Effect of ion irradiation on structural state and mechanical properties of naturally-aged hot-pressed D16 (Al-Cu-Mg) alloy profiles

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Abstract. The effect of irradiation with 20 keV argon ions on the mechanical properties, structure, and phase composition of quenched and then naturally aged, hot-pressed profiles (6 mm thick) from the D16 alloy of the Al-Cu-Mg system has been studied. It was found that short-term irradiation with Ar⁺ ions (E = 20 keV, j = 200 μA/cm², F = 1 × 10¹⁶ cm⁻², irradiation time 8 s) leads to transformation of the microstructure and phase composition of the alloy. The coarsening of the initial subgrain structure occurs near the sample surface. Both in the surface layer and at a distance of ~ 150 μm from it, partial dissolution and fragmentation of complex intermetallic compounds of crystallization origin located along grain boundaries are observed, as well as a decrease in the size and change in the morphology of Al₆(Fe, Mn) intermetallic compounds of crystallization origin are observed too: the distribution density of lamellar precipitations decreases, and equiaxial precipitations disappear. Under the influence of irradiation, the decomposition of the supersaturated solid solution is activated with the formation of a more stable phase S'. As a result of ion-beam treatment in this mode, the plasticity of the alloy increases while maintaining the strength properties.

1. Introduction
The results of earlier studies on the effect of the gas ion beam on aluminum alloys [1-5] indicate that there is a possibility in principle of using ion-beam processing at intermediate stages of the technological process of obtaining sheet metal from these alloys. In this case, a decrease in the labor intensity and energy consumption of the processes can be ensured. It was found that such treatment, in addition to accelerated (within a few seconds) annealing of work-hardened sheets 1-6 mm thick during cold rolling, makes it possible to control the intermetallic composition of the alloys. In this regard, it is of interest to study the possibility of using ion irradiation as an additional final processing operation to improve the functional properties of sheets and profiles made of aluminum alloys.

In this work, a D16 alloy of the Al-Cu-Mg system with Mn additives was chosen as the object of research. This classic duralumin is characterized by high strength in combination with high plasticity.
It is widely used as a structural material in various fields of modern technology, including the aircraft industry.

2. Experimental part

Standard flat specimens for tensile tests were made from hot-pressed profiles PR100-23 with a section in the form of a corner with a thickness of 6 mm from alloy D16 in the state of delivery – after quenching and natural aging [6, 7]. The samples were irradiated with continuous beams of accelerated argon ions on an ILM-1 ion-beam implantation unit equipped with a PULSAR-1M ion source with a hollow cold cathode based on a glow discharge [8]. A ribbon ion beam 100×20 mm² was cut from the cylindrical ion beam using a collimator. Irradiation was carried out on both sides of the samples. The following irradiation parameters were used: ion energy \( E = 20 \text{ keV} \), ion current density \( j = 200 \text{ µA/cm}^2 \), fluence \( F = 2 \times 10^{15} \text{ and } 1 \times 10^{16} \text{ cm}^{-2} \), the maximum temperature to which the samples were heated during irradiation did not exceed 40°C.

Standard mechanical uniaxial tensile tests were performed using flat specimens in accordance with GOST 1497 [9]. The tests were carried out on an INSTRON 8801 servo-hydraulic test setup. The measurement error was ~ 3%.

The study of the structure and phase composition of the alloy was carried out by the method of thin foils using a JEM-200 CX and Philips CM 30 Super Twin electron microscopes, at the Electron Microscopy Center of Collaborative Access of the Institute of Metal Physics, UB RAS. The microstructure was studied in two sections parallel to the irradiated surface: directly near the surface and at a distance of ~ 150 µm from it.

3. Details of exposure, measurements, and results

The results of measuring the mechanical properties of samples cut from extruded profiles of alloy D16 in the initial state and after ion irradiation are given in table 1.

Table 1. Mechanical properties of the D16 alloy samples in the initial state (after aging) and after various modes of Ar⁺ ion beam irradiation.

| №  | Alloy processing                                      | Yield strength \( \sigma_{0.2} \) (MPa) | Ultimate strength \( \sigma_{u} \) (MPa) | Relative elongation \( \delta \) (%) |
|----|------------------------------------------------------|----------------------------------------|----------------------------------------|-------------------------------------|
| 1  | Initial state after quenching and natural aging      | 441.0                                  | 557.3                                  | 9.7                                 |
| 2  | Irradiation mode 1: \( E = 20 \text{ keV}, j = 200 \text{ µA/cm}^2, \) \( F = 2 \times 10^{15} \text{ cm}^{-2}, T = 40\degree \text{C} \) | 477.3                                  | 563.9                                  | 9.5                                 |
| 3  | Irradiation mode 2: \( E = 20 \text{ keV}, j = 200 \text{ µA/cm}^2, \) \( F = 1 \times 10^{16} \text{ cm}^{-2}, T = 40\degree \text{C} \) | 435.4                                  | 548.9                                  | 12.3                                |

In the initial state after quenching and natural aging, the D16 alloy has the following mechanical properties: \( \sigma_{u} = 557.3 \text{ MPa}, \sigma_{0.2} = 441.0 \text{ MPa}, \delta = 9.7\% \). After ion irradiation with a fluence \( F = 2 \times 10^{15} \text{ cm}^{-2} \), the mechanical properties practically do not change. An increase in the dose (fluence) to \( F = 1 \times 10^{16} \text{ cm}^{-2} \) leads to an increase in the relative elongation to \( \delta = 12.3\% \), while the strength characteristics within the above measurement error also remain unchanged.

Electron microscopic examination of the D16 alloy in the initial state indicates that the alloy is characterized by a developed subgrain structure. Most of the subgrains are somewhat elongated, up to 5 µm in length and about 2 µm in width, or nearly equiaxial with a diameter of 2-3 µm (figure 1 (a)).

Along the grain boundaries, large intermetallic particles of a rounded shape with a size of up to 5 µm were revealed, which are various phases of a complex composition based on Al, Cu, Mg, Fe, Mn, Si, formed during crystallization. In addition, both in the volume and along the boundaries of subgrains, there are intermetallic compounds of crystallization origin \( \text{Al}_6(\text{Fe, Mn}) \) of equiaxed or

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lamellar form (figure 1 (b-d)). The intermetallic compounds $\text{Al}_6(\text{Fe}, \text{Mn})$, in contrast to the coarse intermetallic particles described above, are characterized by a high distribution density. The average diameter of equiaxed intermetallic compounds $\text{Al}_6(\text{Fe}, \text{Mn})$ is 0.2-0.7 µm. The length of the plates is 0.5-0.7 µm, the width of the plates is up to 0.1 µm. Against their background, there are also larger plates up to 1.5 µm in length and up to 0.6 µm in width (figure 1 (b)). The dark-field image of these intermetallics is shown in figure 1 (d).

In addition, in the volume of subgrains, contrast is seen from dispersed precipitates of strengthening phases formed during natural aging: $\theta'(\theta'')$ (metastable modifications of the stable $\text{CuAl}_2$ phase), as well as of $S'$ phase (which is a modification of the stable $\text{Al}_2\text{MgCu}$ phase) (figure 1 (e, f)). Phase reflections and characteristic diffraction effects are revealed in the electron diffraction patterns of the aged alloy: strands parallel to the $<001>$ direction with intensity maxima, the centers of which correspond to reflections from the $\theta'(\theta'')$ phase (figure 1 (e)) and reflections from the $S'$ phase.

In the dark-field images in the phase reflections, it can be seen that the precipitates are characterized by a high distribution density. More dispersed particles in the form of flat disks up to 20 nm in diameter are phases $\theta'(\theta'')$ distributed over the $\{001\}_{\text{Al}}$ planes (figure 1 (e)). Larger plate-shaped particles with a diameter of 25-50 nm correspond to the $S'$-phase (figure 1 (f)). A qualitative analysis of the contrast from the appeared dispersed precipitates in dark-field images indicates the predominance of particles of the metastable phase $\theta'(\theta'')$.

Next, let us consider the results of an electron microscopic study of a D16 alloy sample after irradiation in mode 2 ($E = 20$ keV, $j = 200$ µA/cm$^2$, $F = 1 \times 10^{16}$ cm$^{-2}$, $T = 40^\circ$C) when an increase in alloy plasticity is observed.
The study showed that irradiation in the considered regime has only a slight effect on the grain-subgrain structure of the irradiated alloy samples near the surface. After irradiation, a substructure is observed with characteristic dimensions of structural elements from 2 to 5 μm (figure 2 (a, b)).

As a result of irradiation along the grain boundaries, crushing and partial dissolution of phases of the complex composition of crystallization origin occurs. After processing, particles of a round or elongated shape are preserved in the form of separate precipitates or chains of particles (figure 2 (a)), the diameter of intermetallics is 0.6-1.5 μm. After irradiation, the alloy retains Al₆(Fe, Mn) intermetallics in the form of extended sticks up to 0.6 μm in length (figure 2 (c, d)). Comparison of the structure in the naturally aged and irradiated states shows that after irradiation, the distribution density and the size of these precipitates slightly decrease. In addition, the dimensional and morphological homogeneity of Al₆(Fe, Mn) particles increases: in the images of the microstructure of the alloy, previously detected large plates with a length of more than 1 μm become single (figure 2 (c, d)), and the volume fraction of equiaxed particles significantly decreases.

In the irradiated alloy, as well as in the aged one, there are particles of the strengthening phases θ'(θ'') and S' (figure 2 (e, f)). The dark-field images clearly show that irradiation initiates the decomposition of the supersaturated solid solution with the predominant release of the more stable S'-phase (figure 2 (e, f)). After irradiation, the distribution density and the size of the S'-phase plates (figure 2 (f)) increase significantly (length up to 85 nm, width up to 20 nm). Dark-field images show a contrast in the form of "coffee beans", which indicates the formation of particles of the θ'' phase.
coherent with the aluminum matrix (figure 2 (e)). Particle diameters range from 10 to 20 nm, they are characterized by negligible volume fraction and density of distribution (figure 2 (f)).

With distance from the sample surface, the effect of exposure to radiation decreases. The subgrain structure at a distance of ~ 150 µm from the irradiated surface is close to the structure of the initial state. The structure is more uniform, the average diameter of equiaxed subgrains is 2-5 µm (figure 3 (a)). In the volume of grains, increased dislocation density was found in comparison with that for grains near the irradiated surface (figure 3 (b)) and is closer to the dislocation density in the initial state.

![Microstructure of alloy D16 at a distance of 150 µm from the irradiated surface after irradiation in mode 2 (E = 20 keV, j = 200 µA/cm², F = 1×10¹⁶ cm⁻²): a – dark-field image in reflections (200)ₐ₆, (200)ₜₖ, (200)ₜₖ; b-d – bright field images; e, f, g – dark-field images: e – in the reflection (311)ₐ₆(Fe,Mn), f – in the reflection (200)ₜₖ(φ), g – in the reflection (112)ₚₖ.](image)

As well as near the irradiated surface, larger particles of phases of the complex composition of crystallization origin are retained in the bulk of the studied samples, the particle size reaches 1.5 µm (figure 3 (a, c)), there are intermetallic compounds based on Al₆(Fe, Mn) in the form of extended plates. The size and density of distribution of these precipitates in the volume are higher than in the irradiated surface layer (figure 3 (c-e)). But in comparison with the initial state, a decrease in the distribution density and dimensions of the indicated intermetallic compounds is observed both on the
surface and in the volume. Figure 3 (d, e) clearly visualizes a crushed Al₆(Fe, Mn) plate, which proves the effect of accelerated ions on the microstructure of not only the surface but also the volume of the samples under study.

In the dark-field images obtained in phase reflections, at a considerable distance from the surface, a large number of highly dispersed particles (figure 3 (f, g)) are detected, although the volume fraction and sizes of lamellar precipitates representing the S'-phase are lower compared to the surface (in this case the length of the plate reaches 60 nm). To a greater extent, the contrast is visualized from the discs with a diameter of 10-20 nm. Analysis of electron microscopic images and the corresponding electron diffraction patterns showed that this is a mixture of precipitates of the θ'-phase semi-coherent with the aluminum matrix and the coherent θ''-phase.

4. Conclusion
Thus, it was found that short-term (for 8 s) irradiation with Ar⁺ ions \( (E = 20 \text{ keV}, j = 200 \mu\text{A/cm}^2, F = 1 \times 10^{16} \text{ cm}^{-2}) \) leads to the transformation of the microstructure and phase composition of naturally aged hot-pressed alloy profiles D16. It is shown that irradiation leads to the formation of a coarser subgrain structure near the sample surface. In addition, irradiation promotes, both in the surface layer and at a distance of \( \sim 150 \mu\text{m} \) from it, partial dissolution and fragmentation of complex intermetallic compounds of crystallization origin located along grain boundaries, as well as a decrease in the size and change in the morphology of Al₆(Fe, Mn): intermetallic compounds: the density of distribution of lamellar-shaped excretions decreases and equiaxed excretions disappear. Under the influence of irradiation, the decomposition of the supersaturated solid solution is activated with the formation of a more stable phase \( S' \). An increase in the volume fraction of lamellar precipitates of the \( S' \)-phase suppresses the nucleation and growth of metastable aluminum-copper phases \( \theta''(\theta') \).

As a result of irradiation with \( \text{Ar}^+ \) ions with a fluence of \( 1 \times 10^{16} \text{ cm}^{-2} \), an increase in the ductility of the alloy occurs while maintaining the strength characteristics. This result seems to be interesting, since improving the plastic properties while maintaining the strength properties can have a beneficial effect on the resource characteristics.

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