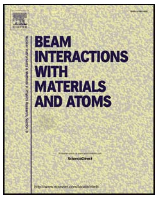
Nuclear Inst. and Methods in Physics Research B 420 (2018) 46–49

Contents lists available at ScienceDirect

Nuclear Inst. and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb



Ion induced millimetre-scale structures growth on metal surfaces

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ARTICLE INFO

Keywords:
Tungsten
Surface morphology
Ion-induced structures
Stresses
Relaxation
Activated surface mobility

ABSTRACT

Polished polycrystalline Plansee tungsten (W) sample with purity 99.99 wt% and 0.75 mm thickness has been exposed to intense argon (Ar) ion beam with average energy of 2 keV and etched through in the centre. As a result, castle-like structures with strong asymmetry and with the height of > 200 µm have been formed. Structures can be observed by naked eyes and with scanning-electron microscopy (SEM). It has been revealed, that the structures have been formed not immediately, but at the later stages of irradiation. Primary factors favouring the formation for the structures are relaxation of the surface stresses and activated surface mobility of atoms.

1. Introduction

Interaction of energetic ions with a surface causes a number of physical phenomena, which is an intensive research area for decades. Among them, there are particle reflection, physical and chemical sputtering, development of surface morphology, etc. A number of reviews were published [1,2], demonstrating deep understanding of these topics. For example, physical sputtering became the subject for numerical simulation and good agreement with experimental data was achieved [1,3], merging the various hypotheses into single widely recognized theory.

In contrast, development of surface morphology is investigated by means of experimental bombardment and scanning electron microscopy. Growing of features and structures on the top of the surface exposed to ion flux is especially intriguing issue. It is generally accepted opinion that development of the structures on metal surfaces is caused by a number of factors like ion sputtering [4], scattering [5] and secondary electron emission [6]. Activated surface mobility of atoms is another mechanism which is especially responsible for the growth of the structures on a surface [7,8]. The superposition of thermally activated surface diffusion and ion bombardment activated surface atom transport is observed in Ref. [8]. Results indicate that it is this superposition, which results in the observed epitaxial microprotrusion growth and its confinement to the axial (1 1 1) W plane. Bulk vacancy diffusion in tungsten becomes significant at 700 K (Q – 1.8 eV). In general, one would expect that the activation energy for surface adatom and/or vacancy diffusion would be less than that for bulk vacancy diffusion. The epitaxial microprotrusion growth at emitter tip temperatures between − 800 and 1300 K is due to the annealing of the surface and microprotrusion which occurs during the growth at these temperatures. This result indicates that bubble formation does not play a role in the microprotrusion growth process. The removal of stresses can be accomplished in two ways: removal of stressed top layer and heating of the sample [9,10]. The propagation of the stress deep under the surface in tungsten and other materials has been observed in a number of investigations [11–13]. The authors of Refs. [14,15] indicate the redeposition of the sputtered particles as one more reason for the structures' growth. In the magnetron sputtering systems, where ion irradiation is combined with negative voltage applied to the surface [16], another important mechanism responsible for the growth of the structures is found. In this case, cones with the height above 10 µm emit electrons, which ionize the sputtered atoms causing their redeposition under the influence of electric field. Other important surface structures, which are intensively investigated in the fusion oriented material research, are blisters and tungsten fuzz. Blister formation occurs typically at relatively low surface temperature exposed to H and/or He ion fluxes below 700 K [17–19]. Fuzz is specific to low-energy He plasma in the 1–100 eV range [20]. The dependence of blistering and deuterium retention upon the exposure temperature was also examined for tungsten exposed at the fluence of 10^{27} D/m^2. [18] In high temperature region (higher than 600 K), the blisters became much sparser with the increasing temperature and disappeared at 1000 K [17]. Stress-induced plastic deformation caused by deuterium supersaturation within the near-surface layer and formation of superabundant deuterium-vacancy clusters are suggested as mechanisms for nucleation and growth of microscopic cavities at depths up to several micrometers. At long-term

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https://doi.org/10.1016/j.nimb.2018.02.002
Received 30 October 2017; Received in revised form 12 January 2018; Accepted 5 February 2018

plasma exposure, the diffusing D atoms recombine on the cavity surfaces, thus increasing the gas pressure inside these cavities, which may support the formation of blister-like structures [19].

Present work is initiated by suddenly discovered structures on metal surface, which size strongly exceed what was reported in literature (1 ÷ 100 μm). Structures are observed on the W surface after exposing to intense Ar ion beam. However, the structures do not start to grow when the sample is half-eroded. The conditions for structures formation and their strongly non-symmetrical shape imply that redeposition processes cannot play major role. Instead, mobility of atoms and relaxation of surface stresses can be the main factors causing the formation and growth of the observed structures.

2. Experimental conditions

Polished polycrystalline Plansee tungsten sample with purity 99.99 wt% was exposed to Ar ion beam generated by FALCON ion source [21,22]. Initial state of the sample was measured with XRD analysis before exposure. Sample size is 15 × 12 × 0.75 mm^3 with the following initial state parameters: grain size 5–20 μm, texture [1 0 0], residual stresses −220 MPa, lattice parameter 3.1645 Å. The sample was exposed to Ar ion beam with current of 30 mA, and ion energy had broad distribution peaking at 2 keV. The sample was exposed twice with collected charge of 500 Coulomb for each exposure. Sample temperature can be calculated from balance of incoming energy and its loss through infrared radiation as described by Stephan-Boltzmann law and compared with thermocouple measurements [23,24]. For given conditions the sample temperature is evaluated to be more than 1000 K.

Following each exposure, the sample surface was studied with SEM. Also, the thickness of the sample and the loss of its weight were measured to evaluate the erosion.

3. Results and discussion

The losses of the material from the sample are summarized in Table 1. The material losses are measured using two methods. Thickness of the sample on the edge is measured with micrometer and characterizes the minimal etch of the sample at the edge of the beam spot. The decrease of the sample weight is also measured which indicates total removal of the material. The resulting sputter yield is calculated using the method described in Ref. [3], it is based on weight-loss measurements. Experimentally obtained results are compared to those published in Ref. [1,3] revealing good agreement with both simulation and previously published experimental data.

The first exposure has modified the surface with morphology typical for sputter erosion, no features have been found during SEM analysis. Typical morphology found on the surface after the first exposure is shown in Fig. 1. The sample has lost about 0.17 mm in thickness, as measured with micrometer at the edge of the sample and as a result of weight-loss measurements. The central part of the sample (the area within which the ion beam was concentrated) has eroded stronger than the edges.

After the second exposure, the sample has been found to be etched through with the hole about 1.5 mm in diameter in the central part, see Fig. 2. The thickness of the sample edge has lost 0.21 mm more. However, the respective losses of the weight are smaller than after the first exposure. This can be explained by the formation of the hole, which area does not contribute to the losses of the weight once the hole is formed. The hole is rounded by pronounced features of the mm-scale, which are visible by naked eye as shown in Fig. 2. Investigation with SEM shows that these dots are the castle-like structures on the surface. Fig. 3a shows the tallest of the structures in the vicinity of the hole. It demonstrates the scale of the structure relatively to the macro-object (the hole). The other two structures are shown together in the same scale in Fig. 3b and c. One can see that the linear sizes of the structures are about 0.2 ÷ 0.3 mm. The structures have essentially asymmetrical shape. The castle-like structures are located away from each other at the distance, which is many times greater than the size of the structures.

In order to measure the height of the structures, the sample was tilted at 30 degrees relatively to the normal to the surface. The obtained SEM images are shown in Fig. 4. As it follows from trivial trigonometry equations, under these conditions the projected height is twice as low as the real one. One can measure that the height of the structure (1) exceeds 200 μm. The structure (2) reaches approximately the same height. Height of the structure (3) is about 50 ÷ 60 μm, which is a little bit

Table 1
Measured losses of the material from the sample.

Exposure #	Fluence (Coulomb)	Sample edge thickness (mm)	Sample weight (g)	Sputter yield (atom/ion)
Initial state	0	0.75	2.413150	0
1	507	0.58	1.770550	0.7
2	500	0.37	1.197935	0.6

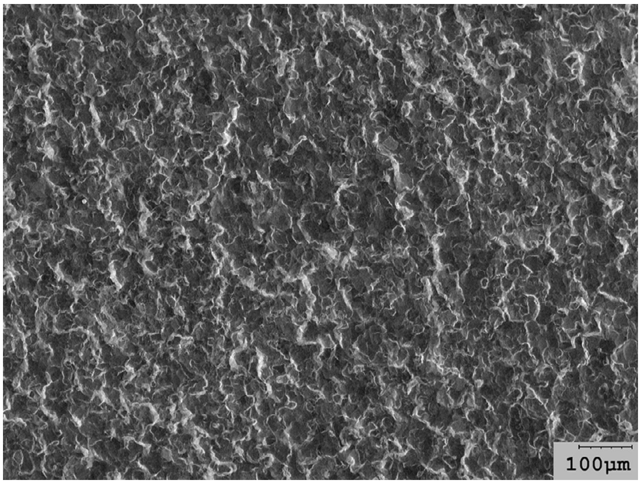

Fig. 1. Typical SEM image of the W surface after first exposure to Ar ions with the fluence of 507 Coulombs.

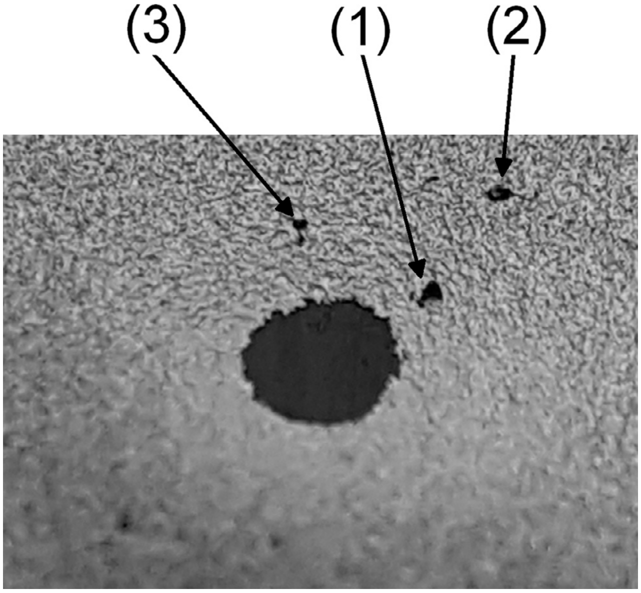

Fig. 2. Optical image of the sample surface after second exposure to Ar ions with the fluence of 1007 Coulombs. Arrows point at millimeter-scale structures: structure (1) – shown in Fig. 3(a); structure (2) – shown in Fig. 3b; structure (3) – shown in Fig. 3c.