KINETICS OF BLISTERS GROWTH IN A DEFORMED α-Fe AT LOW-ENERGY DEUTERIUM PLASMA EXPOSURE

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The results of the study of surface topography evolution and the features of deuterium interaction with α-Fe under glow discharge 1 keV deuterium ions bombardment at T_{room} up to ion fluence of $3.4 \cdot 10^{24}$ D/m^2 are presented. At a threshold irradiation dose the formation of blisters with dimensions ranging from 0.01 to 1 mm is observed. The extent of blistering development is determined by ability of hydrogen to penetrate to the depths exceeding the ions stopping range of 1 keV hydrogen by orders of magnitude and by the density, location depth and mutual arrangement of fissures in the preliminarily deformed material.

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INTRODUCTION

The problem of materials selection for the vacuum chamber and protection of the chamber from the fusion plasma exposure is one of the most important in the creation and design of fusion devices [1, 2]. One of the most promising structural materials for nuclear fusion reactors is ferritic-martensitic steels with a low activation in a fusion neutron spectrum [3]. 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 [1].

Blisters formed on the surface of the ferritic-martensitic steels and α-Fe (bcc) 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 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 [2]. It is established that the surface and near-surface structural-phase state of a particular alloy may affect the various erosion processes. Consequently, 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 up to ion fluencies of $3.4 \cdot 10^{24}$ D/m^2.

1. MATERIAL AND METHODS

The plate of α-Fe with a purity of 99.8 wt % was recrystallized at 1600 K after rolling of bar 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 plate was then deformed by 70 %. Deformation was carried out by cold working via rolling at room temperature.

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

The specimens have been irradiated at room temperature with deuterium ions using glow gas-discharge plasma electrodes at 1000 V, producing an ion flux of $3 \cdot 10^{20}$ D/(m^2·s). The maximum irradiation fluence was $3.4 \cdot 10^{24}$ D/m^2.

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 attached to the lower surface of specimen. Fig. 1 presents a schematic diagram of the experiment.

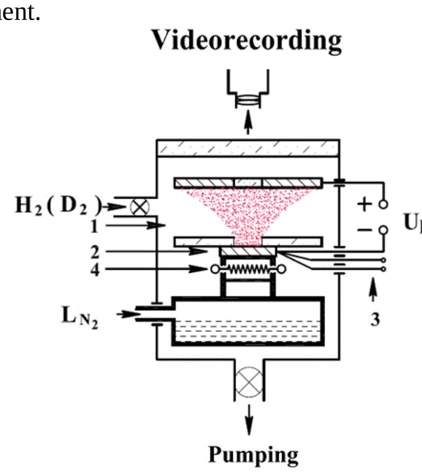

Fig. 1. Diagram of experiment: 1 – glow gas-discharge plasma; 2 – target; 3 – thermocouple; 4 – resistive heater

The central portion of the specimen was irradiated through an aperture, providing an easily-observed boundary between irradiated and unirradiated regions. The kinetics of blister growth was monitored continuously during the irradiation using a video recorder.

2. RESULTS AND DISCUSSION

The images of the α-Fe sample exposed to the fluence of $2.5 \cdot 10^{24}$ D/m^2 at temperatures ~300 K are shown in Fig. 2. The surface is covered by a large number of blisters. Most blisters are not symmetric (spherical) shape, and extend along the rolling direction.

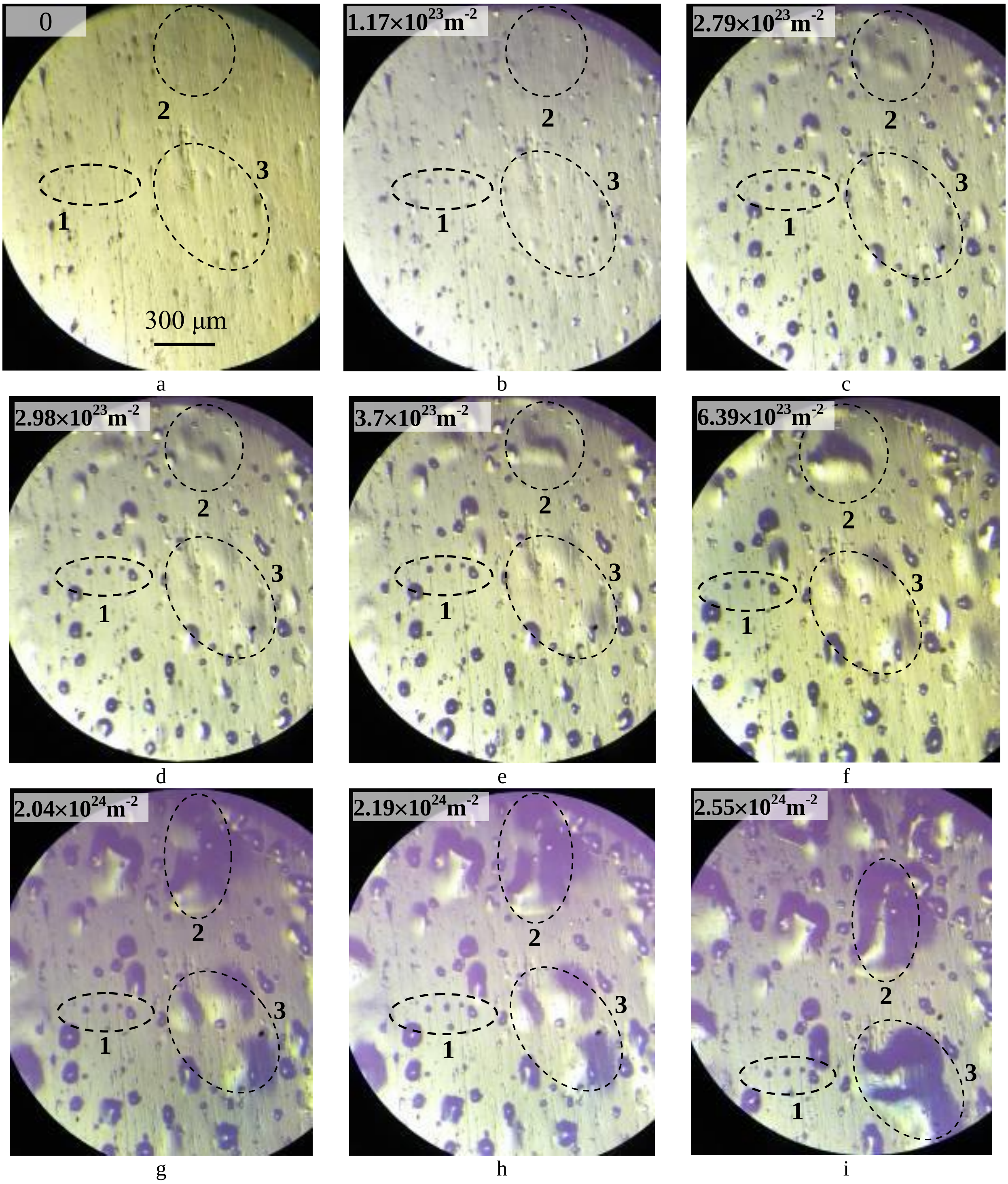

Fig. 2. Optical micrograph demonstrating the kinetics of blister growth in a surface of α-Fe specimen during hydrogen plasma exposure. Selected areas 1-3 in the figures show the characteristic features in the development of blisters. The scale is the same for micrographs a-i

We have previously shown that at the temperatures close to T_{room} blisters appear on the surface after reaching a threshold dose D_n that varied with the initial material microstructure [2]. In the present work video registration shows that small blisters with diameters of ~ 10 microns appear when a dose is reached $1.7 \cdot 10^{23}$ D/m^2 (see Fig. 2,b, area #1). For comparison, in Fig. 2,a the surface of virgin specimen is shown.

The first characteristic feature noted in this study is the presence of blisters, the dimensions of which practically do not change from the moment of their appearance until the irradiation stops (see compare Fig. 2,b and 2i, area #1).

Two blisters in the area #2 appear at the irradiation dose of $2.79 \cdot 10^{23}$ D/m^2 (see Fig. 2,c). They have an irregular shape and practically do not change their size and shape until the dose of $3.2 \cdot 10^{23}$ D/m^2. Upon reaching the dose $3.7 \cdot 10^{23}$ D/m^2 they begin to coalesce (see Fig. 2,e), and then in the dose range $(0.64…2.19) \cdot 10^{24}$ D/m^2 merge with some neighboring blisters (see Fig. 2,f-h), forming an irregular blister with a size of ~0.9 mm along the rolling direction. Further, with an increase in the radiation dose up to $3.4 \cdot 10^{24}$ D/m^2 the parameters of this blister do not change. Many small blisters lying above this blister indicate that the emergence of the gap of an irregular blister occurs at a much greater depth from the surface.

Three blisters in area #3 appear approximately at the same irradiation dose as the blisters in area #2. Analysis of the parameters of blisters in area #3 showed that their average size is 1.5 times larger than blisters in area #2, but the height at the surface is smaller. With increasing irradiation dose from $2.79 \cdot 10^{23}$ to $2.3 \cdot 10^{24}$ D/m^2 the average size of these blisters increases by only a few tens of percent, while their height above the surface is visibly growing. And only at a dose of $2.55 \cdot 10^{24}$ D/m^2 this group of blisters merges, forming a blister of complex shape with an average size of 1 mm (see Figs. 2,i and 3). At the same time, the average blister size over the entire sample surface is about 100 μm.

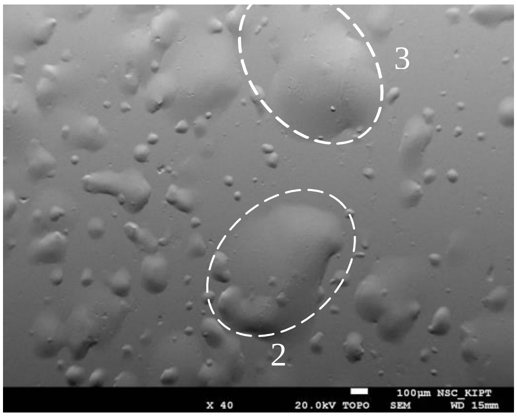

Fig. 3. SEM images of the blister on surface of α-Fe specimen after irradiation to $2.55 \cdot 10^{24}$ D/m^2 (area 2, 3)

In the process of deformation by rolling, especially at temperatures close to room temperature, fissures are formed in the sample (Fig. 4). These fissures can act as embryos of future blisters (Fig. 5). Closely spaced fissures can contribute to the formation of irregularly shaped blisters.

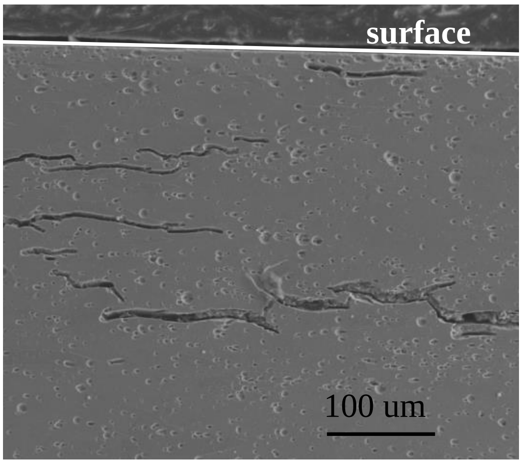

Fig. 4. Morphology of fissures at different depths

Hydrogen activity is one of the key parameters that determine the blister and crack growth rate. The hydrogen penetration depth directly depends on its diffusivity in steel. The assessment of penetration depth can be performed by solving a diffusion equation with a source function:

$$\frac{\partial C(x,t)}{\partial t} = D(T)\frac{\partial^2 C(x,t)}{\partial x^2} + \varphi(x),$$

where C is the concentration of hydrogen; $D(T)=D_0 \cdot \exp(-E_m/k_B T)$ is the hydrogen diffusion coefficient [5].

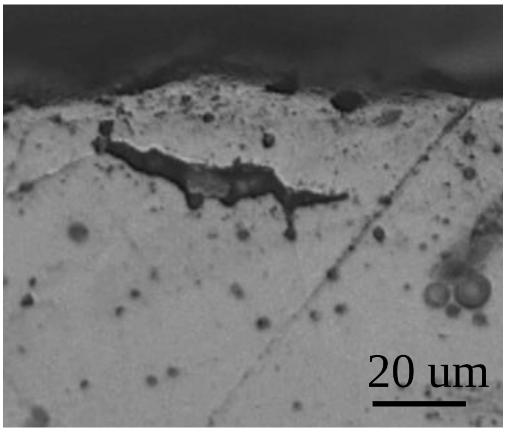

Fig. 5. Cross-section images of typical dome-like blisters

The distribution of hydrogen injection rate over a depth φ(x) simulates the action of a plasma ion source, the intensity of which in the calculations corresponded to the experimental value of $3 \cdot 10^{20}$ m^{-2}s^{-1}. Fig. 6 shows the calculated distributions of hydrogen in α-Fe after irradiation of a glow discharge plasma to doses of $8 \cdot 10^{22}$, $2 \cdot 10^{23}$ and $3 \cdot 10^{23}$ m^{-2}.

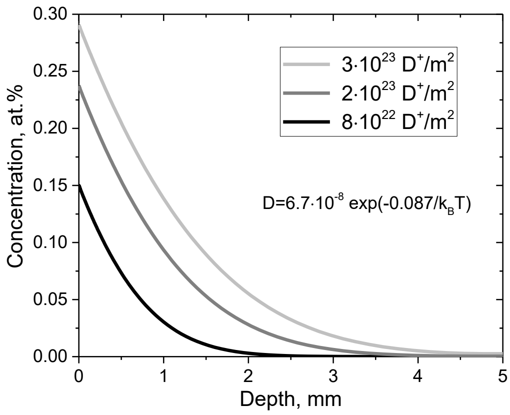

Fig. 6. Calculated distribution profiles of deuterium

As can see the mobility of hydrogen is quite enough to penetrate the distances measured in millimeters. This means that deuterium can reach fissures located fairly deep in the bulk of the material. The second prerequisite for blistering development is the presence of nucleation sites with a certain density, location depth and mutual arrangement in the preliminarily deformed material.

Summarizing the above observations, the following should be noted:

- blisters with irregular shape and a characteristic size of ~1 mm are formed as a result of the merger of a large number of small blisters;
- blisters with sizes of ~10 microns located in the lid of large blisters;
- deformation by rolling, especially at temperatures close to T_{room}, leads to elongation of grain boundaries along the rolling direction. Wherein, the grain boundaries are weakened and on some of them the cracks are formed.

▪ the calculation shows the high mobility of hydrogen and its penetration to depths of order of millimeters. Thus, hydrogen can be easily delivered to the places of its localization in the collectors. Cracks along the grain boundaries can be such collectors.

Based on these results, it can be suggested a simple model picture of the formation of different blisters configurations (Fig. 7).

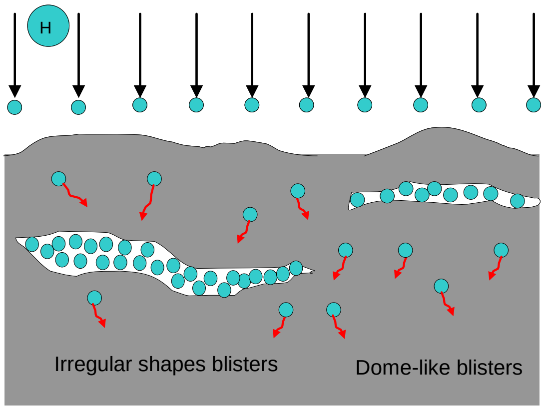

Fig. 7. Schematics of development of high dome or irregular shapes blisters

High H flux loading would increase the H concentration in the saturated layers. Small size cracks near the surface are saturated with H, and blisters with high pressure inside and dome shape are formed.

As the irradiation dose increases, deeper cracks are filled with hydrogen. The hydrogen atoms on the surfaces of the cracks recombine into the molecule resulting in increase of the pressure on the crack walls. When the pressure in the blister nucleus exceeds the iron yield strength, the blister is formed. The dimensions of blister (average diameter and height above the surface) can increase with increasing irradiation dose.

The hydrogen pressure in the blister nuclei can cause not only a lifting of the surface, but also a breaking of barriers between neighboring blisters. Closely located (even in different horizons) cracks merge, thereby forming a blister of irregular shape.

CONCLUSIONS

The kinetic of blisters nucleation and growth during exposure of α-Fe in glow discharge deuterium plasma with ion energies of ~ 1 keV and ion fluencies up to $3.4 \cdot 10^{24}$ D/m^2 have been defined.

The presence of blisters with dimensions that virtually do not change from the moment of their appearance until the irradiation stops is shown.

Some blisters with dimensions an order of magnitude larger than the average size of observed blisters were registered. It is found that large blisters having complex shape and an average size of 1 mm are formed by the merger of several closely spaced groups of blisters.

The development of blistering is determined by ability of hydrogen to penetrate to the depths exceeding the ions stopping range of 1 keV hydrogen by orders of magnitude and by the density, location depth and mutual arrangement of fissures in the preliminarily deformed material.

REFERENCES

1. G. Federici, C.H. Skinner, J.N. Brooks, et al. Plasma-material interactions in current tokamaks and their implications for next step fusion reactors // *Nucl. Fusion.* 2001, v. 41, p. 1967-1981.

2. A.V Nikitin., G.D. Tolstolutskaya, V.V. Ruzhytskyi, et al. Blister formation on 13Cr2MoNbVb ferritic-martensitic steel exposed to hydrogen plasma // *Journal of Nuclear Materials.* 2016, v. 478, p. 26-31.

3. R.L. Klueh, A.T. Nelson. Ferritic/martensitic steels for next-generation reactors // *Journal of Nuclear Materials*. 2007, v. 371, p. 37-52.

4. R. Behrisch. Surface erosion from plasma materials interaction // *Journal of Nucl. Mater*. 1979, v. 85-86, p. 1047-1061.

5. V.I. Bendikov, S.A. Karpov, V.V. Ruzhitsky. *Numerical simulation of thermal desorption spectra of hydrogen implanted into metals*: Preprint KhFTI. Kharkov: NSC KIPT, 2003, p. 16.

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КИНЕТИКА РОСТА БЛИСТЕРОВ В ДЕФОРМИРОВАННОМ α-Fe ПРИ ОБЛУЧЕНИИ НИЗКОЭНЕРГЕТИЧЕСКОЙ ДЕЙТЕРИЕВОЙ ПЛАЗМОЙ

А.В. Никитин, Г.Д. Толстолуцкая, И.Е. Копанец, С.А. Карпов, В.В. Ружицкий

Представлены результаты исследования эволюции топографии поверхности и особенностей взаимодействия дейтерия с α-Fe при бомбардировке ионами тлеющего разряда с энергией 1 кэВ при $T_{комн.}$ до дозы $3{,}4 \cdot 10^{24}$ D/м^2. При пороговой дозе облучения наблюдается образование блистеров с размерами от 0,01 до 1 мм. Степень развития блистеринга определяется способностью водорода проникать на глубины, превышающие на порядки пробег ионов водорода с энергией 1 кэВ, а также плотностью, глубиной и взаимным расположением трещин в предварительно деформированном материале.

КІНЕТИКА РОСТУ БЛІСТЕРІВ У ДЕФОРМОВАНОМУ α-Fe ПРИ ОПРОМІНЕННІ НИЗЬКОЕНЕРГЕТИЧНОЮ ДЕЙТЕРІЄВУЮ ПЛАЗМОЮ

А.В. Нікітін, Г.Д. Толстолуцька, І.Є. Копанець, С.О. Карпов, В.В. Ружицький

Представлено результати дослідження еволюції топографії поверхні та особливостей взаємодії дейтерію з α-Fe при бомбардуванні іонами тліючого розряду з енергією 1 кеВ при $T_{кімн.}$ до дози $3{,}4 \cdot 10^{24}$ D/м^2. При пороговій дозі опромінення спостерігається утворення блістерів з розмірами від 0,01 до 1 мм. Ступінь розвитку блістерінга визначається здатністю водню проникати на глибини, що перевищують на порядки пробіг іонів водню з енергією 1 кеВ, а також щільністю, глибиною і взаємним розташуванням тріщин у попередньо деформованому матеріалі.