

# Development of Amorphized States in Subsurface Metal Regions under Radiation Exposure

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**Abstract**—Modes of radiation exposure for development of amorphized states in subsurface regions of platinum are determined. Diagnostics of the irradiated structure was carried out using the field ion microscopy technique. It is shown that radiation exposure of pure metals with an energy of  $E = 30$  keV under variation of the fluence of the charged argon ion beams by two orders of magnitude ( $10^{16}$  to  $10^{18}$  ions/cm<sup>2</sup>) produces a significant effect on the kinetics of defect formation in the subsurface regions of irradiated materials. It is found that the phenomenon of metal amorphization in the subsurface regions occurs up to a sample depth of 12 nm under an increase in the fluence to  $10^{18}$  ions/cm<sup>2</sup> and the above irradiation energies.

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One high-priority area in condensed-matter physics is related to work in the field of production of new materials via severe exposure. Such types of exposure include irradiation techniques.

Experimental studies of changes in the metal and alloy structure after radiation exposure [1–6] showed that interaction between accelerated charged ions and the substance initiates formation of amorphous, nano-, and submicrocrystalline states in the subsurface regions.

One important problem in radiation material science is determination of the structural state and phase composition of subsurface layers of materials subjected to irradiation. One topical problem consists in studies of interaction between charged particle beams and the material surface in the subsurface regions (at a distance of ~1–100 nm from the irradiated surface). Therefore, in this work, precision studies of changes in the actual structure surface of atomic layers of metals were carried out using the field ion microscopy (FIM) technique. The options of FIM allow studying the subsurface regions of irradiated materials using controlled removal of atoms from the surface and thus analyzing the sample structure in the course of layer-by-layer field evaporation of atoms. Visualization of an atomically pure and atomically smooth surface of the research object at cryogenic temperatures allows obtaining quantitative results of changes in atom positions in the crystal lattice due to ion implantation doping of positive argon atoms.

The aim of this work is to determine the modes of radiation exposure of Ar<sup>+</sup> beams accelerated to 30 keV for development of amorphized states in subsurface metal regions (Pt). Earlier, the FIM technique was

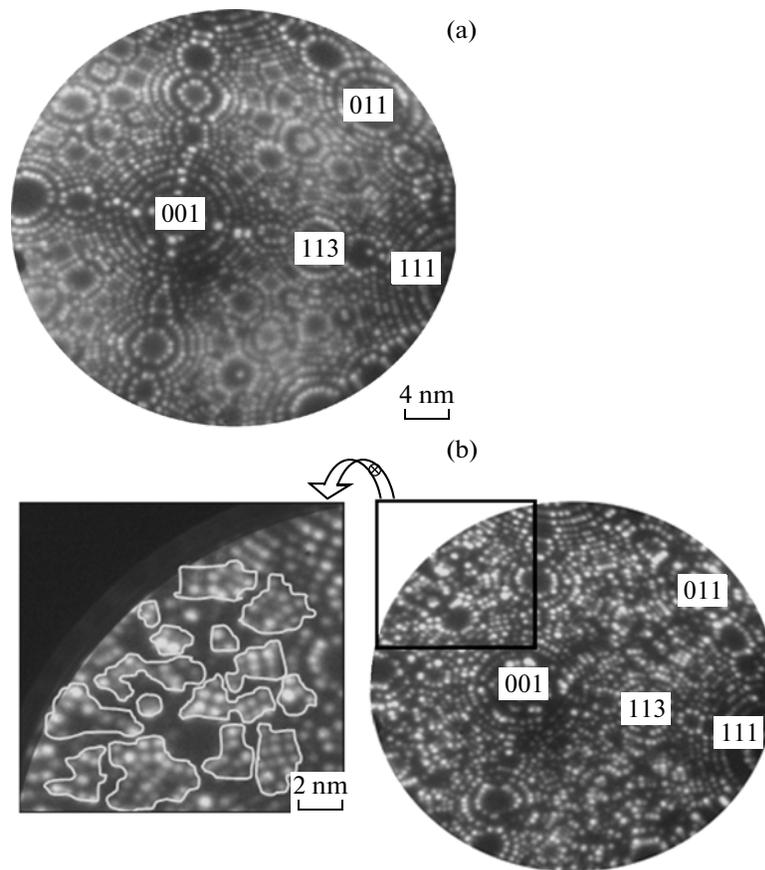
used in [5] to establish the effect of partial amorphization in subsurface regions of the Cu<sub>3</sub>Au alloy.

The object of irradiation was platinum (its purity was 99.99%). Samples intended for study were prepared in the form of needle emitters with a tip curvature radius of 30–50 nm made from metal billets by electropolishing. The samples were then evaluated in a field ion microscope to provide an atomically smooth surface prior to irradiation. The obtained ionic images of the metal surface served as confirmation (Fig. 1a). Implantation of Pt tip samples evaluated in a field ion microscope was carried out using gas ion beams (Ar<sup>+</sup>) accelerated to 30 keV with fluences ( $F$ ) of  $10^{16}$ – $10^{18}$  ions/cm<sup>2</sup> and ion current density  $j = 150$  ( $T = 70^\circ\text{C}$ ) and  $200 \mu\text{A}/\text{cm}^2$  ( $T = 200^\circ\text{C}$ ), respectively.

Bombardment was performed in the direction parallel to the sample tip axis. Implanted tip samples were again placed into the field ion microscope, and field ion surface micropatterns were registered using a photo or video camera with controlled removal of atomic layers and experimental material was obtained for further analysis of the modified structure.

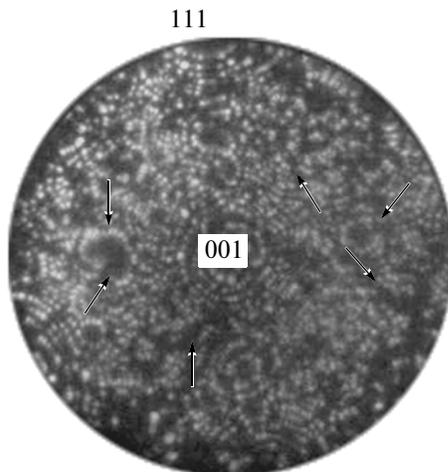
The field ion microscope was equipped with a microchannel ion–electron converter that enhanced the brightness of ion patterns by  $10^4$  times. The cooling agent was liquid nitrogen ( $T = 78$  K); the imaging gas used was spectrally pure neon.

An atomically smooth surface of the emitter tip for the further irradiation was obtained in situ in the course of field evaporation of surface atoms. Ion images of evaluated field emitters registered a practically perfect ring pattern of the pure metal single crystals pointing to the virtual absence of structural defects (Fig. 1a). As shown by analysis of the ion pattern of



**Fig. 1.** Neon images of Pt: (a) ion pattern of the evaluated crystal; (b) ion pattern of the surface after irradiation by  $\text{Ar}^+$  with  $F = 10^{16}$  ions/cm $^2$  ( $T = 70^\circ\text{C}$ ).

pure platinum after irradiation with the fluence of  $10^{16}$  ions/cm $^2$ ,  $j = 100\text{--}150$   $\mu\text{A}/\text{cm}^2$ ,  $T = 70^\circ\text{C}$  (Fig. 1b) and the fluence of  $10^{17}$  ions/cm $^2$ ,  $j = 200$   $\mu\text{A}/\text{cm}^2$ ,  $T = 200^\circ\text{C}$  (Fig. 2), the phase state of the



**Fig. 2.** Neon image of Pt after irradiation by  $\text{Ar}^+$  with  $F = 10^{17}$  ions/cm $^2$  ( $T = 200^\circ\text{C}$ ). Typical ion pattern of nanoblock boundaries and defects is shown by arrows.

metal obviously remains crystalline at the given exposure doses.

In the case of the fluence of  $10^{16}$  ions/cm $^2$ , changes are observed in the ion pattern of the irradiated platinum surface (Fig. 1b) as compared to the surface pattern of the initial evaluated Pt (Fig. 1a). Ion micropatterns manifested irregularities in the ring patterns of the crystal face images. It is such irregularities in the ring ion pattern that allow registering defects in the perfect crystal structure and determining the patterns of any defects appearing in the material as a result of exposure. In this case, changes in the ion pattern of irradiated platinum observed in the layer at a depth of 1.5 nm from the irradiated surface as compared to the pattern of the initial evaluated Pt indicate the presence of a block nanosize structure in the subsurface regions of the material [7] (Fig. 1b).

Analysis of the ion pattern of atom positions in nanoblocks (Fig. 1b) clearly indicates that atoms actually occupy their sites in the crystal lattice of the material, although the blocks themselves are nonoriented.

As a result of irradiation up to a higher fluence ( $F = 10^{17}$  ions/cm $^2$ , Fig. 2), the effect of formation of the block nanocrystalline structure (at the block size of 1–5 nm) is observed in subsurface regions at a depth of at

least 20 nm from the irradiated surface. Studies of the corresponding experimental data allowed determining the lateral and longitudinal dimensions of nanocrystalline blocks and the width of the boundary region between nanoblocks. The estimated width of the boundary region varied from 0.4 to 0.8 nm at different parts of the nanoblock boundaries in ion-irradiated platinum [8].

The ion pattern of the irradiated platinum surface manifests an image typical for grain boundaries and packing defects [9] for practically all micropattern faces (Fig. 2). This means that at  $F = 10^{17}$  ions/cm<sup>2</sup>, the mechanism of formation of the nanoblock structure in the body of the material changes.

Radiation exposure of pure metals with  $E = 30$  keV under variation of the fluence of charged argon ion beams by two orders of magnitude ( $10^{16}$  to  $10^{18}$  ions/cm<sup>2</sup>) produces a significant effect on the kinetics of defect formation in the subsurface regions of irradiated materials. Figure 3 shows the ion pattern of the irradiated platinum surface with the fluence of  $10^{18}$  ions/cm<sup>2</sup>.

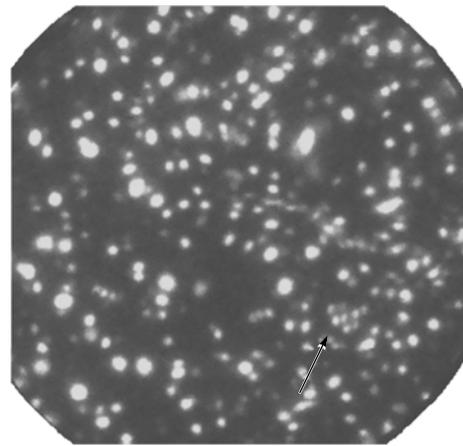
It is obvious, on the basis of the contrast of micropatterns of the atomically smooth platinum surface in analysis of the subsurface regions of the material in the course of controlled elimination of atomic layers, that the phase state of the metal becomes practically amorphous at an increase in the fluence of up to  $10^{18}$  ions/cm<sup>2</sup>. The proof of this is the structureless arrangement of atoms in the subsurface layers. The counterpart of the observed ion pattern corresponds to the ion pattern of amorphous materials obtained by ultrafast cooling. In our estimation, amorphization of the pure metal (Pt) occurs in the subsurface region at the depth of 12 nm from the irradiated surface.

Thus, radiation exposure modes for development of amorphized states in the subsurface regions of platinum were determined in this work.

It is shown that radiation exposure of pure metals with the energy of  $E = 30$  keV under variation of the fluence of the charged argon ion beams by two orders of magnitude ( $10^{16}$  to  $10^{18}$  ions/cm<sup>2</sup>) produces significant effect on the kinetics of defect formation in the subsurface regions of irradiated materials.

It is found that the phenomenon of metal amorphization in the subsurface regions of Pt occurs up to the sample depth of 12 nm under an increase in the fluence to  $10^{18}$  ions/cm<sup>2</sup> and the above irradiation energies. The amorphized phase regions are retained in the metal at a depth of at least 60 nm.

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**Fig. 3.** Ion pattern of Pt surface after irradiation by Ar<sup>+</sup> with  $F = 10^{18}$  ions/cm<sup>2</sup> ( $T = 300^\circ\text{C}$ ) (the arrow denotes the region of the crystalline state of the metal).

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