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The effect of low energy helium ion irradiation on tungsten-tantalum (W-Ta) alloys under fusion relevant conditions

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ABSTRACT

Currently, tungsten remains the best candidate for plasma-facing components (PFCs) for future fusion devices because of its high melting point, low erosion, and strong mechanical properties. However, continued investigation has shown tungsten to undergo severe morphology changes under fusion-like conditions. These results motivate the study of innovative PFC materials which are resistant to surface morphology evolution. The goal of this work is to examine tungsten-tantalum (W-Ta) alloys, a potential PFC material, and their response to low energy helium ion irradiation. Specifically, W-Ta samples are exposed to 100 eV helium irradiations with a flux of 1.15×10^{21} ions m^{-2} s^{-1}, at 873 K, 1023 K, and 1173 K for 1 h duration. Scanning electron microscopy (SEM) reveals significant changes in surface deterioration due to helium ion irradiation as a function of both temperature and tantalum concentration in W-Ta samples. X-Ray Diffraction (XRD) studies show a slight lattice parameter expansion in W-Ta alloy samples compared to pure W samples. The observed lattice parameter expansion in W-Ta alloy samples (proportional to increasing Ta wt.% concentrations) reflect significant differences observed in the evolution of surface morphology, i.e., fuzz development processes for both increasing Ta wt.% concentration and target temperature. These results suggest a correlation between the observed morphology differences and the induced crystal structure change caused by the presence of tantalum. Shifts in the XRD peaks before and after 100 eV helium irradiation with a flux of 1.15×10^{21} ions m^{-2} s^{-1}, 1023 K, for 1 h showed a significant difference in the magnitude of the shift. This has suggested a possible link between the atomic spacing of the material and the accumulated damage. Ongoing research is needed on W-Ta alloys and other innovative materials for their application as irradiation resistant materials in future fusion or irradiation environments.

1. Introduction

Tungsten remains one of the primary materials for plasma-facing components (PFCs) in future fusion devices, *viz.*, ITER and DEMO (DEMOnstration Power Station). This is due to tungsten's good thermomechanical properties, low erosion, and limited tritium retention [1–3]. It has the highest melting point of all metals (~3410 °C) and has good thermal conductivity [3]. Additionally, it has a high threshold for physical sputtering of ~200 eV [1]. Finally, it does not form hydrides or co-deposits with tritium (T) which results in less tritium retention when compared to carbon based PFC [1,4,5]. Despite these inherent advantages recent research on tungsten (W) as PFC material has shown tungsten to undergo severe surface morphology evolution in response to low energy (~100 eV) helium (He) and deuterium (D) irradiation. Mechanisms leading to the formation of bubbles [6–8], blister [9–11], and eventually fuzz [12–14] are still being investigated. Furthermore, the effects of these extreme morphologies on device operation and plasma performance are uncertain. These issues regarding W as PFC prompt the need for investigation of alternative wall materials in order to better understand the fundamental mechanisms and physics behind this extreme morphology evolution and to develop materials better suited to harsh fusion environments.

Tungsten alloys have sparked some interest as a potential PFC material. The alloying of tungsten with certain materials like rhenium (Rh) has been shown to improve ductility [15,16]. Other alloys, such as oxide dispersion strengthened (ODS) heavy alloys, have been shown to exhibit enhanced mechanical properties [17,18]

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and even reduced hardening and embrittlement when exposed to high energy neutron irradiation [19,20]. This result has motivated study of additional W alloys such as W-Ta alloy materials. Recent work [21] investigated the mechanical properties of W-Ta alloys and showed that the alloying of W with Ta does not have the same ductility enhancement as compared to that of the W-Rh. Additionally, they also observed that the toughness for crack propagation may be enhanced for carefully designed PFC W-Ta alloy materials [21]. Other studies on W-Ta alloys have shown increased hardness as a function of neutron damage simulated by ion implantation [22]. Further, research on W-Ta alloy's response to thermal shock *via* transient heat loading has shown a significant improvement when compared to pure W materials [23,24]. Perhaps that most significant enhancement, of W-Ta alloys, for fusion applications may be in regards to retention of hydrogen (H) isotopes. Several recent studies [25–27] have looked exclusively at these issues. Results suggest that W-Ta alloys exhibit significantly reduced D retention under fusion relevant conditions. These advantages suggest W-Ta alloys may be considered as possible alternative PFC material for current and future DEMO reactors.

Despite the extensive work on testing the mechanical properties of W-Ta alloys, very little effort has been done on understanding the surface modification of W-Ta alloys when exposed to fusion relevant He ion fluxes in fusion environment. The work presented here focuses on the surface morphology evolution driven by low energy He^+ ion irradiation of W and W-Ta alloys. Specifically, pure W and W-Ta alloys of 1, 3, and 5 wt.% Ta are exposed to low energy He^+ ion irradiation at various temperatures. Post irradiation characterization using SEM revealed significant surface morphology dependence on both Ta concentration and temperature. Further analysis using XRD linked this behavior with slight crystallographic change induced by the alloying process.

2. Materials and experimental methods

The experimental work detailed here uses four different W based materials: one 99.95% pure W (from Alfa Aesar), and three W-Ta alloys having 1, 3, and 5 wt.% of Ta (from American Elements). High resolution SEM of pristine samples confirmed homogeneous distribution of Ta throughout the sample. When referring to these samples going forward the following name convention will be used: W, W-1Ta, W-3Ta, and W-5Ta corresponding to pure W and, 1, 3, and 5 wt.% of Ta, respectively.

In order to verify the composition of the W-Ta alloy materials *in-situ*, pre-irradiation X-ray photoelectron microscopy (XPS) characterization was performed in the Interaction of Materials with Particles and Components Testing experimental (IMPACT) [28] chamber at Center for Materials Under eXtreme Environment (CMUXE), Purdue University. The XPS results confirmed the Ta concentrations of the W-Ta alloy materials to within 0.3% uncertainty of that reported from the commercial companies. Samples undergoing XPS were sputter cleaned with 1 keV Ar^+ ions to remove oxygen and surface impurities (e.g., carbon) in order to achieve accurate alloy compositional information.

Samples of the W, W-1Ta, W-3Ta, and W-5Ta were cut from large sheets into 10 mm × 10 mm × 2 mm samples. 16 total W and W-Ta samples (4-W, 4-W-1Ta, 4-W-3Ta, and 4-W-5Ta samples) were mechanically polished to a mirror finish prior to He^+ ion irradiation. He^+ ion exposures were conducted in the ultra-high flux irradiation laboratory (UHFI-II) at CMUXE [29]. One of each sample type was cleaned using 1 keV Ar^+ ion irradiation for XPS compositional analysis and 12 of the samples (3-pure W, 3 -1% Ta, 3-3% Ta, and 3–5% Ta) were exposed to 100 eV He^+ ion irradiation with a flux of 1.15×10^{21} ions m^{-2} s^{-1}, at 873, 1023, and 1173 K for 1 h durations in each case. The temperature threshold for fuzz formation in W has been shown to be > 1000 K [12]. Therefore in the present work, three temperatures all near this threshold were selected in order to investigate the temperature effect of the fuzz formation process. Fig. 1 shows a schematic of the experimental setup used during these irradiation experiments.

Surface morphology changes were monitored with field emission (FE) scanning electron microscopy (SEM), using Hitachi S-4800 Field Emission SEM. For the XPS measurements, photoelectrons were excited by an Mg-Kα (energy = 1253.6 eV) X-ray radiation source (SPECS XRC-1000), and the emitted photoelectrons were analyzed with Omicron Argus hemispherical electron analyzer using a round aperture of 0.63 mm (for imaging-XPS) and a second aperture at 6.3 mm (for conventional XPS). No sample charging was observed. All XPS spectra were analyzed with commercial CasaXPS software [30]. Crystalline phase analyses were performed with X-ray diffraction using Bruker D8 Focus X-Ray Diffractometer. Optical reflectivity measurements [29] were performed over a spectrum of incident light wavelength (λ) ranging from 200 to 1100 nm (using a combination of halogen and deuterium light source and a beam diameter of ~1 mm). Maya 2000 Pro Spectrometer from Ocean Optics was used for signal detection. Before the reflectivity measurements began, the spectrometer was calibrated using Spectralon white reference plate having 100% reflectivity. The observed reflection in our system is mainly specular. A specular reflection is a reflection of a mirror like surface (keeping in mind that different surfaces to different wavelengths may or may not be mirror like). Specular reflection will result when the surface roughness is smaller than the applied wavelength of light (and diffuse reflection will result when the surface roughness is larger than the wavelength). A specular reflectance of 100% would correspond to an ideal mirror, typical specular reflectance are less than the maximum value (*i.e.*, 100%). For collecting the reflected light a "*reflection probe*" from Ocean Optics was used which can collect light at the same angle as it illuminates, and can be used for either specular or diffuse reflection measurements. In fact it is made of 6 illumination fibers around a single read fiber (in the center), which results in a 25° full angle field of view. Each illumination fiber projects a cone of light from the source and all of them overlap at the sample in the center, exactly where the central read fiber is situated. Thus, in principle the reflectivity for an ideal mirror will be ~100%. During our measurements the "*reflection probe*" was placed at 90° to the sample surface (along the sample surface normal). The distance between sample and "*reflection probe*" was ~1 mm.

3. Results and discussion

3.1. Field emission (FE) scanning electron microscopy (SEM) studies

Fig. 2 (a) - (d) is a set of SEM images for four W-Ta alloy samples (W, W-1Ta, W-3Ta, and W-5Ta). In each case samples were exposed to 100 eV He^+ ions at 873 K. There is slight variation in the surface morphology for the different samples as a function of Ta concentration. All the surfaces display roughening with notable reduction in observed feature size as Ta concentration is increased. Specifically, there are small pores present in all the images and the largest of these pores are clearly evident in Fig. 2(a) which is the pure W sample. In addition to the pore sizes, the surface appears to be less damaged in the W-Ta samples.

Fig. 2(e)–(h) is another set of four SEM images for four W-Ta alloy samples (W, W-1Ta, W-3Ta, and W-5Ta). As of the previous cases here also samples were exposed to low energy He^+ ions but at 1023 K. Note, although the target temperature difference between the previous set of experiments (Fig. 2 (a) -(d)) and the present one (Fig. 2(e)-(h)) is only 150 K, the difference in surface morphology is significant, leading to very clear surface morphology dependence