Structure and Mechanical Properties of Cold-Deformed Alloy of System Al-Cu-Mg-Mn Irradiated with Ar⁺ Ions

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Abstract – Investigation was carried out into the effect of Ar⁺ irradiation (E=20)keV, *i*=150 ions $\mu A/cm^{2}$, $D=1\cdot10^{15}-7.5\cdot10^{17}$ cm⁻²) on mechanical properties and structural-and-phase state of VD1 strain-hardened aluminum alloy. It was established using the method of transmission electron microscopy that under irradiation with low doses $D=1\cdot10^{15}$ and $1\cdot10^{16}$ cm⁻² (irradiation time ~ 1 and 10 s), there takes place formation of a developed subgrain structure with equiaxial subgrains and disorientation angle not lower than 10°. This leads to insignificant drop of strength characteristics and gradual growth of unit elongation.

At irradiation dose increase to $1 \cdot 10^{17}$ cm⁻² and higher, homogeneous coarse crystal-grain structure is formed, with grain size over 10 μ m, similar to the structure of the same alloy in recrystallized state. Besides, high density of eqiaxial precipitates 10 to 150 nm in diameter of phases $\theta'(\theta') - \text{CuAl}_2$, $\text{Al}_6(\text{Fe},\text{Mn})$, $\text{Al}_8\text{Fe}_2\text{Si}$ is observed. The abrupt increase of plasticity and drop in value of strength characteristics observed in this state (similar to those taking place at annealing), despite the high density of rather uniformly distributed precipitate particles, may be explained by intensive grains growth.

The registered structural changes take place at a high rate within 1-100 s over the entire depth of 3-mm thick samples.

1. Introduction

The role of aluminum alloys as construction materials for modern engineering constantly grows. This stimulates creation of principally new aluminum alloys and processes of their treatment.

One of the most promising trends in modern processes of modification of properties of materials is the application of ion beams. For this reason, the task of establishing the laws governing change of structural-and-phase states and mechanical properties of aluminum-based alloys is a quite topical and important problem of today.

The object under study in the present work was aluminum alloy VD1 of the system Al-Cu-Mg with additions of Mn (duralumin) in strain-hardened, annealed and irradiated states.

The given alloy is an alloy taking deformation and hardening by thermal treatment. The principal dopes

of precipitation hardening alloys of this system are copper and magnesium which most efficiently participate in the processes of ageing, forming secondary θ - and σ -phases (CuAl₂ and CuMgAl₂, respectively) [1]. All duralumins are marked for high strength combined with high plasticity. This has determined their broad application as construction materials in different spheres of modern engineering, including aircraft building.

The alloy chemical composition is given in the Table I.

Table I. Alloy VD1 Chemical Composition (Al-based	1)
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Si	Fe	Cu	Mn	Mg	Ti	Zn
0.7-1.2	0.7	1.8-2.6	0.4-0.8	0.4-0.8	0.1	0.3

2. Principal part

Breaking-test samples of VD1 alloy cut out from clad sheet (3 mm thick) manufactured at the Kamensk-Uralsky Metallurgical Plant were subjected to ion-beam treatment.

Samples were irradiated with continuous Ar⁺ ion beams in the PULSAR ion-beam implanter outfitted with a cold hollow-cathode glow-discharge ion source [2] designed at the Institute of Electrophysics of the Ural Branch of RAS. Ion current density was 150 μ A/cm², with ions energy *E*=20 keV; at that, the irradiation dose varied: *D*=1.10¹⁵-7.5.10¹⁷ cm⁻².

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 under low doses did not exceed 40–60 °C, and under high doses, 250 °C, or the temperature of intermediate annealing conducted in the course of rolling of the given alloy to remove strain-hardening.

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.

Figure 1 illustrates the dependence of mechanical properties of VD1 alloy on irradiation dose. It may be seen that, at low irradiation doses of 10^{15} and 10^{16} cm⁻², ultimate stress σ_{e} and yield strength $\sigma_{0.2}$ decrease insignificantly, while unit elongation grows up to 8 % (6 % in the initial strain-hardened state).

At irradiation dose increase to 10^{17} cm⁻², σ_e and $\sigma_{0.2}$ continue decreasing, while grows to 19 %. At $D=7.5\cdot10^{17}$ cm⁻² (irradiation time 13 min), σ_e decreases – this time to 180 MPa, yield strength $\sigma_{0.2}$ drops to 70 MPa (247 MPa for strain-hardened sample), unit elongation δ grows to 24 % (compared with $\delta=59$ % for annealed during 2 h sample).



Fig. 1. Relationship between dose, ultimate stress σ_e , yield strength $\sigma_{0.2}$ and unit elongation for VD1 alloy under irradiation with Ar⁺ ions: E=20 keV, $j=150 \mu\text{A/cm}^2$)

The electron microscopy study of the initial strain-hardened alloy VD1 testifies to the presence in it of a cellular dislocation structure with narrow boundaries between cells (Fig. 2, *a*). The cells diameter is $0.5-2 \ \mu$ m.

After a 2-hour annealing at temperatures 240–250 °C, a practically homogeneous subgrain structure is formed in VD1 alloy, with subgrain mean diameter of $0.5-2 \ \mu m$ (Fig. 2, b). The subgrain boundaries present mainly dense dislocation walls.



Fig. 2. Microstructure of initial strain-hardened (a) and annealed VD1 alloy (b)

After irradiation of deformed VD1 alloy with Ar⁺ ions with energy E=20 keV, ion current density $j=150 \ \mu\text{A/cm}^2$ and dose $D=10^{15} \text{ cm}^{-2}$ (irradiation time 1 s), a developed subgrain structure was detected in the sample cross-section, parallel to the irradiated surface (Fig. 3, *a*).

The observed subgrains are of predominantly equiaxial shape. Their mean diameter is $0.5-1.5 \mu m$. The subgrain boundaries show a characteristic striped contrast specific for tilt boundaries. Single dislocations in the boundaries are not resolved. This indirectly points to the fact that the disorientation angle between subgrains is at least 10°. An insignificant number of dislocations is preserved in single subgrains only. The above facts show that a structure of a polygonal type was formed in the alloy under irradiation.



Fig. 3. Images of subgrain structure of VD1 alloy irradiated with Ar⁺ ions: E=20 keV, $j=150 \mu$ A/cm², $D=10^{15}$ cm⁻²: a – section parallel to (along) irradiated surface; b to d – cross-section: b – in the vicinity of irradiated surface, c – central part of sample, d – in the vicinity of non-irradiated surface

Analysis of sample cross-section revealed equiaxial subgrains $0.5-1 \ \mu m$ in diameter (Fig. 3. b) in the areas bordering with the irradiated surface. In the central part of the cross-section, subgrains of both equiaxial and elongated shape were noted (Fig. 3, c). On the side opposite to the irradiated one, subgrains have an elongated shape only (Fig. 3, d).

So, structural changes are observed over the entire sample depth, but their character is however determined by the distance from the irradiated surface.

Irradiation of VD1 alloy with a beam of Ar⁺ ions to a dose $D=10^{17}$ cm⁻² results in formation of a crystalline structure with grain size over 10 μ m. Fragments of grains separated with straight-line high-angle borders are shown in Fig. 4, *a*.

Inside the grains (Fig. 4, *a*, *b*), high density of equiaxial precipitates 10 to 150 nm is found. Note that at lower irradiation doses practically no precipitates had been observed in this alloy. It was established on the basis of calculation of inter-planar distances of the formed particles, by additional reflexes on the electron diffraction pattern, that phases Al₆(Fe,Mn), Al₈Fe₂Si and $\theta'(\theta'')$ – CuAl₂ are present in the irradiated alloy. The electron diffraction patterns of VD1 alloy with reflections of such phases rare shown in Fig. 4, *c*, *d*. The distribution of phases $\theta'(\theta'')$ and Al₆(Fe,Mn) particles inside the grain is illustrated in dark-field images in the reflections of such phases (Fig. 4, *b*, *c*). It may be seen that partic-



les in the form of flat disks 10–20 nm in diameter are

distributed evenly. Their density is high.

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Fig. 4. Image of grain fractions (a), dark-field image of particles of $\theta'(\theta')$ -phase (b) and Al₆(Fe,Mn) (c) in VD1 alloy irradiated with Ar⁺ ions: E=20 keV, j=150 μ A/cm², $D=10^{17}$ cm⁻², and respective

electron diffraction patterns: d – single arrow marks elongated reflections of phase $\theta'(\theta')$, double arrows show reflections of phase Al₆(Fe,Mn); e – reflections of phase Al₈Fe₂Si are marked

The diameter of equiaxial particles $Al_6(Fe,Mn)$ varied from 100 to 150 nm. They are also evenly distributed in the sample, but their density is insignificant. The particles of phase Al_8Fe_2Si in the alloy light-field images have a shape close to equiaxial, with the diameter equal to 40–80 nm (Fig. 4, *a*, shown with arrows).

It is quite a difficult task to interpret the measurements of mechanical properties of samples of the irradiated alloy. This particularly relates to such characteristics as yield strength $\sigma_{0,2}$ and unit elongation δ . Despite the high density of uniformly distributed precipitates, the abrupt increase of plasticity and drop of the strength characteristics values may only be explained by intensive grain growth.

Increase of the irradiation dose to $7.5 \cdot 10^{17}$ cm⁻² has no significant influence on the state of the irradiated alloy grain structure. At the same time, there is noted further change of the size and distribution density of the earlier revealed phases: $\theta'(\theta') - \text{CuAl}_2$, Al₈Fe₂Si, Al₆(Fe,Mn). So, the diameter of the principal strengthening phase $\theta'(\theta'')$ grew to 50 nm (Fig. 5, *a*, *b*). Simultaneously, there takes place certain decrease of its density. This is clearly observed in comparing Figures 4, *b* and 5, *a*.

The results of measurements of mechanical properties have shown that irradiation dose increase from $1 \cdot 10^{17}$ to $7.5 \cdot 10^{17}$ cm⁻² promotes further drop of yield strength at some increase of unit elongation. Comparison of structural changes with the alloy properties has revealed that this is mainly connected with decrease of density and increase of the sizes of the principal strengthening phase $\theta'(\theta'')$.



Fig. 5. Dark-field reflection image of phase $(200)_{\theta(\theta')}$ (a), and corresponding electron diffraction pattern (b) of VD1 alloy (reflections of $\theta'(\theta')$ -phase are marked)

3. Conclusion

Thus, its was established using the method of transmission electron microscopy that at irradiation of strain hardened alloy VD1 with small doses $D=1\cdot10^{15}$ and $1\cdot10^{16}$ cm⁻² (irradiation time ~ 1 and 10 s) there takes place formation of a developed subgrain structure with equiaxial subgrains, with disorientation angle not less than 10, over the entire depth of 3-mm thick samples. This leads to insignificant drop of strength characteristics and gradual growth of unit elongation.

At irradiation dose increase to $1 \cdot 10^{17}$ cm⁻² and higher, there is formed a uniform coarse crystal-grain structure with grain size over 10 μ m, similar to the same alloys structure in recrystallized state.

Besides, high density of equiaxial precipitates of phases $\theta'(\theta') - \text{CuAl}_2$, Al₆(Fe,Mn), Al₈Fe₂Si, 10 to 150 nm in diameter, is observed inside the grains.

The abrupt increase of plasticity and drop in value of strength characteristics, despite the high density of rather uniformly distributed precipitate particles, may be explained by intensive grains growth.

Electron microscopy investigation of the alloy structural state in the sample cross-sections parallel and perpendicular to the irradiated surface points to the fact that structural changes under irradiation, even to low doses (10^{15} , 10^{16} cm⁻²), occur in the entire volume of the ~ 3-mm thick sample, while the Ar⁺ ions projected range at 20 keV energy in aluminum alloy VD1 accounts for 40 nm only (according to calculations made by the TRIM method).

The registered structural changes take place at a high rate (within a few seconds at low irradiation doses) at a depth exceeding the ions projected range by a factor of tens of thousands. This is, undoubtedly, an argument is favour of the radiation-dynamic nature of the effect of accelerated ion beams on the structure and properties of solids.

References

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