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Formation of submicron cone—shaped surface morphology under ion-beam sputtering of nanostructured nickel

© *A.M. Borisov*[1], *E.S. Mashkova*[2], *M.A. Ovchinnikov*[2], *R.Kh. Khisamov*[3], *R.R. Mulyukov*[3]

[1] Moscow Aviation Institute, National Research University, Moscow, Russia
[2] Skobeltsyn Institute of Nuclear Physics, Moscow State University, Moscow, Russia
[3] Institute for Metal Superplasticity Problems, Russian Academy of Sciences, Ufa, Russia
E-mail: r.khisamov@mail.ru

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The results of a research on the surface morphology of nanostructured nickel after high—fluence irradiation with 30 keV argon ions have been presented. The nanostructure in nickel was formed by high—pressure torsion deformation. It has been shown that deformation nanostructuring of nickel and subsequent ion—beam sputtering allows receiving a surface uniformly coated with submicron cones. The thermal stability of the obtained cone—shaped structure on nanostructured nickel has been determined.

Keywords: nanostructure, high—pressure torsion, ion irradiation, cones, thermal stability.

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It is known that ion—irradiation sputtering of polycrystalline metals can result in formation of a surface relief with grains of different heights; on the grains there may arise etch pits, ripples, cones and other surface structures [1,2]. Of great practical interest are metals with ion—induced cone—shaped surface morphology; such metals may be considered as, e.g., field cathodes [3,4], discharge device electrodes [5], plasma—facing components in thermonuclear facility [6,7], etc. Formation of cones depends on the radiation fluence, as well as on the material, temperature and crystallographic orientation of grains [8–11]. A significant role is played also by stress states on the metal surface [12,13]. In the ion—beam sputtering experiments, cones arising on the metal surfaces are distributed extremely non—uniformly, which restricts their practical application. It is known that among the preferable places of cone formation there are areas near the grain boundaries [1,11]. It is possible to assume that the deformation nanostructuring of metal which provides increasing the length of grain boundaries by several orders of magnitude [14] will enable obtaining under the ion—beam sputtering a uniform cone—shaped surface morphology.

The goal of this work was to investigate the influence of the deformation nanostructuring on formation of the cone—shaped nickel surface morphology under ion—beam sputtering. In the experiment, the 99.99% pure polycrystalline nickel was used. Nanostructured samples with the average grain size of $0.2\,\mu$m were obtained by severe plastic deformation by high—pressure torsion on a Bridgman anvil at the pressure of 6 GPa and number of revolutions of 10 [15,16]. The prepared samples were shaped as disks 0.5 mm thick and 12 mm diameter. For comparison, samples with the average grain size of 2, 5 and $30\,\mu$m were used. Samples with the average grain sizes of 2 and $5\,\mu$m were obtained by annealing the nanostructured samples at 300 and 500°C. As coarse-grained samples with the grain size of $30\,\mu$m, the initial (prior—to—deformation) samples were taken. The grain size in the samples was estimated by the EBSD analysis (EBSD is the electron backscattering diffraction) using by scanning electron microscopy. Irradiation of the samples was performed at the mass monochromator of the Skobeltsyn Institute of Nuclear Physics, Moscow State University [17]. Prior to irradiation, all the samples were polished. The sample surface roughness R_a prior to irradiation was $0.3\,\mu$m. The irradiation was performed normally to the surface by 30 keV Ar^+ ions at the ion current density of 0.3 mA/cm^2. The irradiation fluence was 10^{18} cm^{-2}. The sample temperature during irradiation did not exceed 45°C. To estimate thermal stability of the ion—induced cone—shaped morphology, the irradiated nanostructured samples were heated in vacuum at 500 and 800°C with keeping for 15 min. Morphology of the sample surfaces was studied with a scanning electron microscope (SEM) Tescan Mira 3LHM.

Irradiation of coare-grained and microcrystalline samples produced on their surfaces a relief with different grain heights. The appearance of difference in grain heights in the case of ion irradiation of polycrystals is caused by the known difference in sputtering yields of different faces of grains (crystallites) [1]. The difference between the grain heights is comparable with the average grain size and equals approximately $10-15\,\mu$m on coarse-grained samples (Fig. 1, *a*) and $1.5-2\,\mu$m on microcrystalline samples (Fig. 1, *b*, *c*). The prominent grains had a smoothed shape. On some grains, etch pits emerged. On the heavier etched grains there arose ion—induced cones; the more preferable places of their emergence were the grain boundary vicinities, which is clearly demonstrated in SEM images of the

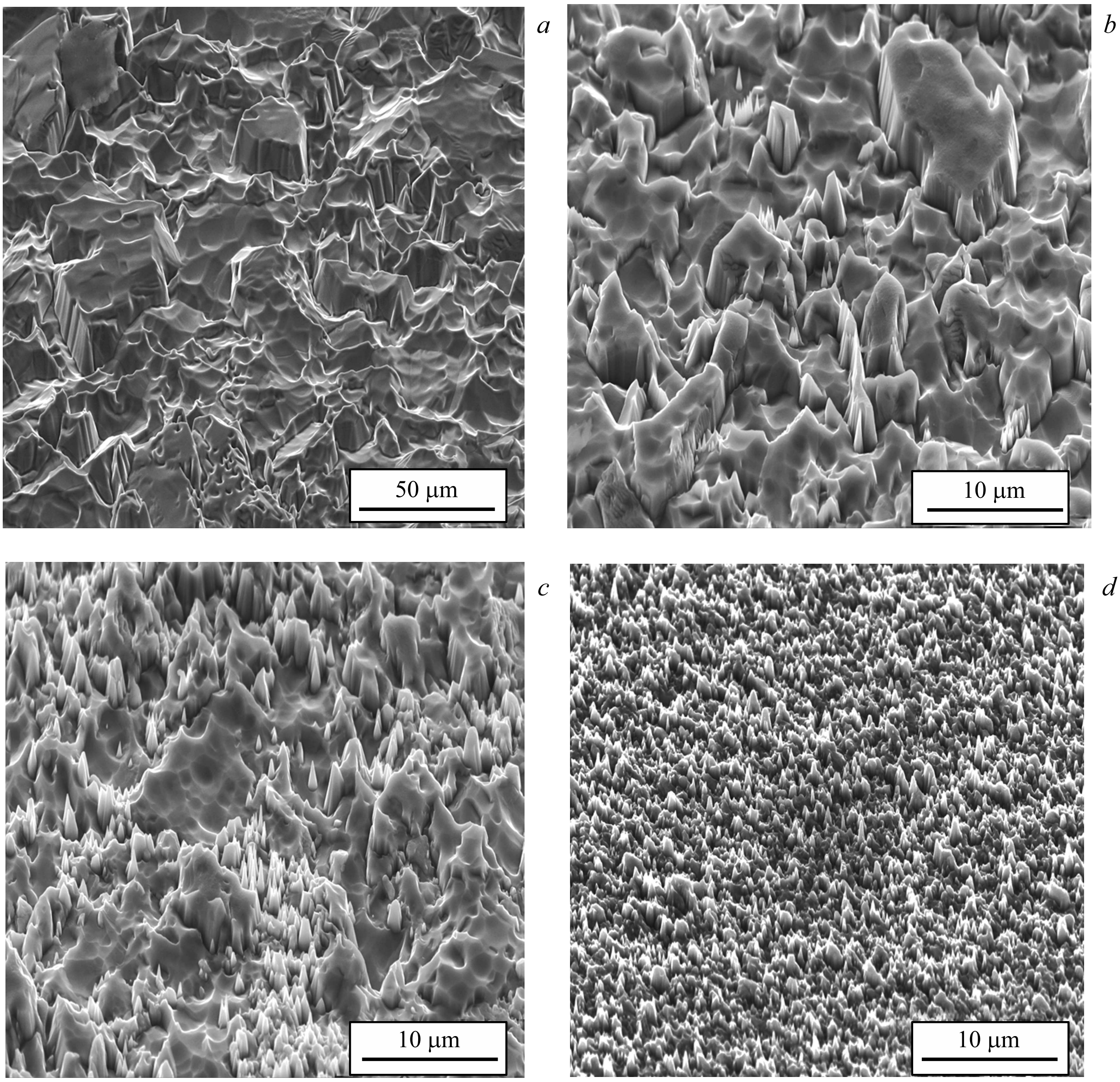

Figure 1. SEM images of the nickel surface with different grain sizes after high−fluence irradiation with 30 keV normally incident argon ions. Grain sizes (in μm) were as follows: *a* — 30, *b* — 5, *c* — 2, *d* — 0.2. The shooting angle is 45°.

microcrystalline samples (Fig. 1, *b*, *c*). Formation of cones on the grain surfaces was noticed in the work [11] where it was associated with the fact that the cones arise at such grain crystallographic orientations in which ion−induced defects have no time to relax during irradiation. The cone heights on coarse-grained and microcrystalline samples are comparable with the difference between the grain heights. The average surface density of the ion−induced cones on the nickel microcrystalline samples was about 10^6-10^7 cm^{-2}, that on the coarse-grained samples was lower than 10^6 cm^{-2}.

After irradiation of the nanostructured samples, the cone−shaped surface morphology developed on their surfaces (Fig. 1, *d*). Contrary to the morphology of coarse-grained and microcrystalline samples, the cone−shaped surface morphology on nanostructured samples is predominant. The cone−shaped surface elements are both grains with acuminate apexes with the rounding radius of about 100 nm and ion−induced cones with the apex rounding radius of 30−50 nm. Heights of the prominent grains and ion−induced cones reach 1 μm. Surface density of the ion−induced cones on the nanostructured sample reaches a value of about 10^8 cm^{-2}.

Heating of the nanostructured sample with the con−shaped morphology up to 500°C with keeping for 15 min and subsequent cooling in vacuum resulted in some modification of the cone shape (Fig. 2, *a*). The cone apexes got blunt, the cone bases got broader; at the same time, the cone height remained almost the same. Some of the cones became curved. Subsequent heating to 800°C led to radical

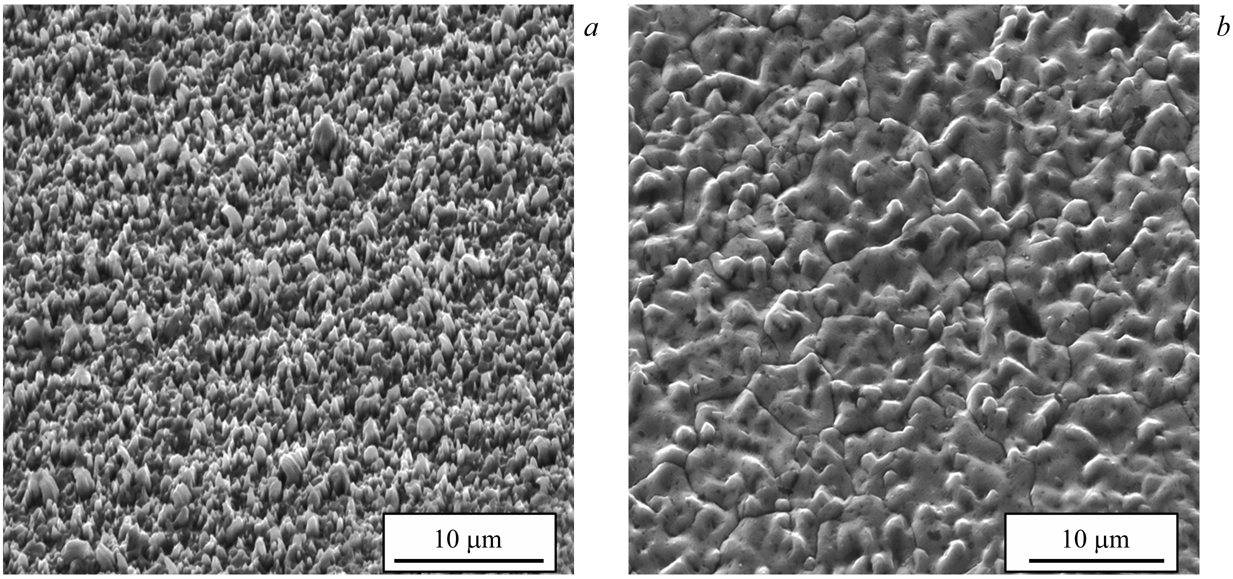

Figure 2. SEM images of the nanostructured nickel after irradiation with 30 keV argon ions and subsequent heating in vacuum with keeping for 15 min at 500 (*a*) and 800°C (*b*). The shooting angle is 45°.

variations in the irradiated surface morphology. The surface got smoothed but had prominent pimples which, most probably, were previously cones (Fig. 2, *b*). In addition, the surface got covered with a net of submicron cracks that may be caused by bulk recrystallization and grain growth up to 10 μm.

Thus, this study showed that the deformation nanostructuring of nickel enables obtaining by high–fluence ion–beam sputtering a surface uniformly coated by submicron cones with high surface density (10^8 cm^{-2}). Being heated to a temperature at least 500°C, the obtained cone–shaped morphology of the nanostructured nickel surface remained stable; the temperature it was heated to was 300°C higher than the thermal stability threshold of the bulk nickel nanostructure [18].

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Conflict of interests

The authors declare that they have no conflict of interests.

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