

# Retention and surface blistering of helium irradiated tungsten as a first wall material

S.B. Gilliam<sup>a,\*</sup>, S.M. Gidcumb<sup>a</sup>, N.R. Parikh<sup>a</sup>, D.G. Forsythe<sup>a</sup>,  
B.K. Patnaik<sup>a</sup>, J.D. Hunn<sup>b</sup>, L.L. Snead<sup>b</sup>, G.P. Lamaze<sup>c</sup>

<sup>a</sup> Department of Physics and Astronomy, University of North Carolina at Chapel Hill, Phillips Hall,  
CB #3255, Chapel Hill, NC 27599-3255, USA

<sup>b</sup> Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6136, USA

<sup>c</sup> National Institute of Standards and Technology, Gaithersburg, MD 20899-3460, USA

## Abstract

The first wall of an inertial fusion energy reactor may suffer from surface blistering and exfoliation due to helium ion irradiation and extreme temperatures. Tungsten is a candidate for the first wall material. A study of helium retention and surface blistering with regard to helium dose, temperature, pulsed implantation, and tungsten microstructure was conducted to better understand what may occur at the first wall of the reactor. Single crystal and polycrystalline tungsten samples were implanted with 1.3 MeV <sup>3</sup>He in doses ranging from 10<sup>19</sup> m<sup>-2</sup> to 10<sup>22</sup> m<sup>-2</sup>. Implanted samples were analyzed by <sup>3</sup>He(d,p)<sup>4</sup>He nuclear reaction analysis and <sup>3</sup>He(n,p)T neutron depth profiling techniques. Surface blistering was observed for doses greater than 10<sup>21</sup> He/m<sup>2</sup>. For He fluences of 5 × 10<sup>20</sup> He/m<sup>2</sup>, similar retention levels in both microstructures resulted without blistering. Implantation and flash heating in cycles indicated that helium retention was mitigated with decreasing He dose per cycle.

© 2005 Elsevier B.V. All rights reserved.

PACS: 24.30.-v; 52.40.Hf; 61.72.Ss; 61.82.Bg

## 1. Introduction

A proposed inertial fusion energy reactor operates at ~10 Hz. Each cycle begins with the injection of a pellet with a deuterium–tritium (DT) core.

Next, multiple high intensity laser beams are focused on this pellet, which leads to implosion and fusion in the core. Immediately following the fusion event, the chamber wall is subjected to intense radiation. X-rays arrive first, then reflected laser light, followed by high-energy neutrons, and finally fast and slow ion debris [1]. Most of the wall heating results from the energy deposition from X-rays and ion fluxes. Simulations of the thermal evolution at the first wall indicate that the maximum

\* Corresponding author. Tel.: +1 919 962 7160; fax: +1 919 962 0480.

E-mail address: [sgilliam@physics.unc.edu](mailto:sgilliam@physics.unc.edu) (S.B. Gilliam).

temperature reached will be 2000–3400 °C with an operating temperature greater than ~700 °C [2]. The intense radiation damage to materials directly facing the plasma, i.e., the first wall, has motivated widespread research. A major concern is erosion of the wall surface due to evaporation, physical and chemical sputtering, as well as blistering due to trapping of gaseous ions. Tungsten is a favorable choice for the material of the first wall because of its lower physical and chemical sputtering yields and high melting point of 3410 °C [1–3].

Implantation of helium, with energies on the order of 1 MeV, can give rise to the formation of He bubbles about 1 µm beneath the surface. As the helium bubbles grow they cause blistering of the surface, which leads to repeated surface exfoliation of ~1 µm thick layers [4]. In previous studies it was observed that blistering occurs in most helium-implanted materials at doses around  $10^{21} \text{ m}^{-2}$  and exfoliation occurs around  $10^{22} \text{ m}^{-2}$  [5]. If we consider, for example, a He flux of  $\sim 3 \times 10^{18} \text{ ions/m}^2 \text{ s}$ , then a 1 µm thick layer would exfoliate about once per hour resulting in unacceptable surface erosion over the course of a year.

The objective of this study was to investigate the helium retention and surface blistering characteristics of tungsten with regard to helium dose and temperature. Ultimately, the goal was to determine if helium retention and its damaging effects can be mitigated by the cyclic nature of the helium irradiation and high temperature thermal spikes within the IFE reactor. Helium implantation and annealing conditions were chosen in an effort to imitate conditions at the first wall. In reality, the helium ion bombardment and heating would occur at much faster time scales. Also, the exact timing of the helium bombardment within the thermal evolution of the first wall is not well known. Tungsten samples were implanted at a temperature near the expected base operating temperature (850 °C) followed by flash annealing at 2000 °C. Implanting  $^3\text{He}$  ions allowed the measurement of helium retention by  $^3\text{He(d,p)}^4\text{He}$  nuclear reaction analysis and  $^3\text{He(n,p)}\text{T}$  neutron depth profiling.

The study presented here consists of three major components as follows: (i) determination of the critical helium dose for which surface blistering occurs, (ii) investigation of the effects of microstructure (single crystal vs. polycrystalline) on helium retention, and (iii) a study of how helium retention is affected by cyclic implantation and flash annealing.

## 2. Experimental

The size of single crystal and polycrystalline tungsten samples were  $\sim 8 \times 50 \text{ mm}^2$  and  $\sim 1.0 \text{ mm}$  thick. Preparation of the single crystal tungsten samples involved extensive grinding and polishing with a final step of 3 µm diamond polishing. All tungsten samples were implanted with a 1.3 MeV beam of  $^3\text{He}$  with an incident angle of 4.5° from the surface normal. The slight tilt of the sample was to avoid accidental channeling of He in single crystal tungsten. According to SRIM-2000.40 code [6], the projected range of the  $^3\text{He}$  ions in tungsten was 1.73 µm with a longitudinal straggle of 0.21 µm.

Use of a  $5 \times 5 \text{ mm}^2$  aperture in the beam line allowed selection of the implantation beam size. Due to beam spread between the aperture and target, the actual  $^3\text{He}$  implantation area was approximately a  $6 \times 6 \text{ mm}^2$ . Targets were implanted at a temperature of 850 °C. After implantation high temperature heating was conducted at 2000 °C. The helium doses ranged from  $10^{19} \text{ He/m}^2$  to  $10^{22} \text{ He/m}^2$ . The  $^3\text{He}$  beam currents used were 0.1–1.0 µA, depending upon the implantation dose. The effect of dose rate was not considered in this study. All implantation, flash heating, and analysis were conducted in an ultra high vacuum environment without breaking the vacuum.

A 2.5 MV Van de Graaff accelerator was used to generate the ion beams. A beam profile monitor (BPM) was used extensively for helium implantations to ensure that the beam profile was uniform over the  $6 \times 6 \text{ mm}^2$  implantation region. The water-cooled sample holder used in these experiments did not allow for measurement of the beam current hitting the target. Therefore, a Faraday cup located between the BPM and the target chamber (~0.5 m away from the target) was used to calibrate the BPM output. In addition, a surface barrier Si detector placed at 160° was employed to monitor the beam fluctuations. The BPM output maintained proportionality to the backscattered ion yield in the monitor. Error in the dosimetry was estimated to be ~10%.

The computer controlled implantation sequence involved a custom computer program with two separate threads running in parallel, one for temperature control, and the other for dosimetry control. The dosimetry control thread read the signals from two separate digital current integrators to calibrate the BPM/Faraday cup ratio, then removed the Faraday cup from the beam path until the proper dose

was implanted. This was repeated as many times as necessary to implant the total dose in the number of steps desired. Resistive heating of the tungsten samples was accomplished by passing an AC current through the sample, and the temperature was monitored by an infrared thermometer. The temperature control thread used a PID control algorithm with the infrared thermometer as a feedback signal, and an analog control signal going to a solid state power controller to adjust the temperature. During implantation the temperature was held at  $850\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ , and after implantation the temperature was raised to  $2000\text{ }^{\circ}\text{C} \pm 50\text{ }^{\circ}\text{C}$  in approximately 10 s. The sample was held at high temperature for 2 s and cooled down in  $\sim 10\text{ s}$  to  $850\text{ }^{\circ}\text{C}$  for the next implantation.

Nuclear reaction analysis (NRA) was conducted at room temperature using the  ${}^3\text{He}(\text{d},\text{p}){}^4\text{He}$  reaction ( $Q = 18.352\text{ MeV}$ ) [7]. The cross section for this reaction has a broad maximum at a deuteron energy of  $\sim 425\text{ keV}$  [8]. Energy loss calculation by SRIM code [6] showed that deuterons incident on tungsten with an energy of 780 keV reduce to  $\sim 425\text{ keV}$  at the implantation depth of  $1.73\text{ }\mu\text{m}$ . The calculation was verified experimentally by implanting a tungsten sample with  $1.3\text{ MeV } {}^3\text{He}$  to a dose of  $5 \times 10^{20}\text{ ions/m}^2$  and then measuring the proton yield with deuteron energies ranging from 700 to 920 keV in 20 keV increments.

A 2 mm diameter aperture was used to constrain the analyzing deuteron beam. Due to beam spread between the aperture and target, the actual analyzed region on the target was  $\sim 2.3\text{ mm}$  in diameter, which was considerably smaller than the  ${}^3\text{He}$  implantation area. This ensured that the deuteron beam was striking the tungsten target well within the implanted area. A silicon detector with a  $1500\text{ }\mu\text{m}$  depletion depth was set at a scattering angle of  $155^{\circ}$  and presented a solid angle of 3 msr to the target. The reaction products included both protons and alpha particles. At a scattering angle of  $155^{\circ}$ , the protons and alpha particles are emitted at energies 13.3 MeV and 2.1 MeV, respectively [8,9]. A  $12.6\text{ }\mu\text{m}$  thick aluminized Mylar foil was placed in front of the detector to stop the backscattered deuterons and alphas.

A typical NRA spectrum is shown in Fig. 1. In addition to the protons of interest near 13 MeV from the  ${}^3\text{He}(\text{d},\text{p}){}^4\text{He}$  reaction, the deuteron beam triggers other nuclear reactions which emit protons. These are (d,p) reactions with carbon and oxygen impurities on the surface. Also, the D(d,p)T reaction occurs because the incident deuterons react

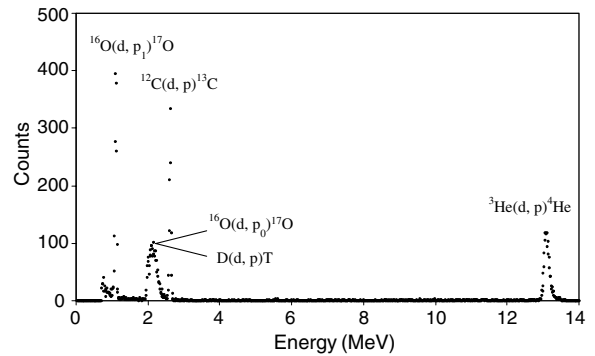


Fig. 1. Typical NRA spectrum resulting from 780 keV deuterons incident on tungsten implanted with  $1.3\text{ MeV } {}^3\text{He}$ . All peaks are protons emitted by the denoted reactions.

with previously implanted deuterons. All of these additional protons have much lower energies ( $< 3\text{ MeV}$ ) and do not interfere with the peak near  $13\text{ MeV}$ .

Deriving a  ${}^3\text{He}$  concentration vs. depth profile from NRA data requires deconvolution of the raw proton spectrum, considering the energy dependence of the reaction cross section and energy loss of the incoming deuterons and emitted protons. Instead of using this deconvolution procedure to get the absolute profile, we have tried to compare the relative  ${}^3\text{He}$  retained in the samples under the different experimental conditions. The proportionality of the total proton yield from the  ${}^3\text{He}(\text{d},\text{p}){}^4\text{He}$  reaction to the amount of  ${}^3\text{He}$  in the sample is likely a good approximation provided the  ${}^3\text{He}$  profile has not changed significantly in depth or distribution. In order to verify this, selected samples were analyzed by neutron depth profiling at the National Institute of Standards and Technology (NIST) NDP facility [10,11].

Since the neutrons lose negligible energy on penetrating the sample, the  ${}^3\text{He}(\text{n},\text{p})\text{T}$  reaction can be used to obtain a relative  ${}^3\text{He}$  concentration profile. Comparison to a known standard gives the absolute scale of the concentration profile. The reaction produces protons with an initial energy of 572 keV [10], which were detected by a surface barrier detector placed normal to the sample surface. Background radiation due to electrons and photons was subtracted from the proton spectra by measurements of unimplanted single crystal W under identical conditions. The  ${}^3\text{He}$  concentration profiles were determined by considering the proton stopping power of tungsten as compiled by Ziegler [12]. The depth scale is obtained by deriving stopping power

values,  $S(E) = dE/dx$ , calculated with the computer code PROFILE for each layer in the sample. Mathematically, the relationship between the depth traveled in the medium,  $x$ , and residual energy,  $E(x)$ , can be expressed as

$$x = \int_{E(x)}^{E_0} dE/S(E), \quad (1)$$

where  $E_0$  is the initial energy of the particle, which is determined solely by the reaction kinematics as the incoming neutron contributes negligible kinetic energy and momentum. The concentration is normalized by comparing the  $^3\text{He}$  measurements with  $^{10}\text{B}(n,\alpha)$  measurements made in the identical geometry. The well-known cross section ratio of the two reactions (independent of energy at thermal energies and below) is used to determine the absolute  $^3\text{He}$  concentration. A boron implanted standard with a concentration known to 0.6% accuracy was used in the comparison.

### 3. Results and discussion

#### 3.1. Surface blistering at a critical dose of $^3\text{He}$

Four polycrystalline tungsten samples were implanted with  $^3\text{He}$  (doses ranged from  $10^{21}$   $\text{He}/\text{m}^2$  to  $10^{22}$   $\text{He}/\text{m}^2$ ) in an effort to determine the critical dose required for surface blistering. Visual observation of surface blistering was noted after flash heating the samples at 2000 °C. Scanning electron microscopy (SEM) images (see Fig. 2) indicated that the size of the blister formations varied significantly ( $\sim 20$ – $150$   $\mu\text{m}$ ). Helium irradiation studies on various materials [5] have revealed that the size of the blisters is less dependent on implanted helium dose and more dependent on the helium ion energy (range) in the target material.

The sample implanted with  $10^{22}$   $\text{He}/\text{m}^2$  (see Fig. 2(a)) blistered quite severely. In fact, visual observation and SEM imaging indicated that the surface was flaky and exfoliating. Subsequent experiments were conducted by systematically reducing the implanted helium dose. The remaining three samples were implanted with  $5 \times 10^{21}$   $\text{He}/\text{m}^2$ ,  $2 \times 10^{21}$   $\text{He}/\text{m}^2$  (Fig. 2(b)), and  $1 \times 10^{21}$   $\text{He}/\text{m}^2$  (Fig. 2(c)). All of these samples exhibited surface blistering over the entire  $6 \times 6$   $\text{mm}^2$  helium implantation area except for the sample implanted with  $1 \times 10^{21}$   $\text{He}/\text{m}^2$ . This sample blistered only slightly in a small region near the center of the implantation area, possibly due to

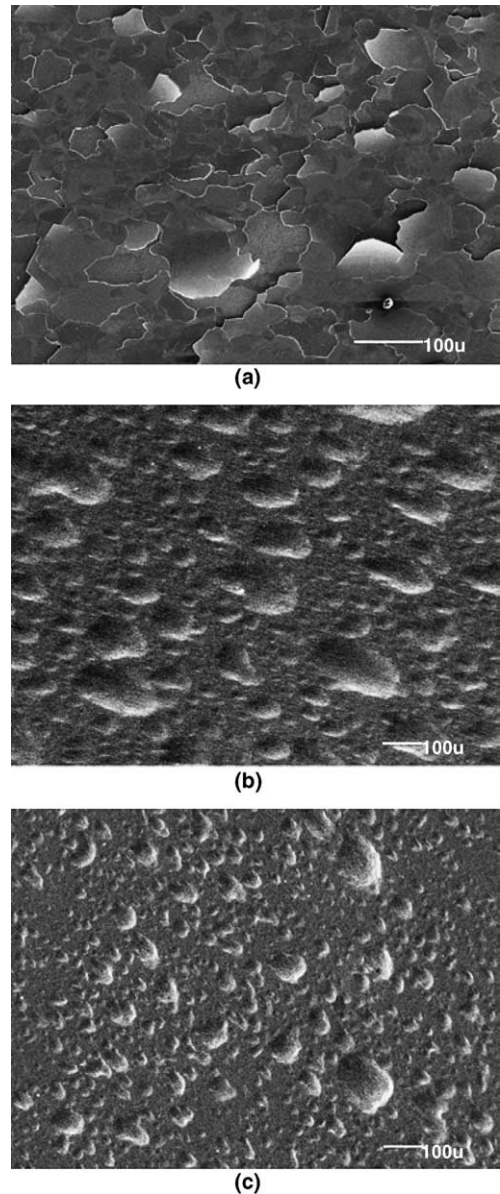


Fig. 2. SEM images of polycrystalline tungsten implanted with (a)  $10^{22}$   $\text{He}/\text{m}^2$ , (b)  $2 \times 10^{21}$   $\text{He}/\text{m}^2$ , and (c)  $1 \times 10^{21}$   $\text{He}/\text{m}^2$  at 850 °C and then flash heated at 2000 °C.

a slight non-uniformity in the beam profile. From these observations, it was concluded that the critical dose for surface blistering under these implantation and annealing conditions was  $\sim 10^{21}$   $\text{He}/\text{m}^2$ .

The SRIM simulation of 1.3 MeV  $^3\text{He}$  implanted into tungsten indicated a value of  $\sim 25000$  (atoms/ $\text{cm}^3$ )/(atoms/ $\text{cm}^2$ ) at the peak helium concentration. Multiplying this value by the implanted helium dose gives the peak atomic density of helium in the

implanted target. The atomic density of tungsten is  $6.34 \times 10^{22}$  atoms/cm<sup>3</sup>. From these high dose experiments it was concluded that polycrystalline tungsten experiences surface blistering when the peak He concentration is greater than  $\sim 4$  at.%. Recall that the sample implanted with  $10^{22}$  He/m<sup>2</sup> was observed to suffer from surface flaking and exfoliation, but the sample implanted with  $5 \times 10^{21}$  He/m<sup>2</sup> did not. Thus, one can conclude that the peak helium concentration necessary to cause surface exfoliation is between 20 and 40 at.%.

An atomic force microscopy (AFM) scan provided a measure of the typical blister cap height. The blister caps were approximately 1.9  $\mu\text{m}$  tall, which is comparable to the projected range (1.7  $\mu\text{m}$ ) of the implanted <sup>3</sup>He ions. A correspondence between blister cap height and implanted ion range has been observed for a variety of materials and helium irradiation energies [5]. This would suggest that helium bubbles form near the depth of peak helium concentration. Annealing to 2000 °C enhances the bubble growth and increases the bubble pressure leading to surface blistering.

### 3.2. Comparison of <sup>3</sup>He retention in single crystal and polycrystalline tungsten

In these experiments, single crystal and polycrystalline tungsten samples were each implanted with  $5 \times 10^{20}$  He/m<sup>2</sup> at 850 °C. Since this dose was about half of the critical dose for blistering, it was expected that significant helium trapping and bubble growth would occur. It was also expected that at this dose level and implant temperature the as-implanted samples would retain nearly 100% of the implanted helium.

Immediately after implantation, the samples were cooled to room temperature and analyzed by nuclear reaction analysis with a 780 keV deuteron beam. The samples were then flash heated at 2000 °C and reanalyzed with the same dose of 780 keV deuterons. Since the implanted helium dose, analyzing deuteron dose, and expected helium profile were the same for all three samples, the total proton yield was used as a relative measure of helium retention.

The NRA data collected for the single crystal and polycrystalline tungsten samples both before and after the 2000 °C anneal were remarkably similar. Considering the estimated 10% error in dosimetry and the statistical error in counting, the total proton yields were the same within experimental error.

Thus, it was concluded with certainty that the helium retention levels were identical.

The data indicates that the single crystal and polycrystalline tungsten samples retained the same amount of helium. Annealing at 2000 °C did not affect the total proton yields within the bounds of experimental uncertainty. These sample surfaces did not blister at any time during the experiments. From these observations it was concluded that the implantation conditions resulted in strong enough helium trapping such that heating to 2000 °C would not allow detrapping of helium.

### 3.3. Effect of implanting <sup>3</sup>He and annealing in multiple cycles to reach the same total dose

The set of experiments conducted on single crystal and polycrystalline tungsten at a dose of  $5 \times 10^{20}$  He/m<sup>2</sup> indicated that the implanted helium was strongly trapped and immobile. Annealing at 2000 °C resulted in no discernible change in helium retention based on NRA data. One of the objectives of this research was to determine if helium retention could be reduced under certain implantation and annealing conditions. A new set of experiments was performed at a lower helium dose, and a different approach to the implantation and annealing process was introduced.

Single crystal and polycrystalline tungsten samples were implanted with 1.3 MeV <sup>3</sup>He at 850 °C to a dose of  $10^{19}$  He/m<sup>2</sup>. In these experiments the total helium dose was implanted in multiple steps, heating to 2000 °C between each step. Computer controlled automation made these experiments possible. This experimental approach was developed as an effort to reproduce conditions in an IFE reactor since the first wall is subjected to cycles of helium bombardment and high temperatures.

Samples were implanted and annealed in 1, 10, 100, and 1000 steps to reach a total implanted dose of  $10^{19}$  He/m<sup>2</sup>. The target was heated to 850 °C, implanted with the appropriate fraction of the total dose, flash-annealed to 2000 °C, returned to 850 °C, and the process was repeated. After the desired number of cycles completed, the sample was quickly brought to room temperature. The time required for these experiments varied between  $\sim 0.5$  h for one cycle to  $\sim 24$  h for 1000 cycles.

For the samples implanted with the total helium dose in a single step, NRA data was collected before and after heating at 2000 °C. This was done to determine if a single step dose of  $10^{19}$  He/m<sup>2</sup> led

to helium trapping and bubble formation significant enough that heating would not affect the helium retention. Fig. 3(a) and (b) shows the proton spectra collected for the various step sizes of helium implanted into the single crystal and polycrystalline samples, respectively. The difference in proton yields between the two figures is due to differing deuteron doses. It is clear that the implantation step size between annealing cycles affects the amount of retained helium.

The total proton yields from the  $^3\text{He}(d,p)^4\text{He}$  reaction were used as a relative measure of the retained helium. For the single step implantations of helium in both single crystal and polycrystalline tungsten, flash annealing at 2000 °C produced no measurable change in helium retention. The total number of proton counts before and after annealing were the same within about ~5%. The helium retention is strongly dependent on the number of cycles, and hence the size of the helium dose in each cycle. Single crystal tungsten exhibits the most pronounced response to a change in the number of implantation and annealing cycles.

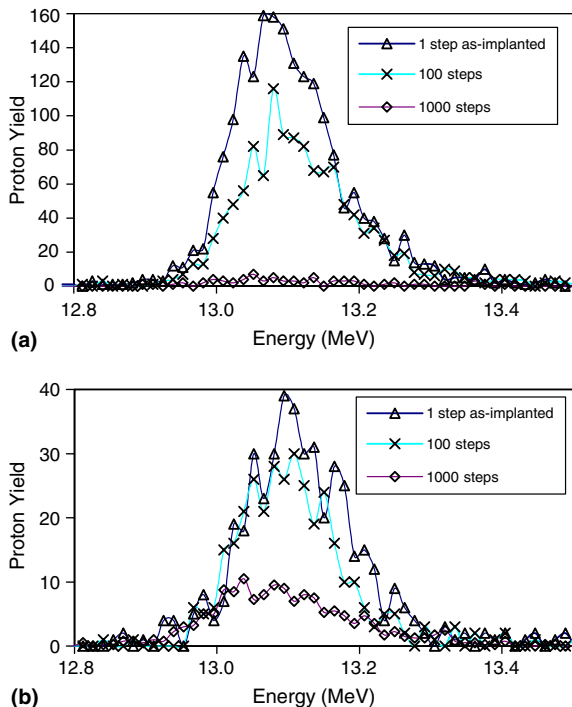


Fig. 3. Proton spectra for (a) single crystal and (b) polycrystalline tungsten implanted at 850 °C and flash heated at 2000 °C in 1, 100, and 1000 cycles to reach a total dose of  $10^{19}$  He/m<sup>2</sup>. The sample implanted with the total dose in one cycle was analyzed before and after the 2000 °C heating.

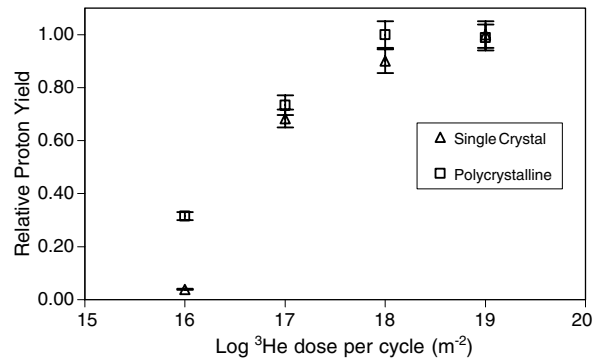


Fig. 4. Relative proton yields for single crystal and polycrystalline tungsten samples implanted at 850 °C and flash heated at 2000 °C in 1, 10, 100, and 1000 cycles to reach a total dose of  $10^{19}$  He/m<sup>2</sup>. Vertical axis represents the percentage of retained helium compared to the sample implanted and heated in a single cycle.

Fig. 4 shows the proton yield, which is the measure of helium retention, relative to that measured for the sample implanted and annealed in a single cycle. Both single crystal and polycrystalline tungsten showed a similar reduction in helium retention as the helium dose per cycle decreased. When the helium dose implanted in each cycle was  $10^{16}$  He/m<sup>2</sup>, polycrystalline tungsten retained ~30% of the implanted helium while single crystal tungsten retained only ~5%. The difference in helium retention behavior for single crystal vs. polycrystalline may be due to trapping at the grain boundaries in the polycrystalline material. In addition to the vacancies created by implantation, grain boundaries act as nucleation sites for helium bubble growth. The extent of helium trapping and bubble growth is apparently affected quite considerably by the tungsten microstructure.

Helium bubble formation may be important in the retention of helium. The hypothesis is that once helium bubbles form, the helium will not diffuse away at 2000 °C. For small implant doses, helium can diffuse away during the anneal stage before the local helium concentration becomes high enough to increase the probability of the formation of complex He-vacancy clusters which have a higher trapping energy. This would explain why the magnitude of the helium dose per implantation cycle seems to affect helium retention significantly.

For an implanted helium dose of  $10^{16}$  He/m<sup>2</sup>, the peak concentration of helium in the tungsten target is calculated to be  $3.9 \times 10^{-5}$  at.%. If the peak helium concentration is around  $10^{-5}$  at.%, then

helium retention in single crystal tungsten can be significantly reduced under these implantation and annealing conditions.

Another set of experiments was conducted on single crystal tungsten implanted with a total dose of  $10^{20}$  He/m<sup>2</sup>. One sample was implanted and annealed in a single cycle, whereas the other sample was implanted and annealed in 1000 cycles. The implantation, heating, and analysis conditions were the same as before. The sample implanted with  $10^{20}$  He/m<sup>2</sup> in a single step was analyzed before and after the 2000 °C anneal. Once again the proton yields measured before and after annealing varied by less than 10%.

For the sample implanted in 1000 cycles ~75% of the implanted helium was retained. When a total dose of  $10^{19}$  He/m<sup>2</sup> was implanted in 100 cycles, the percentage of retained helium was ~70% (see Fig. 4). In both cases the <sup>3</sup>He dose per cycle was  $10^{17}$  He/m<sup>2</sup>, which provides support for the conclusion that retained helium is dependent on the implanted helium dose per cycle. Table 1 presents the helium retention results for single crystal and polycrystalline tungsten samples for various total helium doses and numbers of implant/anneal cycles. The data is quite compelling and makes it clear that helium retention is strongly dependent on dose per cycle.

Two single crystal W samples, one with a single step dose of  $10^{20}$  He/m<sup>2</sup>, followed by flash heating at 2000 °C, and another with the same total dose implanted in 1000 steps with flash heating at 2000 °C between steps, were analyzed using the NDP technique. Fig. 5 shows the derived concentra-

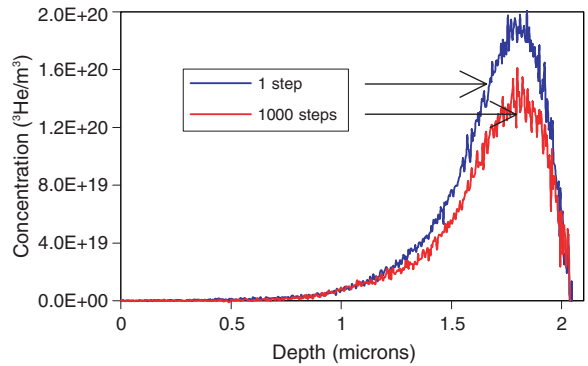


Fig. 5. Helium depth profile for single crystal W implanted at 850 °C and flash heated at 2000 °C in 1 and 1000 cycles to reach a total dose of  $10^{20}$  He/m<sup>2</sup>.

tion vs. depth profiles for these two samples. The peak helium concentration for both of these spectra are at a depth of ~1.7 μm, as expected from SRIM simulations and NRA measurements. Comparison of single step vs. multiple step implants showed that the implanted <sup>3</sup>He profile neither shifted in depth nor did it change shape significantly. This supports using the total proton yield obtained from NRA measurements at the fixed deuteron energy as a relative measure of <sup>3</sup>He retained in the samples. It is surprising that the <sup>3</sup>He profile in a sample implanted in a single step is so similar to that of a sample implanted and annealed in 1000 steps. One would expect the profile in the latter case to be perhaps more spread out due to heat-driven diffusion. Some future plans involve studying thermal desorption and diffusion of helium in tungsten which may offer some explanations.

Table 1

Helium retention results for (a) single crystal and (b) polycrystalline tungsten implanted and annealed in various numbers of cycles to reach total doses ranging from  $10^{19}$  He/m<sup>2</sup> to  $10^{20}$  He/m<sup>2</sup>

Dose/step	Total dose			
	1E19/m <sup>2</sup>	3E19/m <sup>2</sup>	5E19/m <sup>2</sup>	1E20/m <sup>2</sup>
<i>(a) Single crystal</i>				
	100% (1)	100% (1)	100% (1)	100% (1)
1E18/m <sup>2</sup>	85% (10)			
1E17/m <sup>2</sup>	65% (100)	70% (300)	75% (500)	75% (1000)
1E16/m <sup>2</sup>	5% (1000)			
<i>(b) Polycrystalline</i>				
	100% (1)	100% (1)	100% (1)	100% (1)
1E18/m <sup>2</sup>	100% (10)			
1E17/m <sup>2</sup>	75% (100)	75% (100)	75% (500)	75% (1000)
1E16	30% (1000)	30% (3000)		

The number in parentheses indicates the number of cycles, and the percentage retained is compared to the sample implanted in a single step.

$^3\text{He}$  concentration measured by NDP gave values of  $[8.8 \pm 0.9] \times 10^{19} \text{ He/m}^2$  for the single step implant and  $[6.7 \pm 0.7] \times 10^{19} \text{ He/m}^2$  for the 1000 step implant. Analysis of the single step implant indicated  $\sim 12\%$  lower dose compared to the intended implant dose. This difference can be accounted for by a combination of uncertainty in implantation dosimetry and in the NDP analysis. NDP analysis indicated that  $^3\text{He}$  retention by the 1000 step implant process was  $\sim 76\%$  compared to the single step implant. These results are comparable to those obtained by NRA analysis.

Based on the data and results presented thus far, it was concluded that implanting single crystal tungsten at  $2000^\circ\text{C}$  would result in almost no helium retention because this approximates an infinitesimally small helium dose per cycle. This prediction was confirmed by implanting single crystal samples with  $10^{19} \text{ He/m}^2$  and  $10^{20} \text{ He/m}^2$  in a single step at  $2000^\circ\text{C}$ . Both cases resulted in no detectable helium retention.

To evaluate the effect of post-implantation heating on the helium retention a single crystal tungsten sample was implanted with a straight dose of  $10^{20} \text{ He/m}^2$  at  $850^\circ\text{C}$ . NRA data was collected on the as-implanted sample. Afterwards, the sample was flash heated to  $2000^\circ\text{C}$  1000 times and analysis was repeated. Data indicated less than 10% reduction in helium retention compared to the as-implanted sample. These results suggest that repeated heating does not affect helium retention significantly after a sufficiently high dose has been implanted at lower temperature.

#### 4. Conclusions

In this study of helium irradiated tungsten, the experimental conditions were chosen as an attempt to imitate conditions at the first wall of an IFE reactor. The results of irradiation with  $^3\text{He}$  doses in the range of  $10^{19}$ – $10^{22}$  ions/ $\text{m}^2$  were investigated, either in a single step or in multiple cycles of implantation and flash annealing. Helium retention in the irradiated samples was analyzed by the  $^3\text{He}(\text{d,p})^4\text{He}$  reaction at a single deuteron energy corresponding to the maximum yield of protons and by the  $^3\text{He}(\text{n,p})^3\text{H}$  reaction.

Single crystal and polycrystalline tungsten samples implanted with  $5 \times 10^{20} \text{ He/m}^2$  at  $850^\circ\text{C}$  exhibited similar helium retention characteristics. A subsequent flash anneal at  $2000^\circ\text{C}$  had no effect on the retention of helium. Thus, the conclusion is that this dose was low enough to avoid surface blis-

tering, but high enough to result in strong helium trapping and bubble growth.

Surface blistering of polycrystalline tungsten samples occurred at helium doses greater than or equal to  $10^{21} \text{ He/m}^2$ , which corresponds to a peak helium concentration of  $\sim 4 \text{ at.}\%$ . Helium doses that resulted in surface blistering ranged between  $10^{21} \text{ He/m}^2$  and  $10^{22} \text{ He/m}^2$ . In all cases the largest blisters were  $\sim 100 \mu\text{m}$  in diameter, which indicated that the size of the blisters was independent of the helium dose. Also, the height of a typical blister cap was  $\sim 1.9 \mu\text{m}$ , which is comparable to the  $1.7 \mu\text{m}$  projected range of the implanted helium ions. This supports reports by others [5] that blister size closely corresponds to the implantation depth.

It was found that helium retention may be mitigated by cyclic helium implantation and high temperature heating. Implantation at  $850^\circ\text{C}$  and annealing at  $2000^\circ\text{C}$  in cycles to reach total doses of  $10^{19} \text{ He/m}^2$  and  $10^{20} \text{ He/m}^2$  produced interesting conclusions. When  $10^{19} \text{ He/m}^2$  was implanted into single crystal tungsten in 1000 cycles ( $10^{16} \text{ He/m}^2$  per cycle), the observed helium yield dropped to  $\sim 5\%$  compared to  $\sim 30\%$  for polycrystalline tungsten under the same conditions. Single crystal tungsten experiences greater losses of helium at these low doses and high temperature annealing due to the lack of grain boundaries in the single crystal structure. Experimental data also indicated that helium retention may be a function of dose per implant/anneal cycle with little dependence on total implanted dose.

Considering all the findings of this study, the first wall of an IFE fusion reactor will potentially suffer from significant damage due to high fluences of helium ions and intense temperatures. Helium trapping and bubble formation just below the surface of the first wall material may result in surface blistering and exfoliation at critical helium doses. However, the data here suggests that maintaining the helium ion flux per fusion event below a threshold level may diminish the damaging effects of helium radiation. Further research is necessary to investigate the effects of variable helium energy and simultaneous lattice damage (from neutrons and other fusion reaction ion debris) on helium retention and blistering.

#### Acknowledgements

This work was supported under the US Department of Energy High Average Power Laser



Program managed by the Naval Reactor Laboratory through subcontract with the Oak Ridge National Laboratory. Also, the authors would like to thank Dr Hugon Karwowski for his helpful discussions with this project.

## References

- [1] A. Hassanein, V. Morozov, Fusion Eng. Des. 63&64 (2002) 609.
- [2] A.R. Raffray, G. Federici, A. Hassanein, D. Haynes, Fusion Eng. Des. 63&64 (2002) 597.
- [3] A. Hassanein, V. Morozov, V. Tolkach, V. Sizyuk, I. Konkashbaev, Fusion Eng. Des. 69 (2003) 781.
- [4] D. Kaminsky, S.K. Das, J. Nucl. Mater. 76&77 (1978) 256.
- [5] G.M. McCracken, Rep. Prog. Phys. 38 (1975) 241.
- [6] Available from: <<http://www.srim.org/>>.
- [7] G. Vizkelethy, in: J.R. Tesmer, M. Nastasi (Eds.), Handbook of Modern Ion Beam Materials Analysis, Materials Research Society, Pittsburgh, 1995, p. 141.
- [8] W.H. Geist, The  $^3\text{He}(d,p)^4\text{He}$  Reaction at Low Energies, Doctoral Dissertation, UNC-Chapel Hill, 1998.
- [9] S.T. Picraux, F.L. Vook, J. Nucl. Mater. 53 (1974) 246.
- [10] R.G. Downing, G.P. Lamaze, J.K. Langland, S.T. Hwang, NIST J. Res. 98 (1993) 109.
- [11] G.P. Lamaze, H.H. Chen-Mayer, J.K. Langland, R.G. Downing, Surf. Interface Anal. 25 (1997) 217.
- [12] J.F. Ziegler, The Stopping and Ranges of Ions in Matter, vol. 4, Pergamon, New York, 1977.