

# Implantation of He<sup>+</sup> in candidate fusion first wall materials

R.F. Radel, G.L. Kulcinski \*

*Fusion Technology Institute, University of Wisconsin, 1500 Engineering Drive, Madison, WI 53706, United States*

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## Abstract

The effect of high temperature (700–1200 °C) implantation of helium in candidate fusion first wall materials was studied in the University of Wisconsin Inertial Electrostatic Confinement device. Powder metallurgy tungsten, single crystal tungsten, and a W–25% Re alloy were irradiated with 30–50 keV He<sup>+</sup> ions. Scanning electron microscopy was used to evaluate changes in surface morphology for various ion fluences at temperature ranges comparable to first wall temperatures. Helium fluences in excess of  $1 \times 10^{18}$  He<sup>+</sup>/cm<sup>2</sup> produce extensive pore formation at 1150 °C. These changes will have an impact on the lifetime of thin tungsten coatings on the first walls and divertors of inertial and magnetic confinement fusion reactors.

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## 1. Introduction

The durability and lifetime of thin tungsten or refractory metal coatings on the first walls of inertial and magnetic confinement fusion reactors is a key issue for the feasibility of such devices. Work as early as 1974 by Thomas and Bauer showed blistering in vanadium as the result of helium implantation at high temperature [1]. Past studies at UW-Madison and ORNL have shown that when tungsten is subjected to He<sup>+</sup> fluences in excess of  $4 \times 10^{17}$  He<sup>+</sup>/cm<sup>2</sup>, it exhibits extensive pore formation at 800 °C [2,3]. The current study has investigated alternative forms of tungsten for future use in fusion devices.

Scientists working in the High Average Power Laser (HAPL) program [4] are currently focusing

on tungsten as a potential material to act as a shield to protect the first wall from light ions, such as helium whose energy ranges from tens of keV to a few MeV. This study examines the lower portion of that spectrum, as shown in Fig. 1 [5].

## 2. Experimental procedure

Scientists at the University of Wisconsin have been studying the performance of Inertial Electrostatic Confinement (IEC) fusion devices for more than a decade [6]. One of these devices has been recently used to test the response of fusion related materials to high temperature bombardment by helium. The device used in this study (Fig. 2) is an aluminum chamber 65 cm tall and 90 cm in diameter [6]. A base pressure of  $\sim 10^{-7}$  Torr is maintained in the device using a 1000 L/s turbo pump. The outer stainless steel anode grid is 50 cm in diameter and is kept at ground potential. The inner target is the W sample and it is connected to a 200 kV power

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\* Corresponding author. Tel.: +1 608 2632308; fax: +1 608 2634499.

E-mail address: [kulcinski@engr.wisc.edu](mailto:kulcinski@engr.wisc.edu) (G.L. Kulcinski).

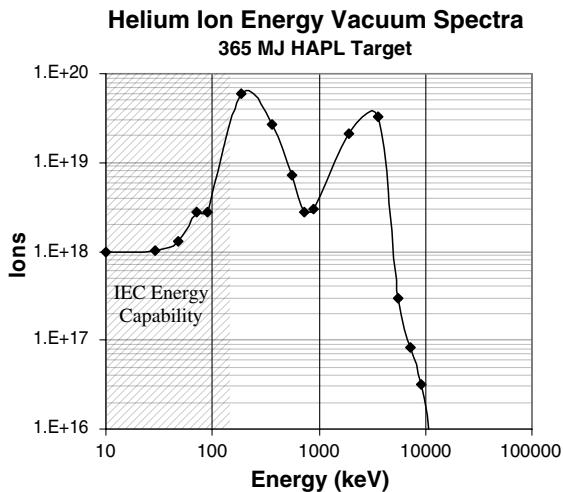


Fig. 1. Calculated helium threat spectra for the HAPL chamber [5].

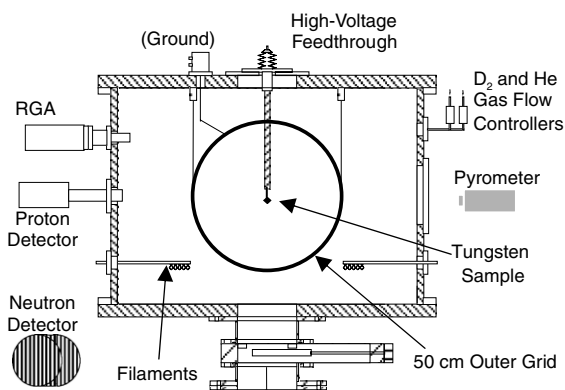


Fig. 2. The University of Wisconsin IEC fusion device. The inner cathode has been replaced with a tungsten sample.

supply through a high voltage feed thru. During operation, deuterium or helium gas is fed into the chamber to produce a background pressure of 0.5 mTorr. This gas is then ionized using electron bombardment from standard light bulb filaments. A negative voltage is applied to the target (cathode), and the positively charged ionized gas is attracted to the target. In addition to damaging the target, these ions also heat the sample to temperature relevant to fusion first walls. It is assumed that target cooling is due entirely to radiation losses.

Samples have been received both from ORNL and from ESPI, a high-purity metal alloy supplier. SEM micrographs were taken of all samples before irradiation in the IEC. Each of the samples was then mounted in the IEC device and irradiated to various

Table 1

Summary of irradiation history and pore density (P = polycrystalline, SC = single crystal, W-Re = W-25% Re)

Sample	Ions	Fluence (#/cm <sup>2</sup> )	Temp (°C)	Pore density (pores/cm <sup>2</sup> )
P-11	<sup>4</sup> He <sup>+</sup>	1 × 10 <sup>20</sup>	~1150	2.8 × 10 <sup>9</sup>
P-1	<sup>4</sup> He <sup>+</sup>	1 × 10 <sup>19</sup>	~1150	1.9 × 10 <sup>9</sup>
P-2	<sup>4</sup> He <sup>+</sup>	6 × 10 <sup>18</sup>	~1120	3.7 × 10 <sup>9</sup>
P-3	<sup>4</sup> He <sup>+</sup>	3 × 10 <sup>18</sup>	~1125	7.7 × 10 <sup>9</sup>
P-4	<sup>4</sup> He <sup>+</sup>	1 × 10 <sup>18</sup>	~1150	1.2 × 10 <sup>9</sup>
P-5	<sup>4</sup> He <sup>+</sup>	6 × 10 <sup>17</sup>	~1130	8.9 × 10 <sup>7</sup>
SC-4	<sup>4</sup> He <sup>+</sup>	1 × 10 <sup>19</sup>	~1100	1.7 × 10 <sup>9</sup>
SC-2	<sup>4</sup> He <sup>+</sup>	6 × 10 <sup>18</sup>	~1100	1.6 × 10 <sup>9</sup>
SC-3	<sup>4</sup> He <sup>+</sup>	3 × 10 <sup>18</sup>	~1100	2.4 × 10 <sup>9</sup>
SC-5	<sup>4</sup> He <sup>+</sup>	1 × 10 <sup>18</sup>	~1100	2.5 × 10 <sup>7</sup>
SC-6	<sup>4</sup> He <sup>+</sup>	6 × 10 <sup>17</sup>	~1100	0
P-6	<sup>4</sup> He <sup>+</sup> and D <sup>+</sup>	1 × 10 <sup>19</sup>	~1150	2.5 × 10 <sup>9</sup>
P-7	<sup>4</sup> He <sup>+</sup> and D <sup>+</sup>	6 × 10 <sup>18</sup>	~1150	1.7 × 10 <sup>9</sup>
P-9	<sup>4</sup> He <sup>+</sup> and D <sup>+</sup>	3 × 10 <sup>18</sup>	~1150	1.2 × 10 <sup>9</sup>
P-10	<sup>4</sup> He <sup>+</sup> and D <sup>+</sup>	1 × 10 <sup>18</sup>	~1150	3.2 × 10 <sup>8</sup>
W-Re-1	<sup>4</sup> He <sup>+</sup>	1 × 10 <sup>19</sup>	~1100	1.3 × 10 <sup>9</sup>
W-Re-2	<sup>4</sup> He <sup>+</sup>	6 × 10 <sup>18</sup>	~1100	1.5 × 10 <sup>9</sup>
W-Re-3	<sup>4</sup> He <sup>+</sup>	3 × 10 <sup>18</sup>	~1100	2.5 × 10 <sup>9</sup>
W-Re-4	<sup>4</sup> He <sup>+</sup>	1 × 10 <sup>18</sup>	~1100	1.7 × 10 <sup>9</sup>
W-Re-5	<sup>4</sup> He <sup>+</sup>	6 × 10 <sup>17</sup>	~1100	5.9 × 10 <sup>7</sup>

All samples were irradiated at 30 kV.

fluence levels at temperatures ranging from 700 to 1200 °C. Temperatures were measured using a Raytek® Marathon MR pyrometer. A summary of the fluence history of the samples used in this set of experiments is given in Table 1. Irradiation times in the IEC device ranged from approximately 20 s to 6 h. Temperature, pressure, voltage, and current were constantly monitored during the irradiation to ensure a constant flux. After irradiation samples were again imaged using a LEO 1530 Field Emission Scanning Electron Microscope. Selected samples were also imaged using a Zeiss Crossbeam focused ion beam (FIB) mill.

### 3. Results

Previous work by Cipiti et al. investigated the effects of temperature, ion voltage, and fluence up to 10<sup>19</sup> ions/cm<sup>2</sup> for deuterium and helium implantation in polycrystalline tungsten [2]. Recent work has expanded that investigation to 10<sup>20</sup> ions/cm<sup>2</sup> and evaluated two new materials – single crystal tungsten and a W-25% Re alloy. Table 1 provides a summary of the temperature, fluence, and post-irradiation pore density for these experiments.

Increasing the helium fluence to 10<sup>20</sup> ions/cm<sup>2</sup> produced an increase in the pore density and a significant increase in surface roughness of the

polycrystalline samples. Fig. 3 shows the surface morphology of tungsten irradiated at 1150 °C to  $10^{18}$ ,  $10^{19}$ , and  $10^{20}$  ions/cm<sup>2</sup>. Not only can an increased damage be seen on the surface, but pore formation is also observed beneath the surface. As the fluence is increased, the creation of helium bubbles forms and extends a semi-porous surface layer. Changes in surface morphology on the  $10^{20}$  He<sup>+</sup>/cm<sup>2</sup> sample are observed at multiple magnifications in Fig. 4. Though the sample had been polished prior to irradiation, grain boundaries are clearly seen in the first image in Fig. 4. The second image indicates that different erosion behavior may be occurring at these grain boundaries. At higher magnification, the surface can be seen to contain not only a high pore density, but also many small protrusions jutting from the surface.

In addition to the morphological changes, this sample also experienced a measurable change in mass. Theoretical calculations based on physical sputtering rates predict that the sample would lose ~2.1 mg after  $10^{20}$  He<sup>+</sup>/cm<sup>2</sup>. However, the sample actually lost 10.2 mg, or roughly 2.6 μm uniformly from the surface, suggesting that other mechanisms of mass loss must be taking place. One possible explanation is increased sputtering coefficients for

non-perpendicular ion incidences. However, this would not increase the physical sputtering loss enough to account for the observed loss, even assuming the forward-sputtered material is not re-deposited on the sample [7]. Another possible explanation is the loss of the small protrusions during the irradiation process.

Polycrystalline samples were also evaluated with Elastic Recoil Detection (ERD) analysis in the University of Wisconsin Tandem Particle Accelerator Laboratory. The analysis was performed with 8 MeV (4<sup>+</sup>) oxygen. Initial ERD results indicate up to 40% atomic fraction helium within the tungsten matrix. This fraction was observed to saturate at helium fluences above  $1 \times 10^{18}$  He<sup>+</sup>/cm<sup>2</sup>.

Single crystal tungsten samples were irradiated at 1100 °C to fluences ranging from  $6 \times 10^{17}$  to  $1 \times 10^{19}$  ions/cm<sup>2</sup>. These samples were then compared to polycrystalline tungsten irradiated at the same temperatures. The top two micrographs in Fig. 5 show single crystal ((001)) and polycrystalline samples irradiated to  $1 \times 10^{18}$  He<sup>+</sup>/cm<sup>2</sup>. While pores have begun to develop on the polycrystalline sample, the single crystal does not appear to have received a threshold level at  $1 \times 10^{18}$  He<sup>+</sup>/cm<sup>2</sup>. When similar samples are further irradiated to  $3 \times 10^{18}$  He<sup>+</sup>/cm<sup>2</sup>,

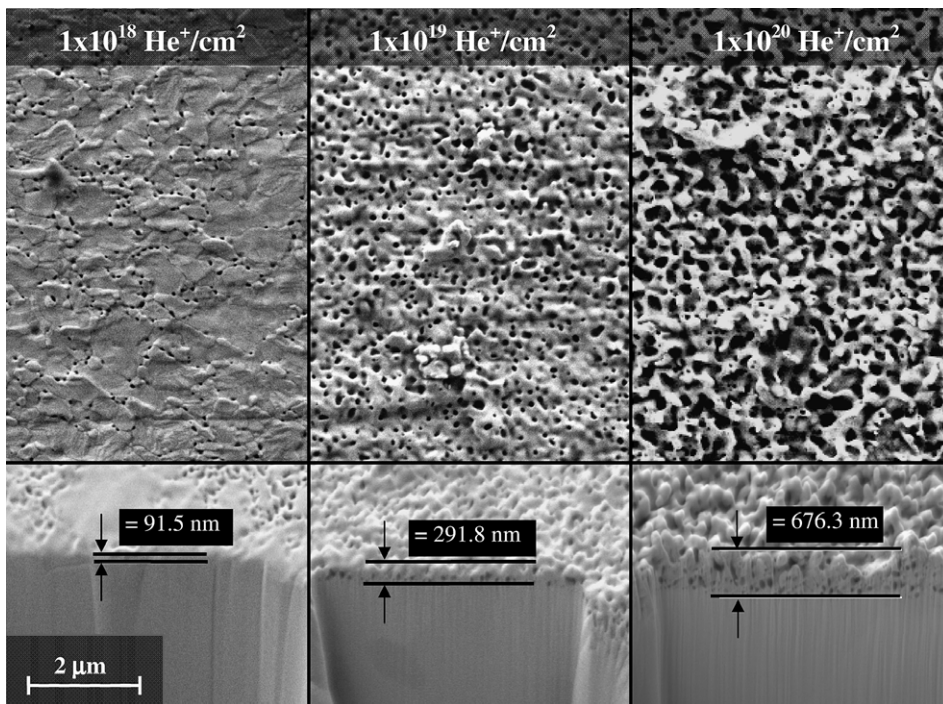


Fig. 3. Polycrystalline tungsten samples irradiated with 30 keV He<sup>+</sup> at 1150 °C to  $10^{18}$ ,  $10^{19}$ , and  $10^{20}$  He<sup>+</sup>/cm<sup>2</sup>, respectively. The lower micrographs show depth profiles of bubbles.

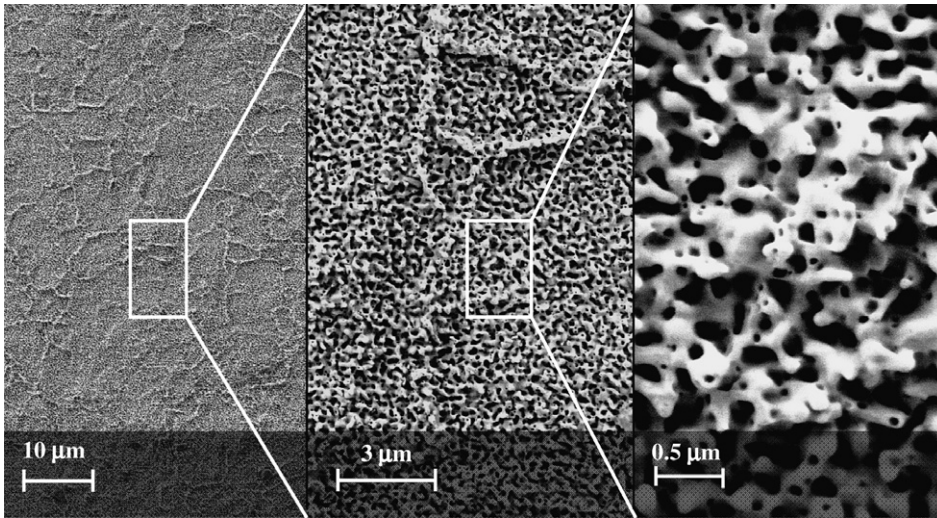


Fig. 4. Polycrystalline tungsten sample irradiated with 30 keV He<sup>+</sup> at 1150 °C to 10<sup>20</sup> He<sup>+</sup>/cm<sup>2</sup>. Each magnification of the W sample reveals new surface features.

it was observed that the single crystal samples had approximately one-third the pore density of the polycrystalline sample.

The effect of alloying tungsten with 25% rhenium was also studied in the IEC device. The W–25% Re alloy has a melting point 300 °C lower than pure

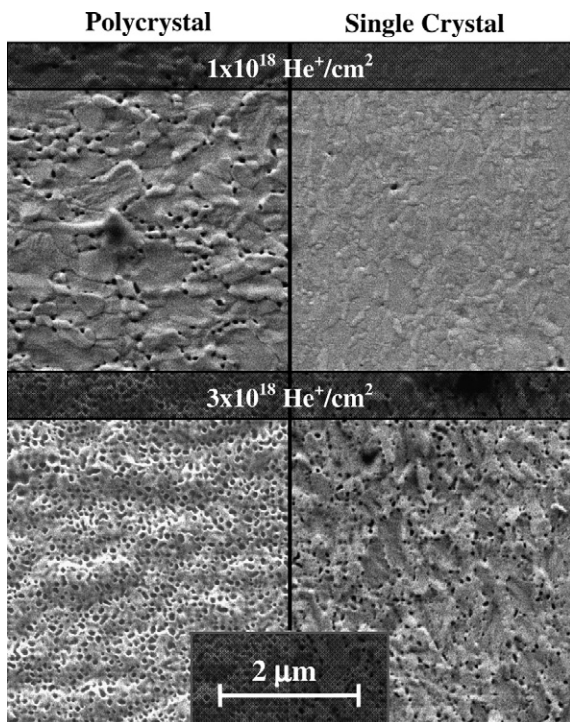


Fig. 5. Polycrystalline and single crystal samples irradiated with 30 keV He<sup>+</sup> at ~1100 °C.

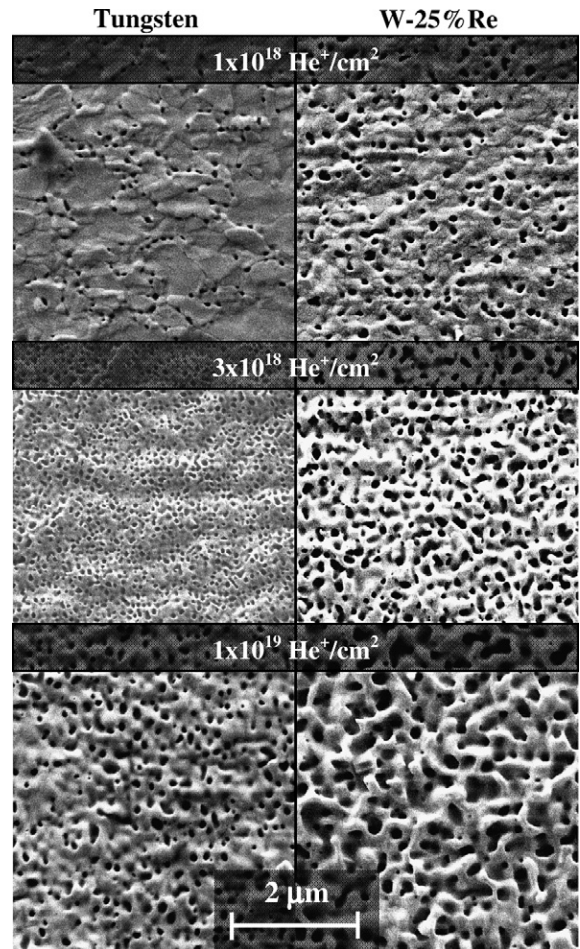


Fig. 6. Pure tungsten and W–25% Re alloy samples irradiated with 30 keV He<sup>+</sup> at 1150 °C.



tungsten, but the added rhenium improves many of the mechanical properties [8]. The first images in Fig. 6 compare pure and alloy samples irradiated to  $1 \times 10^{18}$  He<sup>+</sup>/cm<sup>2</sup>. The pure tungsten sample appears to have just reached its threshold fluence, with pores beginning to collect at grain boundaries, while the alloy sample already has fairly uniform coverage with a pore density of  $1.7 \times 10^9$  pores/cm<sup>2</sup>. The second micrographs, each representing samples subjected to a fluence of  $3 \times 10^{18}$  He<sup>+</sup>/cm<sup>2</sup>, indicate that both samples have achieved a uniform pore density, although the average pore diameter is larger on the W–25% Re sample, perhaps due to the material's lower melting point. Similarly, the samples that received fluences of  $1 \times 10^{19}$  ions/cm<sup>2</sup> also show uniform coverage of larger pores. Again, the W–25% Re alloy sample has an average pore diameter larger than the pure tungsten sample.

#### 4. Discussion

Extending the fluence scan to  $1 \times 10^{20}$  He<sup>+</sup>/cm<sup>2</sup> revealed further degradation of the tungsten surface and an extension of the porous layer deeper into the bulk material, compared to earlier results with a maximum fluence of  $10^{19}$  ions/cm<sup>2</sup> [2]. Mass loss on the high fluence sample was found to be nearly five times greater than the loss calculated due to physical sputtering alone. Future work will attempt to determine the loss mechanism. The ERD analysis revealed that the tungsten samples saturate at a 40% atomic fraction of helium for fluences of  $1 \times 10^{18}$  He<sup>+</sup>/cm<sup>2</sup> or greater.

Two new materials were evaluated using the IEC device and compared to polycrystalline tungsten samples. Single crystal tungsten was found to have a higher fluence threshold for the formation of surface pores than the polycrystalline tungsten. In addition, the single crystal samples had a lower density of surface pores at higher fluences. Alloying tungsten with 25% rhenium does not appear to improve the resistance to damage from helium implantation. The W–25% Re sample showed a lower threshold for the formation of surface pores. These samples also had a larger average pore diameter than the polycrystalline, pure tungsten at all fluences, perhaps due to the lower melting point of the alloy.

The creation of micro-pores, as well as the changes in surface morphology of the tungsten surfaces, may have an impact on the lifetime of W coatings on the first walls of inertial and magnetic

confinement fusion reactors. The high density of small pores could act as nucleation sites for cracking under the repeated shock loading environment of ICF operation. The loss of the small protrusions could result in micron-size radioactive 'dust' particles. Finally, the pores may act as release sites for He in the tungsten surface layer, potentially increasing the life of the material. Further study, both to extend the ion energy range and to evaluate the thermo-mechanical properties of these materials, will be required to evaluate the feasibility of using tungsten-coated foams for first wall use.

#### 5. Conclusions

Several conclusions have been drawn from the data presented above:

- Helium implantation of tungsten and W–25% Re to  $10^{20}$  He<sup>+</sup>/cm<sup>2</sup> created substantial surface degradation and resulted in mass loss greater than that expected from physical sputtering.
- Single crystal tungsten has a higher fluence threshold for the formation of surface pores than the polycrystalline tungsten and had a lower density of surface pores at higher helium fluences.
- Alloying tungsten with 25% rhenium does not appear to improve the resistance to damage from helium implantation. In fact, the W–25% Re samples showed a lower threshold for the formation of surface pores and had a larger average pore diameter than the polycrystalline material.

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