function of irradiation conditions is shown in Figure 5. With the increase in irradiation dose and temperature, the entire distribution moved to larger sizes.

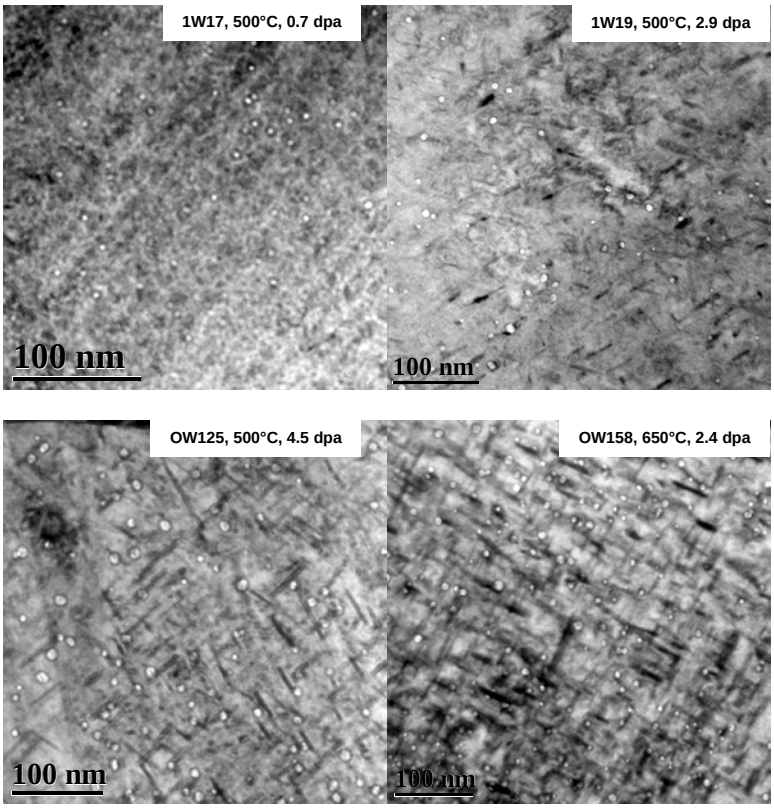

Figure 4. TEM images showing voids in neutron irradiated tungsten.

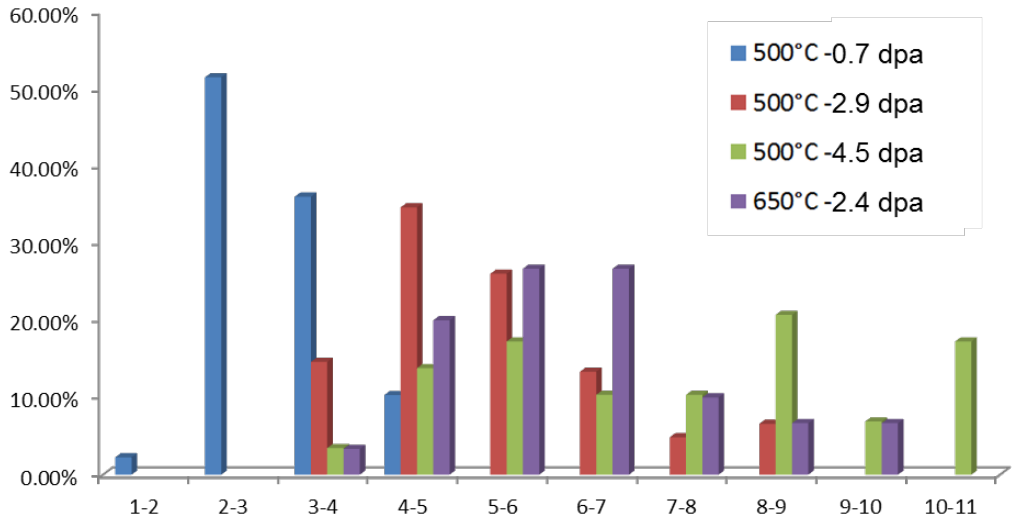

Figure 5. Void distribution in irradiated tungsten.