Effect of Ar^+ Ions Implantation on Phase Composition, Microstructure and Strength Characteristics of Al-Mg Alloy

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Abstract – Investigation was carried out into the effect of Ar^+ ions irradiation (E=20, 40 keV, j=100–400 μA/cm^2, D=1·10^{15}–1.7·10^{17} cm^{-2}) on mechanical properties and structural-and-phase state of AMg6 strain-hardened aluminum alloy. As a result of uniaxial tension tests, it was established that irradiation with ions to doses of 1·10^{17} cm^{-2} and higher leads to increase of plasticity and loss of strength characteristics of the alloy. Transmission electron microscopy data show that the effect is due to transformation of the cellular structure, formation of a subgranular structure with low-angle boundaries, as well as refinement and dissolution of intermetallic compounds Al_6(Mn, Fe).

The registered structural transformations take place under irradiation not only in the thin subsurface layers of AMg6 alloy with a thickness comparable with the Ar^+ ions projected range (~40 nm), but also over the entire depth of 3-mm thick samples.

1. Introduction

Investigation of the effect of ion-beam treatment on structure, phase composition and properties of commercial aluminum alloys is of great interest from the scientific and practical points of view. A number of research works was carried out along this line, being dedicated mainly to the effect of high-energy (from tens to several hundred MeV) ions. Unfortunately, there exist serious difficulties preventing high-energy ion implantation from being used to modify the properties of construction materials, despite that this type of implantation finds application in microelectronics, where no treatment of large-area surfaces or high ion current densities and irradiation doses are required.

In view of the above, investigations of changes in the structure and properties of industrial aluminum alloys at medium- and low-energy ions implantation arouse special interest.

The object under study in the present work was aluminum alloy AMg6 in strain-hardened, annealed and irradiated states in the form of clad sheet 3 mm thick manufactured at the Kamensk-Uralsky Metallurgical Plant.

The AMg6 alloy of Al-Mg system belongs to the group of alloys which take no heat hardening. The combination of satisfactory strength, high plasticity, very good corrosion resistance and weldability determine value of Al-Mg system alloys.

The alloy chemical composition is given in the Table I.

Table I. Alloy AMg6 Chemical Composition (Al-based)

Mg	Mn	Si	Fe	Cu	Zn	Ti	Li
6.4	0.69	0.1	0.1	< 0.1	< 0.1	0.04	0.0008

2. Principal part

Standard samples for tension tests were cut out from AMg6 alloy sheets. Samples were irradiated with continuous Ar^+ ion beams in the PULSAR ion-beam implanter outfitted with a cold hollow-cathode glow-discharge ion source [1] designed at the Institute of Electrophysics of the Ural Branch of RAS. Irradiation was carried out with varying parameters: ions energy E=20 and 40 keV, ion current density j=100–400 μA/cm^2, irradiation dose D=1·10^{15}–1.7·10^{17} cm^{-2}. The conditions of irradiation of breaking-test samples were so selected as to provide opportunities for most complete investigation of the effect of accelerated Ar^+ ions on the structure and properties of aluminum alloys, including the kinetics of the process. It was interesting to see which of the parameters (from the number of E, j, D and T) are critical in initiating the processes taking place under irradiation.

In the course of irradiation, the target temperature was kept under permanent control with the help of a chromel-alumel thermocouple. The maximum temperature of samples heating did not exceed 320 °C, which is the temperature of intermediate annealing conducted in the course of rolling of the given alloy to remove strain-hardening. The duration of such annealing is between 30 minutes to 2 hours.

Samples were subjected to static uniaxial tension tests in the initially deformed, annealed and irradiated states at room temperature using a standard procedure as per GOST 1497-84. The measurement error was 1 %.

The electron microscopic study was carried out by the thin foils method with the use of the JEM-200 CX transmission electron microscope. The irradiated samples structure was analyzed in two sections: parallel and perpendicular to the sample surface.

It was established by the mechanical tests data that irradiation of hardened alloy samples with Ar+ ions in certain conditions results in substantial changes of mechanical properties.

At ion current density j=150 μA/cm^2, energy E=20 keV and dose 10^{17} cm^{-2}, plasticity grows, accompanied with significant drop of plasticity characteristics (Fig. 1, *a*). The samples maximum temperature at D=10^{17} cm^{-2} was 220 °C. The irradiation time was 107 s.

With high values of ion current density (200 and 400 μA/cm^2), the maximum irradiation doses were D=1.7·10^{17} cm^{-2} and D=6.8·10^{16} cm^{-2} respectively, under which the samples were noticeably heated, but never in excess of the temperature of intermediate annealing (320 °C for the given alloy). It was established that, upon reaching of the above doses, even more significant increase of unit elongation and drop of strength characteristics are observed (Fig. 1, *b*) as compared with the previous sample. The higher the irradiation dose, the higher the loss of the alloy strength. Mechanical properties obtained at j=400 μA/cm^2, D=1.7·10^{17} cm^{-2}: $\sigma_в$=334 MPa, $\sigma_{0.2}$=172 MPa, δ=28 %, are close to those obtained after intermediate annealing ($\sigma_в$=328 MPa, $\sigma_{0.2}$=178 MPa, δ=28 %). The treatment time was 140 s, which is 13 times less than the time required for strain-hardening removal annealing.

Thus, under irradiation to doses of 10^{15}, 10^{16} cm^{-2}, no drop of strength characteristics occurs at all values of j, while increase of the irradiation dose to 10^{17} cm^{-2} results in a drop of strength and growth of plasticity characteristics. At D=1.7·10^{17} cm^{-2}, the loss of alloy strength is close to that reached at intermediate annealing.

In the initial strain-hardened state, a developed cellular structure is observed in AMg6 alloy, with broad boundaries between individual cells (Fig. 2, *a*). These boundaries present dense dislocation tangles. The width of boundaries is commensurate with the central regions free from dislocations. It may be seen that the cells are of equiaxial or elongated shape. The equiaxial cells diameter is 1–2 μm.

The alloy contains a great number of coarse intermetallic compounds $Al_6(Fe,Mn)$ of crystallization origin of rounded or ellipsoid shape with a mean diameter of ~ 0.5–1 μm (Fig. 2, *b*).

Annealing of strain-hardened AMg6 alloy at temperatures of 310–325 °C during 2 h leads to formation of a uniform recrystallized structure with grain size over 10 μm (Fig. 2, *b*). Inside the grains, high density of dislocations is observed.

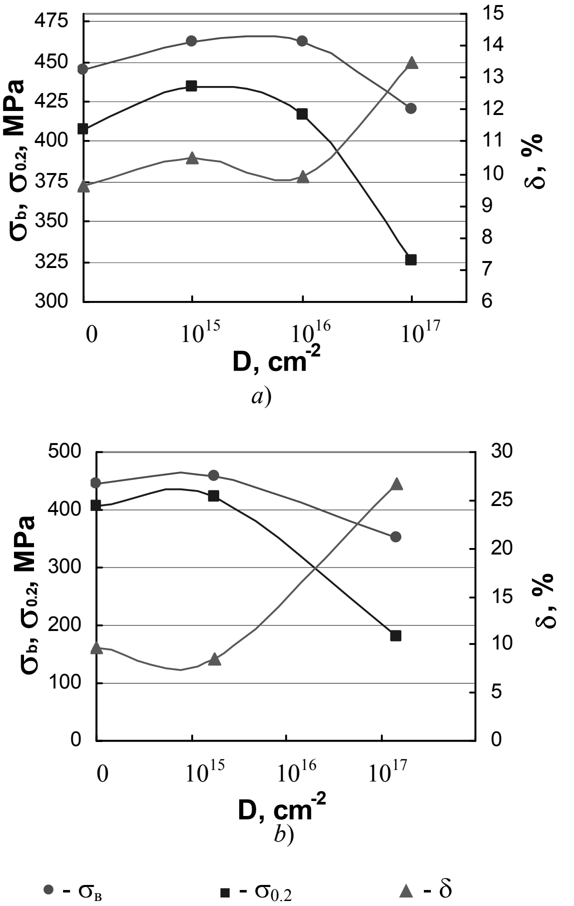

Fig. 1. Relationship between dose, ultimate stress $\sigma_в$, yield strength $\sigma_{0.2}$ and unit elongation for AMg6 alloy under irradiation with Ar$^+$ ions: a – E=20 keV, j=150 μA/cm^2; b – E=40 keV, j=200 μA/cm^2

It may be seen that a great number of intermetallic compounds Al_6(Fe, Mn), which were observed in the initial state after cold deformation, is preserved in the annealed alloy.

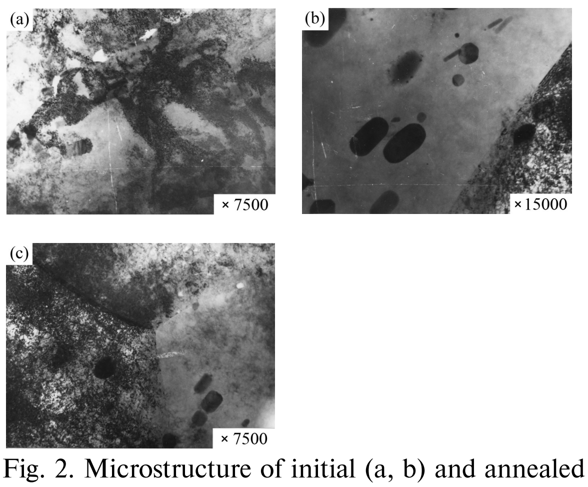

Fig. 2. Microstructure of initial (a, b) and annealed (c) AMg6 alloy

Study of the cross-section (parallel to the irradiated surface) of samples irradiated with 20-keV Ar^+ ions at relatively low ion current density j=150 μA/cm^2 to dose D=10^{15} cm^{-2} has shown that irradiation in the described regime does not exert any significant effect on the structure of the deformed AMg6 alloy. After irradiation it retains the cellular structure illustrated in Fig. 3, *a*. The structure has nevertheless undergone some transformation compared with the one observed in the initial deformed state. This is manifested mainly by decrease of the cell boundaries width, the cells presenting, as before, close dislocation tangles up to 0.1–0.5 μm in size.

The majority of cells have elongated shape. The length of cells central regions free from dislocations is, on the average, 1–2 μm, at 0.1–0.5 μm width.

It was also established that the intermetallic compounds of crystallization origin found in the alloy after its deformation and annealing are practically not observed the irradiated alloy.

At alloy AMg6 irradiation to a higher dose D=10^{17} cm^{-2}, with the same as above energy and density of ion current, the alloy cellular structure is also preserved. At the same time, with the irradiation dose increase some transformation is stimulated: the number of cells in which the diameter of dislocations-free central regions exceeds 2 μm at boundaries width less than 0.5 μm has grown (Fig. 3, *b*). The number of cells in which the boundaries width is commensurate with the width of their central region has increased respectively. At certain sections of the sample there are no clearly formed cells, the broad dislocations-free regions are separated by "fragmented" boundaries (Fig. 3, *b*). It has been also established that irradiation dose increase facilitates dislocations redistribution, with consequent formation of "looser" boundaries. Within some boundaries such redistribution is accompanied with formation of dislocation walls.

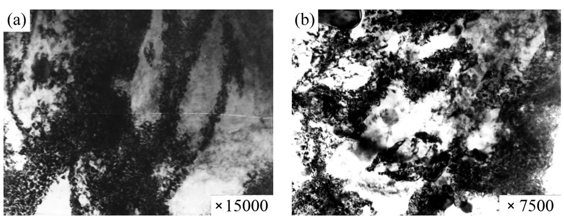

Fig. 3. Image of cellular structure of AMg6 alloy irradiated with Ar^+ ions at E=20 keV, j=150 μA/cm^2: a – D=10^{15} cm^{-2}; b – D=10^{17} cm^{-2}

In some sections of the sample with broad cell boundaries intermetallic compounds Al_6(Fe, Mn) of crystallization origin were detected. The increase of irradiation dose has no influence on the structure of intermetallic compounds, but causes further decrease in their amount.

Measurements of mechanical properties of the irradiated alloy have shown that irradiation with an increased dose enhances its plasticity and loss of strength. This may be connected with the increase of the size of cells, decrease of their boundaries width and redistribution of dislocations within their boundaries. At that, the boundaries turn out to be less efficient barriers for the moving dislocations.

Irradiation of AMg6 alloy with Ar^+ ions with 40 keV energy at higher ion current density j=200 μA/cm^2 and to maximum feasible (due to temperature limitations) irradiation dose D=1,7·10^{17} cm^{-2} promotes abrupt change of the alloy structural state: a subgrain structure has formed in it in the process of irradiation (Fig. 4).

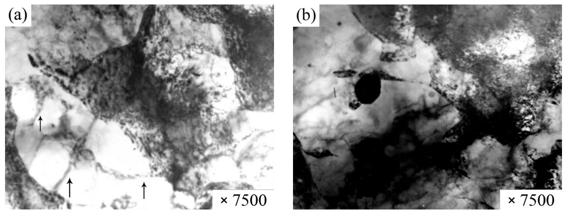

Fig. 4. Image of subgrain structure of AMg6 alloy irradiated with Ar^+ ions: E=40 keV, j=200 μA/cm^2, D=1.7·10^{17} cm^{-2}

It may be seen in the presented Figures that the formed structure is quite irregular. So, the subgrains mean diameter varies from 1 to 6 μm. Larger subgrains are mainly free from dislocations. Smaller subgrains (less than 2 μm in diameter) have preserved dislocation tangles. In some large subgrains dislocations redistribution has taken place. It is accompanied with formation of dislocation-free microregions not over 1 μm in diameter separated with narrow poorly shaped boundaries (marked with an arrow in Fig. 4, *a*).

The subgrain boundaries, like their sizes, are irregular. In some boundaries striped contrast is observed, which is inherent to tilt boundaries and testifying to the fact that disorientation between adjacent subgrains is at least 10°. At the same time, the majority of subgrain boundaries present dislocation networks or walls, with different degree of density of the latter. In this case, the disorientation angle varied from 1 to 10°.

The alloy contains a very insignificant amount of intermetallic compounds (Fe, Mn)Al_6 of equiaxial shape 0.5–1.5 μm in diameter. Thus, increase of the irradiation dose has resulted in further decrease of the amount of intermetallic compounds.

The results of mechanical tests of samples of irradiated AMg6 alloy showed that increase of irradiation dose to 1.7·10^{17} cm^{-2} promotes abrupt increase of plasticity and drop of strength characteristics of the alloy, the yield strength in particular. It was established, on the basis of comparison of these data with the electron microscopy study results, that change of the mechanical properties is caused mainly by formation of a subgrain structure with low-angle boundaries.

It is known from the results of earlier studies, including those described in [2] that metal alloys yield strength depends on the angle of grain boundaries disorientation and grows with its increase. This is due to the fact that efficiency of boundaries as barriers for the moving dislocations grows with the increase of the angle of their disorientation. As a result, the low-angle boundaries, similar to those existing in irradiated AMg6 alloy, present a barrier for dislocations, which move easily from one subgrain to another.

Electron-microscopy investigation of samples cross-section has shown that transformation of cellular structure, as well as refinement and dissolution of intermetallic compounds Al_6(Mn, Fe) in alloy under the action of Ar^+ ions takes place not in the surface layer only, but in the whole volume of the 3-mm thick sample, which exceeds the ions projected range 10^5 times.

3. Conclusion

Thus it was established that at irradiation of strain-hardened AMg6 alloy with Ar^+ ions (E=20–40 keV, j=100–400 μA/cm^2) to a dose of $1{\cdot}10^{17}$ cm^{-2}, increase of plasticity occurs at noticeable drop of strength characteristics. This is connected with increase of the size of cells, decrease of their boundaries width and dislocations redistribution at boundaries.

At further increase of the irradiation dose to $1.7{\cdot}10^{17}$ cm^{-2}, the processes of polygonization take place with formation of a subgrain structure with low-angle boundaries, which promotes abrupt increase of plasticity and drop of the alloy strength characteristics, the yield strength in particular.

Besides, under ion irradiation, there takes place refinement and dissolution of coarse intermetallic compounds Al_6(Fe,Mn) of crystallization origin, which are observed in the initially deformed state and cannot be removed by usual annealing. According to [3], decrease of density of such intermetallic compounds also promotes the growth of plasticity of AMg6 alloy.

Analysis of samples cross-section has shown that cellular structure transformation, as well as intermetallic compounds Al_6(Mn, Fe) refinement and dissolution under irradiation occur not in the thin subsurface layer of the AMg6 alloy only, but also over the whole 3-mm thick sample, which exceeds the projected ions range 105 times.

Internal stress removal by the beam takes place within ~100 s, which is 20 times faster than in the process of annealing carried out for stresses removal.

References

[1] N.V. Gavrilov, S.P. Nikulin, G.V. Radkovsky, Pribory i tekhnika eksperimenta 1, 93 (1996).

[2] Honeycomb R., *Plasticheskaya deformatsiya metallov*. Moscow, Mir, 1972, p. 407.

[3] L.I. Kaigorodova, V.M. Zamyatin, V.I. Popov, FMM, 98, No. 4, 75 (2004).