Effect of Irradiation with Powerful Ion Beams on Microstructure of Cold-Worked Aluminum–Lithium Alloy 1441

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Abstract – In this paper, the results of electron-microscopic investigation of alloy 1441 of the Al–Cu–Mg–Li–Zr–Mn system, after cold rolling ($\varepsilon = 72\%$) and subsequent exposure to a 180 keV proton-carbon ion beam (70% C^+ + 30% H^+) are considered. It is shown that exposure of cold-worked alloy 1441 to low dose to pulsed irradiation with a powerful ion beam of 70% C^+ + 30% H^+ causes noticeable transformation of the existing cellular structure of the alloy. There was revealed a dependence of the degree of transformation on ion current density: at $j = 200$ A/cm^2, more intensive changes in the alloy microstructure are observed than at $j = 100$ A/cm^2.

1. Introduction

It is shown in a series of papers by the authors that the radiation-dynamic effect of accelerated ions may initiate massive (martensitic) transformations [1], formation of short- and long-range atomic order at low temperatures [2, 3], activation (acceleration) and reduction of temperature of the ageing processes [4], and transformation of dislocation and granular structure in the alloys [5]. The result is formation of unique electrical [2], magnetic [1, 6], mechanical [7] and other properties of materials.

In some cases, the radiation-dynamic affect may cause effects similar to those observed in the course of thermal annealing. So, in [8, 9], it was established that, in a cold-worked alloy 1441 of the system Al–Li–Mg–Cu–Zr–Mn, when irradiated with continuous $E = 20$ keV Ar^+ ion beams at ion current densities of 150–400 μA/cm^2 to doses of $1 \cdot 10^{15} - 1 \cdot 10^{16}$ cm^{-2} (respective irradiation time ~1–10 s), a transfer from initial cellular to subgranular structure, similar to polygonal, is observed. Under irradiation doses of $5 \cdot 10^{16}$ cm^{-2} and higher, uniform granular structure with grains 5–10 μm in diameter and over is formed, leading to a significant yield point drop and increase of plasticity of the alloy. Changes in the dislocation and granular structure of the alloy observed after irradiation are similar to those observed after annealing of the same alloy in a furnace at $T = 380$–400 °C during 2–3 h.

Changes in the dislocation and granular structure are observed not in the surface layer adjoining the ions implanting zone only (the 40-keV Ar^+ ions projected range is ~ 40 nm), but over the whole thickness of the 1-mm thick layer, which is about 10^5 times greater than the ions projected range. The nature of such effects of long-range kind under ion irradiation still remains underinvestigated.

It is therefore interesting to see how the sort and energy of ions, the ion current density and the change-over from continuous to pulsed-periodic irradiation regime, including nanosecond ion pulses, affect the nature of changes in the structure and properties of alloys.

With this purpose in view, there was conducted electron-microscopic investigation of the effect of irradiation with ion beam of 180-keV accelerated (70% C^+ + 30% H^+) at high ion current density of 100–200 A/cm^2 in a pulsed-periodic regime (pulse duration 80 ns, frequency 0.1 Hz) on the structure and phase composition of alloy 1441.

2. Experimental part

The object under investigation was cold-worked alloy 1441 ($\varepsilon = 72\%$) which in the form of clad sheets (~ 1 mm thick) were manufactured at the Kamensk-Uralsky Metallurgical Plant.

One-side irradiation of alloy 1441 samples with proton-carbon beam (70% C^+ + 30% H^+) in pulsed-periodic regime was carried out in the TEMP accelerator (TPU, Tomsk), at pulse duration $\tau = 80$ ns and pulse repetition frequency $f = 0.1$ Hz. The ions power was 180 keV.

At ion current density of 100 A/cm^2, samples were irradiated to a dose of $2 \cdot 10^{15}$ cm^{-2}, and at 200 A/cm^2, to $1 \cdot 10^{14}$ cm^{-2}. In all cases, the temperature of samples under irradiation did not exceed 30 °C.

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 over the cross-section and over the section from that parallel to the irradiated surface and located ~ 150 μm away

2.1. Cold-worked state

In the section parallel to the irradiated surface of the initially cold-worked alloy 1441 the presence of non-uniform cellular dislocation structure was revealed (Figs. 1, *a* and *b*).

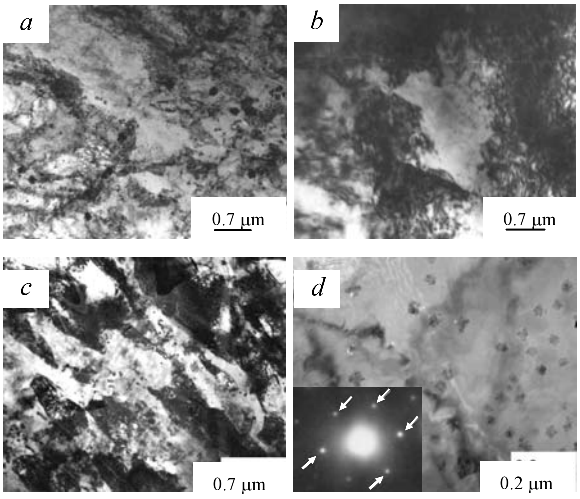

Fig. 1. Image of cellular structure (*a*, *b*), slip bands (*c*) and intermetallics Al_3Zr (*d*) in cold-worked alloy 1441; *a*, *b*, *d* – section parallel to irradiated surface; *c* – cross-sections

Cells diameter and their boundaries width differ from section to section approximately 2–4 times. The average diameter of dislocation-free central regions of cells varies between 0.5–2 µm. The cell boundaries present dense tangles of dislocations. In some sections of the sample, cell boundaries width exceed dislocation-free regions the diameter.

Electron microscopic images of the sample cross-section show slip bands 0.3–0.5 µm wide (Fig. 1, *c*). Inside the bands, dislocation tangles are present. They either occupy the entire volume of the bands, or serve as boundaries between individual blocks.

Electron microscopic images of the deformed alloy show equiaxial-shaped particles 20–30 nm in diameter. Type 100, 110 superstructure reflections have been noted in respective electron diffraction patterns. It is known that the presence of superstructure reflections in electron diffraction patterns of aluminum-lithium alloys with addition of Zr is connected with Al_3Zr phase particles formation in the course of casting and subsequent homogenizing. A dark-field image of Al_3Zr particles is given in Fig. 1, *d*.

2.2. One-sided pulsed irradiation with 70%C^+ + 30%H^+ ions to 10^{14} cm^{-2} dose at j = 200 A/cm^2

After irradiation, the alloy retains the cellular structure (Figs. 2, *a* and *b*). However, analysis of different sections of the sample points to its non-uniformity. So Fig. 2, *a* demonstrates a rather regular structure: the cells are separated with narrow boundaries, the average size of dislocation-free central regions is 0.5–1 µm. Some sections of cell boundaries have acquired a banded structure, which points to the beginning of dislocations rearrangement at boundaries.

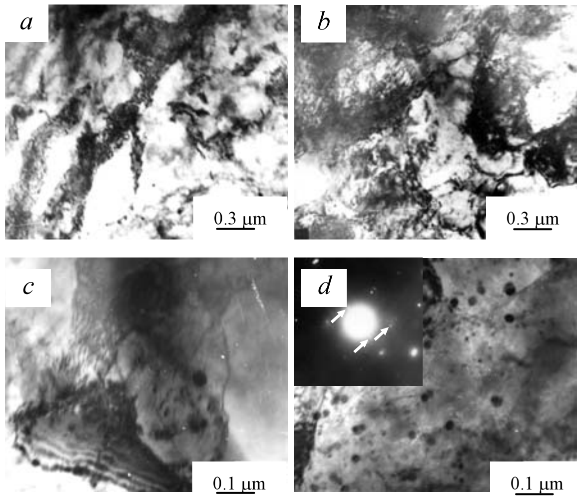

Fig. 2. Microstructure in section parallel to irradiated surface of sample of cold-worked alloy 1441 after pulsed irradiation with 70% C^+ + 30% H^+ ions, j = 200 A/cm^2, $D = 10^{14}$ cm^{-2}: *a*, *b* – cellular structure; *c* – disperse subgrains; *d* – intermetallics of β'- and θ'(θ")-phases

In other regions, the width of dislocation tangles presenting cell boundaries exceeds 1 µm, while the diameter of central regions of cells is not over 0.1–0.5 µm (Fig. 2, *b*). The density of dislocations in tangles is high (single dislocations are not resolved in electron microscopic images).

Alloy structure examination at high magnification has revealed formation of disperse subgrains against the cellular structure background (Fig. 2, *c*). Their average diameter does not exceed 0.5 µm.

Inside the cells and subgrains, equiaxial particles 20–30 nm in diameter and fine platelets up to 10 nm long are observed (Fig. 2, *d*). Based on analysis of respective electron diffraction patterns, it was established that equaxial particles are β'-phase (Al_3Zr) with ordered $L1_2$-type structure, and plate-shaped particles are θ'(θ")-phase ($CuAl_2$). The distribution of β'-phase particles in the sample volume is non-uniform: in some sections their density is high, while in others they are practically not found.

Examination of the sample cross section confirmed that irradiation had produced an effect on the alloy structure, however not resulting in polygonization and formation of subgrains in the whole volume of the material. There is observed formation of single elongated subgrains inside which dislocation tangles are found (Fig. 3, *a*). Subgrains decrease in quantity with increase of distance from the irradiated surface, and the presence of cellular structure becomes more visible inside slip bands (Fig. 3, *b*). In the vicinity of the non-irradiated surface, the area of dislocation-free sections strongly decreases, the dislocation tangles occupy practically the whole of the volume, and subgrains are rare.

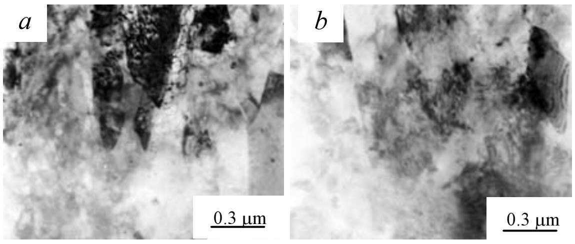

Fig. 3. Microstructure observed in cross-section of cold-worked sample of alloy 1441 after pulsed irradiation with 70% C^+ + 30% H^+ ions, j = 200 A/cm^2, $D = 10^{14}$ cm^{-2}: *a* – in the vicinity of irradiated surface; *b* – in the vicinity of non-irradiated surface

Thus irradiation has had a noticeable effect on transformation of the substructure formed in the sample under deformation, however not leading to dissolution of β′-phase particles. At the same time, it facilitated realization of the initial stage of supersaturated solid solution decomposition accompanied with precipitation of copper-bearing θ′(θ″)-phases.

2.3. One-side pulsed irradiation with 70%C^++30%H^+ ions to $D = 2 \cdot 10^{15}$ cm^{-2} at j = 100 A/cm^2

Irradiation to a high dose, but at smaller current density has resulted in uniform cellular structure being retained in the alloy (Figs. 4, *a* and *b*). The cells diameter varies from 0.2 to 2 and over µm. In a few cases disperse subgrains, revealed at high magnification only, were formed inside cells. Their diameter does not exceed 0.1–0.4 µm (Fig. 4, *c*).

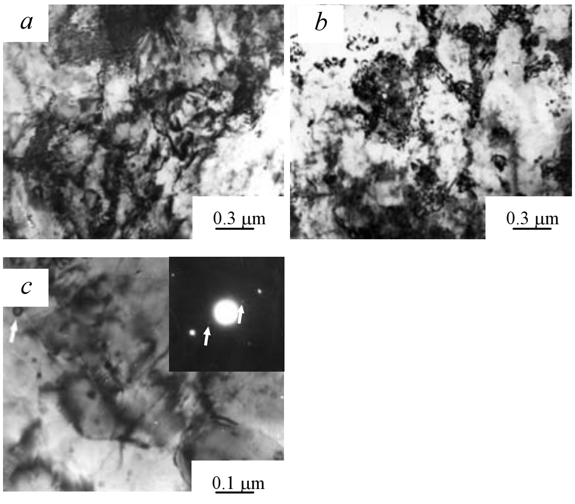

Fig. 4. Image of cellular structure (*a*, *b*), disperse subgrains (*c*) in cold-worked alloy 1441 after pulsed irradiation with 70% C^+ + 30% H^+ ions, j = 100 A/cm^2, $D = 2.25 \cdot 10^{15}$ cm^{-2}. Composite σ'/β' particle is shown with an arrow

Inside the cells, equaxial particles of β'-phase (Al_3Zr) 20–30 nm in diameter are observed (Fig. 4). The respective electron diffraction patterns show superstructural reflections. It should be noted that after irradiation in the said regime, particles show uniform distribution over the entire sample volume, but their density is insignificant. No decomposition of supersaturated solid solution with precipitation of metastable θ′- and θ″-phases was found, but precipitation of metastable σ′-phase (Al_3Li) on the inter-phase surface β′-phase was registered, the latter phase also featuring a cubic lattice with ordered $L1_2$-structure.

But composite particles like σ′/β′ are very rarely found in irradiated alloy (marked with an arrow in Fig. 4, *c*).

Thus decrease of beam current value has resulted in that the alloy has practically retained the structure observed in it after deformation, which is witnessed by the presence of a developed uniform cellular structure and the β'-phase particles.

3. Conclusion

It was established that irradiation of cold-worked alloy 1441 in pulsed regime with 70% C^+ + 30% H^+ ion beams produces a noticeable effect on transformation of the initial cellular dislocation structure. There were found signs of the beginning of rearrangement of dislocations within cell boundaries and formation of disperse subgrains in the alloy.

Electron microscopic investigation of alloy 1441 samples in the section parallel to the irradiated surface was carried out at a distance approximately 150 µm from it, which testifies to the fact that the depth at which structural changes take place is thousands of times larger that the ions projected range in this alloy.

Investigation of the sample cross section has shown that changes in the dislocation structure take place over the entire sample thickness, however more significant changes occur closer to the irradiated surface.

There was revealed an effect of ion current density, since more intensive changes in the alloy microstructure were taking place at higher ion current density (j = 200 A/cm^2), despite that the irradiation dose at such density was lower, $D = 10^{14}$ cm^{-2} (at j = = 100 A/cm^2, the irradiation dose was $D = 2 \cdot 10^{15}$ cm^{-2}).

The causes of absence of the process of polygonization with formation of a subgranular structure in the whole volume of alloy 1441 sample, which was observed under irradiation in continuous regime already at a dose of $1 \cdot 10^{15}$ cm^{-2}, requires additional analysis. It may be that, together with the radiation-dynamic action of the beam, some thermal stimulation (heating) is required. Practically no heating under the pulsed irradiation regime used in this work had been applied.

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