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Field ion microscopy of radiation defects in FCC materials at atomic-level spatial

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ABSTRACT

Using the method of field ion microscopy, we studied radiation induced defects on an atomically clean surface and within a subsurface volume of platinum initiated by the interaction of neutron (E >0.1MeV) and Ar^+ beams (E = 30 keV). It is shown that the interaction of fast neutrons (E > 0.1 MeV) F = $6.7 \cdot 10^{21}$ m^{-2}, F = $3,5 \cdot 10^{22}$ m^{-2} with matter leads to the formation in the amount of platinum such as radiation damage which occur after ion irradiation by beams of charged Ar^+ ions with E = 30 keV, F = 10^{20} ion/m^2. They are observed at a depth of about 1.5–2 nm, irradiated under the surface of Pt byAr^+ions.Thus, we have carried out modeling of neutron impact with matter when replacing the neutron beam by an ion beam that causes the same radiation damage in the bulk of the material. Experimental results on atomic–spatial investigation of radiative defect formation in surface layers of materials, initiated by neutron bombardment (of Pt, E > 0.1 MeV) and ion implantation (in Cu_3Au: E = 40 keV, $F = 10^{20}$ ion/m^2, $j = 10^{-3}$ A/cm^2), are considered. Quantitative estimates were obtained for the size, shape, and volume fraction of cascades of atomic displacements formed under various types of irradiation in the surface layers of the materials. It is shown that the average size of radiation clusters after irradiation of platinum to a fast neutron fluence of 6.7×10^{22} m^{-2} (E > 0.1 MeV) is about 3.2 nm. The experimentally established average size of a radiation cluster (disordered zone) in the alloy after ion bombardment is 4 × 4 × 1.5 nm.

Keywords: *radiation defects,cascades of atomic displacements, fast neutrons,Ar^+ beams, field ion microscopy.*

1. INTRODUCTION

Investigating the interaction mechanisms of accelerated particles with matter and studying the atomic rearrangement and, therefore, formation of crystal lattice defects and changing the phase state of the material are important tasks in radiation physics of solids. Radiation clusters formed under irradiation during the evolution of cascades of atomic displacements are the regions of strong elastic distortions, which affect the motion of dislocations under straining, induce radiation hardening, reduce plasticity and change the characteristics of elasticity. Information on the characteristics of radiation clusters such as concentration, size, internal structure and the number of point defects contained in them is essential for the quantitative analysis of the effect of irradiation on the structure and physico-mechanical properties of alloys. This study is devoted to the experimental investigation of fundamental physical processes in solids, which are initiating by the interaction of flows of charged gas ions (Ar^+) and neutron beams with the substance.

The main goal was the analysis of radiation defects on an atomically pure surface and in the bulk of materials. Materials were induced by neutron bombardment of Pt (99.99) with E > 0.1 MeV and ion implantation in Pt and Cu_3Au alloy in the ordered state (E = 30-40 keV) by the methods of field ion microscopy (FIM).

One of the tasks of this work was to establish the adequacy of the effects of different forms of radiation affecting on the same material (Pt) when analyzing radiation damage of the same type. For this purpose, using the methods of FIM, we have studied radiation defects on an atomically clean surface and within a subsurface volume of platinum that are created because of neutron and ion beam bombardment (E > 0.1 MeV and E = 30 keV, respectively). FIM allows precise studying to be carried out of the changes in the real crystal lattice structure of metals and alloys occurring as a result of irradiation on the atomic scale. At the same time, this method allows one to analyze the structure in the bulk of the sample by means of consecutive removal of surface atoms by the electric field.

2. EXPERIMENTAL SECTION

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3. RESULTS SECTION

The initial (FIM-attested) samples prior to irradiation had an atomically smooth surface of the emitter tip with a nearly hemispherical shape. This surface was obtained *in situ* due to field evaporation of the surface atoms. Ion images of the field emitters exhibited an almost perfect ring pattern characteristic to pure metal single crystals, which was indicative of the absence of structural defects (Figure 1).

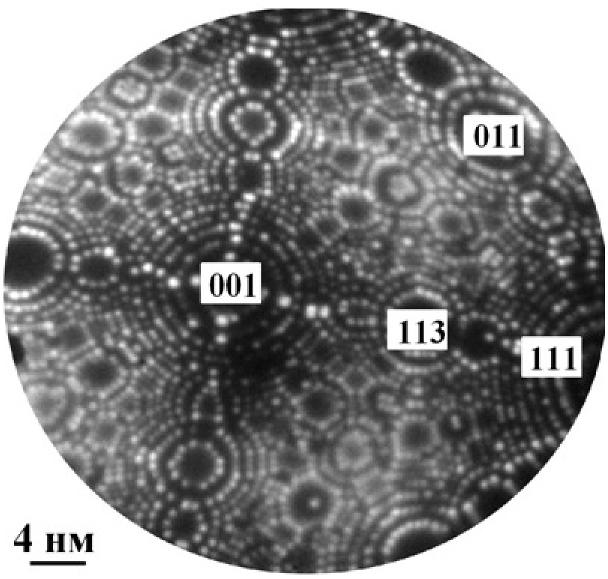

Figure 1. Neon image of the surface of platinum before irradiation.

As a result of studying structural state of pure Pt when irradiating by neutrons up to fluence of $6.7 \cdot 10^{21}$ m^{-2} ($E > 0.1$ MeV), a great amount of crystal lattice damage was found, Figure 2 [1]. Among them, we observed isolated point defects, vacancies, displaced interstitial atoms and vacancy clusters.

Figure 2 shows the ion contrast of typical spatial radiation damage distribution in the crystal lattice of Pt upon irradiation by the beam of neutrons. Micro image of the surface of platinum that was subjected to irradiation by fast neutrons is nearly similar to that of the unirradiated surface. However, in some parts of the irradiated platinum surface, violations in the ion contrast and regularity in the annular picture were observed.

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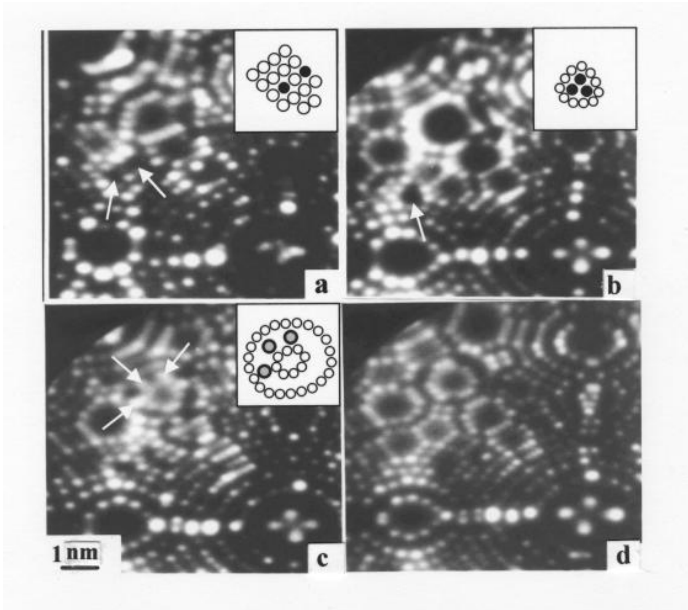


Figure 2. Micrographs of platinum surface areas irradiated by neutrons with $F = 6.7 \cdot 10^{21}$ m^{-2} obtained using Ne as the imaging gas. The arrows show: (a) isolated vacancies, (b) a tetrahedral vacancy cluster, (c) interstitial atoms, and (d) the same surface region without defects. The corresponding schemes of defects are shown in the right upper corner of the figure: ○ – atom, ● - vacancy, and O - interstitial atom.

However, in some parts of the irradiated platinum surface, violations in the ion contrast and regularity in the annular picture were observed. It is the violation of the ion contrast of the annular picture determines the imperfection of crystal structure. One or another type of defect appearing in material after a certain external irradiation is identified according to the known contrast [2]. In this case, variation in the ion contrast of the irradiated platinum as compared to the initial Pt is caused by radiation damages, which appear as a result of interaction of neutrons with crystal lattice atoms. The defect structure was analyzed in the volume during the process of controlled removal of platinum atoms by the electric field. As a rule, the observed radiation damage was represented by individual point defects (vacancies and interstitial atoms), or small vacancy complexes with the dimensions comparable to the interatomic distances (figure 2).

The depleted zones with the locally higher concentration of vacancies and annular zones of interstitial atoms were found with increasing neutron fluence up to 3.5 10^{22} m^{-2}, Figure 3.

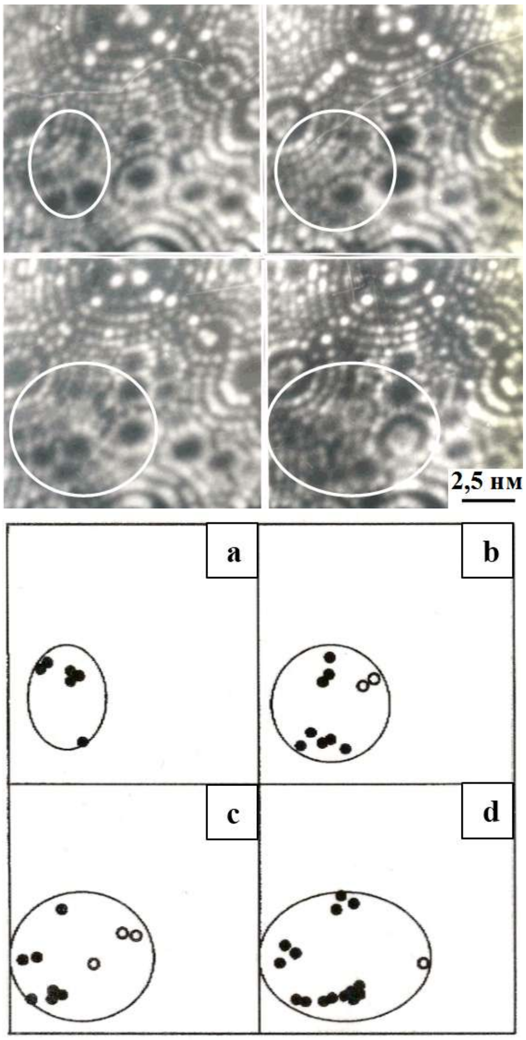


Figure 3. Regions of sequential field ion images of the platinum surface bombarded by neutrons and corresponding scheme of spatial distribution of defects. The depleted zone formed as a result of sequential controllable removal of atomic layers (a. l.): (a) initial distribution; (b) differs from (a) by one a. l.; (c) differs from (b) by two a. l.; (d) differs from (c) by two a. l.: ● - vacancy; ○ - interstitial atom.

This observation supports the hypothesis of [3], according to which the development of the cascade in metal occurs in such way that a great number of atoms are removed from the central part of cascade (the most disturbed area) by means of substitution chains. According to our estimates, the average concentrations of vacancies and interstitial atoms in the depleted areas proved to be equal to 9 and 1.5%, respectively. An attempt to clarify spatial arrangement of the depleted zones in platinum when irradiated by neutrons of high and intermediate energies (E > 0.1 MeV) with a fluence of 3.5 10^{22} m^{-2} was made in [1]. The shape of these depleted zones was analyzed in the standard regime by controlled electric field evaporation of atomic layers to determine the characteristic anisotropy. As a result of analyzing the contrast in defect areas, we did not find anisotropy of shape in the depleted areas. As follows from the data, the configuration of the zones does not correspond to any simple geometrical figure, since the vacancies are arranged extremely irregularly.

Studying a great number of micro images of the irradiated platinum allowed us to measure longitudinal and transverse dimensions of separate depleted areas. The estimated average radiative cluster diameter was equal to 3.2 nm, Figure 4. The preliminary ion images of the field emitters obtained before the ion irradiation showed an almost perfect annular contrast for single crystals of pure metal, indicating the absence of structural defects, Figure 5a [4].

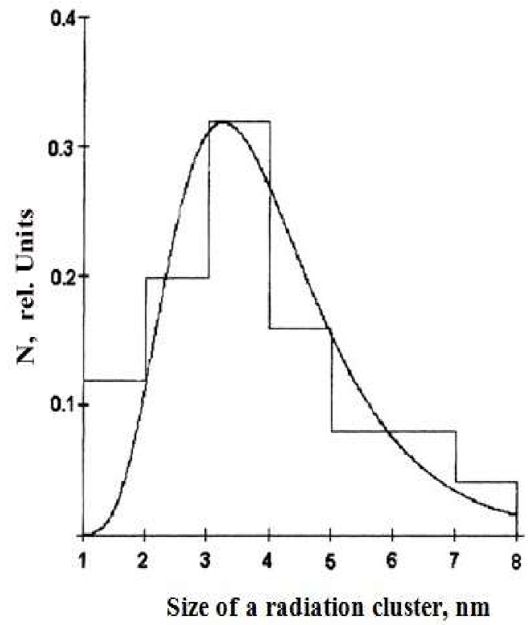
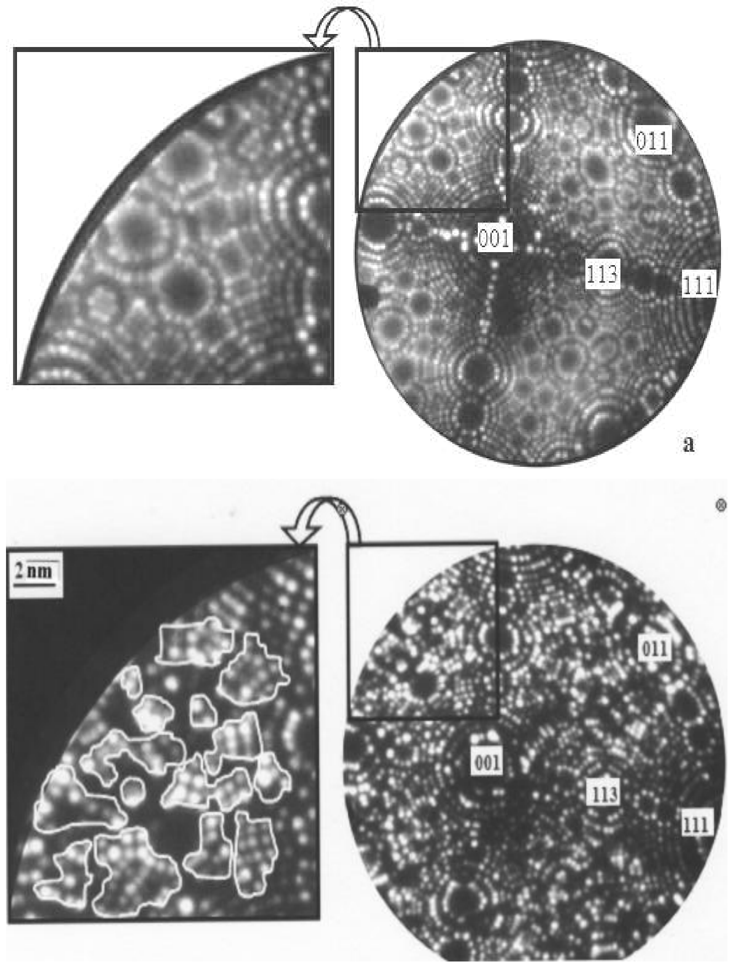


Figure 4. Size distribution of radiation clusters in platinum bombarded by neutrons (F=6.7 × 10^{22} m^{-2}).



b

Figure 5. Neon micrographs of the Pt surface: a - ion contrast certified crystal; b – ion contrast surface after irradiation by Ar^+ ions with $F = 10^{20}$ ions/m^2 (T = 343 K). Nano blocks are indicated.

Figure 5b shows a neon image of an atomically clean platinum surface irradiated by Ar^+ ions with E =30 keV and F = 10^{20} ion/m^2.

Violations in the annular contrast on the ion micrographs of the crystal faces were found. Precisely these violations in the annular ion contrast point to the imperfectness of an ideal crystal structure and determine the contrast from various defects arising in the material after external irradiation. In this case, the changes in the ion contrast of irradiated platinum as compared to the contrast of the initially certified Pt are observed within a surface layer of 1.5 nm thickness and indicate the presence of a block like structure in the subsurface layer of the material [4].

As we can see, the ion contrast of radiation damage after the ion irradiation (Figure 5b) differs from thecontrast after the neutron interaction with material (Figure 2).

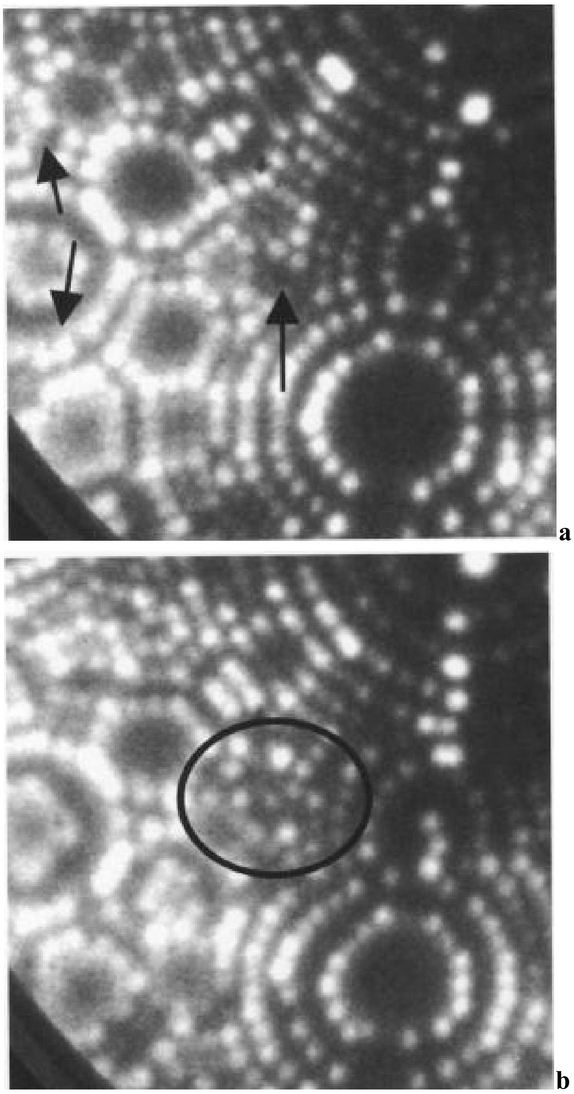


Figure 6. Neon micrographs of the regions of Pt surface after irradiation by Ar^+ ions with F = 10^{20} ions/m^2 (T = 343 K): a - at a depth of 2 nm from the irradiated surface (arrows indicate isolated vacancies and interstitial atoms); b - at a depth of 1.5 nm from the surface (the depleted zone is shown).

Upon further study of the surface of the ionirradiated Pt by the consecutive field evaporation of atomic layers, radiation damage was found at a depthof 2 nm, (Figure 6a) and was identical to that shown in Figure 2. The ion contrast of such radiation damage is shown in Figure 2 (F= 6.7 10^{21} m^{-2}) leads to the formation of the same amount of radiation damages which is observed at a depth of 2 nm after the Ar^+ irradiation (E = 30 keV, F = 10^{20} ions/m^2).

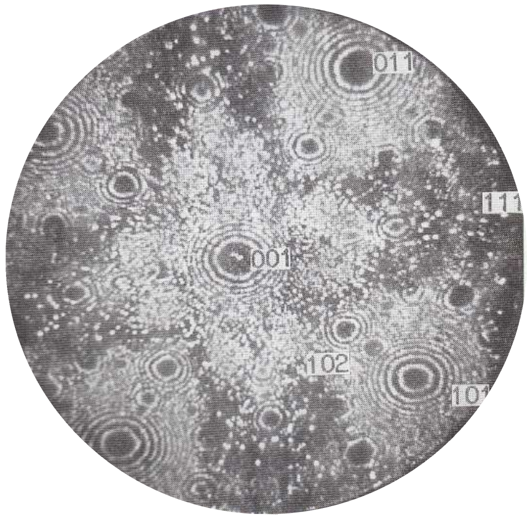


Figure 7. Neon image of the ordered Cu3Au alloy before irradiation.

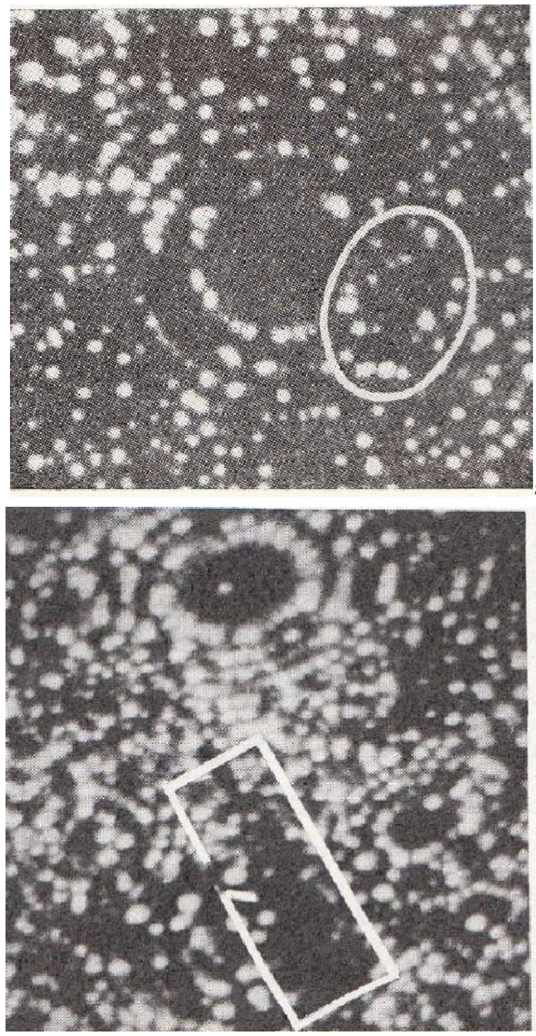


Figure 8. Neon surface images of the reogins of ordered alloy Cu_3Au after radiation: a – ion contrast of disordered zone near crystal face (011); b - ion contrast of a Cu atoms segregation.

The radiation damage (depleted zone) at a depth of 1.5 nm, (Figure 6b) was identical to that shown in Figure 3. From this, we can conclude that all the types of radiation defects arising in the bulk of the material after neutron irradiation depend only on the energy and fluence of neutrons. In contrast to this, upon the interaction of charged ion beams with matter, the type of radiation damage, apart from the parameters of radiation, depends on the distance from the irradiated surface. At a different depth of the material, one can observe transition from one type of the radiation damage to the other. Therefore, one can correctly simulate the impact of fast neutrons when replacing the neutron beam by a beam of ions only at a certain depth of a subsurface layer of material irradiated by the charged particles, given the corresponding fluence.

For studying the atomic structure of defects formed in the region of single displacement cascades, atomically ordered alloy Cu_3Au, (Figure 7), was irradiated perpendicularly to the needle axis with E = 40 keV; for ion current density $j = 10^{-3}$ A/cm^2 and pulse duration $\tau = 10^{-3}$ s, the radiation dose was F~ 6 × 10^{20} ion/m^2.

This ensured (on the average) the incidence of one ion on an area element 4 × 4 nm in size. Analysis of resultant ion microphotographs of the surface revealed radiation defects such as disordered zones, (Figure 8a), and segregations of copper atoms, (Figure 8b), [5].

The average size of disordered zones determined from analysis of four samples was 4 × 4 × 1.5 nm. Disordered zones were identified as violations of the annular structure of the image of the surface of the atomically ordered alloy, whose contrast is analogous to the ion contrast of the surface of the pure metal. The ion contrast of the surface of Cu_3Au alloy in the ordered state formed only by gold atoms.

The contrast of radiation defects was preserved upon field evaporation of surface atoms to the depth of distorted zones. The experimentally established average size of the distorted zones coincided in order of magnitude with the calculated diameter of a displacement cascade (5–11 nm), which was estimated as the mean free path of the primary knocked-on atom and in the approximation of the spherical shape of the cascade region. In the course of investigation, we succeeded in detecting only the segregations of copper atoms, (Figure. 8b).

Since copper atoms do not form a visible image on ion microphotographs of the surface, the contrast from a copper segregation is observed as a dark region. Aggregates or segregations of copper atoms are three dimensional and contain 200–500 atoms as a rule.

The presence of vacancies in these aggregations cannot be determined experimentally, although analysis of the ion contrast of the boundaries of the dark regions shows that their possible number is insignificant.

4. CONCLUSIONS

We have obtained the results of direct analysis of radiation induced defect formation in the subsurface bulk of the Pt, Cu_3Au by the FIM method. The sizes, shapes, and volume fractions of clusters formed under various types of irradiation in the surface layers of the materials have been estimated. On atomic scale, we have analyzed various types of defects formed in ordered solid solutions as a result of implantation of gas ions as well as upon the interaction of metals with the neutron beam and appearing as a result of evolution of single cascades of atomic displacements and radiation stimulated diffusion and segregation processes. It is shown that the average size of radiation clusters after irradiation of platinum to a fast neutron fluence of 6.7 × 10^{22} m^{-2} (E > 0.1 MeV) is about 3.2 nm.

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6. ACKNOWLEDGEMENTS

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