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Deuterium retention in tungsten simultaneously damaged by high energy W ions and loaded by D atoms

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ABSTRACT

Deuterium retention was for the first time measured in tungsten samples simultaneously irradiated by W ions and exposed to D atoms at five different temperatures from 450 K to 1000 K. In order to obtain information on the defect concentration, samples were afterwards exposed to D atoms at 600 K to populate the created defects. The results were compared to different sequential damaging/exposure experiments. Synergistic effects were observed, namely, higher D concentrations were found in the case of simultaneous damaging and D-atom loading as compared to sequential damaging at elevated temperatures and populating the defects afterwards. However, the deuterium retention is still lower as compared to sequential damaging at room temperature and post-damaging annealing. The observations are explained by stabilization of defects by the presence of solute hydrogen in the bulk that would annihilate at high temperatures without the presence of hydrogen. Results of simultaneous W-ion damaging and D exposure at elevated temperatures were also compared to a sequential experiment of W-ion damaging at room temperature and then D-atom loading at high temperatures showing that thermal D de-trapping dominates deuterium retention at high temperatures.

1. Introduction

Tungsten or advanced tungsten alloys are considered to be the most suitable material for plasma-facing components in future fusion reactors such as DEMO. In these nuclear devices tritium retention in neutron-damaged tungsten will become a more significant issue. Namely, the predicted neutron damage for DEMO produced by neutrons from the fusion D-T reaction is more than one order of magnitude higher than in ITER, 2–6 dpa/fpy [1]. In order to study the influence of material displacement damage on fuel retention, high energy ions are used [2]. It was shown that fuel retention both in neutron-damaged [3] and in W-ion-damaged tungsten (so-called self-damaged W) is strongly increased as compared to undamaged tungsten (e.g. [4]). Until now all retention studies were performed by sequential high energy ion damaging and subsequent plasma/gas/atom loading of the material by hydrogen isotopes. However, in a real fusion reactor environment both implantation of energetic hydrogen ions and neutrals as well as damage creation by neutron irradiation will take place at the same time. The consequences of synergistic effects for retention are unknown. It is well known that in some metals impurities such as hydrogen, change the behaviour of defect creation and recovery, e.g. influence vacancy migration during the recovery stage [5]. Moreover, theory predicts defect stabilization in the presence of hydrogen atoms in tungsten [6,7].

In this paper we present the first experimental results on simultaneous defect creation by high energy self-ion implantation and D-atom-beam loading in tungsten. In order to sort out the observed effects comparison to a series of sequential damaging/annealing/exposure experiments is made. Namely, three sequential experimental series were performed in addition with different damaging/exposure procedures, that help to separate the processes: i) W-ion damaging at elevated temperatures + D-atom exposure at 600 K to determine the trap concentration; ii) W-ion damaging at room temperature + samples post-damaging annealing at different temperatures for one hour + D-atom loading at

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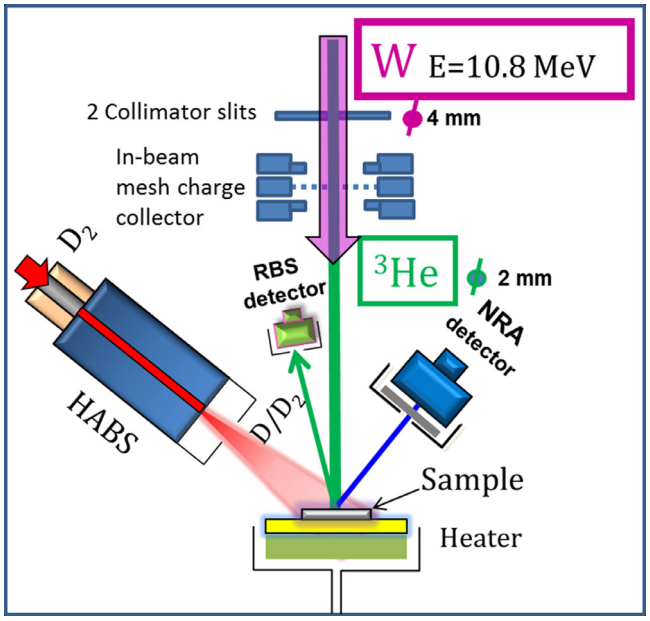

Fig. 1. Schematic figure of the INSIBA experimental set-up for simultaneous W-ion irradiation and D-atom exposure, together with the detector for Nuclear Reaction Analysis (NRA detector) and Rutherford Backscattering Spectroscopy (RBS detector).

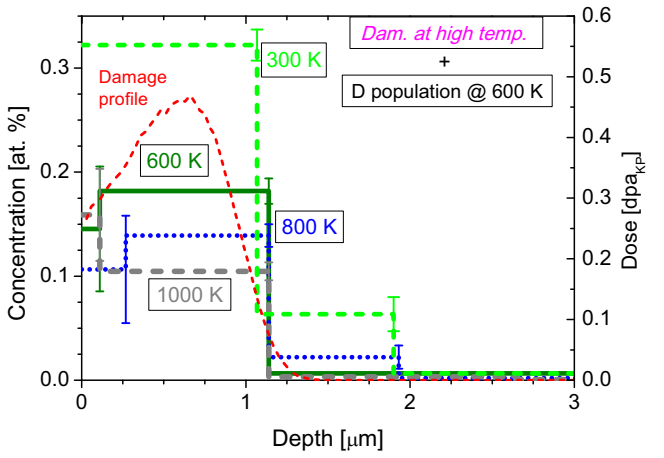

Fig. 2. Deuterium concentration depth profiles obtained on samples damaged by 10.8 MeV W^{6+} ions at different temperatures indicated in the figure and afterward exposed to D atoms for 24 h at 600 K, atom fluence 4.7×10^{23} D/m^2, for defect population. The calculated damage depth profile as obtained by SRIM is also shown in the figure by the red dashed line. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

500 K to determine the trap concentration; and iii) W-ion damaging at room temperature + D-atom exposure at elevated temperatures. We describe in detail the simultaneous W-ion damaging and D-atom loading and W-ion damaging at elevated temperatures whereas details of ii) and iii) were given in more detail in [8] and [9,10] respectively.

2. Experiment

We have used polycrystalline 99.997 wt. % hot-rolled tungsten samples (PW) manufactured by Plansee, 12×15 mm^2 in size and 0.8 mm thick. Grains are oriented parallel to the surface. Samples were chemo-mechanically polished to a mirror-like finish at Max-Planck-Institut für Plasmaphysik (IPP), Garching [11]. After polishing, samples were heated for 2 min in vacuum at 2000 K for recrystallization. This procedure enlarges the grain size to 10–50 μm [11].

The samples were mounted in the INSIBA chamber at the 2 MV Tandem accelerator at Jožef Stefan Institute. The set up shown in Fig. 1 enables in situ Nuclear Reaction Analysis (NRA) measurements before, during or after the D-atom loading (see details in [12]), and irradiation of the samples by a high energy ion beam such as W^{6+} ion beam with energy up to 10.8 MeV. An electrostatic quadrupole lens is positioned in the beam line (not shown in Fig. 1), 4.5 m before the sample, in order to gather and focus the ion beam on the sample. The ion beam is shaped by two apertures that can be changed for each irradiation/analysis. They are positioned before the ion beam mesh charge collector [13], mounted at the entrance to the experimental vacuum chamber. The ion beam mesh charge collector was specially redesigned for this experiment, enabling us to retract it out of the ion beam or placing it in the beam for current measurement.

The sample is mounted by two Ta clamps on a holder with a temperature-controlled heater, capable of heating the samples up to 1200 K. Hydrogen atom beam source (HABS) for sample exposure to D atoms is mounted on the vacuum chamber at an angle of 51° with respect to the sample surface normal. It enables us to expose samples to a beam of neutral D atoms with a flux density of 5.4×10^{18} D/m^{2}s at the NRA ion beam position. The flux density was determined by eroding an a-C:H film and measuring the crater by ellipsometry [14,15]. The atom beam is created by thermal dissociation of D_2 gas on a hot tungsten capillary that is heated to 2170 K ± 20 K for all cases. By assuming that the kinetic energy of the atoms is determined by the capillary temperature the mean energy of neutral atoms (E = 3/2 kT) in the beam is 0.28 eV. The deuterium gas pressure in the gas supply side was controlled by an all-metal leak valve and measured by an absolute capacitance manometer, Baratron by MKS. The average driving pressure during the atom exposures of tungsten samples was 26 ± 1 Pa for D_2.

In the case of the experiment simultaneous W-ion irradiation and D-atom loading and damaging at elevated temperatures samples were irradiated by W^{6+} ions with an energy of 10.8 MeV. The beam size was defined by two 4 mm circular apertures. Before starting the irradiation the beam was checked for homogeneity by observing the glow (by ionoluminescence) on a quartz glass inserted into the irradiating beam. The quartz can be placed instead of the first collimating aperture and a blue glow is observed from the side by a mirror when it is irradiated by the beam. The ion current was first set to the proper value by measuring it on the ion mesh charge collector with 77.4% geometrical transmission. The ion mesh charge collector was then retracted in order not to have a mesh-print on the sample. The stability of the current was checked every 20–30 min. The ion current was stable within 5–10% yielding an uncertainty for the total ion fluence of about 5% for all irradiated samples. The corresponding W-ion irradiation current on the sample was 1.2 nA and irradiation time was 4 h yielding a W-ion fluence of $(1.4 \pm 0.07) \times 10^{18}$ W/m^2 on the irradiated 4 mm diameter spot. The displacement damage profile as calculated by SRIM is shown in Fig. 2. With a fluence of 1.4×10^{18} W/m^2 we create a damage dose of 0.47 dpa_{KP} (Kinchin–Pease calculation, 90 eV displacement damage energy, evaluating the "vacancy.txt" output) at the peak maximum yielding a displacement rate of 3.3×10^{-5} dpa/s.

For damaging with the W-ion beam a sputter ion source is used and in the case of NRA analysis with the ^{3}He beam a duoplasmatron source is used. Therefore the NRA analysis can only be performed about one hour after the W-ion irradiation since one has to switch between these two ion sources for the tandem accelerator when changing from self-implantation to ^{3}He analysis. NRA analysis is performed by detecting protons from D(^{3}He,p)α nuclear reaction using a 1500 μm thick partially depleted Passivated Planar

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