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# Orientation dependence of cones formation under ion-beam physical sputtering of polycrystalline tungsten

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A strong dependence of cone formation on the crystallographic orientation of tungsten grains on the surface was experimentally observed under irradiation with a 30 keV argon ion beam. It was found that grains with the (114) orientation were the most susceptible to cones formation. The existing concepts of cone formation under physical sputtering are supplemented by taking into account ion channeling in crystals.

**Keywords:** tungsten, ion irradiation, channeling, ion-induced cones, sputtering yield.

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Physical ion-beam sputtering of the surface of mono- and polycrystalline metals is known to often give rise to cones, pyramids and other similar structures [1–4]. Obtaining a surface uniformly covered with ion-induced cones is of interest for numerous applications [5–8]. However, the reasons for formation of cones under ion irradiation still remain controversial. Ion-induced cones may be formed due to either impurities in metal or impurities introduced in metal by implantation, which results in local sputtering yield  $Y$  reduction [9,10] necessary for the cone growth. Investigation of pure-metal sputtering with separated ion beams has shown that cone formation is associated not only with impurities. For instance, emergence of ion-induced cones on crystals may depend on their crystallographic orientation with respect to the ion beam. So, paper [11] convincingly shows that orientation of the copper single crystal surface which enables formation of a uniform forest of ion-induced cones is the (11 3 1) face.

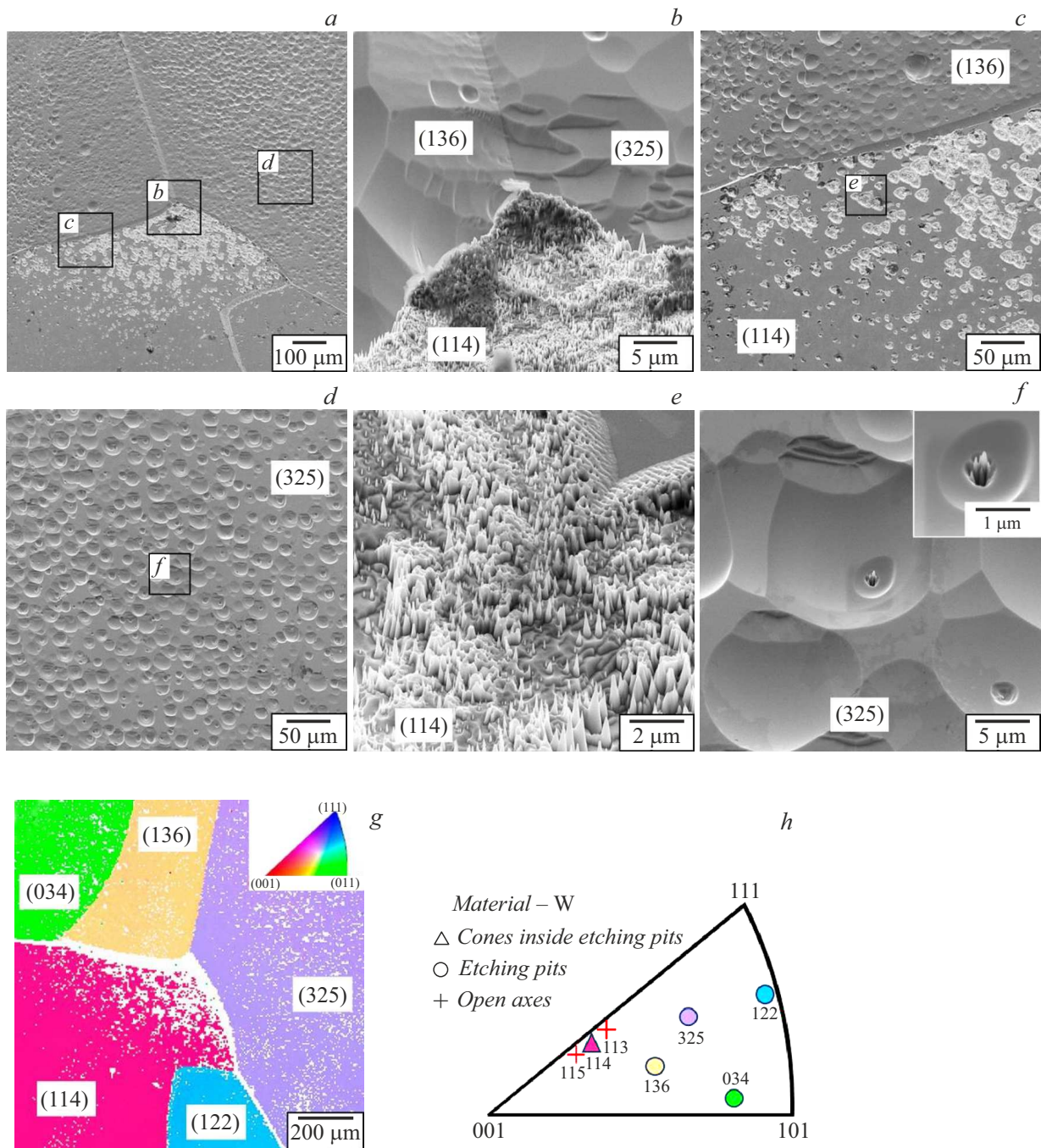
In this study, a strong orientation dependence of cone formation has been revealed for coarse-grained tungsten with different grain orientations with respect to the 30 keV argon ion beam. The existing concepts of cone formation under physical sputtering are supplemented by taking into account ion channeling in crystals.

In the experiment, we used as initial samples rolled 99.95 wt.% pure tungsten plates with a fine-grained structure and predominant grain orientation (001) [12]. To eliminate the texture and obtain a sample with a larger grain size, tungsten plates were remelted twice by the argon-arc method in a cooled copper mold. As a result of remelting, we obtained a coarse-grained sample consisting of crystalline grains larger than  $500\text{ }\mu\text{m}$  [8,12]. X-ray diffraction analysis was performed using diffractometer Rigaku Ultima IV based on  $\text{CuK}\alpha$  radiation in the angular range

$2\theta = 20\text{--}155^\circ$ . The size of the coherent X-ray scattering region and crystal lattice microstrain  $\langle \varepsilon^2 \rangle^{1/2}$  estimated using code Total Pattern Analysis Solution [13] were found to be about 140 nm and 0.114%, respectively.

Ion irradiation was performed using the SINP MSU mass-monochromator which allows irradiating materials with mass-separated monoenergetic ions [14]. The coarse-grained sample was mechanically secured on a copper holder in the mass-monochromator's collision chamber. To provide the sample with a mirror-smooth surface, it was mechanically pre-ground and electrochemically polished. The chamber was evacuated to the pressure of  $\sim 10^{-5}$  Pa with a turbomolecular pump. The beam of 30 keV argon ions  $0.3\text{ cm}^2$  in cross section was directed normal to the sample surface. The ion current was 0.1 mA, relevant ion flux density was  $2 \cdot 10^{15}$  ion/( $\text{cm}^2 \cdot \text{s}$ ). The sample was irradiated for 80 min with the fluence of  $1 \cdot 10^{19}$  ion/ $\text{cm}^2$ . The irradiated sample temperature controlled by a chromel-alumel thermocouple did not exceed  $100^\circ\text{C}$ . The surface morphology and structure were examined with a Tescan Mira 3LHM scanning electron microscope (SEM).

As a result of sputtering, grains with different ion-induced morphologies emerged on the surface of the coarse-grained sample (see Figure, *a–f*). The structure analysis by electron backscattered diffraction (scan step of  $5\text{ }\mu\text{m}$ ) using code Channel 5 [15] showed that on the sample surface there were grains with crystallographic orientations identified as (114), (034), (136), (325) and (122) (see Figure *g*). Etch pits were observed on all the grains. The pits' diameter and concentration depended on their orientation. Etch pit concentration on the grains with orientations (136), (114) and (325) was relatively high: they covered the entire grain surface; in some regions, the pits overlapped with each other



SEM images of the coarse-grained tungsten surface after irradiation with 30 keV argon ions. *a–f* — secondary electron contrast with the sample tilt of 45°, *g* — grain orientation map constructed based on electron backscattered diffraction measurements, *h* — inverse pole figure with marked orientations in which there were formed etch pits, etch pits with cones inside them and with directed orientations of axial ion channeling (*open axes*) with lower sputtering yields.

(see Figure *a–f*). Pit density on the (122)-oriented grain was low.

Comprehensive examination of etch pits on the (114)-oriented grain shows that a large number of ion-induced cones have emerged inside them (see Figure *b, e*). The (114)-oriented grain at the triple junction was slightly lower in level than the grains with orientations (325) and (136) (see Figure *b*). In some regions of the grain surface

where etch pits overlapped each other, the grain surface was completely covered with cones (see Figure *b, e*). The cones were up to 1  $\mu\text{m}$  high and did not exceed the level of surrounding etch pits. The cone base diameter was 100 to 200 nm, double apex angle was about 20°. A few ion-induced cones were also observed on the (325)-oriented grain. They were observed not in each pit and, as, e.g. in Figure *f*, secondary etch pit.

The cone-formation dependence on orientation may be understood by taking into account strong orientation dependence of crystal sputtering, which is associated with the effect of ion channeling. In terms of the channeling effect, there exist directional orientations of the crystal with respect to the ion beam, whose sputtering yield may be several times lower than those for random orientations [16]. For bcc-lattice metals, such directional orientations in order of reducing the axial channeling effect are [111], [001], [011], [113], [133], [012], [112], [115], [135] and [122]. The presence on the surface of microcrystallites with such orientations will cause a local reduction of sputtering and, similar to the case of a difficult-to-sputter impurity, will lead to the cone emergence and growth. Preferential formation of cones on the (114)-oriented grain may be explained by analyzing the inverse pole figure of the tungsten bcc lattice with marked orientations of grains on which etch pits and pits with cones inside them have been formed, and directional orientations with a lower sputtering yield (see Figure, *h*). One can see that orientation (114) is close in position to directional orientations (113) and (115). A grain with a random (in terms of channeling) orientation (114) allows in turn the presence in it of nanoblocks with directional orientations (113) and (115), if misorientation angles between the nanoblock and entire grain do not exceed  $5^\circ$  [17]. In our case, angles between the normals for orientation (114) and directional orientations (113) and (115) are  $5.8^\circ$  and  $3.7^\circ$ , respectively. A local decrease in sputtering yields of such nanoblocks was experimentally shown to initiate the cone emergence and further development. This model for the cone formation is also supported by the fact that the size of coherent X-ray scattering region of the sample under study was approximately 140 nm, which correlates with the cone apex diameter of 100 nm.

In the experiment, such a practically important parameter as the sample's mass loss under sputtering was also measured, and relevant sputtering yield was determined via the relation given in [16]:

$$Y = \Delta m N_A q / M I t,$$

where  $\Delta m$  is the target mass variation,  $N_A$  is the Avogadro number,  $q$  is the ion charge,  $M$  is the target molar mass,  $I$  is the average ion current,  $t$  is the irradiation time. The mass measurement accuracy was 0.01 mg. As a result of irradiating the coarse-grained sample with 30-keV argon ions at the fluence of  $1 \cdot 10^{19}$  ion/cm<sup>2</sup>, its mass loss amounted to 3.07 mg. The found  $Y$  values are given in the Table together with the data on sputtering of fine- and ultrafine-grained tungsten previously obtained in [12] under identical irradiation conditions. Almost identical  $Y$  values for coarse- and fine-grained tungsten supplement the conclusions of [12] about a weak sputtering yield dependence on grain size and differences in ion-induced morphologies. Significant discrepancies revealed in [3,12] between the  $Y$  calculations *via* empirical formulas and

High-fluence sputtering yields  $Y$  for various-grain-size tungsten under normally incident 30 keV argon ions

| Structure         | Grain size, $\mu\text{m}$ | $Y$ , atom/ion     |
|-------------------|---------------------------|--------------------|
| Coarse-grained    | $> 500$                   | $3.4 \pm 0.1$      |
| Fine-grained      | 1–25                      | $3.4 \pm 0.1$ [12] |
| Ultrafine-grained | 0.04–1                    | $3.6 \pm 0.2$ [12] |

computer simulation, as well as between those calculations and experimental data, makes topical the issues of adequate sputtering simulation [18–21].

Thus, the proposed physical model accounting for the presence in the grain structure of nanoblocks whose crystallographic orientations cause reduction in sputtering yields due to ion channeling and explaining the experimentally observed strong dependence of cone formation on grain orientations supplements the existing mechanisms of ion-induced cone formation.

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## Conflict of interests

The authors declare that they have no conflict of interests.

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