



Surface modification and deuterium retention in reduced-activation steels exposed to low-energy, high-flux pure and helium-seeded deuterium plasmas

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ABSTRACT

Surface topography of and deuterium (D) retention in reduced activation ferritic-martensitic Eurofer'97 and ferritic oxide dispersion strengthening ODS-16Cr steels have been studied after exposure at 600 K to low-energy (70 and 200 eV), high-flux ($\sim 10^{22}$ D/m²s) pure D and D-10%He plasmas with D fluence of 2×10^{25} D/m². The methods used were scanning electron microscopy, energy-scanning D(³He,p)⁴He nuclear reaction, and thermal desorption spectroscopy. As a result of the plasma exposures, nano-sized structures are formed on the steel surfaces. After exposure to pure D plasmas, a significant fraction of D is accumulated in the bulk, at depths larger than 8 μ m. After exposures to D-He plasmas, D is retained mainly in the near-surface layers. In spite of the fact that the He fluence was lower than the D fluence, the He retention in the steels is one order of magnitude higher than the D retention.

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1. Introduction

Reduced Activation Ferritic-Martensitic (RAFM) steels including Oxide Dispersion Strengthened Reduced Activation Ferritic/Martensitic and Ferritic steels (RAFM/RAF-ODS) are considered to be promising candidates for advanced fission and fusion structural materials [1–6]. The conventional RAFM and RAFM/RAF-ODS steels were developed to reduce the activation of structural materials and simplify special waste storage of fusion reactor components after service. With this objective, some alloying elements such as Mo, Nb and Ni present in the commercial martensitic steels have been replaced by other elements which exhibit faster decay of induced radioactivity such as Ta, W, and V [7].

RAFM/RAF-ODS steels belong to the special group of steels with very good creep properties and oxidation resistance. This is due to

the addition of Y₂O₃ nanoparticles, which stabilize the grain size and dislocation motion at elevated temperature [8]. These ODS steels are also known as YWT alloys, since they contain Y, W and Ti particles. The W causes solid solution hardening, whereas the Y and Ti form complex mixed oxides and cause material strengthening [6].

In early papers [2,9,10], RAFM steels were considered for use as the first wall and structural material for the DEMO reactor. At present, the most promising plasma-facing material for the main chamber wall of a fusion reactor is tungsten while RAFM and RAFM/RAF-ODS steels are considered as reference structural materials. Nevertheless, it was suggested to consider RAFM and RAFM/RAF-ODS steels as first wall materials in certain areas of the main chamber wall in DEMO where the particle and power flux are not prohibitive [11]. These certain areas will be mainly subjected to fluxes of energetic deuterium (D) and tritium (T) as well as helium (He) particles at energies in the range from a few eV to several keV. Additionally, both plasma-facing and structural materials will be

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bombarded with 14 MeV neutrons from the D–T fusion reaction.

Therefore, the evaluation of hydrogen isotope retention in undamaged and damaged RAFM and RAFM/RAF-ODS steels (i.e., in steels as-received and with ion-induced displacement damage simulating neutron-induced defects) is an important issue for safety assessment of fusion reactors.

In the last few years, experiments have provided a considerable database on the D retention in undamaged and damaged RAFM and RAFM/RAF-ODS steels irradiated with D ions and exposed to D plasmas [12–27]. In Refs. [12–19,25,26], the D retention was studied with the help of thermal desorption spectrometry (TDS). However, the TDS technique provides only information about the total retention of hydrogen isotopes. In order to reveal what fractions of hydrogen isotopes are accumulated in the near surface layers and in the bulk of materials, measurements of hydrogen depth profiles are required. In Refs. [20–27], the depth distributions of D concentration in undamaged and damaged steels were determined by nuclear reaction analysis (NRA) at depths of up to several micrometers.

It has been reported recently [22,24,25,28–30] that exposure of multicomponent RAFM and RAFM/RAF-ODS steels to low-energy (40–200 eV) deuterium plasmas with fluxes $>10^{19}$ ions/m²s leads to the formation of nano-structured near-surface layers enriched with W. Along with the base medium-Z (or mid-Z) iron (Fe), RAFM and RAFM/RAF-ODS steels contain typical mid-Z steel-composing elements: Cr, V, Mn (conventional RAFM); Ti (RAFM/RAF-ODS) as well as 1–2 wt percents of high-Z W (conventional RAFM and RAFM/RAF-ODS) and smaller amounts of high-Z Ta (conventional RAFM) and Y (RAFM/RAF-ODS). At low particle bombarding energy, the sputtering yield of the mid-Z elements including Fe is significantly higher than that of high-Z elements. As a result, the surface composition is changed due to preferential sputtering of mid-Z elements, resulting in an enrichment of high-Z elements at the surface. For example, a high concentration of tungsten is observed on tips of nano-sized fibers forming the nano-structured layer on the surface of the RAFM F82H steel [28]. As reported in Ref. [25], the exposure of EUROFER'97 and ODS steels to D plasma with D ion energy of 200 eV (i.e., at D ion energy exceeding the sputtering threshold) results in a decrease in the D retention.

It should be noted that seeding of He ions into the D plasma reduces the D retention in RAFM steels F82H and Eurofer'97 as compared with that after exposure to a pure D plasma [19,27].

In spite of intensive studies of D and He interaction with RAFM steels, the data are still scarce and there are uncertainties in the interpretation of the experimental data. Moreover, there is no direct comparison of data obtained under similar experimental conditions, and there is still a lack of understanding of the mechanisms of deuterium trapping in steels with different surface modifications. The objective of this work is to study the surface modification of and deuterium retention in conventional RAFM steel EUROFER'97 and RAF steel ODS-16Cr under exposure to low-energy, high-flux D and He-seeded D plasmas at the sample temperature of 600 K. This temperature was chosen because the minimum temperatures of about 600 K and about 773 K are expected at the plasma-facing side of the RAFM steel used as first wall for Water Cooled Lithium Lead (WCLL) and Helium Cooled Pebble Bed (HCPB) blanket concepts of DEMO power reactor, respectively [2–4,9,10]. Therefore, the minimum sample temperature of 600 K was chosen to obtain data for assessment of expected maximum tritium retention in RAFM/RAF-ODS steels.

2. Experimental

Two types of reduced activation steels were used in this work: conventional RAFM steel EUROFER'97 (hereinafter EU'97)

manufactured in the EU [3] and RAF-ODS steel ODS-16Cr manufactured in China [31]. The compositions of the EU'97 steel, in weight percent, is 0.09–0.12 C, 8.5–9.5 Cr, 1.0–1.2 W, 0.2–0.6 Mn, 0.15–0.25 V, 0.10–0.14 Ta, 0.015–0.045 N₂, balance Fe [32]. The composition of the ODS-16Cr steel, in weight percent, is 0.008 C, 15.5 Cr, 1.67 W, 0.19 Si, 0.49 Ti, 0.18 Y, 0.13 O, 0.47 N, balance Fe [33].

Rectangular-shaped EU'97 and ODS-16Cr samples, 10 × 10 mm² in size, were cut from slabs of each material followed by mechanical polishing to a mirror-like finish and cleaning in an ultrasonic bath. The EU'97 samples were 1 mm thick, whereas a thickness of the ODS-16Cr samples was 0.5 mm.

The steel samples (targets) were exposed to low-energy, high-flux pure and helium-seeded deuterium plasmas at a sample temperature of 600 K. The linear plasma generator (LPG) used for producing plasma beams is described in Ref. [34]. Each of the steel samples was fixed on the LPG holder with the help of rings manufactured from 304 type stainless steel consists of (in weight percents) 18–20 Cr, 8–12 Ni, <2 Mn, balance Fe.

To generate a pure D plasma, the D₂ working pressure was kept at about 1 Pa. As a result, a plasma beam with species of D₂⁺ (about 70% of the ion flux) and D⁺ (about 30%) was obtained. Bias voltages of –74 and –204 V were applied to the samples, resulting in incident energies of 70 and 200 eV for mixture of D₂⁺ and D⁺, respectively, taking into account the plasma potential of about –4 V as measured by a Langmuir probe.

The incident D particle flux was fixed in the range from 0.8×10^{22} to 1.2×10^{22} D/m²s, whereas the D fluence was fixed at 2×10^{25} D/m². It should be noted that the D particle flux was calculated as the monitored D ion flux multiplied by 1.7 (i.e., $0.3 + 2 \times 0.7$). The D fluence was determined by integrating the D particle flux over the exposure time.

The required exposure temperature was set by the thermal contact between the sample and the water-cooled holder and by slight variation of the incident D ion flux. The temperature was monitored using a type K thermocouple tightly pressed to the rear of the sample. It should be noted that after plasma exposure termination, the sample temperature cooled down to 373 K in 5–7 min.

To generate a helium-seeded D plasma (D-He plasma), ⁴He gas was injected into the plasma source region and the He partial pressure was kept at 10^{–1} Pa. The concentration of He ions in the D-He plasma was about 10%, as determined in separate experiments by measuring the sputtering yields of a W sample exposed to D-He plasmas with various He percentages [35]. Thus, the ion composition of the D-He plasma was determined to be D₂⁺ (about 63%), D⁺ (about 27%), and He⁺ (about 10%). The samples were exposed to the D-He plasma to a D fluence of 2×10^{25} D/m². In these conditions the He fluence was estimated to be 1.3×10^{24} He/m².

The surface morphology of the steel samples exposed to the D and He-D plasmas was examined with a field-emission scanning electron microscope (SEM) JEOL JSM-6710F.

The deuterium depth profiles in the plasma-exposed samples were determined by nuclear reaction analysis (NRA) at IPP Garching. The D(³He,p)⁴He reaction was utilized, and both the α particles and protons were analyzed at reaction angles of 102° and 135°, respectively. To determine the D concentration at larger depths, an analyzing beam of ³He ions with energies varied from 0.69 to 4.0 MeV was used. This energy-scanning NRA technique allowed measurements of the D depth profiles to depths of 8 μm [36]. The beam spot area was ~1 × 1 mm² at normal incidence. For the deconvolution of the proton yields measured at different ³He ion energies, the program SIMNRA [37] was used. A deuterium depth distribution was assumed taking into account the near-surface depth profile obtained from the α particle energetic spectrum, and the proton yield was calculated as a function of incident ³He

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