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Change in Dislocation Structure and Phase Composition of 1565Ч Alloy Exposed to Ion Irradiation (Cu + Pb)

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Abstract

Background/Objectives: Using electron microscopy, it was revealed that exposure to ion irradiation (Cu + Pb) with an energy of 35 keV of samples sized 25 x 25 x 3 mm of 1565ч industrial aluminum alloy after cold rolling, leads to a significant narrowing of the boundaries of intragranular cellular dislocation structure and increase in the extent of its regularity. **Methods/Statistical Analysis**: We have studied samples of 1565ч alloy sized 25 x 25 mm^2 cut from non-clad sheets of a thickness of 3 mm, a change in the structure of cold-worked 1565ч alloy exposed to the beams of accelerated ions (Cu + Pb) with an energy of 30 keV at the densities of ion current of 100, 200 and 300 µA/cm^2 and irradiation doses of 1016 and 1017 cm-2. Irradiation with pulsed beams of ions (Cu + Pb) was conducted at the setup for ion beam implantation equipped with ion source based on plasma arc in vacuo. Electron microscopic study by the method of thin foils was conducted at JEM-2100 transmission electron microscope. **Findings:** It was found that irradiation of cold-worked clad 1565ч alloy with copper and lead ions does not lead to a fundamental change in its structure compared to the structure existing in the deformed condition: cellular dislocation structure remains. With increase in ion current density and irradiation dose, the cellular structure becomes more regular – well-formed cells are observed throughout almost the entire volume of the sample. The average width of dislocation-free regions reaches 2.5 µm at the width of the boundaries not exceeding 0.6 µm. The effect of accelerated ions (Cu + Pb) (E = 30 keV, j = 100 µA/cm2) on the alloy without the cladding layer even at a dose of 1016 cm-2 leads to formation of the developed subgrain structure in initially deformed alloy. The average size of subgrains with interlacements of dislocations varies in the range of 0.6 – 2.2 µm. In other words, a polygonal structure forms in the alloy similar to the structure forming at the initial stages of recrystallization annealing. **Applications/Improvements:** This applies to many industries associated with the development of high-tech and energy-saving technologies as well as addressing environmental problems.

Keywords: Aluminum Alloy, Ion Implantation, Ions (Cu + Pb), Microstructure, Phase Composition

1. Introduction

The significance of aluminum alloys as structural materials of modern technology is constantly increasing. This applies to many industries associated with the development of high-tech and energy-saving technologies as well as addressing environmental problems[1,2].

In recent years, in addition to high requirements for static strength, corrosion resistance, fracture toughness, high stability of the properties of semi-finished products, strict requirements are set for the cost of semi-finished products from aluminum alloys and to the level of costs for their production. This is an incentive to develop new compositions of aluminum alloys and processing technologies for semi-finished products from them.

To improve the physical and mechanical properties of aluminum alloys, in addition to traditional methods, such as plastic deformation, heat and thermomechanical

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treatment, new methods of external influence are offered. Some methods allow effecting on the collapse of a supersaturated solid solution, to form a special defect structure, the state of the interphase boundaries, etc. This can be a high static pressure, quenching from the liquid state, ultrasonic deformation[3] or the use of concentrated energy flows, including plasma flows, microwave radiation, beams of charged particles (in particular ion beams)[4-6].

It was found that the use of ion beams is one of very promising areas of the modern material processing technology. The structural condition and physical properties of the substances subjected to ion-beam effect differ significantly from the corresponding condition and properties of the substances after common heat treatment or other traditional types of effects[7-9]. In some cases it is possible to obtain unique electrical, magnetic and mechanical, tribological, contact-chemical and other properties of materials[8,9].

In a number of works[10-12], to modify the properties of commercial aluminum alloys, high-energy ion implantation (E>10 MeV) was used. Interest in it is due to fundamentally new opportunities to effect on the structure and accordingly physical and mechanical properties of metals and alloys.

In particular, due to the transfer of energy to the electron subsystem (and its subsequent pumping to the atomic subsystem), there is a rapid heating of microscopic amounts of material around tracks of high-energy ions, up to $T>T_{melt}$, followed by rapid quenching, which promotes formation of defects, phase transformations and other metallurgical processes.

In[10,11], the effect of high-energy ion implantation of neon on the structure and properties of aluminum alloys was studied. It has been shown that in thermally non-hardened alloys AMr2 and AMц, radiation-induced hardening occurs due the increase in disorientation angle of the blocks until transformation of the structure to fine-grained. A changes in strength characteristics of aging alloys Д16, AK-4 are controlled by both the processes of dynamic recovery during implantation and radiation-stimulated processes of phase aging. The dose dependence of the strength properties of these alloys is non-monotonic by nature, and initial loss of strength of the implanted alloys associated with polygonization or recrystallization during the subsequent growth of the implantation dose changes to a significant strengthening as a result of emergence of fine segregations of strengthening phases.

Because of the long run of high-energy ions with energies of tens and hundreds of MeV in the matter, the thickness of the modified layer reaches up to several tens of micrometers.

The basis of so-called implant metallurgy[6] is setups allowing to generate powerful ion beams of low and medium energy (usually in the range of 10-100 keV) for implantation of high doses of ions (10^{16}-10^{18} cm^{-2}) to modify construction materials. In this regard, in the light of possible practical applications, the studies of the changes in the structure and properties of commercial aluminum alloys implanted with ions of medium and low energies are of special interest. Unfortunately, the number of such studies, especially on bulk samples of industrial alloys is extremely limited.

In implantation of Sb and As ions with energies of 20-100 keV, loss of strength of aging aluminum alloys was observed, and with a growth of implantation dose, this effect intensified[12-14].

It was found that exposure with gas (Ar^+) and metal (Al^+, Cu^+) of the alloy of Al-Cu system with ions of low and medium energy (radiation dose of $2\bullet10^{15}$ to $2\bullet10^{18}$ cm^{-2}) of supersaturated solid solution alloy Al + 4 wt. % Cu[15], causes its decomposition with separation of the particles of second phases *(θ', θ)* at low temperatures <60 °C (at which in the absence of irradiation, only zonal aging stage occurs). In this case the depth of phase separation is much greater than the penetration depth of ions in this alloy. The rate of aging increases by several orders of magnitude (compared to the common thermally stimulated aging even at higher temperatures of ~200 °C)[15,16].

In this paper, 1565ч alloy of aluminum-magnesium system (with additional doping with zinc) was selected as subject of research, belonging to the group of alloys not hardened by heat treatment, in spite of the strong temperature dependence of the limiting solubility of magnesium in aluminum[15-17].

The value of Al-Mg system alloys is characterized by the combination of satisfactory strength, high ductility, very good corrosion resistance and weld ability. These characteristics allow successfully using these alloys as a structural material in construction, transport engineering and other industries, as well as in aircraft engineering and rocket technology[2,18].

Alloy 1565ч has great prospects as the most durable among the number of thermally non-hardened aluminum alloys[2]. The disadvantage of the alloys of Al-Mg system is the relatively low yield limit. To increase its, AMr5 and 1565ч alloys and subjected to cold deformation of 20-30%[15]. Cold hardening is currently the main way to improve the mechanical properties of these alloys[19-21].

2. Materials and Methods of the Experiment

We have studied samples of 1565ч alloy sized 25x25 mm^2 cut from non-clad sheets of a thickness of 3 mm. Chemical composition of the alloy is given in Table 1.

We have studied a change in the structure of cold-worked 1565ч alloy exposed to the beams of accelerated ions (Cu + Pb) with an energy of 30 keV at the densities of ion current of 100, 200 and 300 $\mu A/cm^2$ and irradiation doses of 10^{16} and 10^{17} cm^{-2}.

Irradiation with pulsed beams of ions (Cu + Pb) was conducted at the setup for ion beam implantation equipped with ion source based on plasma arc in vacuo. The modes of treatment of the samples of 1565ч alloy with a thickness of 3 mm (with cladding aluminum layers) and 3 mm thick samples without cladding are shown in Table 2.

Target temperature was controlled during irradiation using a thermocouple. Sample temperature did not exceed 170 °C (Table 2).

The values of ion current density and irradiation dose were varied so that during implantation the samples were not significantly heated, and correspondingly the role of thermally induced processes would be minimized.

Electron microscopic study by the method of thin foils was conducted at JEM-2100 transmission electron microscope. The structure of the irradiated samples was analyzed in two sections – perpendicular and parallel to the sample surface.

Foils were made of bulk samples: 0.4 mm thick plates were cut at the electrospark machine, parallel to the surface of the sample (from the side of the irradiated surface) and perpendicular to this surface. Then they were mechanically thinned on sandpaper to a thickness of 0.1 mm. Final thinning of samples to a thickness suitable for transmission of electron beam (about 0.1 m) was conducted electrolytically at a voltage of 20-40 V in the chlorine-acetic 23% $HC1O_4$ electrolyte and 77% CH_3COOH. Electrolyte temperature did not exceed 4-5 °C.

3. Results and Discussion

In electron micrographs of the initial cold-worked 1565ч alloy, the developed cellular structure can be observed with wide boundaries between individual cells (Figure 1, *a*). These boundaries are dense interlacements of dislocations (Figure 1, *b*). The width of boundaries is comparable to the dislocation-free central regions. It can be seen that the cells have equiaxed or elongated shape. The diameter of the equiaxed cells is 1.5-2 μm.

The alloy has a large number of rounded or ellipsoidal intermetallic compounds with an average diameter of ~0.6-1.3 μm (Figure 1, *c*). On the basis of the calculation

Table 1. Chemical composition of 1565ч alloy sheets

Mass fraction of elements, %								
Al	Mg	Mn	Zn	Cu	Zr	Cr	Fe	Si
Basic	5,80	0,63	0,68	0,08	0,10	0,090	0,20	0,10

Table 2. Parameters of irradiation of 1565ч alloy samples at ion energy of 30 keV

Condition of samples	Density of ion current, *j*, $\mu A/cm^2$	Dose, D, ion/cm^2	Irradiation time, *t*, sec,	Sample temperature during irradiation, *T*, °C
With cladding	100	10^{16}	40	Less than 100
	200	10^{17}	250	Less than 140
	300	10^{17}	300	Less than 170
Without cladding	100	10^{16}	40	Less than 100
	200	10^{17}	250	Less than 140
	300	10^{17}	300	Less than 170

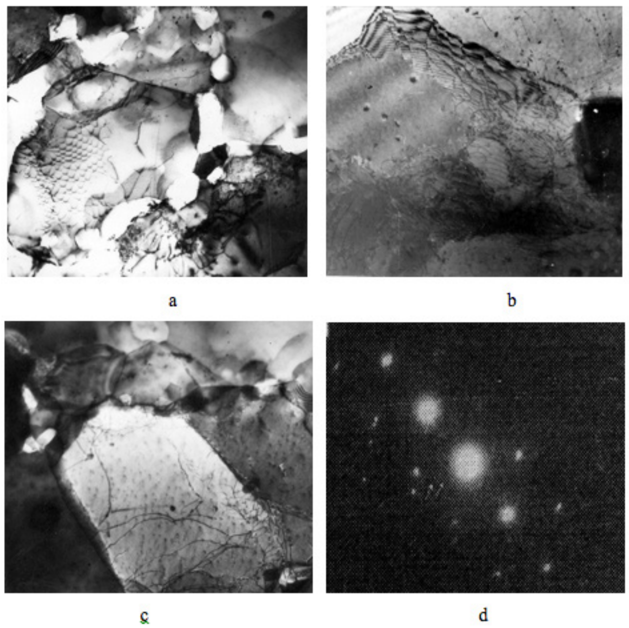


Figure 1. Microstructure of the original deformed 1565ч alloy: a) image of the cellular structure; x15,000; b) dislocation structure of the cell boundaries; x75,000; c) bright-field image of intermetallic compounds (Fe, Mn) Al_6; x30,000; d) electron diffraction pattern (reflexes of intermetallic compounds ((Fe, Mn)Al_6 are shown)).

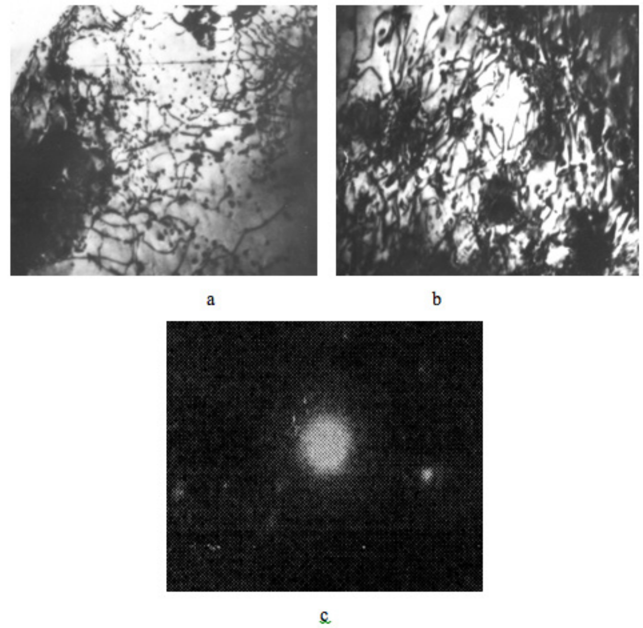


Figure 2. Microstructure of annealed 1565ч alloy: a) image of triple junctions of recrystallized grains; x15,000; b) dark-field image of intermetallic compounds (Fe, Mn) Al_6; x75,000; c) electron diffraction pattern, reflexes of intermetallic compounds Fe, Mn)Al_6 are shown.

of the corresponding electron diffraction patterns, one of which is shown in Figure 1, *d*, it was found that intermetallic compounds have the composition of (Fe, Mn) Al_6.

Annealing of cold-worked 1565ч alloy at temperatures of 330-350 °C for 1 hour leads to formation of a uniform recrystallized structure. Equilibrium triple junctions of the recrystallized grains are shown in Figure 2, *a*. Grain diameter exceeds 12-15 microns. High density of dislocations is observed inside the grains.

In Figure 2, *a*, it is clear that a large number of intermetallic compounds remains in the annealed alloy, which have been observed in the initial state after cold rolling deformation. According to the calculation of electron diffraction patterns (Figure 2, *c*), they have the same composition: (Fe, Mn) Al_6. Dark-field image of intermetallic compounds in the phase reflex is shown in Figure 2, *b*.

The study of the sample section parallel to the irradiated surface irradiated with a dose of 10^{16} cm^{-2} with ion current density of 300 μA/cm^2, showed that after irradiation, a well developed cellular structure is observed in the alloy, in general, similar to that which is typical for deformed state. However, a careful comparison of the structures of irradiated and deformed samples allows concluding that as a result of irradiation, a clear transformation of the cellular structure occurred in the alloy. This manifests itself mainly in that the fact that it became more regular (Figure 3, *a*) and, in addition, cell boundaries narrowed (their average width is about 0.4 microns), a diameter of the central regions of cells free from dislocation that varies in the range of 1.8-3.4 μm significantly increased.

It was also found that in the irradiated alloy, the number of intermetallic compounds of crystallization origin that have been seen in it after deformation and annealing, decreased. Diameter of intermetallic compounds is predominantly 0.1-0.3 μm (Figure 3, *b*). A small number of intermetallic compounds of up to 0.5 microns remained only in certain parts of the sample. Based on the analysis of electron diffraction patterns, one of which is shown in Figure 3, *c*, we have found that intermetallic compounds have the same composition (Fe, Mn) Al_6.

After irradiation with a higher dose (of 10^{17} cm^{-2}) at ion current density of 200 μA/cm^2, non-uniform cell structure is observed in the alloy (Figure 4, *a, b*). The

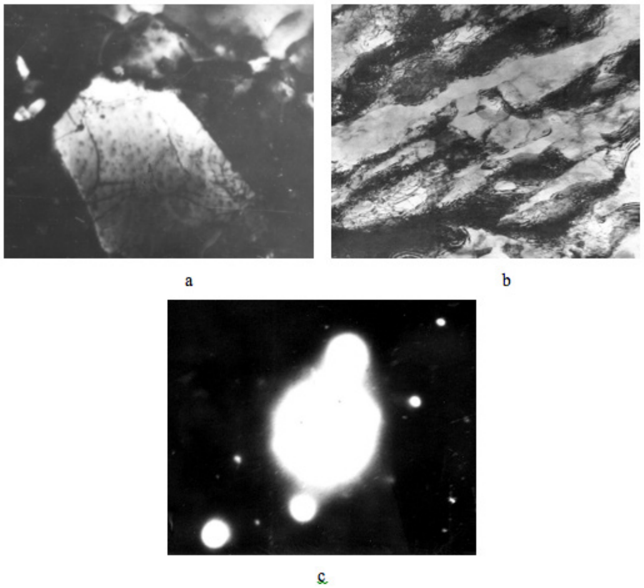


Figure 3. Microstructure of 1565ч alloy irradiated with ions (Cu + Pb) with E = 30 keV, j = 300 μA/cm^2, D = 10^{16} cm^{-2}; a) bright-field image of the cellular structure; x15,000; b) Image of intermetallic compounds (Fe, Mn)Al_6; x30,000, c) electron diffraction pattern, reflexes of (112), (021) (Fe, Mn)Al_6 are shown.

Figures show that the cells have both equiaxed and elongated shape. The diameter of dislocation-free central regions of the cells is 1-2 μm. the width of their boundaries ranges from 0.3 to 1.3 μm and more.

The presence of intermetallic compounds (Fe, Mn) Al_6 is registered in the irradiated samples but, as shown by a quantitative analysis, they become less in number than in deformed and annealed condition. The average diameter of intermetallic compounds is 0.2-0.5 μm.

Equiaxed cellular structure (Figure 4, *c*) is found at the cross section in the areas adjacent to the exposed surface. The average diameter of dislocation-free central regions of the cells is up to 2.5 μm. The width of the cell boundaries being very dense interlacements of dislocations is 0.4-0.6 μm. With the distance from the irradiated surface, slip bands begin to be detected (Figure 5, *d*). In the central part of the sample, their width reaches 0.8 μm (Figure 5, *e*). When approaching the unirradiated surface, the bands narrow to 0.4 μm. The figures show that the interlacements of dislocations are present inside the bands that break them into separate blocks.

After irradiation of the cold-worked alloy AMr6 with irradiation dose of 10^{17} cm^{-2}, but with a higher density of

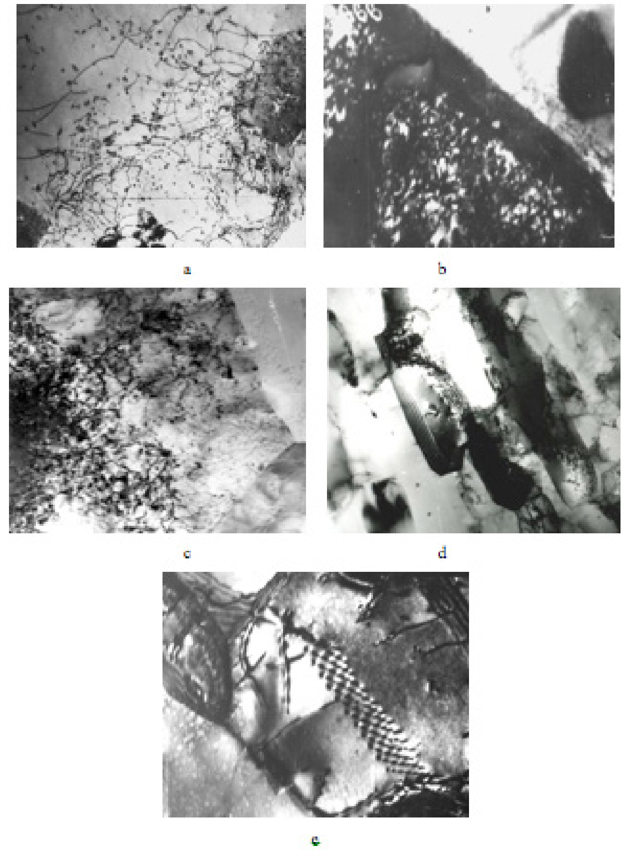


Figure 4. Image of the cellular structure in the sample of 1565ч alloy irradiated with ions (Cu + Pb) with E = 30 keV, j = 200 μA/cm^2, D = 10^{17} cm^{-2}; a, b) a section parallel to the irradiated surface; x30,000; c, d) cross-section; x15,000: e) near the irradiated surface; f) in the central part of the sample; g) near unirradiated surface.

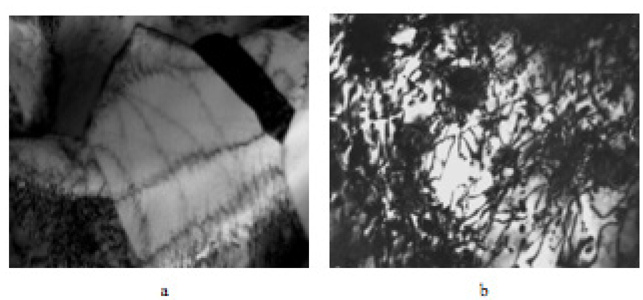


Figure 5. Image of the cellular structure of the sample of 1565ч alloy irradiated with ions (Cu + Pb) with E = 30 keV, j = 300 μA/cm^2, D = 10^{17} cm^{-2}; a) a section parallel to the irradiated surface; x30,000; b) cross-section; x30,000.

ion current (300 μA/cm^2) it preserves a cellular structure (Figure 5, *a*), but it becomes more uniform. Well-formed cells are observed almost throughout the entire volume of the sample. The average width of dislocation-free regions is up to 3 μm and a width of the boundaries of not more than 0.4 μm. The main difference of the observed structure from the alloy structure after irradiation with a lower ion current density (200 μA/cm^2) is reduction of the amount of intermetallic compounds. In some regions, they have not been detected at all.

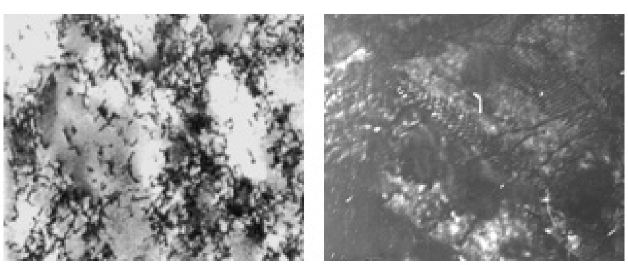
In the cross section of the sample, a cellular structure with elongated cells is observed (Figure 5, *b*). Slip bands are not clearly identified. This structure is preserved throughout the depth of the sample.

Thus, it was found that irradiation of 1565ч alloy using different modes does not lead to a fundamental change in its dislocation structure compared to a structure specific for a deformed state. Only its remarkable transformation is found after irradiation dose of 10^{17} ion/cm^2 with ion current density of 300 μmA/cm^2.

To evaluate the exposure effect from the presence of cladding layer, samples with a thickness of 3 mm were irradiated after removal of this layer by the electric spark method. Irradiation parameters were chosen so that the temperature of the sample during irradiation did not exceed the temperature registered in previous experiments.

Irradiation of the cold-worked sample after removal of cladding layer from it with a dose of 10^{16} cm^{-2} at ion current density of 100 μA ion/cm^2 caused formation of the developed subgrain structure in it. The average size of subgrains was 0.7-2.5 μm (Figure 6, *a*, *b*). The interlacements of dislocations were found inside subgrains.

Thus, under the effect of accelerated ions on the cold-worked 1565ч alloy without cladding layer, a polygonal structure formed in it similar to that formed at the early



a b

Figure 6. Image of subgrain structure of the sample of 1565ч alloy irradiated with ions (Cu + Pb) without the cladding layer, E = 30 keV, j = 100 μA/cm^2, D = 10^{16} cm^{-2}; x30,000.

stages of recrystallization annealing. Another difference of structural states of 1565ч alloy formed after annealing and after irradiation with accelerated ions (Cu + Pb) (with removed cladding layer), is the absence of coarse intermetallic compounds (Fe, Mn) Al_6 of crystallization origin in the irradiated alloy.

These data indicate that the cladding layer significantly reduces the effect of irradiation on the structure of 1565ч alloy but does not inhibit it completely.

It was established experimentally that in implantation of ions (Cu + Pb) even at low irradiation doses, starting from 10^{16} cm^{-2} intermetallic compounds (Mn, Fe)Al_6 of crystallization origin dissolve at a depth of tens of thousands of times greater than the projected runs of ions (calculated value of the projected runs of ions (Cu + Pb) with the energy of 30 keV in 1565ч alloy does not exceed 30 nm).

It is known that the most powerful effect on the properties of thermally non-hardened intermetallic alloys is due to intermetallic compounds of crystallization origin that form the atoms of elements introduced to modify the cast structure and increase the recrystallization temperature[2]. These include ferriferous intermetallic compounds with impurities of additional elements, which are practically insoluble in aluminum. Such phases especially (Mn, Fe)Al_6 crystallize as coarse plates and adversely affect the properties of alloys, in particular, reduce their plasticity. The reduction of the density of such intermetallic compounds after homogenization contributes to increase in plasticity index of 1565ч alloy approximately 1.8-2.2 times.

Using the method of electron microscopy, it was found that after standard mode of homogenizing annealing at 500 °C for 12 hours, in 5083 alloy (an alloy of Al-Mg system with 4,6% Mg) various intermetallic compounds of crystallization origin are present, including (Mn, Fe)Al_6 in the form of flat needles[22]. An increase in the annealing temperature from 500 to 535 °C promotes dissolution of such phases as $Al_{18}Cr_2Mg_3$ and Mg_2Si, while only the change in the original morphology is characteristic for intermetallic compounds (Mn, Fe)$A1_6$: some particles obtain predominantly equiaxed shape or a shape close to it. In this case, the total density of intermetallic compounds remains unchanged.

Dissolution of coarse intermetallic compounds $Al_{18}Cr_2Mg_3$ and Mg_2Si also occurs during hot deformation. At the same time, deformation does not lead to dissolution of particles (Mn, Fe)Al_6, but merely contributes to some their slight breaking-down.

The results of this study evidence the fact that dissolution of intermetallic compounds (Mn, Fe)Al_6 occurs with irradiation of ions (Cu + Pb) (30 keV) for 150-300 seconds, and in this case the temperature of the samples does not exceed 170 °C.

It should be noted that transformation of the cellular structure manifesting in narrowing of the cellular boundaries and increase in the diameter of their central dislocation-free regions and breaking-down and dissolution of intermetallic compounds (Mn, Fe)Al_6 in the studied alloy as a result of irradiation with accelerated ions, occurs not only in the surface layer, but also at a depth of 2-3 mm, by several orders of magnitude greater than projected runs of ions. Attempts to theoretically explain the anomalously large depth of the effect of ion beams of low and medium energy on the structure and properties of metastable mediums, i.e., mediums with high stored energy, are made[22-25].

The dissolution of intermetallic compounds was previously observed only in irradiation with high energy ions. Thus, it was found that irradiation with high-energy ions of krypton of D16 alloy in aged condition results in partial dissolution of metastable segregations that increases their dispersity, and further aging of the alloy[12]. The effect of dispergation of strengthening phases was also observed when irradiating by xenon ions with energy of 10 MeV of $CuA1_{25}$ alloy containing 0.25 wt. % of fine grained inclusions of $A1_2O_3$ [23-27].

4. Conclusion

It was found that irradiation of cold-worked clad 1565ч alloy with copper and lead ions does not lead to a fundamental change in its structure compared to the structure existing in the deformed condition: cellular dislocation structure remains. There is only a partial structure transformation manifesting in narrowing of the cell boundaries and increase in the diameter of their central dislocation-free regions. With increase in ion current density and irradiation dose, the cellular structure becomes more regular – well-formed cells are observed throughout almost the entire volume of the sample. The average width of dislocation-free regions reaches 2.5 µm at the width of the boundaries not exceeding 0.6 µm.

The effect of accelerated ions (Cu + Pb) (E = 30 keV, j = 100 µA/cm^2) on the alloy without the cladding layer even at a dose of 10^{16} cm^{-2} leads to formation of the developed subgrain structure in initially deformed alloy. The average size of subgrains with interlacements of dislocations varies in the range of 0.6-2.2 µm. In other words, a polygonal structure forms in the alloy similar to the structure forming at the initial stages of recrystallization annealing.

These results show that the cladding layer reduces the effect of irradiation on the structure of 1565ч alloy, but does not inhibit it completely.

It is established that in case of irradiation of 1565ч alloy with ions (Cu + Pb) both with the cladding layer and without it, there is breaking-down and dissolution of coarse intermetallic compounds of crystallization origin, which are observed in the initial deformed state and are not eliminated by common annealing.

Analysis of the cross section of the samples showed that transformation of the cellular structure, and breaking-down and dissolution of intermetallic compounds (Mn, Fe)Al_6 under irradiation occurs not only in a thin near-surface layer of 1565ч alloy, i.e. in the area of ion implantation and its immediate surroundings, but also at a depth substantially greater than projective runs of ions. If under irradiation dose of 10^{16} cm^{-2} with ion current density of 200 µA/cm^2, the structure changes non-uniformly along the depth of the sample, then by increasing ion current density to 300 µA/cm^2, well-formed cells are observed throughout the entire depth of the sample.

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