Investigation of the Effect of Ion Irradiation on Microstructure of Alloy 1960 (Al–Zn–Mg–Cu) in Different Initial States

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Abstract – The method of transmission electron microscopy was used to investigate the effect of irradiation with Ar^+ ions [E = 40 keV, D = = (1–3) · 10^{17} cm^{-2}] on structural-phase state of alloy 1960 of Al–Zn–Mg–Cu system after different kinds of treatment: hot deformation, natural and artificial ageing. Structural and phase transformation taking place in the alloy under irradiation were revealed.

1. Introduction

The object under investigation was alloy 1960 of Al–Zn–Mg–Cu system. Depending on composition, the alloys based on this system are related either to high-strength aircraft alloys, or to medium-strength general application alloys [1]. Their service characteristics are determined by both the structural factors (grain and subgrain size, dislocations density and their distribution), and the phase composition, with account for shape, density and nature of distribution of strengthening phases formed in the process of ageing. The role of intermetallics of crystallization origin should also be taken into account [2].

In the recent years, data have been published on the effect of ion irradiation on the structure and phase composition of aluminum alloys based on Al–Li–Mg and Al–Cu–Mg–Mn systems [3, 4]. To efficiently vary the properties of alloys by accelerated ion beams, it is necessary to thoroughly study the regularity the effect of irradiation regimes on the formation of structure and phase composition in the alloys, and to find out the role of their chemical composition and initial state in these processes.

The purpose of the present investigation was to find out whether the nature of changes in the structure and phase composition of alloy 1960 under ion irradiation depends on its initial structural state. The effect of Ar^+ ions irradiation on alloy 1960 in hot-deformed and naturally and artificially aged states was investigated.

2. Principal part

Alloy 1960 was quenched in water from 465 °C. The period of natural ageing after quenching was 3 months. Artificial ageing to maximum strength was carried out at 130 °C during 16 h.

Irradiation of alloy 1960 samples with accelerated Ar^+ ion beams with energy of 40 keV and at ion current density of 200–400 μA/cm^2 was carried out in the PULSAR ion implanter with an Ar^+ ion glow-discharge source with a cold hollow cathode. In the course of irradiation, a dose of (1–3) · 10^{17} cm^{-2} was accumulated (respective irradiation time 75–240 s), the temperature of samples at the end of irradiation reached 400 °C. It should be noted that such short-term heating in the absence of irradiation causes no changes in the structure and phase composition of aluminum alloys.

Electron microscopic investigation by the thin foils method was carried out in a JEM-200 CX transmission electron microscope. The irradiated samples structure was studied in the section located ~150 μm away and parallel to the irradiated surface.

Hot-deformed state. After hot deformation, non-uniform subgranular structure with 1 to 5 and over μm grain size was revealed in the alloy (Fig. 1, *a*). Inside subgrains, dense dislocation tangles are found, and film-like precipitates are observed along separate boundaries.

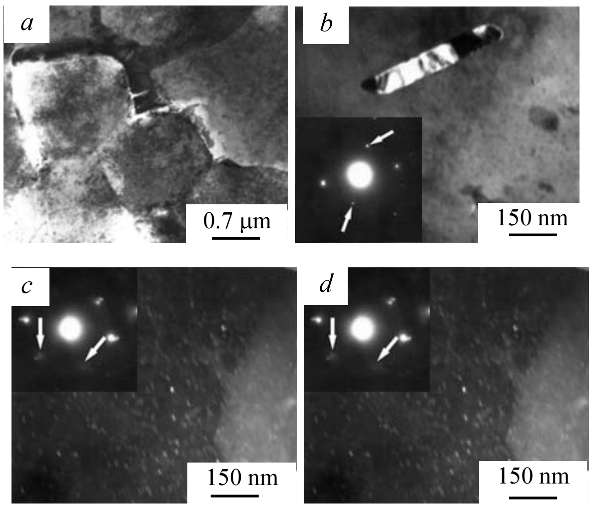


Fig. 1. Image of subgranular structure (*a*) and intermetallics Al_7Cu_2Fe (*b*), Al_3Zr (*c*), η'-phase (*d*) in hot-deformed alloy 1960

Aggregates of randomly distributed intermetallic particles 0.3 to 1.5 μm long are present in subgrains

(Fig. 1, *b*). Based on estimation of their interplanar distances by additional reflections in the electron diffraction patterns their composition was established as Al_7Cu_2Fe.

Beside coarse platele-shaped intermetallics, equiaxial β′-phase (Al_3Zr) particles not over 15 nm in diameter with type $L1_2$ ordered structure are found (Fig. 1, *c*). The quantity of Al_3Zr particles is insignificant. Their small volume fraction may be due to the short-term period of homogenizing. During keeping of the hot-deformed alloy at room temperature its natural ageing takes place, being accompanied by precipitation of fine-disperse particles of metastable η'-phase ($MgZn_2$) in the form of thin disks 5–10 nm in diameter (Fig. 1, *d*). Besides, an insignificant quantity of θ′′-phase (Al_2Cu) was found.

Alloy 1960 irradiation with Ar^+ *ions in hot-deformed state* to a dose of $2 \cdot 10^{17}$ cm^{-2} causes formation of new subgrains along separate small-angle boundaries (Fig. 2, *a*, shown with an arrow). After irradiation, the grain and subgrain boundaries are mainly free from precipitates (which were observed after hot rolling). A great quantity of intermetallics in the form of sticks, strips and elongated plates 0.4–0.5 μm long is observed inside the subgrains. Part of intermetallics retain the Al_7Cu_2Fe composition, but their sizes and quantity are smaller. At the same time, particles of phase T ($Al_{49}(Zn, Mg)_{32}$) in the form of strips and phase S (Al_2CuMg) in the form of coarse elongated plates appear. A dark-field image of particle Al_2CuMg in shown in Fig. 2, *b*. Particles of phases T and S were not observed in the initial deformed state, hence their formation has been initiated by irradiation.

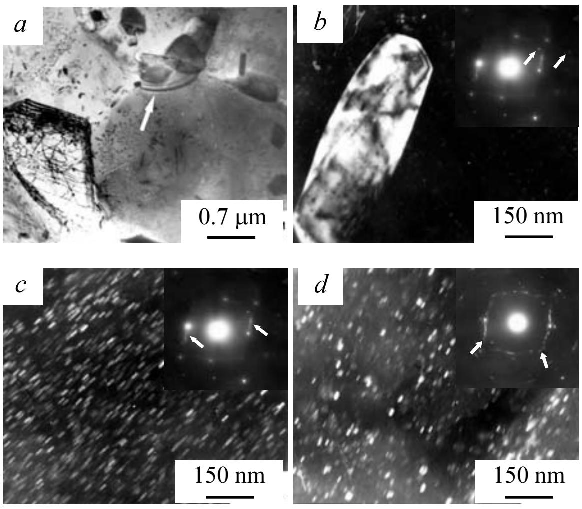


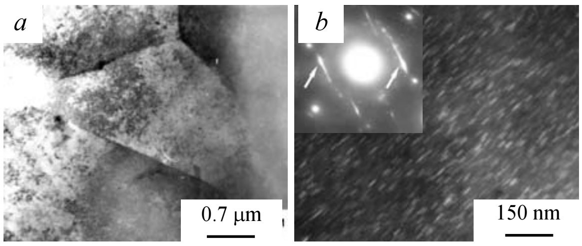
Fig. 2. Image of subgranular structure (*a*) and intermetallics Al_2CuMg (*b*), θ′′(θ′)-phase (*c*), η′- and η-phases (*d*) in hot-deformed alloy 1960 after irradiation with Ar^+ ions, $D = 2 \cdot 10^{17}$ cm^{-2}

Phases η′ and η are also found in the form of thin disks, with diameter not over 10 nm in case of metastable η′-phase precipitation, and 20 nm in case of stable η-phase. A dark-field image of these phases, with a respective electron diffraction pattern, is given in Fig. 2, *d*.

Under irradiation, decomposition of supersaturated solid solution also takes place, with precipitation of fine-disperse strengthening particles of phases η′, η, and θ′′(θ′). It has been noted above that in deformed alloy 1960, formation of these phases takes place in the process of natural ageing. Ion irradiation accelerates the process of decomposition. Particles of θ′′(θ′)-phase in the form of thin disks are up to 20 nm in diameter and are present in high enough density (Fig. 2, *c*).

Thus irradiation of deformed alloy to a dose of $2 \cdot 10^{17}$ cm^{-2} results in decrease of the quantity of intermetallics Al_7Cu_2Fe of crystallization origin, but initiates formation of coarse intermetallics of T- and S-phases. Besides, formation of phase Al_3Zr is suppressed, and precipitation of particles of θ′′(θ′), η′- and η-phases is stimulated.

Natural aged state. After quenching and natural ageing, a subgranular structure was revealed, but with the subgrain sizes larger than in the hot-deformed state. The diameter of single subgrains reaches 10 μm (at average diameter of 4–5 μm, Fig. 3, *a*). As a result of quenching, high density of dislocations is formed in the subgrains, being caused by quenching stresses.



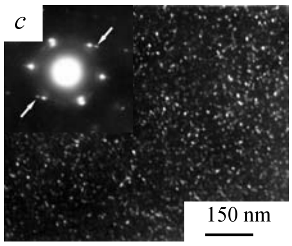


Fig. 3. Image of subgranular structure (*a*) and intermetallics of θ''(θ')-phase (*b*), η'-phase (*c*) in naturally aged alloy 1960

The alloy retains an insignificant quantity of intermetallics of crystallization origin, but of much smaller size and distribution density than in the deformed state. The observed particles length does not exceed 0.2 μm. Analysis of respective electron diffraction patterns testifies to the fact that intermetallics have retained their original composition, Al_7Cu_2Fe.

The size and distribution density of particles of metastable β'-phase (Al_3Zr) are approximately the same as in the hot-deformed state.

As a result of natural ageing, particles of metastable θ′′- and η′-phases in the form of thin disks were formed. The diameter of θ′′-particles reaches 15–20 nm, and that of η′-particles, not over 10 nm. Dark-field images of particles in reflections of both phases, and respective electron diffraction patterns, are given in Figs. 3, *b* and *c*.

Alloy 1960 irradiation with Ar^+ *ions in naturally aged state* to a dose of $1 \cdot 10^{17}$ cm^{-2} leads to transformation of the structure from subgranular to recrystallized: grains of predominantly equiaxial shape with large-angle boundaries are observed, with grain size over 10 μm (Fig. 4, *a*). The grain boundaries are free from precipitates. Irradiation has also caused complete dissolution of intermetallics Al_7Cu_2Fe of crystallization origin and β'-phase particles.

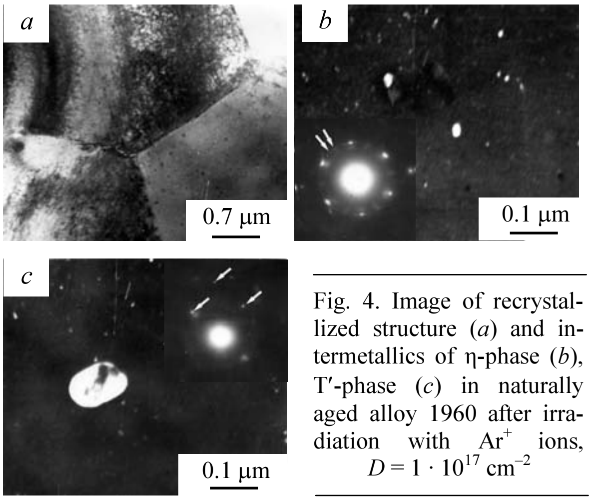


Fig. 4. Image of recrystallized structure (*a*) and intermetallics of η-phase (*b*), T′-phase (*c*) in naturally aged alloy 1960 after irradiation with Ar^+ ions, $D = 1 \cdot 10^{17}$ cm^{-2}

At the same time, formation of new phases is observed: the particles of stable η-phase up to 30 nm in diameter (Fig. 4, *b*) and metastable T′-phase ~ 200 nm in diameter (Fig. 4, *c*) appear. No significant effect of irradiation on the process of metastable θ′′ and η′-phases is observed.

Artificially aged state. The subgranular structure of alloy 1960 aged during 16 h at 130 °C is similar to the structure observed in a quenched state: the subgrains average size varies within 4–5 μm.

Inside the grains bright-field images, a contrast from uniformly distributed particles of strengthening phases is observed (Fig. 5, *a*). Analysis of respective electron diffraction patterns and dark-field images shows that these particles present metastable η′- and stable η-phases. The volume fraction of disk-shaped η-phase with ~15 nm disk diameter is insignificant (Fig. 5, *b*), while the density of metastable η′-phase less than 10 nm in diameter is large (Fig. 5, *c*). Such ratio of strengthening phases (prevalence of fine-disperse metastable phase and small quantity of stable phase) precipitated in artificial ageing is due to the low ageing temperature.

The metastable θ′′(θ′)-phase is not precipitated at artificial ageing of this alloy.

Particles of η′ and η-phases are uniformly distributed within the grains volume; in particular, there are no precipitation-free zones close to the boundaries.

In the aged alloy, particles of metastable β′-phase (Al_3Zr) of equiaxial form are also present with an ordered internal structure, 15–20 nm diameter and low distribution density (Fig. 5, *d*).

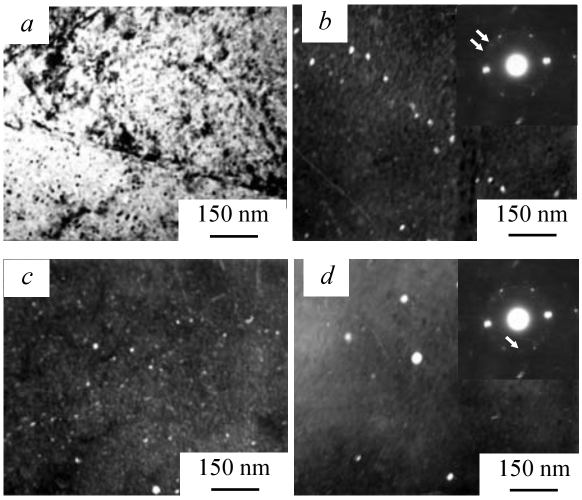


Fig. 5. Bright-field (*a*) и and dark-field images of particles of η′ (*b*) and η-phases (*c*), and of β′-phase (*d*) in artificially aged alloy 1960

Alloy 1960 irradiation with Ar^+ *ions in artificially aged state* to a dose of $3 \cdot 10^{17}$ cm^{-2} had a strong effect on its structure: under irradiation, recrystallization took place. This is testified to by the absence of small-angle boundaries and increase in the density of rectilinear large-angle boundaries. A detail of such boundary is shown in Fig. 6, *a*.

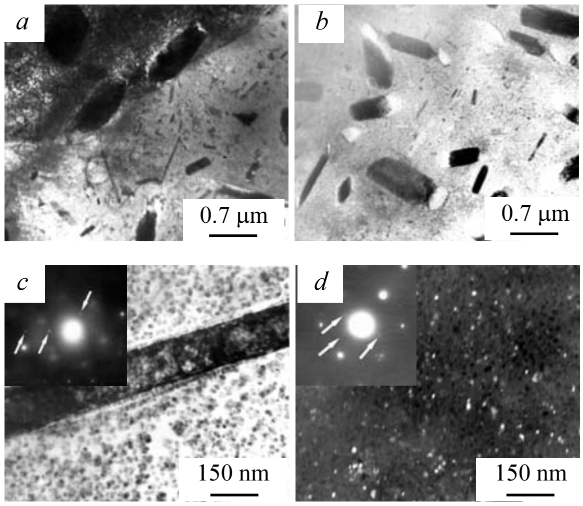


Fig. 6. Images of intermetallics close to large-angle boundary (*a*) and inside grain (*b*), particles of T′-phase (*c*) and η- and η′-phases (*d*) in artificially aged alloy 1960 after irradiation with Ar^+ ions, $D = 3 \cdot 10^{17}$ cm^{-2}

It is seen that precipitates have nucleated at the boundaries either in the form of isolated particles, or in the form of a continuous film.

A characteristic feature of the irradiated sample is the presence of randomly distributed very coarse precipitates in the form of plates and strips inside the grains (Fig. 6, *b*). The length of single particles reaches several μm.

Based on the analysis of electron diffraction patterns it was established that the composition of elongated strip-shaped intermetallic particles is $Al_{49}(Zn, Mg)_{32}$ (Fig. 6, *c*), and that of plate-shaped intermetallics is Al_2CuMg. Note that intermetallics Al_2CuMg of similar morphology had also been detected after ion irradiation of a *hot-deformed* sample to a high dose ($D = 2 \cdot 10^{17}$ cm^{-2}). However, in the case of a preliminarily aged sample, they are of a coarser size (about 2 times longer).

Intermetallics $Al_{49}(Zn, Mg)_{32}$ (strip-shaped) and Al_2CuMg (plate-shaped) were not found in *artificially aged* samples. This allows us to suggest that change of phase composition as a result of formation of the named intermetallics was initiated by irradiation to a dose of $3 \cdot 10^{17}$ cm^{-2}. After irradiation, the alloy retained disperse particles of stable η- and metastable η′-phases in the form of flat disks which had precipitated in the course of artificial ageing. Irradiation had practically no effect on their morphology and did not lead to change in the volume fraction. The diameter of particles of both phases is 10–15 nm (Fig. 6, *d*).

3. Conclusion

Thus, as a result of electron-microscopic investigation, it was established that:

1. After irradiation of *hot-deformed* alloy 1960 with accelerated Ar^+ ions to a dose of $2 \cdot 10^{17}$ cm^{-2}, disperse grains are formed along separate small-angle boundaries. Under irradiation, partial dissolution of coarse intermetallics Al_7Cu_2Fe and layers of excess phases along grain boundaries formed in the course of crystallization takes place. Also dissolution of particles of the metastable β′-phase (Al_3Zr) is observed. There is observed formation of coarse particles of T′ and S-phases in the form of strips and plates. Irradiation accelerates the processes of decomposition of supersaturated solid solution in a deformed alloy, with precipitation of fine-disperse strengthening η′-, η- and θ″(θ′)-phases.
2. As a result of irradiation of *naturally aged* alloy 1960 with accelerated Ar^+ ions to a dose of $1 \cdot 10^{17}$ cm^{-2}, a recrystallized structure is formed, and complete dissolution of intermetallics of crystallization origin Al_7Cu_2Fe and particles of β'-phase takes place. At the same time, irradiation initiates precipitation of stable η- and metastable T'-phases.
3. Irradiation of *artificially aged* alloy to a dose of $3 \cdot 10^{17}$ cm^{-2} initiates primary recrystallization and dissolution of intermetallics of crystallization origin, and along with that facilitates formation of coarse plate- and strip-shaped particles of T′- and S-phases both inside grains and along grain boundaries. At the same time, the volume fraction and the nature of η′ and η particles do not vary as compared with the artificially aged state.

References

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