Rev.Adv.Mater.Sci. 10 (2005) 422-426

EFFECT OF SEVERE PLASTIC DEFORMATION ON ATOMIC STRUCTURE OF METALS AT STUDY OF FIELD ION MICROSCOPY METHOD

V. Varyukhin[1], B. Efros[1], V. Ivchenko[2], N. Efros[1] and E. Popova[2]

[1]Donetsk Physics and Technology Institute of National Academy of Science of Ukraine, 72 R. Luxembourg St., Donetsk, 83114, Ukraine
[2]Institute of Electrophysics of Ural of branch of Russia Academy of Science, 106 Amundsen St., Ekaterinburg, 620016, Russia

Received: March 02, 2005

Abstract. It has been revealed that UFG structure is formed (the grain size of 20-30 nm) in Iridium influenced by SPD; but there are practically no defects of structure in the bodies of grains. However, a subgrain structure (subgrain size of 3-5 nm) is formed after irradiation and defects are observed in the bodies of subgrains. The subgrain structure was also found in UFG Nickel and Copper after SPD (subgrain size of 3-15 nm), but in the latter case the observed boundary region is broader and subgrains are highly disoriented.

1. INTRODUCTION

Lately there has been an increased interest to ultrafine grained (UFG) materials, among which there are submicrocrystalline (the average grain size ~ 100 - 200 nm) and nanocrystalline (the average grain size ~ 10 - <100 nm) materials. This is because the physical and mechanical properties of UFG materials are much differ from those of usual coarse-grained materials [1-3].

This fact opens new possibilities for the production of materials with the present and record properties. A significant part in the formation of specific properties in UFG materials is played by a large portion of grain boundaries in their volume and their peculiar nonequilibrium state [4].

UFG materials are produced by powder methods, ball milling, and quick cooling of the melt and by severe plastic deformation (SPD). The latter methods [3,5,6] turn out well for the production of UFG specimens having no pores and contaminations. Such specimens are suitable for defect-structure investigations. Besides, from the strain hardening point of view of interest are experimental studies of radiation-induced defects in irradiated materials when there are no radiation-stimulated phase transitions, and the increased density of implantation defects can result in an essential changes in structure state and properties.

In this work, the field ion microscopy (FIM) has been used to study the atomic-spatial structure of

Corresponding author: B. Efros, e-mail: efros@ukr.net; efros@hpress.dipt.donetsk.ua

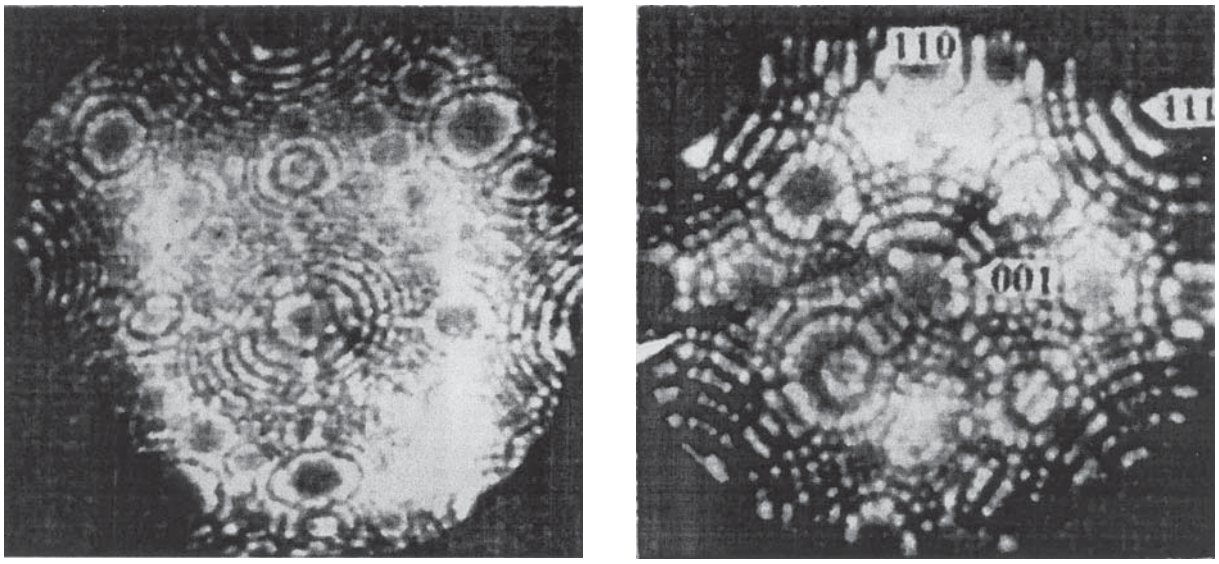

Fig. 1. Field ion image of Iridium surface (V = 10 kV): a – initial state; b – UFG state after SPD ($e \approx 6$). (The arrows show the grain boundaries).

defects in different metals subjected to severe external influences. FIM method potentialities make it possible to investigate the real structure of the crystal lattice of solids at the atomic level, to work with the atomically pure surface at cryogenic temperatures, and, at the same time, to analyze the atomic structure of an object in the volume by a controllable consecutive removal of surface atoms by the electric field.

The paper is aimed not only at the comparison of parameters of the defect structure of metals on the atomic level, but at the analysis of the type of action that has induced the particular faults in the crystal lattice of the material.

2. MATERIALS AND EXPERIMENTAL METHODS

The investigation objects were polycrystalline Iridium, Tungsten, Nickel, and Copper of 99.95-99.99% purity (with the initial grain size ≈ 20-50 μm).

The atomic structure of defects of different *n*-dimensionality (preferentially the in-plane type) was studied after SPD and ion irradiation of the metals. The UFG structures in Iridium, Tungsten. and Copper were formed by SPD method of shear (logarithmic deformation $e \approx 7$) under quasi-hydrostatic pressure by using a plant of the Bridgman anvils type [5].

The Iridium samples were irradiated by argon ions with the energy E = 20-24 keV, irradiation dose D = 10^{18} ion/cm ^{2}, and current density j = 300 μA/cm ^{2}.

SPD of Nickel with the aim of obtained UFG structure was realized by the method of packet hydrostatic extrusion (PHE) (with the maximal logarithmic deformation $e \approx 12$) under the room-temperature conditions [7].

The samples of metals investigated by FIM were in the form of needle-like emitters, with the radius of curvature at the apex of 30-50 nm; they were prepared from the radiation and SPD-treated billets by electrochemical polishing. The installation for FIM-investigation consisted of a field ion microscope constructed in Institute of Electrophysics of Ural of branch of RAN fitted with a micro-channel ion-electron converter providing a 10 times gain in brightness of micro-images. Liquid nitrogen (T = 78K) was the coolant and the image-making gas was neon of spectral purity [8].

3. EXPERIMENTAL RESULTS AND DISCUSSION

Pure Iridium preliminarily certified in the field ion microscope (the initial state) had, prior to the external influences, atomically smooth surface *in situ* prepared by field evaporation of surface atoms. The ion images of the certified field emitters (Iridium samples) fixed a truly ring-like pattern typical of single crystals (Fig. 1a) pointing to the absence of the atomic lever structure defects in the grain body.

After the ion irradiation, the implanted samples were placed in FIM once again. The field ion images

of the surface were registered by a video or a photographic camera under a controllable removal (evaporation) of one atomic layer after another; next the state of metal in the near-surface layer was analyzed. As a result, there was a high density of point, linear, and volume structure defects (Fig. 2) in the implanted pure Iridium.

A comparative analysis of structure defects revealed in Iridium after SPD ($e \approx 6$) (Fig. 1b) and in irradiated Iridium (see Figs. 2a-2c) showed that their structure is essentially different and depends on external influence type. In the deformed Iridium, there were the deformation grain boundaries, $d_g \approx 20$-30 nm, and in the grain bodies there were practically no crystal structure defects (Fig. 1b). On the contrary, a sub-grain structure (subgrain size $d_{sg} \approx 3$-5 nm) was revealed in the irradiated Iridium (Figs. 2a-2c). The angular misorientation ω of sub-grain was approximately equal to 0.5-1°. Different structure defects were observed (in shown by arrows in Fig. 2) down to the micro-pores (Fig. 2a) in subgrain bodies. After the external influence action, the width of the boundary region in Iridium was of the order of the inter-atomic distance, the same as in thermally treated metals and alloys [9].

The ion contrast of implanted Iridium sub-grain structure was revealed by a slight increasing of voltage on emitter sample (with a 500 V difference with respect to the most clear image) at a moment when the field induced evaporation of surface atoms has not been yet taken place. The boundary contrast was easily seen in the form of more bright lines surrounding the subgrains (Fig. 2c). The complete correspondence between the break in the circular pattern of Fig. 2b and the contrast of boundaries of structure subgrains of Fig. 2c is clearly seen; note that microphotographs of the same Iridium surface are shown in Figs. 2b and 2c, but the microphotograph in Fig. 2c was taken with a 500 V increase of voltage at the sample. It should be stressed that it is the break in circular pattern of the ion contrast that points to faults in perfect crystal structure and determines the contrast from some or another defects occurring in the metal after the action of external influences.

The analysis of the near-surface volume of the argon ions-implanted Iridium in course of a successive controllable removal of surface atoms has shown that the defected structure is preserved at a distance to 50 nm from the irradiated surface. It is known [10] that the projective path of argon ions in metals, in particular in Iridium, makes not more that 10 nm for the implantation regimes used. Hence, it can be assumed that the observed deformation ef-

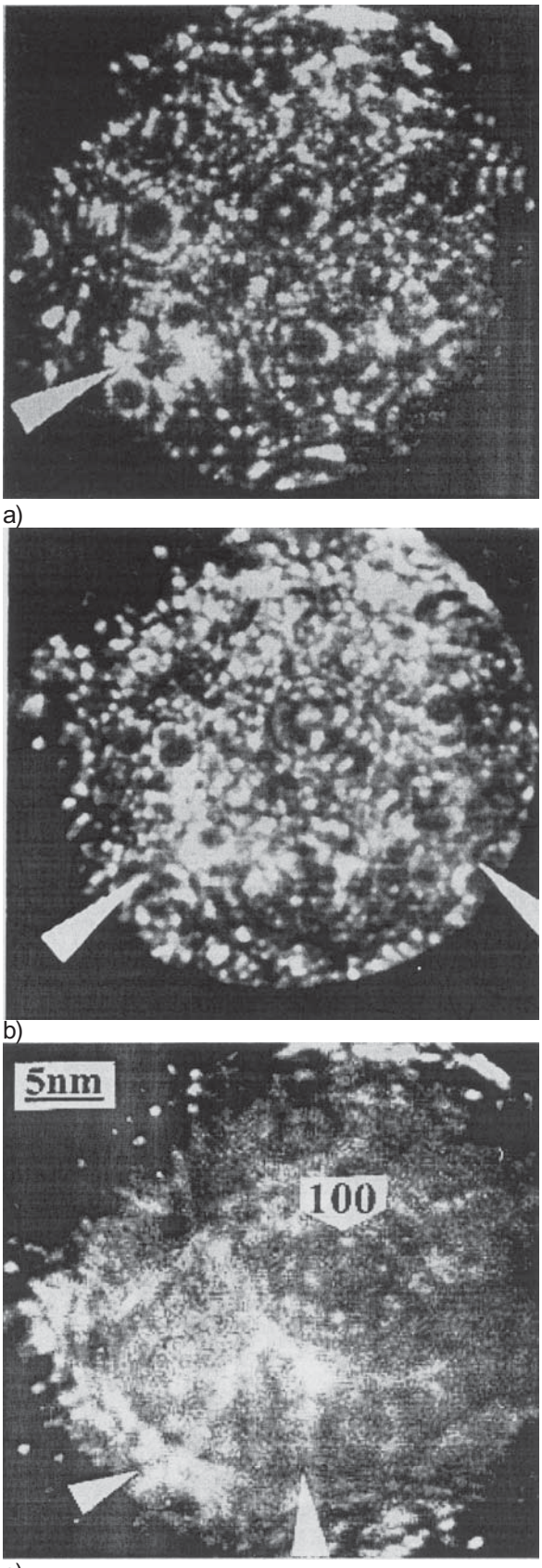

Fig. 2. Field ion image of Iridium surface after the argon ion implantation (E = 20 keV, D = 10^{18} ion/cm^2, j = 300 μA/cm^2): a – V = 7.2 kV (A micro-pore is shown); b = V = 8.4 kV (The arrows shown crystal-lattice defects); c - V = 8.9 kV.

fects are, evidently, due to the impact action of the ion beam, distribution of the elastic waves in the material and their interaction with irradiation induced lattice defects as well as with the implanted argon ions. Moreover, a definite role in the process of defect formation can be played by a high density of the implantation current.

SPD of coarse-crystalline Nickel by PHE (logarithmic deformation $e >> 12$) results in the formation of UFG state with the average size of microcrystalline-grain $d_g \approx 100$ nm [11]. Fig. 3a shows the ion images of UFG Nickel surface sections depending on the quantity of removed layers of surface atoms under a controlled evaporation. This is adequate to UFG structure change through the thickness of Nickel sample in the investigated near-surface volume, so the boundaries of ultra-disperse subgrains, which make the substructure of separate grains of UFG nickel, can be fixed clearly. Dimensions of the revealed subgrains d_{sg} were estimated both on the surface of the ion micropattern for UFG Nickel and during the removal one atomic layer after another. They were ranging from ≈ 3 to ≈ 10 nm (see Fig. 3a). The performed analysis has shown that the subgrain bodies are perfect microcrystallites, which are not practically misorientation relative each other. During the examination of UFG Nickel atomic structure, at the sub-grain interface some dislocations were seen to emerge. The width of boundary region was the distance comparable with the interatomic distance.

The obtained results are identical in a first approximation to those of UFG Tungsten atomic structure after SPD ($e \approx 7$) with the average grain size $d_g \approx 100$ nm [12]. The analysis of field ion image of UFG Tungsten surface section having the interface has shown that its width approximately equals 0.6-0.8 nm. We note that in the initial (underformed) coarse-crystalline Tungsten, the boundary is ≈ 0.3-0.4 nm wide [12].

The formation of ultra-disperse sub-grains was also noted in UFG Copper after SPD by torsion ($T_{def} \approx 400$ °C, $e \approx 7$), see Fig. 3b. The ion contrast from the subgrain boundaries in UFG Copper shows a wider boundary region (of the order of 3-4 interatomic distances), see Fig. 3b, as compared to UFG Nickel, Fig. 3a. It should be also noted that after SPD of such a type, in samples of UFG Copper the subgrains ($d_{sg} \approx$ 8-15 nm) are much more misorientation relative each other as compared to the sub-grains in UFG Nickel.

In such a way, the study of the defect structure of metals on the atomic level by FIM method has revealed for the first time the formation of subgrains

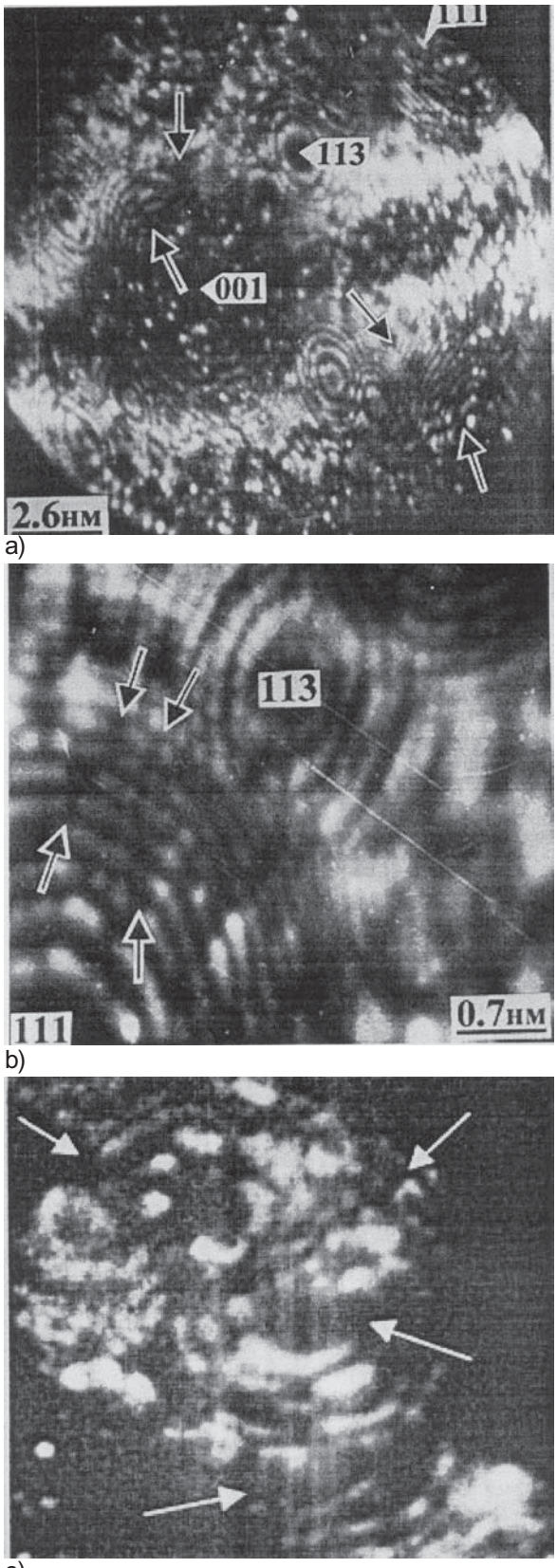

Fig. 3. Field ion image of UFG Nickel (a, b) and Copper (c) surface after SPD (Ni: $e \approx 12$ and Cu: $e \approx 7$, $T_{def} \approx 25$ °C, $V = 15$ kV): a – after removal of 73 atomic layers (26 nm) ($V = 20$ kV); b – 622 atomic layers (240 nm) ($V = 12.5$ kV). (The arrows show the boundaries of ultra-disperse subgrain).

and different structure of their boundary region during the external influences. The nature of their crystal structure essentially depends on external influence type and determines, in the end, physical and mechanical properties of the investigated metals.

4. CONCLUSIONS

The direct FIM method has evidenced on the atomic level that the density of defects of different *n*-dimensionality is high in metals subjected to external influences (ion radiation and severe plastic deformation).

It has been revealed for the first time that in the volume of UFG Nickel and UFG Copper microcrystallite -grains an ultra-disperse subgrain structure ($d_{sg} \approx$ 3-15 nm) is formed after SPD. The dislocation nature of the boundaries of the observed subgrains has been determined.

The ultra-disperse subgrain structure in the surface and near-surface volumes of Iridium was for the first time found as resulting from the implantation of argon ions at distances, which are the order of magnitude larger than the projective path of argon ions from the irradiated surface.

REFERENCES

[1] I.D. Morokhov, L.I. Trusov and V.I. Lapovok, *Physical phenomena in ultra-disperse media* (Nauka, Moscow, 1984), in Russian.

[2] V.A. Teplov, V.P. Pilyugin and G.G. Talutz // *Metals* **2** (1992) 109.

[3] R.Z. Valiev and I.V. Aleksandrov, *Nanostructural materials produced by severe plastic deformation* (Logos, Moscow, 2000).

[4] O.A. Kaibyshev and R.Z. Valiev, *Grain boundaries and properties of metals* (Metallurgia, Moscow, 1987), in Russian.

[5] B.M. Efros, V.P. Pilyugin and S.V. Gladkovskii // *Def. & Diffus. Forum* **208** (2002) 263.

[6] N.A. Akhmadeyev, R.Z. Valiyev, V.I. Kopylov and R.R. Mulyukov // *Metals* **5** (1992) 96.

[7] B.M. Efros, S.G. Synkov and V.G. Synkov // *Phys. & Tech. of High Press*. **12** (2002) 27.

[8] V.A. Ivchenko, V.V. Ovchinnikov and S.O. Cholakh, *Crystallographic identification of the field ion images of crystals* (UGTU-UPI, Ekaterinburg, 2001), in Russian.

[9] V.A. Ivchenko, In: *Radiation physics of a solid*, ed. by G.G. Bondarenko (MGIEM: Moscow, 2002), p. 18.

[10] A.F. Burenkov, F.F. Komarov, M.A. Kumakhov and M.M. Temkin, *Spatial distribution of energy liberated in a cascade of atomic collisions in solids* (Energoatomizdat, Moskow, 1985), in Russian.

[11] V.A. Ivchenko, B.M. Efros, E.V. Popova and N.B. Efros // *Phys. & Tech. of High Press.* **13** (2003) 109.

[12] R.R. Mulyukov, Yu.M. Yumagizim, V.A. Ivchenko and L.R. Zubairov // *Zh. Exper. Teor. Phys. Pis'ma* **72** (2000) 377, in Russian.