TEMPERATURE DEPENDENCE OF SURFACE TOPOGRAPHY AND DEUTERIUM INTERACTION WITH A PURE α-Fe EXPOSED TO LOW-ENERGY HIGH-FLUX D PLASMA

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Surface topography and deuterium interaction with α-Fe under glow discharge hydrogen (deuterium) ions bombardment with energy of ~1 keV at ion fluencies of $(0.02…1)\cdot10^{24}$ D/m^2 and various temperatures have been examined. The methods used were scanning electron microscopy, thermal desorption spectroscopy and the D(^{3}He,p)^{4}He nuclear reaction. Formation of blisters was observed in the temperature range 230…340 K. Round-shaped cavities contained small nonmetallic particles Fe_xO_y (x=1…2, y=1…4) were founded on the irradiated surface of α-Fe. Temperature dependence of average blister diameter, the deuterium depth profile and temperature of deuterium retention were studied.

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INTRODUCTION

Sputtering-induced erosion of plasma-facing materials (PFMs) is a serious concern for future commercial reactors, not only for low Z materials, but also for structural materials (ferritic steels, vanadium alloys, etc.) [1].

Ferritic steels are currently considered as promising materials for structural elements of both fission and fusion reactors due to their higher resistance to void swelling and irradiation creep as compared with austenitic steels.

However, ferritic steels are known to be particularly susceptible to nucleation of both blisters and associated subsurface cracks arising during exposure to glow discharge hydrogen plasma with ion energies of ~ 1 keV [2]. Such low ion energies are known to be characteristic of near-wall plasma fluxes in fusion reactors [3].

Blisters formed on the surface of the ferritic steel look similar to blisters seen on the surface of metals such as Nb, Cu, Ni, and stainless steel caused by light ion bombardment (H, D, He) [4]. But the dimensions of plasma-induced blisters are two orders of magnitude higher than the average dimensions of blisters produced at ion beam energy of ~1 keV. The majority of blisters in the current study have a dome-like shape, with the ratio of height to diameter around 0.02…0.05.

In glow discharge experiments it was shown that the critical fluence of first blister formation strongly depends on the target temperature and the deformation level of the alloy [1]. There is insufficient understanding concerning the influence of hydrogen plasma on the concurrent formation of cracks and blisters in ferritic-martensitic steels and requires further investigation.

It is anticipated that the surface and near-surface structural-phase microstructure of a particular alloy may affect the various erosion processes. Accordingly it is desirable to investigate a material with approximately uniform structure and monatomic composition, e.g. α-Fe.

The goal of this paper is to investigate the evolution of surface topography and the features of deuterium interaction with α-Fe under glow discharge deuterium ions bombardment with energy of ~ 1 keV at ion fluencies of $(0.02…1)\cdot10^{24}$ D/m^2 and various temperatures.

1. MATERIAL AND METHODS

The samples of α-Fe (bcc) with a purity of 99.9 wt% were recrystallized at 1600 K after rolling and cutting. It contains impurities of more than a dozen elements. The carbon and copper concentration are of about 0.02% and 0.1 %, respectively. The remaining elements are in thousandths of a percent.

The specimens with dimensions of 10 × 7 mm were cut from a sheet with thickness of 1 mm. The surface of each sample was polished mechanically and then electropolished in a standard electrolyte to remove any mechanically damaged near-surface layer.

The specimens have been irradiated at various temperatures with deuterium ions using glow gas-discharge plasma electrodes at 1000 V, producing an ion flux of 10^{19} D/(m$^2\cdot$s). In this study we chose D in order to easily measure the depth dependence of the implanted and diffused hydrogen. The maximum irradiation fluence was $1\cdot10^{24}$ D/m^2.

The main parameter changed in the experimental series was the temperature during plasma exposure, which was varied between 240 and 300 K. The specimen was placed in a resistively-heated holder. The specimen temperature was continuously monitored using a thermocouple in the base of the specimen holder and was attached to the lower surface of specimen. Temperature maintenance on the steel samples in this device was achieved either by resistive heating or liquid nitrogen cooling. The temperature was maintained to within ±2.5 K. A detailed schematic diagram of the experimental setup is presented in Ref. [1].

The D concentration in the plasma-exposed Fe samples was measured by means of the D(^{3}He,α)H reaction, where protons were analyzed. To determine the D concentration at larger depths, an analyzing beam of ^{3}He ions with energies varied from 0.3 to 1.4 MeV was used. The proton yields measured at different ^{3}He ion energies allow measuring the D depth profile at depths of up to 2 μm.

Total deuterium retention in the Fe samples was monitored ex-situ using thermal desorption spectrometry (TDS). A resistive heater was used to heat the samples at a ramp rate of 6 K/s and the sample temperature was raised to 1300 K. D_2 molecules released during TDS run were monitored by monopole mass spectrometer.

A JEOL JSM-7001F 00 scanning electron microscope was used to study the surface morphology. Investigations of surface microstructure were performed using a MMO-1600-AT metallographic microscope.

2. RESULTS AND DISCUSSION

Fig. 1 shows SEM images of surface morphology of initial α-Fe after etching in 5 % nitric acid with water. Etching pits at the exit of dislocations on the surface, and pits with inclusions were observed.

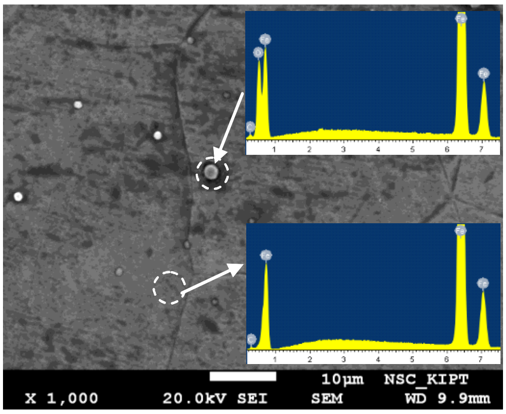

Fig. 1. SEM image of α-Fe initial surface after etching and EDS X-ray spectrums of different part of surface

The inclusions can be introduced during the preparation of samples for the experiment (cutting, rolling, annealing and the mechanical and electrolytic polishing) or the accumulation of impurities that have "slided" to the dislocation core. EDS analyses revealed that these particles are Fe_xO_y, where x=1…2, y=1…4.

The SEM images of the α-Fe sample exposed to the fluence of $1{\cdot}10^{24}$ D/m^2 at temperatures in the range 240…300 K are shown in Fig. 2. The surface is covered by a large number of blisters having an irregular shape.

Video registration shows that small blisters with diameters of 2 microns grow in the initial stage and then later blisters having diameters on the order of 60 microns began to develop. All blisters are limited by the grain boundaries.

Fig. 3 shows the temperature dependence of average diameters and density of blisters formed under deuterium plasma. A minimum of two samples were used at each temperature. The average diameter and density of blisters increases monotonically (density of blisters increases weakly) with increasing temperature of irradiation up to 250…260 K and then decrease.

The large blisters showed *a* multi-layered structure like steps (Fig. 4). This effect is most pronounced at the irradiation temperature of 270 K.

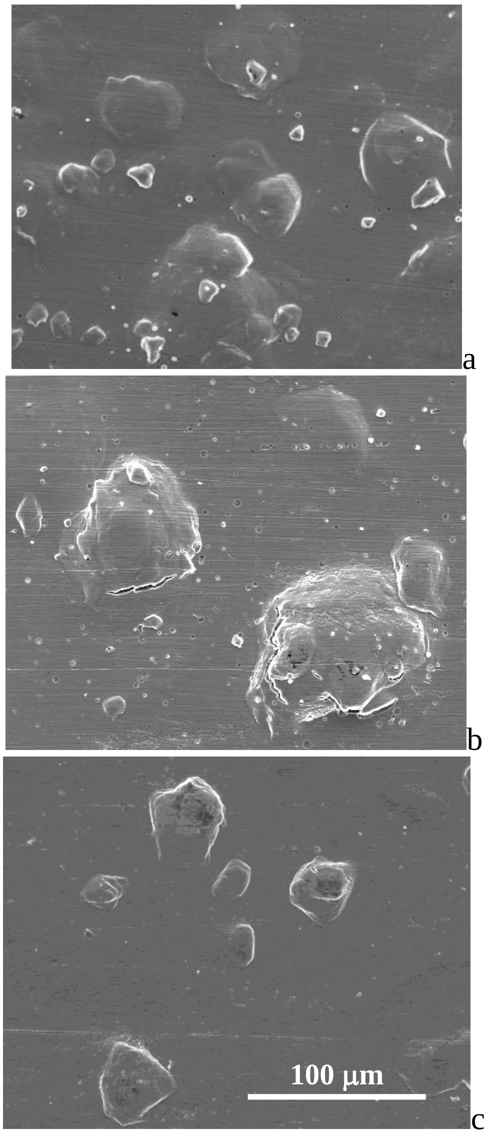

Fig. 2. SEM images of blisters on the surface of α-Fe after irradiation with deuterium plasma to $1{\cdot}10^{24}$ D/m^2 at 240 (a), 270 (b) and 285 (c). The scale is the same for all micrographs (a – c)

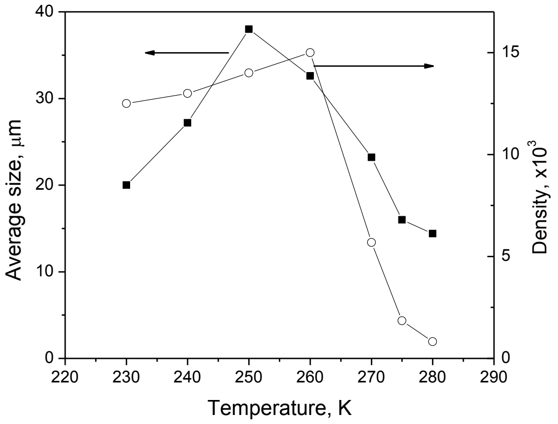

Fig. 3. Temperature dependence of average diameter and density of blisters formed under deuterium plasma irradiation to $1{\cdot}10^{24}$ D/m^2 for α-Fe

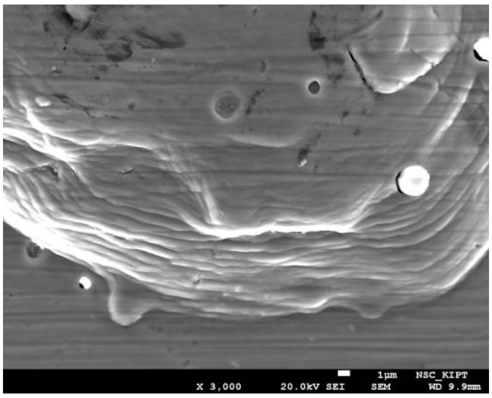

Fig. 4. SEM images of a multi-layered structure like steps (45° tilt) appearing on α-Fe exposed to the fluence of $1\cdot10^{24}$ D/m^2 at 270 K

It has been considered that the high-dome blisters are formed by deuterium-promoted local superplasticity [5]. First, deuterium-induced vacancies are generated due to the lowering of vacancy formation energy caused by trapping of deuterium [6]. Subsequently deuterium-vacancy clusters are formed and diffuse deeply into the bulk such as somewhere near the surface in the grains and grain boundaries and agglomerate, resulting in blisters. In addition, every agglomeration of the deuterium-vacancy clusters results in a step print on the blister [7].

The penetration of deuterium on the depth orders of magnitude greater than the calculated ion range confirms the data obtained by the NRA. Fig. 5 presents the depth distribution profiles of deuterium in α-Fe exposed to 1 keV deuterium plasma.

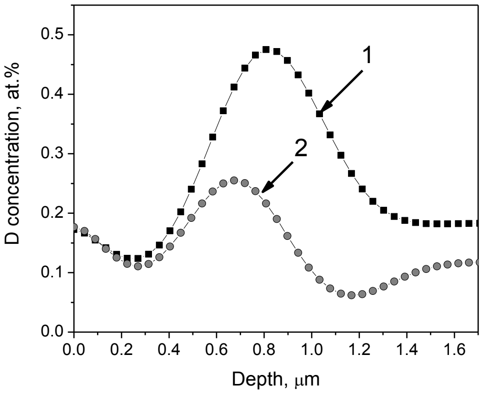

Fig. 5. Profiles of deuterium in α-Fe exposed to deuterium plasma at 300 K to a doses of $5\cdot10^{23}$(1) and $4\cdot10^{22}$ (2) D/m^2

The calculated normal-incident range of 0.5 keV D^+ in iron is about 7 nm. Detection of deuterium at a depth of 1.7 μm confirms that the implanted D migrates into the bulk far beyond the ion range and thereby promotes nucleation of blister gaps at this depth. At fluence $\geq 5\cdot10^{23}$ D_2^+/m^2 the deuterium concentration in sub-surface layer reaches 0.5 at.% and becomes enough for blister formation.

Glow gas-discharge D plasma irradiation simultaneously with blisters causes the formation of large round-shaped cavities on the surface of Fe. Fig. 6 shows that almost all of large cavities contained small nonmetallic particles. EDS analyses revealed that these particles are Fe_xO_y, where x=1…2, y=1…4. The diameter of the cavities (~ 1 micron) correlates with the size of the inclusions. The discarded material – "plug" consists of pure Fe.

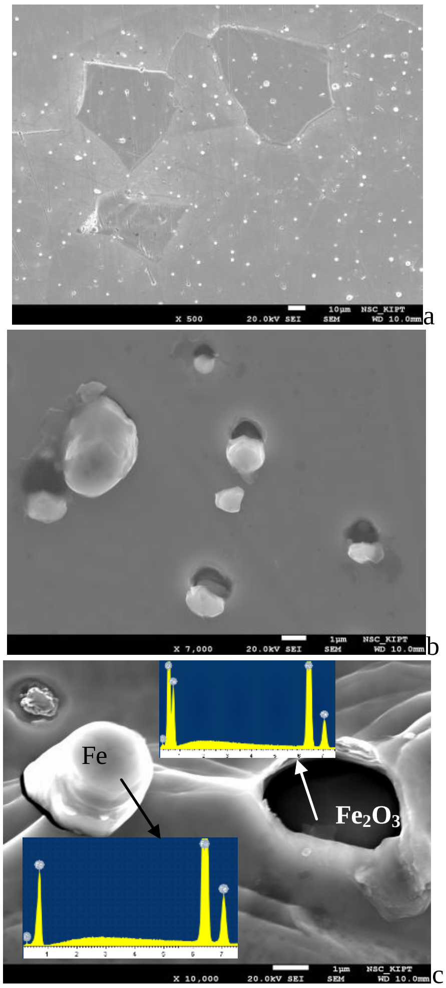

Fig. 6. SEM images of round-shaped cavities on the surface of α-Fe after irradiation with deuterium plasma to $1\cdot10^{24}$ D/m^2 at 270 K with different scale for micrographs a – c. EDS X-ray spectrums of inclusion and extruded material are shown in the insert of Fig. 6,c

It can be assumed that the deuterium trapping by these inclusions promotes gas atoms combination to form hydrogen molecules that will exert a pressure on the surrounding α-Fe, resulting in extrusion of material and the formation of round-shaped cavities.

The strong trapping of deuterium on the inclusions may indicate the data obtained by TDS. Fig. 7 shows desorption of deuterium from α-Fe exposed to deuterium plasma at 300 K to a dose of $1\cdot10^{24}$ D/m^2. The release of deuterium from iron sample starts at ~ 330 K. The maximum of desorption peak is observed at 500 K.

As can be seen from Fig. 2, blisters formed at different temperatures of irradiation have bursting covers whereby the deuterium should release from fissures. According to [8] deuterium is not retained in solution or nor radiation-induced point defects near room temperature irradiation in α-Fe. Deuterium release at T~500 K may indicate gas de-trapping from detected inclusions. To confirm the observed effect additional studies are needed.

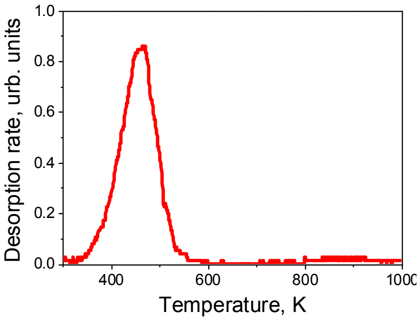

Fig. 7. TDS spectra of D_2 from α-Fe exposed to deuterium plasma to a dose of $1\cdot10^{24}$ D/m^2 at 300 K

CONCLUSIONS

Surface topography and deuterium interaction with α-Fe under glow discharge deuterium ions bombardment and various temperatures have been examined. Real-time monitoring of the target surface was performed with a set of *in-situ* optical surface diagnostics that allows detection of the first appearance of blisters and their subsequent growth. The conclusions to be drawn from this work are as follows.

Exposure of α-Fe to low-energy (~ 1 keV) and high flux (10^{19} D/(m^2·s)) plasma to fluence of ~ $1\cdot10^{24}$ D/m^2 leads to the formation of blisters.

It is suggested that hydrogen atoms diffuse to low free energy locations such as the interface of matrix and inclusions. At these locations, the hydrogen atoms can combine to form hydrogen gas molecules that exert pressure on the surrounding metal, thereby forming a blister.

Round-shaped cavities contained small nonmetallic particles Fe_xO_y (x=1…2, y=1…4) were founded on the irradiated surface of α-Fe. This phenomenon was not observed for EP-450 F/M steel under the same conditions of plasma exposure.

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ТЕМПЕРАТУРНАЯ ЗАВИСИМОСТЬ ПОВЕРХНОСТНОЙ ТОПОГРАФИИ И ВЗАИМОДЕЙСТВИЯ ДЕЙТЕРИЯ С ЧИСТЫМ ЖЕЛЕЗОМ ПРИ ВОЗДЕЙСТВИИ НИЗКОЭНЕРГЕТИЧЕСКОЙ ПЛАЗМЫ ДЕЙТЕРИЯ ВЫСОКОЙ ПЛОТНОСТИ

А.В. Никитин, Г.Д. Толстолуцкая, В.В. Ружицкий, И.Е. Копанец, С.А. Карпов, Р.Л. Василенко, А.Ю. Ростова, Н.Д. Рыбальченко

Изучена топография поверхности и взаимодействие дейтерия с α-Fe под воздействием тлеющего разряда ионов водорода (дейтерия) с энергией ~ 1 кэВ при ионных флюенсах (0,02…1)·10^{24} D/м^2 и различных температурах. Использовались методы сканирующей электронной микроскопии, термодесорбционной спектроскопии и ядерных реакций D(^{3}He,p)^{4}He. В интервале температур 230…340 К наблюдалось образование блистеров. На облучённой поверхности α-Fe были обнаружены полости округлой формы, содержащие небольшие неметаллические частицы Fe_xO_y (x=1…2, y=1…4). Обсуждаются: температурная зависимость среднего диаметра блистеров, распределение дейтерия по глубине образца и особенности удержания дейтерия.

ТЕМПЕРАТУРНА ЗАЛЕЖНІСТЬ ПОВЕРХНЕВОЇ ТОПОГРАФІЇ І ВЗАЄМОДІЇ ДЕЙТЕРІЮ З ЧИСТИМ ЗАЛІЗОМ ПРИ ДІЇ НИЗЬКОЕНЕРГЕТИЧНОЇ ПЛАЗМИ ДЕЙТЕРІЮ ВИСОКОЇ ГУСТИНИ

А.В. Нікітін, Г.Д. Толстолуцька, В.В. Ружицький, І.Є. Копанець, С.О. Карпов, Р.Л. Василенко, Г.Ю. Ростова, Н.Д. Рибальченко

Вивчено топографію поверхні і взаємодію дейтерію з α-Fe під впливом тліючого розряду іонів водню (дейтерію) з енергією ~ 1 кеВ при іонних флюенсах (0,02…1)·10^{24} D/м^2 і різних температурах. Використовувалися методи скануючої електронної мікроскопії, термодесорбційної спектроскопії і ядерних реакцій D(^{3}He,p)^{4}He. В інтервалі температур 230…340 К спостерігалось утворення блістерів. На опроміненій поверхні α-Fe були виявлені порожнини округлої форми, що містять невеликі неметалічні частки Fe_xO_y (x=1…2, y=1…4). Обговорюються: температурна залежність середнього діаметра блістерів, розподіл дейтерію по глибині зразка і особливості утримання дейтерію.