

Peculiarities of High Dose Ion Implantation of Polycrystalline Materials under Irradiation by Ion Beams with a Wide Energy Spectrum

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Abstract – The investigation results on redistribution of Be, Al, Ti, Fe, Cu, Zr, Mo, and W atoms implanted into polycrystalline metal samples under irradiation by beams of He^+ , $\text{He}^+ + \text{Ar}^+$, and Ar^+ ions with a wide energy spectrum and a mean energy of 10 keV at irradiation doses of up to $1 \cdot 10^{21}$ ion/cm² are presented in the work.

It has been found that irradiation at a dose higher than $1 \cdot 10^{19}$ ion/cm² results in formation in the surface layer of a substrate of local fine crystalline areas with typical sizes less than 1–5 μm and a density up to $(1-100) \cdot 10^3$ cm⁻². They contain both implanted and impurity atoms with a concentration changing in the 0.1–10 at.% range.

Their subsequent irradiation to doses of $1 \cdot 10^{20}$ ion/cm² and higher results in their elimination predominantly at the expense of sputtering processes.

1. Introduction

Ion alloying of articles with complex geometry (for example, the presence of cavities, fillets etc.) demands the use of additional methods guaranteeing, first of all, the specified distribution of implanted atoms both over the treated surface and in depth in regard to the surface level. In a number of cases it is advisable to irradiate materials by an ion beam with a wide energy spectrum (IB-WES, when the ratio σ of the energy spectrum width ΔE to the mean energy $\langle E \rangle$ of ions in a beam is equal to $\sigma = 10-50\%$), for example, to obtain the distribution of implanted atoms in a matrix close to square or with a specified concentration gradient in the surface layer [1–3].

The experiments show that in case of simultaneous implantation of ions with various energies and atomic masses, distinctions are observed in comparison with irradiation by a single-component monoenergetic beam of the same ions. These distinctions are conditioned, first of all, by peculiarities of the energy-release F_q and the gradient of internal stresses in the surface layers of materials [4]. At that, an important condition of effective implantation of the atoms of multilayer films into polycrystalline matrixes is carrying out implantation using the regime of “ion polishing”, which makes it possible to form uniform multicomponent modified surface layers at a depth up to 0.3–0.5 μm at the expense of radiation-stimulated processes.

The present work shows investigation results of the redistribution of implanted atoms in polycrystalline matrixes under the action by a two-component beam of $\text{He}^+ + \text{Ar}^+$ ions with a wide energy spectrum changing in the irradiation dose range of $\Phi = (0.5-100) \cdot 10^{18}$ ion/cm².

2. Experimental techniques

To study the behavior of film atoms in a substrate under IB-WES-irradiation, materials with various ratios of the atomic radii of the “film-substrate” system were chosen. Al, Si, Ti, Fe, Cu, Ni, Mo were used as film atoms and Be, Al, Fe, Cu, Mo, Zr, W as substrate atoms ($\alpha = 0.8-0.9, \sim 1.0, 1.1-1.3$, where $\alpha = r_1/r_2$, r_1 is the atomic radius of an implanted atom, and r_2 is the atomic radius of substrate-target atoms).

The substrate samples were exposed to a standard treatment which consisted of mechanical treatment using abrasive papers and diamond pastes, electro-polishing in corresponding electrolytes, and vacuum annealing at $P < 10^{-6}$ Pa for 1 h.

The substrates were alloyed by film atoms using ion mixing under irradiation of the “multilayer film – substrate” system by He^+ and Ar^+ ions. The energy of primary knocked-out (*pka*) atoms was determined by the formula: $E_{pka} = \mu_0 \langle E \rangle$, where $\mu_0 = 2M_0M_1 / (M_0 + M_1)^2$ (M_0 is the Ar-ion atomic mass; M_1 is the atomic mass of a film atom). One-, two-, three-, and five-layer films of various combination were sputtered in vacuum at the pressure of residual gases of $P < 10^{-4}$ Pa. The depth of a film layer was determined on condition of full braking at the starting stage of irradiation by He^+ and Ar^+ ions with a mean energy $\langle E \rangle = 10$ keV and it was equal to $X_i = 30-50$ nm.

Irradiation of the “multilayer film–polycrystalline substrate” systems in the $1 \cdot 10^{18} - 5 \cdot 10^{19}$ ion/cm² dose range was carried out on the installation “VOKAL” (Fig. 1) generating ion beams with a wide continuous gauss-like energy spectrum (Fig. 2).

With a purpose to increase the alloying efficiency, irradiation of a “film-substrate” system by a two-component beam of $\text{He}^+ + \text{Ar}^+$ ions ($C_{\text{He}^+} : C_{\text{Ar}^+} = 1 : 1, 1 : 2, 1 : 5, 1 : 10$) was carried out under the following parameters: the mean ion energy in a beam $\langle E \rangle = 10$ keV, the ion energy range was 3–15 keV, the beam current $j = 25-45$ $\mu\text{A}/\text{cm}^2$, the pressure of residual gases in the area of a sample $p \leq 1 \cdot 10^{-5}$ Pa, the irradiation temperature $T < 150$ °C [5].

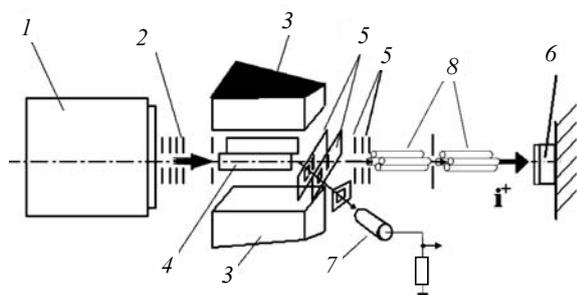


Fig. 1. A scheme of the installation "VOKAL": 1 – the ion source generating an ion beam; 2 – the accelerating tube; 3 – the type Wien-filter electromagnet poles; 4 – the type Wien-filter electrodes generating a transverse electric field; 5 – the limiting diaphragm; 6 – specimen for irradiation; 7 – the detector for determining the current of ions; 8 – quadruple lens ($f = 5$ MHz, $U = 1$ kV)

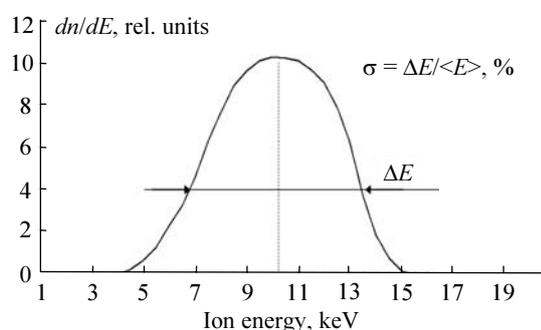


Fig. 2. A typical energy spectrum of He^+ ions in a beam: $\langle E \rangle = 10$ keV; the energy range is 1–17 keV; $\sigma = 40 \pm 5$; the current density of a beam is higher than $35 \mu\text{A}/\text{cm}^2$; the pressure in a working chamber is less than $1 \cdot 10^{-6}$ Pa

Changes of the surface relief of samples were studied by PP-210 and α -Step (the indenter point radius $\approx 1.0 \mu\text{m}$; the sensitivity threshold is 5 nm) profilographs and by scanning electron (OXFORD-LINK (UK)) and ion (Strata-235) microscopes.

The distribution of implanted atoms in multilayer films and in the surface layer of samples was determined by the VIMS-method on the device HP-660, Strata-235 (with a resolution of 50–100 nm in depth and a sensitivity threshold of the content of elements $C \approx 0.001$ at%) and on a spectrometer SA-2000 (Firm LECO) guaranteeing a simultaneous measurement of a signal from five implanted elements with a depth resolution of 1–5 nm and a sensitivity threshold of about $(1-3) \cdot 10^{-2}$ at%.

To obtain more precise distribution $C(x)$ of implanted atoms in depth x , the sputtering rate $V(x)$ of the "film-substrate" system was calibrated for each sample. The sputtering rate was determined by the depth Δx of a crater formed as a result of sputtering of a studied layer for the time Δt . It was determined by measuring a change of the sample weight using the following formula:

$$V(x) = \frac{\Delta x}{\Delta t} = \frac{P_0 - P_t}{\Delta t S_A \rho},$$

where P_0 and P_t are the sample weight before and after the measurement, respectively; S_A is the area of an irradiated (sputtered) zone; ρ is the specific density of a substrate material.

As was shown by an analysis of the experimental results obtained, the measurement precision of a mass change (or a weight change) of the investigated sample (or the matrix) gives the main contribution to the measurement precision of the Δx -value. Therefore, the type GH-252 electronic scales of the Firm A&D (Japan) having a sensitivity threshold of 0.01 mg and a typical error no more than $\pm 10 \mu\text{g}$ were selected as a measuring tool.

3. Experimental results

Surface topography. Normalized histograms of the distribution of deviations $n(x)$ from the mean surface line of polycrystalline Cu-samples irradiated by a beam of Ar^+ ions ($\langle E \rangle = 10$ keV) in the dose range of $(0.5-2) \cdot 10^{18}$ ion/ cm^2 (with an irradiation angle $\alpha = 0^\circ$) are shown in Fig. 3 as an example. It is seen from the Fig. 3 that the surface of polished samples in their initial state has imperfections of the order of $0.2 \mu\text{m}$ (histogram 1).

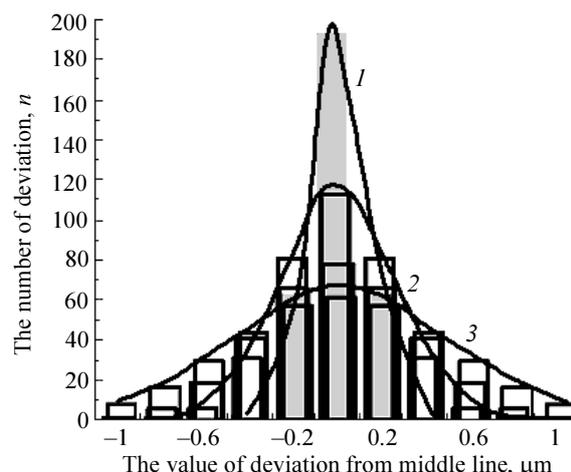


Fig. 3. A change of the surface geometry of polycrystalline Cu-samples under irradiation by an Ar^+ ion beam with a wide energy spectrum, a mean energy $\langle E \rangle = 10$ keV, and irradiation doses of: 1 – $\Phi = 0$; 2 – $0.5 \cdot 10^{18}$; 3 – $5 \cdot 10^{18}$ ion/ cm^2

The $n(x)$ -distribution widens (histograms 2–4) with the irradiation dose.

At the same time, the number of $0.2 \mu\text{m}$ imperfections (irregularities) significantly decreases ($\Phi = 0$; 0.5; 1.0; $5.0 \cdot 10^{18}$ ion/ cm^2 – 196; 117; 78; 63, respectively), but the number of $0.5-1.0 \mu\text{m}$ imperfections increases ($\Phi = 0$; 0.5; 1.0; $5.0 \cdot 10^{18}$ ion/ cm^2 – 0; 25; 33; 41, respectively).

The experimental results of the surface topography obtained by the SEM-method (Fig. 4, a) at irradiation angles less than 45° show that the surface relief practically corresponds to that obtained at a normal irradiation angle ($\alpha = 0^\circ$). However, in case of irradiation

angles more than 50–60°, there is formation of terraces on the surface of samples and both boundaries of individual crystallites and structural defects of the surface layers are revealed more intensively (Fig. 4, b).

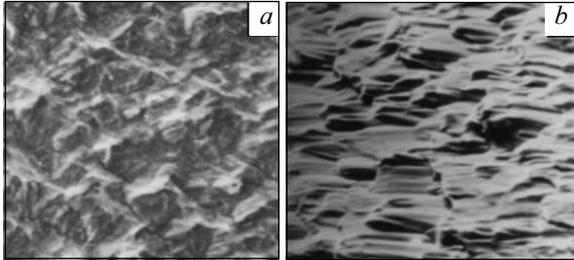


Fig. 4. A photograph of the surface of W-samples irradiated by an Ar^+ ion beam with a mean energy of ions in a beam $\langle E \rangle = 10 \text{ keV}$ up to a dose $\Phi = 1.2 \cdot 10^{18} \text{ ion/cm}^2$: a – $\alpha = 45^\circ$; b – 65°

The experimental results on changing the surface roughness R_a from the dose (for the range of $(0.1\text{--}12) \cdot 10^{19} \text{ ion/cm}^2$) of irradiation by beams of He^+ , Ar^+ and $\text{He}^+ + \text{Ar}^+$ ions ($C_{\text{He}^+} : C_{\text{Ar}^+} = 1 : 1, 1 : 2, 1 : 5, 1 : 10$) with a wide energy spectrum at a mean energy of 10 keV made it possible to find the most optimal irradiation conditions (Fig. 5).

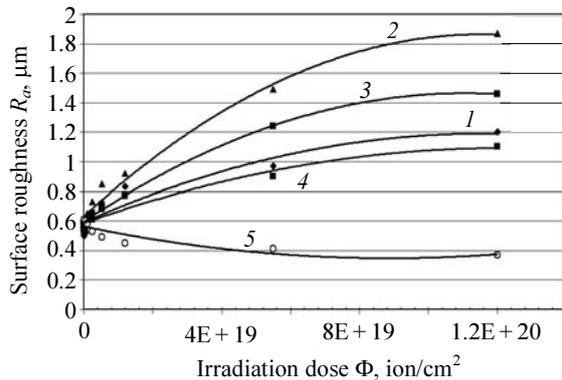


Fig. 5. A change of the surface roughness of E635-alloy samples from the dose of irradiation by ion beams: 1 – He^+ ; 2 – Ar^+ ; 3 – $\text{He}^+ + \text{Ar}^+$ ($C_{\text{He}^+} : C_{\text{Ar}^+} = 1 : 1$); 4 – $\text{He}^+ + \text{Ar}^+$ ($C_{\text{He}^+} : C_{\text{Ar}^+} = 1 : 5$); 5 – $\text{He}^+ + \text{Ar}^+$ ($C_{\text{He}^+} : C_{\text{Ar}^+} = 1 : 10$)

For example, irradiation by a two-component beam of $\text{He}^+ + \text{Ar}^+$ ions with a content of He^+ ions in a beam of the order of 10% (Fig. 5, curve 5) forms favorable conditions for uniform sputtering of the surface of zirconium and zirconium alloys using the regime of “ion polishing”.

Using this regime, it is possible to irradiate the “multilayer film–polycrystalline substrate” system at higher doses and guarantee conditions of simultaneous multicomponent alloying of the Zr-matrix by ion mixing. This regime minimizes distortions in the distribution of implanted atoms formed because of various sputtering rates of separate crystallites [4, 6].

Scanning microscopy of the surface state of samples alloyed from three- and five-layer films and irradiated up to doses higher than $1.3 \cdot 10^{19} \text{ ion/cm}^2$ has

shown that there is formation of local areas (Figs. 6 and 7) with typical sizes of 1–5 μm and a density to $(1\text{--}10) \cdot 10^3 \text{ cm}^{-2}$.

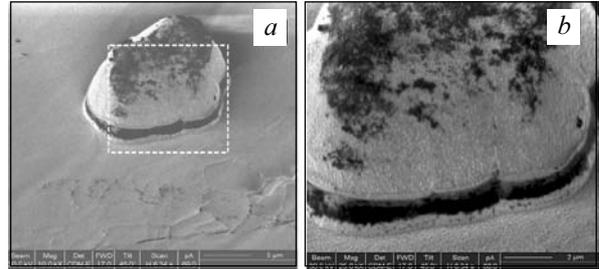


Fig. 6. Photographs of the surface of a Be-sample with a sputtered Ni-film 40 nm in thickness and irradiated by a beam of $\text{He}^+ + \text{Ar}^+$ ions at $\langle E \rangle = 10 \text{ keV}$, $\Phi = 1.3 \cdot 10^{19} \text{ ion/cm}^2$: a – a formation on the surface of a Be-sample; b – a magnified photograph of a formation located on the surface of a Be-sample

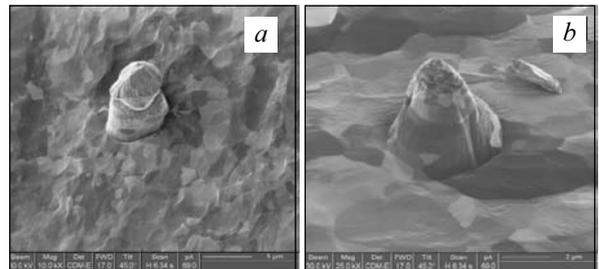


Fig. 7. Photographs of the surface of W-samples with a “cone-like” formation under irradiation by a beam of $\text{He}^+ + \text{Ar}^+$ ions at $\langle E \rangle = 10 \text{ keV}$, $T < 150^\circ\text{C}$: a – $\Phi = 2.3 \cdot 10^{18}$; b – $7.5 \cdot 10^{19} \text{ ion/cm}^2$

They predominantly consist of substrate atoms with a small content of implanted and impurity atoms. Their subsequent irradiation ($\Phi \leq 1.3 \cdot 10^{20} \text{ ion/cm}^2$) results in their elimination at the expense of sputtering.

An analysis made of the surface state of technically pure Be, Ti, Fe, Cu, Mo, Zr, and W samples (Fig. 8) after irradiation by Ar^+ ions to high doses of $1.3 \cdot 10^{20} \text{ ion/cm}^2$ did not reveal the presence of similar formations.

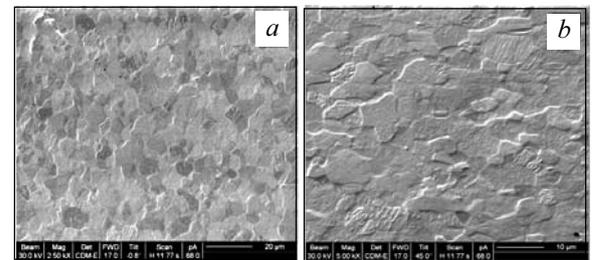


Fig. 8. Photographs of the surface of polycrystalline Zr-samples irradiated by an Ar^+ ion beam with a mean energy of 10 keV, $T < 150^\circ\text{C}$: a – $\Phi = 3.2 \cdot 10^{19} \text{ ion/cm}^2$; b – $1.3 \cdot 10^{20} \text{ ion/cm}^2$

It was found that at high irradiation doses $\Phi > 2.3 \cdot 10^{18} \text{ ion/cm}^2$, a high content of He^+ ions in a two-component beam of $\text{He}^+ + \text{Ar}^+$ ions resulted in

a significant decrease of microhardness and formation of micropores on the irradiated surface of a number of samples. In this connection, irradiation by a two-component beam ($\text{He}^+ + \text{Ar}^+$; $C_{\text{He}^+} : C_{\text{Ar}^+} = 1 : 10$) with a He^+ -content in the beam of the order of 10% was chosen.

Distribution of implanted atoms. Typical profiles of the $C(x)$ -distribution of implanted atoms from films into substrate material (Al, Ni, Sn in Cu) are shown in Fig. 9.

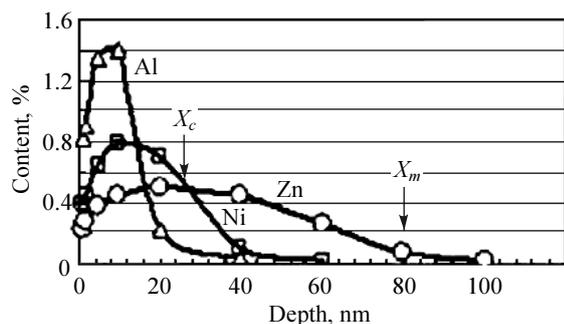


Fig. 9. The $C(x)$ -distribution of implanted Al, Ni, and Zn atoms in a Cu-substrate under irradiation by an Ar^+ ion beam with a wide energy spectrum and a mean energy $\langle E \rangle = 10$ keV, at a dose $\Phi \approx 1 \cdot 10^{18} \text{ cm}^{-2}$, and at the irradiation angle $\alpha = 0^\circ$

It is seen from the figure that the $C(x)$ -distribution has a cone-like dependence. The distribution maximum of implanted atoms X_c is located at the following depths: 11 nm for Al, 20 nm for Ni and Mo. The concentration maximum $C(X_c)$ of implanted atoms is in the 0.5–1.4 at % range. It decreases with an increase of the atomic mass of implanted atoms: Al – 1.4, Ni – 0.8, and Mo – 0.5 at %.

Of the most interest is the maximum penetration depth X_m of implanted atoms which, as shown in Fig. 9, increases with the mass of film atoms and attains, for example, 60–80 nm for Sn at the concentration of implanted atoms of 0.05–0.1 at %.

The $C(x)$ -distribution of Si and Mo atoms is a monotonous decreasing dependence. As to the distribution of Al atoms, it is dome-shaped (asymmetric). The distribution form of implanted Fe-atoms is more complicated. It has two areas of a monotonous concentration decrease; the first one is 40–160 nm and the second one is 180–280 nm. Their appearance can be due to radiation processes of action with a simultaneous mixing (the first segment) and radiation-stimulated diffusion under irradiation (the second segment).

In case of irradiation of the “three-layer film–substrate” system by a two-component beam of $\text{He}^+ + \text{Ar}^+$ ions with a dose of $2.1 \cdot 10^{18} \text{ ion/cm}^2$ at $T \leq 100\text{--}150^\circ \text{C}$, the distribution of implanted atoms slightly changes. For example, dependences of the $C(x)$ -distribution of implanted Al, Ti, and Fe atoms onto Zr are characterized by several distribution maximums: 0–40 nm for Al, 10–30 and 80–120 nm for Ti, and 40–60, 200–220, and 380–420 nm for Fe-

atoms. A deep penetration of Fe-atoms up to 500 nm is observed.

The same is typical under multicomponent penetration from a MoFeAlTiSi film into a metal (Fig. 10).

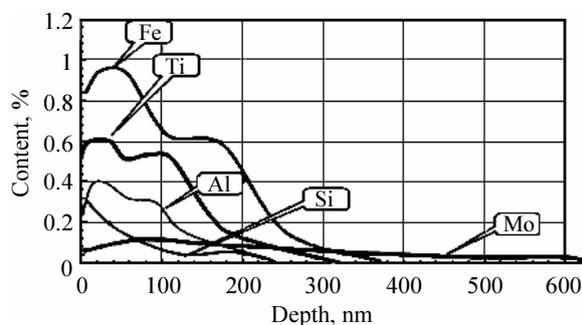


Fig. 10. Distribution of Si, Al, Ti, Fe, and Mo atoms in a polycrystalline Zr-100 sample after irradiation of multilayer MoFeAlTiSi-Me films by $\text{He}^+ + \text{Ar}^+$ ions ($C_{\text{He}^+} : C_{\text{Ar}^+} = 1 : 10$) with a dose of $5.5 \cdot 10^{18} \text{ ion/cm}^2$ at $T \leq 100\text{--}150^\circ \text{C}$

There are secondary concentration maximums on the $C(x)$ -distributions of Al, Ti, and Fe atoms. They are located deeper: 80–100 nm for Al, 100–120 nm for Ti, and 160–180 nm for Fe. At that, the penetration of Si and Mo atoms at depths of 200–220 nm for Si and 600–620 nm for Mo is observed.

4. Discussion of the experimental results

State of an irradiated surface. The experiments carried out on investigation of the dependence between a change of the surface relief R_a and the dose of irradiation by beams of He^+ , Ar^+ , and $\text{He}^+ + \text{Ar}^+$ ions ($C_{\text{He}^+} : C_{\text{Ar}^+} = 1 : 1, 1 : 2, 1 : 5, 1 : 10$) ranging from $0.1 \cdot 10^{19}$ to $12 \cdot 10^{19} \text{ ion/cm}^2$ with a wide energy spectrum (and a mean energy of 10 keV) allowed revealing the most optimal irradiation conditions. It follows from them that irradiation by a two-component beam of $\text{He}^+ + \text{Ar}^+$ ions with a content of He^+ ions in the beam of the order of 10% (Fig. 5, curve 5) creates favorable conditions for uniform sputtering of the surface of samples by “ion polishing”. Using this regime, multicomponent alloying of a matrix can minimize distortions in the distribution of implanted atoms which arise owing to various sputtering rates of separate crystallites. This is especially important under irradiation at high doses [1, 7].

It has been found that in case of long-term irradiation ($\Phi = 1.3 \cdot 10^{19} \text{ ion/cm}^2$), formation of local areas on a modified surface of polycrystalline samples is possible. The areas predominantly consist of substrate atoms with a small content of implanted atoms of multilayer films. Their predominant location close to grain boundaries makes it possible to suppose that their origination is due to redistribution of implanted atoms over the surface under irradiation and additional investigations are needed.

Sputtering and implantation of film atoms. As was shown by earlier experiments [6], to increase the implantation efficiency of film atoms, it is necessary to

decrease the sputtering rate of films ($V_{Spt} = dx_f/dt$) and try to increase the implantation (penetration) rate of their atoms into the matrix ($V_{impl} = dx_m/dt$). In the framework of a cascade theory of sputtering and isotropic mixing, it can be shown that these values are connected by the following equations [1, 8, 9]:

$$V_{Spt} = \frac{A}{\pi^2 \rho N_A s_0} \frac{\Delta x j F_q}{U_0} \quad \text{and} \quad V_{impl} \cong \sqrt{\frac{B j F_q}{4t}},$$

where A is the gram-mole; ρ is the specific weight of a film material; N_A is the Avogadro number; s_0 is the irradiated area; U_0 is the binding energy of atoms on a surface; B is the coefficient of ion mixing; F_q is the energy release in a substrate material; j is the current density of an ion beam; t is the irradiation time.

The following conclusions can be made from the aforesaid:

- decreasing the sputtering rate of films is possible at the expense of decreasing the energy release F_q and increasing the binding energy of atoms U_0 ;

- increasing the implantation rate can be realized in increasing the energy release F_q and the coefficient of ion mixing B .

Increasing the efficiency of ion mixing can, in its turn, be attained in decreasing the irradiation dose F (i.e., the thickness of a sputtered layer) and the energy release F_q in a substrate, where $F_q \approx \langle E \rangle / R_p$, $\langle E \rangle$ is the mean energy of ions in a beam, R_p is the projective track of ions in a matrix. This condition is fulfilled in experiments on irradiation by a two-component beam of $\text{He}^+ + \text{Ar}^+$ ions with a wide energy spectrum. In this case, the efficiency coefficient of ion mixing attains the value $B = (5-20) \cdot 10^{-3} \text{ nm}^5/\text{eV}$.

The optimal mutual location of film layers themselves can also be one of significant factors of increasing the value B [9]. For example, it is expedient to sputter heavier atoms onto the external layer of multilayer films and lighter atoms onto the internal layer adjacent to the surface of a matrix. In this case, favorable conditions for intensification of atom mixing are formed and the sputtering rate of a film decreases.

In case of irradiation of a five-layer film (Mo, Fe, Al, Ti, Si) by a two-component beam of $\text{He}^+ + \text{Ar}^+$ ions ($C_{\text{He}^+} : C_{\text{Ar}^+} = 1 : 10$), a deeper penetration of implanted Fe and Mo atoms takes place and there is appearance of the secondary distribution maximums of Fe, Al, and Ti atoms with a high concentration gradient which can be explained by processes of ion mixing, radiation-stimulated migration, and physicochemical interaction of implanted atoms both between themselves and with matrix atoms [10, 11]. The ratio of atomic radii of a film and a substrate and their electronegativities, which take the formation possibility of solid solutions (an implanted atom in the matrix) into consideration, i.e., the condition of Darken–Guri–Sood (DGS) is fulfilled, can be used as parameters which

promote the radiation-stimulated migration of implanted atoms in polycrystalline substrates [1, 13, 14].

5. Conclusions

Experiments have been carried out on multicomponent (up to five components) alloying of the surface layers of technically pure polycrystalline samples by ion mixing under irradiation by a beam of $\text{He}^+ + \text{Ar}^+$ ions with a wide energy spectrum and doses to $1 \cdot 10^{20} \text{ ion/cm}^2$ (at the temperature $< 150-250 \text{ }^\circ\text{C}$).

It has been found that at high irradiation doses, the energy transfer from the ions of a beam and primarily knocked out atoms to subsequent displacement cascades is the base of a deep penetration of the atoms of multilayer films into polycrystalline substrates. Multicomponent alloying makes it possible to form layers with a high concentration gradient of implanted atoms.

It has been revealed that at irradiation doses higher than $1 \cdot 10^{19} \text{ ion/cm}^2$, local fine-crystalline areas with a concentration of implanted atoms of 0.1–10 at.% are formed in the surface layer of a substrate.

The penetration efficiency of implanted atoms to high depths B is determined by:

- the energy release in a modified layer of a substrate F_q which is expedient to be decreased;

- radiation-stimulated migration of implanted atoms and their physicochemical interaction with matrix atoms which can be described in the framework of the isotropic mixing model and formation of solid solutions.

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