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Field ion microscopy of radiation damage on an atomically clean surface of materials

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Abstract. The defects induced by neutrons ($E > 0.1$ MeV) and Ar^+ beams ($E = 30$ KeV) on the atomically pure surface and in the near-surface volume of platinum were studied using field ion microscopy. Experimental results of radiation defect formation in surface layers of materials initiated by neutron bombardment (Pt, $E > 0.1$ MeV) and ion implantation (in Cu_3Au : $E = 40$ keV, $F = 10^{20}$ ion/m², $j = 10^{-3}$ A/cm²) are considered. Quantitative estimates of the size, shape, and volume fraction of atomic displacement cascades formed during various types of irradiation in these materials are obtained. It is shown that the average size of radiation clusters after irradiation of platinum with a fast neutron fluence of $6.7 \cdot 10^{22}$ m⁻² ($E > 0.1$ MeV) is about 3.2 nm. The experimentally established average size of a radiation cluster (disordered zone) in an alloy after ion bombardment is $4 \cdot 4 \cdot 1,5$ nm.

1. Introduction

This work is devoted to the experimental study of surface modification and subsurface volumes in solids initiated by the interaction of charged gas ion beams (Ar^+) and fast neutrons with matter.

The main goal of the study was experimental analysis of radiation defects on atomically clean and atomically smooth surfaces and in the volume of materials. For this purpose, direct methods of studying the structure of materials at the atomic level – field ion microscopy (FIM) were used.

Radiation defects were obtained by bombardment with fast neutrons and Ar^+ beams ($E > 0.1$ MeV and $E = 30$ KeV, respectively). With the help of FIM, it is possible to directly observe changes in the real crystal lattice of metals and alloys as a result of irradiation at the atomic scale. At the same time, the FIM method allows analyzing the structure of a sample in volume by sequentially and controlled removal of surface atoms by an electric field.

2. Experimental part

Experimental samples were prepared in the form of points with a radius of rounded tip of the vertex of 30-50 nm by electrochemical polishing.

Field emitters certified for ion implantation had an atomically smooth surface prepared by field evaporation of surface atoms in situ. For platinum, the modes of irradiation with charged particle beams Ar^+ were used at $E = 30$ KeV, $F = 10^{20}$ ion/m². Cu_3Au alloy was irradiated perpendicular to the sample axis with $E = 40$ KeV; at the ion current density $j = 10^{-3}$ A / cm² and pulse duration $\sim 10^{-3}$ s, the irradiation dose was $F \sim 6 \cdot 10^{20}$ ion/m². Samples implanted after pre-certification were placed in a field ion microscope to study surface modification.



After neutron bombardment of the samples, the emitters are already produced from irradiated preforms. The billets were irradiated in the IBB-2M reactor at $T = 310$ K for an hour at neutron energies $E > 0.1$ MeV and fluences $F = 6.7 \cdot 10^{21} \text{ m}^{-2}$ and $F = 3.5 \cdot 10^{22} \text{ m}^{-2}$. Emitter samples with a radius of curvature of 30-50 nm were obtained by electrochemical polishing and then placed in a FIM chamber.

The structural state of the alloy in volume was analyzed using ion images of the surface obtained by a recording device (photo or video camera) during controlled removal of atomic layers. This allowed us to analyze the structural state of the alloy in volume. The field ion microscope was equipped with a microchannel ion-electronic converter that allows increasing the brightness of the ion image by 10^4 times. Liquid nitrogen ($T = 78$ K) was used as the refrigerant, and spectrally pure neon was used as the imaging gas.

3. Results and discussion

The initial samples, certified by the FIM before irradiation, had an atomically smooth surface with an almost hemispherical shape. Such a surface was obtained in situ in the process of sequential controlled removal of surface atoms by an electric field. Field ion images of the sample surface had a clear, almost perfect ring character, which meant that there were almost no structural defects.

When studying the structure of platinum irradiated by fast neutrons to a fluence of $6.7 \cdot 10^{21} \text{ m}^{-2}$ ($E > 0.1$ MeV), a large amount of radiation damage to the crystal lattice was found, figure 1 [1]. Vacancies, displaced embedded atoms, and clusters of vacancies were registered.

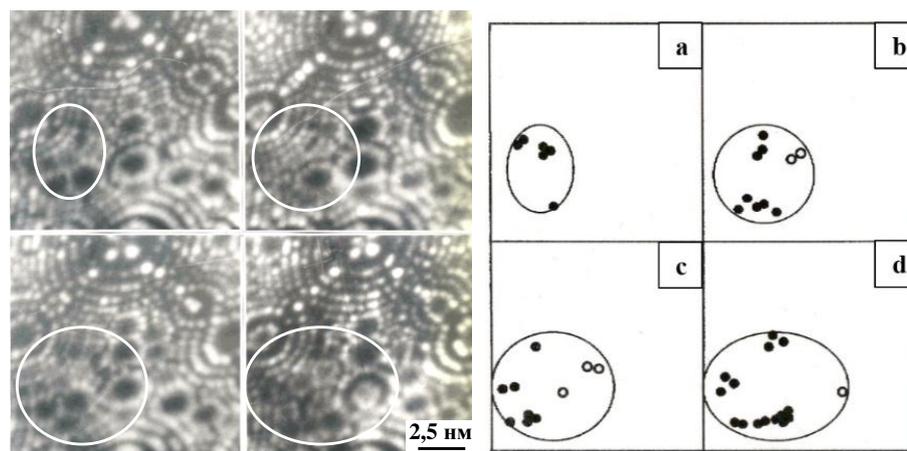


Figure 1. The regions of successive field ion images of the platinum surface after neutron bombardment, and the corresponding scheme of spatial distribution of defects. Depleted zone formed as a result of sequential controlled removal of atomic layers (at. l.): (a) initial distribution; (b) differs from (a) by one at. l.; (c) differs from (b) by two at. l.; (d) differs from (c) by two at. l.: ● - vacancy; ○ - interstitial atom.

The typical ion contrast of the spatial distribution of radiation defects in the Pt crystal lattice after neutron irradiation is shown in figure 1. In some areas of the irradiated surface, violations of the ring ion contrast were observed. The defect of the atomic structure is determined by the change in the ring ion contrast. A particular type of defect in the material after a certain external influence is identified by a known ion contrast [2]. The change in the type of ion contrast of irradiated platinum compared to pre-certified contrast is due to radiation damage. Radiation defects are the result of the interaction of fast neutrons with lattice atoms. The atomic structure of the defect in volume was analyzed by controlled removal of surface platinum atoms by an electric field. The registered radiation defects were either point defects (vacancies and interstitial atoms) or vacancy complexes.

From the analysis of the ion contrast of the studied defect region, it was found that this is a depleted zone (an area with a locally increased vacancy concentration) with a "belt" of interstitial atoms formed when the fluence was increased to $3.5 \cdot 10^{22} \text{ m}^{-2}$.

Experimental analysis confirms this hypothesis [3]. Following [3], the development of the cascade in the metal occurs in such a way that a large number of atoms are removed from the central part of the cascade (the most damaged zone) using substitution chains. From the experimental data obtained, it follows that the average concentrations of vacancies and embedded atoms in the depleted regions were equal to 9 and 1.5%, respectively. In [1], the spatial location of depleted zones in platinum after irradiation with high- and medium-energy neutrons ($F > 0.1 \text{ MeV}$) with a fluence of $3.5 \cdot 10^{22} \text{ m}^{-2}$ was clarified. To determine the characteristic anisotropy, the shape of these depleted zones was analyzed in the standard mode of controlled evaporation of atomic layers by an electric field. Analysis of the ion contrast of the defective regions did not reveal anisotropy of the shape of the depleted zones. From the experimental data obtained, it follows that the configuration of zones does not correspond to any simple geometric figure, since the vacancies of their components are located extremely irregularly. Statistics of a large number of individual depleted zones on ion images of irradiated platinum allowed to measure their longitudinal and transverse. The calculated average diameter of the depleted zone was 3.2 nm. Ion images of the surface of certified platinum showed almost perfect ring contrast for single crystals of pure metal, which indicates the absence of structural defects, figure 2a, [4]. For figure 2b shows a neon image of atomically pure platinum surfaces after irradiation with Ar^+ ions with $E = 30 \text{ keV}$ and $F = 10^{20} \text{ ion/m}^2$.

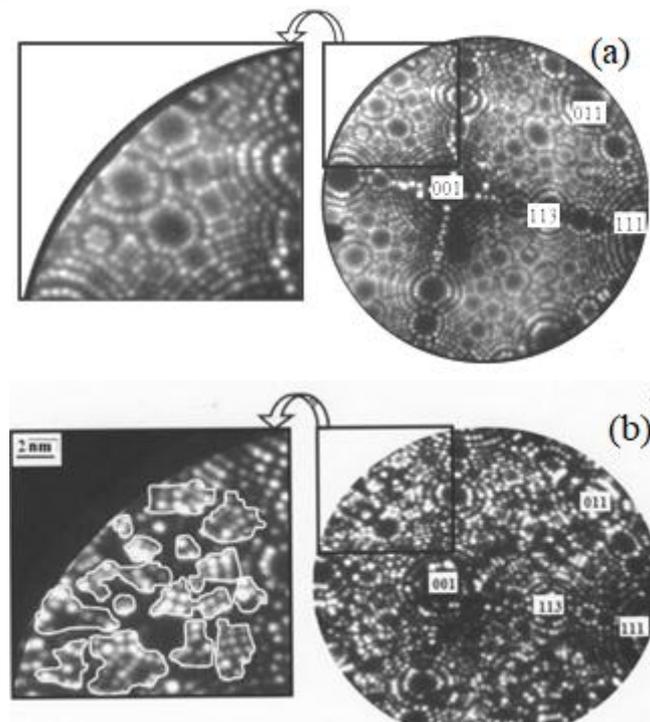


Figure 2. Neon micrographs of the Pt surface: (a) – ion contrast of the certified crystal surface ; (b) – ion contrast of the surface after irradiation with Ar^+ ions with $F = 10^{20} \text{ ion/m}^2$ ($T = 343 \text{ K}$). Nanoblocks are highlighted.

To study the atomic structure of defects formed in the regions of single displacement cascades, an ordered Cu_3Au alloy was irradiated perpendicular to the axis of the sample with an energy of $E = 40 \text{ keV}$, with an ion current density of $j = 10^{-3} \text{ A / cm}^2$ and a pulse duration of $\tau = 10^{-3} \text{ s}$, the dose was $F \sim 6 \cdot 10^{20} \text{ ion/m}^2$. This provided an average hit of one ion per $4 \cdot 4 \text{ nm}$ surface area. When

analyzing ion micro images of the surface, radiation disturbances such as disordered zones (DZ) and segregation of copper atoms were detected, figure 3, [5].

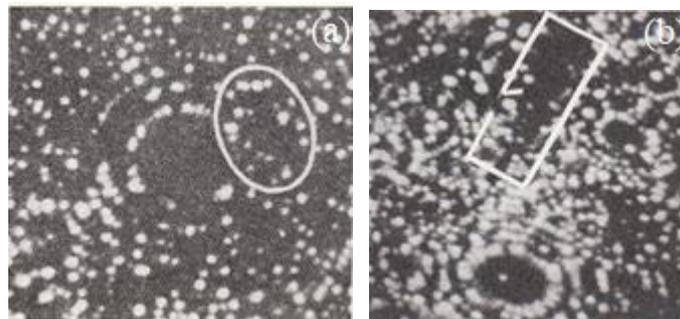


Figure 3. Neon images of surface areas of an ordered Cu_3Au alloy after irradiation with Ar^+ beams: (a) - ion contrast of the disordered zone near (011); (b) - ion contrast of Cu atom segregation.

The average size of the disordered zone (DZ), determined by the results of the study of four samples, was $4 \cdot 4 \cdot 1,5$ nm [5]. DZ were detected as violations of the ring pattern of the image of the surface of an ordered alloy, the contrast of which is similar to the ion contrast of the surface of a pure metal, since it is created only by atoms of one grade - gold. The contrast of radiation disturbances was preserved during the field evaporation of surface atoms to the depth of DZ. The experimentally determined average size of the DZ coincided with the calculated value of the displacement cascade diameter (5-11 nm), which was estimated as the average PVA run in the approximation of the spherical shape of the cascade region.

4. Conclusions

The results of radiation-induced defect formation in the near-surface volume of Pt, Cu_3Au by the FIM method are obtained. The size, shape, and volume fraction of clusters formed by various types of irradiation on an atomically clean surface and in the surface layers of materials are estimated. Various types of defects formed in ordered solid solutions as a result of implantation of gas ions are analyzed on an atomic scale. The evolution of single cascades of atomic displacements and radiation-stimulated diffusion and segregation processes that occur when metals interact with fast neutron beams is analyzed. It was found that the average size of radiation clusters after irradiation of platinum by fast neutron fluence $6.7 \cdot 10^{22} \text{ m}^{-2}$ ($E > 0.1$ MeV) is about 3.2 nm.

Acknowledgments

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