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Isolation of peaks in TDS spectra of deuterium from ion irradiated tungsten

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Abstract

Interaction of deuterium with radiation defects in tungsten was studied by means of thermal desorption spectroscopy. The displacement damage in a recrystallized tungsten foil was produced by irradiation with 10 keV/D ions to the fluences in the range of 3-8×10^{19} D/m^2 at room temperature. The resulted thermal desorption spectra consisted of three wide overlapping peaks. It was shown that annealing of the damaged sample at 550 K and subsequent implantation with 0.67 keV/D ions allows to isolate the peak corresponding to deuterium release from vacancies.

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Keywords: deuterium; tungsten; radiation defects; thermal desorption spectroscopy.

1. Introduction

Tungsten (W) will be used as a plasma-facing material in the divertor region of ITER, and its use in future fusion devices is also considered. Although tungsten has very low hydrogen (H) solubility, presence of lattice defects, such as vacancies and vacancy clusters, significantly increases H isotope retention in W (e.g., Roth and Schmid (2011), T. Tanabe et al. (2014)). Consequently, determination of parameters of H interaction with defects in W is an important

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issue. Thermal desorption spectroscopy (TDS) is a commonly used method for such purposes. The parameters of trapping sites are usually determined either from fitting of numerically simulated TDS spectra to experimental ones (e.g., Eleveld and Van Veen (1992), Pisarev et al. (2003), Ogorodnikova et al. (2008)) or from a shift of a desorption maximum in a series of TDS measurements of identical samples performed with different heating rates [Zibrov et al. (2015)]. To make the interpretation of TDS results easier, spectra with well-resolved peaks are desirable in both approaches.

However, even experiments with W single crystals irradiated by mass-separated ion beams (with energies of several keV) to low fluences at room temperature demonstrate that the TDS spectra consist of two overlapping peaks with the second peak attributed to H release from vacancies (e.g., Eleveld and Van Veen (1992), Alimov and Scherzer (1996)). Although, under these conditions H release mainly from single vacancies created during irradiation is expected. In order to resolve the peak corresponding to H release from vacancies in W, several approaches have been used. Eleveld and Van Veen (1992) used covering of the surface by oxygen impurities prior to irradiation in order to avoid H trapping by adsorption sites. In the experiments of Varava (1993), irradiation was performed at elevated temperatures. Both approaches introduce additional uncertainties and make the interpretation of the results more complicated.

In the present work another approach to resolve a peak corresponding to H release from vacancies in W will be presented.

2. Experimental details

All experiments have been performed in the MEDION ion-beam facility. A detailed description of the setup can was given by Efimov et al. (2013). The duoplasmatron-type ion source is used for generation of deuterium ions with energies in the range of 0.5-10 keV/D. After extraction from the source, the ion beam goes through the lens system, a separating magnet, and then directed onto the sample through a 3 mm diaphragm in order to reduce the spatial beam flux variations. The target chamber is separated from the ion source by a differential pumping stage and has a base pressure below 5×10^{-7} Pa which increases to about 10^{-6} Pa during ion implantation.

A polycrystalline W foil with a thickness of 25 μm was used as a sample. One sample was used for the whole series of experiments. It was annealed at the temperature of 1800 K for 30 minutes before each experiment in order to minimize the amount of natural defects before the experiments and to remove radiation defects from previous irradiation between experiments. There were no any memory effects from the previous irradiations.

The sample (10×50 mm^2) was mounted on two water cooled current feedthroughs. It was linearly heated up to 1800 K during TDS measurements with the heating ramp of 2 K/s. The temperature was measured by a W-Re thermocouple spot-welded to the sample close to the beam strike area. Release of gases from the sample was monitored by a quadrupole mass-spectrometer (QMS).

All irradiations in this series of experiments were done at the room temperature. The lag time between irradiation and TDS was always 3 hours, when the signals of all monitored masses returned to their initial values before the implantation.

3. Results and discussion

Fig. 1 shows TDS spectra from recrystallized W irradiated by 10 keV/D ions to various fluences. At low fluences ($\leq 4\times10^{19}$ D/m^2) the spectra consist of a broad peak near 430 K, a shoulder near 610 K, and a faint shoulder near 720 K. With increasing fluence the amplitudes both of the 430 K peak and of the shoulder near 600 K increase, and also the shoulder near 720 K becomes more pronounced. As was shown by Efimov et al. (2013) the wide peak near 430 K consists of several more narrow peaks. These peaks at temperatures below 550 K are usually ascribed to D release from natural defects (grain boundaries, dislocations) [Ogorodnikova et al. (2008), Rusinov et al. (2011)] and from adsorption sites on the surface [Eleveld and Van Veen (1992), Tamm and Schmidt (1971)]. Rise of the shoulder near 720 K with increasing fluence can be associated with growth of vacancy clusters due to increase in concentration of vacancies in the damaged zone with increasing fluence. Similar tendencies were also observed by Pisarev et al. (1995) and Rusinov et al. (2011).

At all fluences used the peak near 610 K, which is usually ascribed to D release from vacancies in W, is not well resolved (i.e. overlapping with low-temperature peaks). Consequently, it is difficult to use such TDS spectra for determination of parameters of radiation defects using a set of experiments with different heating rates, as a precise knowledge of the peak position is essential for correct determination of the binding energy.

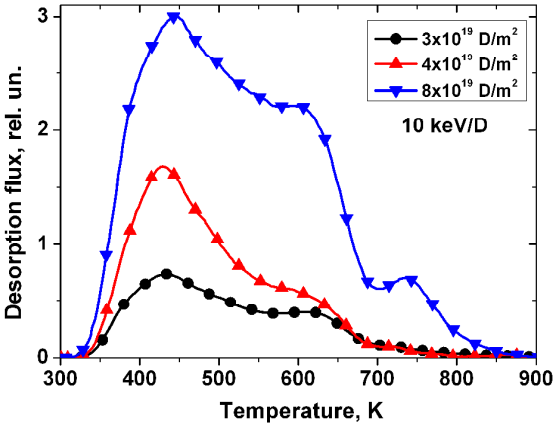

Fig. 1. Thermal desorption spectra of D_2 molecules from recrystallized W irradiated by 10 keV/D ions to different fluencies.

To isolate this peak the following procedure was proposed. Irradiation of a recrystallized W sample by 10 keV/D ions to low fluences (at which the fraction of formed vacancy clusters is quite small) was utilised for creation of single vacancies in the near-surface region of the sample. The fluence of 3×10^{19} D/m^2 was chosen for damaging, as, according to Fig. 1, at this fluence the shoulder at 730 K is still faint, thus, the concentration of vacancy clusters is expected to be small. According to SDTrimSP [Mutzke et al. (2011)] calculations using the threshold displacement energy of 90 eV, under such conditions the damage profile is approximately Gaussian with a maximum located at a distance of 23 nm from the surface and the corresponding maximum damage level is 6.3×10^{-3} dpa. Then the sample was annealed at 550 K for 5 minutes. This temperature is high enough to remove trapped D from vacancies, and, concurrently, the vacancies are still immobile at this temperature, therefore growth of vacancy clusters will not take place [Eleveld and Van Veen (1994), Debelle et al. (2008)]. Afterwards, in order to fill the created vacancies with D without producing additional displacement damage, the sample was implanted with 0.67 keV/D ions (2 keV D_3^+). This energy is below the threshold for Frenkel pair formation. As one can see from Fig. 2, the peak near 610 K is well resolved after this procedure.

Concurrently, implantation of an undamaged sample with 0.67 keV/D ions to the same fluence results in appearance of only a low-temperature peak in the TDS spectrum, which indicates that no displacement damage is produced during this implantation and confirms the fact that the peak near 610 K corresponds to D release from radiation defects (presumably vacancies).

After annealing of the damaged sample at 550 K, only a very small amount of D remains in the sample (<10 % of the total D retained in a recrystallized W after irradiation by 10 keV/D ions to the fluence of 3×10^{19} D/m^2). A subsequent TDS revealed that D release takes place at temperatures above 550 K, which indicates that most of the D trapped in vacancies was removed during this annealing, and that the fraction of D retained in traps with higher binding energies (e.g. vacancy clusters) is relatively small. In addition, the amplitude of the shoulder near 730 K in the spectrum of the damaged sample annealed at 550 K and subsequently implanted with 0.67 keV/D ions is the same as that on the spectrum from the sample only irradiated by 10 keV/D ions to the fluence of 3×10^{19} D/m^2 (Fig. 2). This demonstrates that no significant clustering of vacancies in W took place during annealing at 550 K. The situation will be changed drastically, if one will increase the temperature. In this case, cluster formation will be significant.

It is also remarkable that the low-temperature peak in the spectrum obtained by using the described procedure was narrower compared to that obtained solely after irradiation with 10 keV/D ions to the fluence of 3×10^{19} D/m^2.

This may be connected with decrease in the density of dislocation loops (which may act as trapping sites for D) in the damaged region after annealing at 550 K, as was shown by Sakamoto et al. (1995).

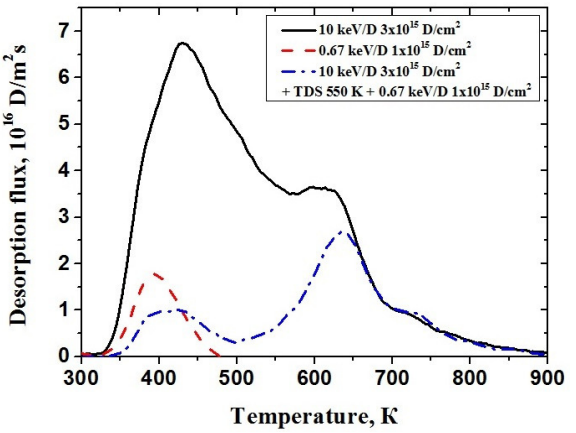

Fig. 2. A comparison of thermal desorption spectra of D_2 molecules from recrystallized W irradiated by 10 keV/D ions to the fluence of 3×10^{19} D/m^2; recrystallized W implanted with 0.67 keV/D ions to the fluence of 1×10^{19} D/m^2; recrystallized W irradiated by 10 keV/D ions to the fluence of 3×10^{19} D/m^2, subsequently annealed at 550 K for 5 min, and then implanted with 0.67 keV/D ions to the fluence of 1×10^{19} D/m^2.

4. Summary

Interaction of deuterium with radiation defects in polycrystalline tungsten has been studied by means of thermal desorption spectroscopy. The damage has been produced by 10 keV D^+ ions with the fluence of 10^{19}-10^{20} D/m^2 at room temperature. TDS spectra have three wide peaks overlapping each other at these conditions. For easier interpretation it is necessary to isolate the peaks in the spectra. It was shown that heating up to 550 K and subsequent irradiation by 0.67 keV/D ions to the fluence of 1×10^{19} D/m^2 allows to isolate the second peak corresponding to single vacancies.

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References

Alimov V.Kh., Scherzer B.M.U., 1996. Deuterium retention and re-emission from tungsten materials. Journal of Nuclear Materials, 240, 75-80.

Debelle, A., Barthe, M.F., Sauvage, T., 2008. First temperature stage evolution of irradiation-induced defects in tungsten studied by positron annihilation spectroscopy. Journal of Nuclear Materials 376, 216-221.

Efimov, V.S., Gasparyan, Y.M., Pisarev, A.A., 2013. Investigation of a fine structure of deuterium thermal desorption spectra from tungsten. Journal of Surface Investigation. X-ray, Synchrotron and Neutron Techniques 7, 472-478.

Eleveld, H., van Veen, A., 1992. Deuterium interaction with impurities in tungsten studied with TDS. Journal of Nuclear Materials 191–194(Part A), 433-438.

Eleveld, H., van Veen, A., 1994. Void growth and thermal desorption of deuterium from voids in tungsten. Journal of Nuclear Materials 212–215(Part B), 1421-1425.

Mutzke, A., Schneider, R., Eckstein, W., Dohmen, R., 2011. SDTrimSP Version 5.00, Report IPP 12/8, Max-Planck-Institut für Plasmaphysik, Garching, Germany.

Ogorodnikova, O.V., Roth, J., Mayer, M., 2008. Ion-driven deuterium retention in tungsten. Journal of Applied Physics 103, 034902.

Pisarev A.A., Varava A.V., Zhdanov S.K., 1995. Ion implantation of deuterium in tungsten. Journal of Nuclear Materials, 220–222, 926-929.

Pisarev, A.A., Voskresensky, I.D., Porfirev, S.I., 2003. Computer modeling of ion implanted deuterium release from tungsten. Journal of Nuclear Materials 313–316, 604-608.

Roth, J., Schmid, K., 2011. Hydrogen in tungsten as plasma-facing material. Physica Scripta T145, 014031.

Rusinov, A., Gasparyan, Y., Trifonov, N., Pisarev, A., Lindig, S., Sakamoto, M., 2011. Investigation of hydrogen-defect interaction in tungsten by the probe fluence method. Journal of Nuclear Materials 415, S645-S648.

Sakamoto, R., Muroga, T., Yoshida, N., 1995. Microstructural evolution induced by low energy hydrogen ion irradiation in tungsten. Journal of Nuclear Materials 220–222, 819-822.

Tanabe, T., 2014. Review of hydrogen retention in tungsten. Physica Scripta T159, 014044.

Tamm, P.W., Schmidt, L.D., 1971. Binding states of hydrogen on tungsten. Journal of Chemical Physics 54, 4775-4787.

Zibrov, M.S., Shubina, A.S., Gasparyan, Yu.M., Pisarev, A.A., 2015. On the possibility of determination of the hydrogen binding energies with defects from thermal desorption measurements with different heating rates. Problems of atomic science and technology: Thermonuclear fusion 38(1), 32-41.