Fusion Engineering and Design 109–111 (2016) 1179–1182

Contents lists available at ScienceDirect

Fusion Engineering and Design

journal homepage: www.elsevier.com/locate/fusengdes

# Tritium desorption and tritium removal from tungsten pre-irradiated with helium

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## HIGHLIGHTS

- Helium pre-irradiation effects on tritium desorption/retention in tungsten was investigated.
- Tritium in helium-irradiated tungsten was desorbed at higher temperatures.
- Long term tritium release was significantly delayed in helium irradiated tungsten.
- Helium effects on tritium desorption/retention depended on pre-irradiated helium fluence.

## ARTICLE INFO

*Article history:*
Received 31 August 2015
Received in revised form
18 December 2015
Accepted 23 December 2015
Available online 7 January 2016

*Keywords:*
Tritium retention
Tungsten
Ion irradiation
Thermal desorption
Isochronal heating

## ABSTRACT

In this study, 1 keV $DT^+$ ion irradiation was performed on tungsten pre-irradiated with helium. The thermal desorption behavior and the reduction of tritium retention during vacuum preservation at room temperature, as well as isochronal annealing were investigated using an IP technique, taking advantage of the fact that tritium detection is nondestructive and is highly sensitive. At a pre-irradiated helium fluence of $1 \times 10^{17}$ He/cm$^2$, retained tritium tended to be desorbed at higher temperatures when compared to no helium irradiation case. Tritium retention during preservation in vacuum and during isochronal annealing became smaller with increasing helium fluence up to $1 \times 10^{17}$ He/cm$^2$. At a helium fluence of $1 \times 10^{18}$ He/cm$^2$, the reduction of tritium retention was found to be greater compared to $1 \times 10^{17}$ He/cm$^2$. The results indicate that helium irradiation has a significant influence not only on the thermal desorption temperature of tritium but on longtime tritium reduction at room and elevated temperatures.



## 1. Introduction

Tritium retention behavior in plasma-facing materials is a crucially important issue for tritium safety and removal. Tungsten is a primary candidate as a plasma-facing material in ITER and as a DEMO reactor because of its high melting point, low physical sputtering yield and relatively high thermal conductivity [1]. In a fusion device using deuterium and tritium as a fuel, energetic tritium particles are implanted into the plasma-facing material and some of them are retained in the material. Thus, the retention behavior of implanted tritium needs to be studied. In most cases, studies on hydrogen retention behavior are done using linear heating methods, mainly thermal desorption spectroscopy (TDS), just after hydrogen uptake. In TDS analysis, it is unclear that how much of the retained tritium is released during long period of time at room and elevated temperatures after irradiation. Also, the measurements of hydrogen and deuterium retention with TDS can be done only once since TDS is a destructive analysis. On the other hand, measurement of tritium retention, such as using an imaging plate technique, is a non-destructive analysis, which allows tritium retention measurements to be performed many times. Using these advantages in tritium detection, the authors have studied tritium retention behavior and found that the long term tritium release in tungsten irradiated with energetic tritium occurs more rapidly as compared with carbon materials [2]. In order to precisely predict tritium retention and tritium removal, long term tritium release at room and elevated temperatures needs to be studied. In fusion devices, helium particles produced by deuterium-tritium nuclear reaction are also implanted into plasma-facing materials together with hydrogen. Irradiation defects produced by helium irradiation, such as helium bubbles and vacancy clusters, will influence the tritium retention behavior.

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http://dx.doi.org/10.1016/j.fusengdes.2015.12.059
0920-3796/

In this study, tritium ion irradiation experiments were performed for helium pre-irradiated tungsten, and the tritium release behavior while preserved in vacuum at room temperature and also at elevated temperatures were investigated. In addition, tritium TDS analysis was done after tritium irradiation in order to investigate the effect of helium irradiation on tritium retention behavior.

## 2. Experimental

Samples used in this study were 99.9% pure polycrystalline tungsten (Nilaco co.) Before tritium irradiation, helium ions with an energy of 5 keV were bombarded onto samples using an ECR ion source at Hokkaido University [3]. The helium ion flux were estimated by measuring the sample current to be $\sim 2 \times 10^{13}$ He/cm$^2$/s, and the helium ion fluence ranged between $1 \times 10^{16}$ He/cm$^2$ and $1 \times 10^{18}$ He/cm$^2$. The sample was at room temperature during the helium irradiation. Afterwards, the samples were transported to the University of Toyama, where tritium irradiation was performed. A schematic diagram of the apparatus used for tritium ion irradiation is found elsewhere [2]. In this apparatus, Tritium-containing gas is released by heating a tritium source containing deuterium (99.5%) and tritium (0.5%). Since the fraction of tritium contained in the tritium source is much smaller than deuterium, the primary tritium-containing molecule released is considered to be the DT molecule. These DT molecules are ionized in an ion gun's ionization chamber and then are accelerated in an electric field and implanted into samples with a DT± ion energy of 1.0 keV (0.6 keV for sole T). The ion fluence estimated by measuring the sample current was $4.5 \times 10^{14}$ T/cm$^2$ ($9.0 \times 10^{16}$ D + T/cm$^2$) and the ion irradiation lasted approximately 30 min. The sample was at room temperature during the irradiation. Just after tritium irradiation, tritium desorption behavior was investigated with TDS analysis. Fig. 1 shows a schematic diagram of the TDS apparatus. In the TDS analysis, samples were heated from room temperature to 1093 K with a ramp rate of 0.5 K/s. In this analysis, tritium containing samples were heated in argon stream (100 ccm) containing methane (50 ccm) and tritium desorption was measured using a proportional counter located downstream of the sample. Since the tritium fluence in the irradiation was pretty small, tritium detection by using a quadrupole mass spectrometer in the TDS analysis was impossible. Therefore, the authors applied a proportional counter to detect tritium.

Some of tritium irradiated samples were preserved in a desiccator pumped by a rotary pump. In order to evaluate the reduction of tritium retention during the vacuum preservation, the change of tritium retention in the samples was periodically measured using an IP technique. For these measurements, the samples were attached to an IP sheet for 1 h at room temperature and a two dimensional distributed dose of β-rays was obtained by using an imaging analyzer to measure the intensity of the resulting photo-stimulated luminescence (PSL). The ion irradiation areas estimated from the IP images were 0.2 cm$^2$. The tritium-irradiated samples were simultaneously measured with standard samples containing a known quantity of tritium and a reduction of tritium in the tritium-irradiated samples was estimated by comparing the PSL intensity of the two types of samples. In order to investigate tritium reduction during baking, isochronal annealing at temperatures ranging from 423 K to 873 K in vacuum was also performed on tungsten samples just after tritium irradiation and after long-term preservation in vacuum.

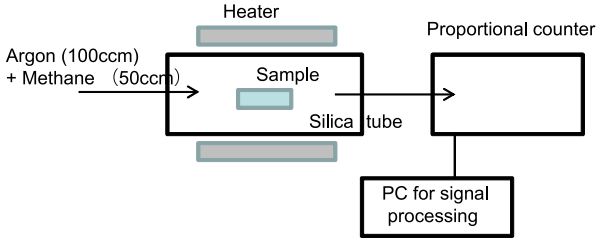


**Fig. 1.** Schematic diagram of tritium TDS apparatus.

## 3. Results and discussion

Fig. 2 shows thermal desorption spectra of tritium in tungsten, including that in graphite for comparison. Tritium retained in tungsten with no helium pre-irradiation started to desorb at around 350 K and the major peak was seen at around 800 K. This broad desorption spectra indicates that the tritium retained in the tungsten was trapped at various trap sites originally existing within the tungsten, such as grain boundaries, dislocations and point defects, with various trap energies and at irradiation defects caused by the tritium ion irradiation. The thermal desorption spectra of tritium in helium pre-irradiated tungsten with a helium fluence of $1 \times 10^{17}$ He/cm$^2$ showed the development of a significant desorption rate at around 700 K. Helium implantation with an energy of 5 keV can knock on tungsten atoms and create cascade collisions, producing irradiation defects, such as vacancy clusters, dislocation loops and helium bubbles, in the region of implanted depth [4] and the density of the defects would be much higher than that produced by only DT ion irradiation. Since this type of irradiation defects could act as a trap site for hydrogen [5]. Also, the average implantation depth of helium with an energy of 5 keV as estimated by SRIM is 19.7 nm, which is relatively close to the implantation depth of tritium with an energy of 0.6 keV, 8.5 nm. Therefore, the implanted tritium could be preferentially trapped in the defects caused by helium irradiation. For these reasons, the development of a desorption rate around 700 K could be attributed to tritium trapping in helium irradiation defects. In the case of graphite, a desorption peak was seen at a much higher temperature, around 1000 K, similar to earlier studies [6]. This is owing to the much higher trap energy of C-T chemical bonds.

The reduction of tritium retention during the TDS analysis is shown in Fig. 3, which was basically calculated based on thermal desorption spectra (Fig. 2). In this figure, the amount of tritium just before the TDS analysis is normalized to unity, then this figure indicates how much of the retained tritium decreased as the sample temperature increased during the TDS analysis. In the case of no helium irradiation, the tritium retention started to decrease at around 400 K. On the other hand, in the case of tungsten pre-irradiated with helium at a fluence of $1 \times 10^{17}$ He/cm$^2$, little reduction of tritium retention was seen until 600 K and the tritium

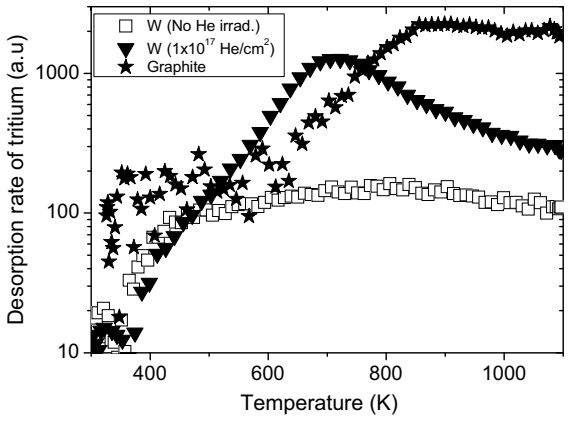


**Fig. 2.** Thermal desorption spectra of tritium for tritium irradiated tungsten and graphite.