Structure Of The Al$_{85}$Ni$_7$Fe$_4$La$_4$ Polycrystalline Alloy After Complex Impacts Of Deformation And Flash Lamp Annealing

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Abstract. On polycrystalline alloy Al$_{85}$Ni$_7$Fe$_4$La$_4$ the effectiveness of complex processing was shown, including intensive plastic deformation (IPD) by shear under pressure and flash lamp annealing (FLA), to improve the strength characteristics of cast aluminum alloys.

1. Introduction
An effective way to strengthen aluminum-based alloys is their alloying with slightly soluble elements, which, reacting with aluminum and other alloying elements, form intermetallics of various types. The last have a high hardness, which increases the strength characteristics of the total alloy. Formation of intermetallic compounds of various types during crystallization of alloys along with hardening leads to the material embrittlement, which reduces the efficiency of its use in real structures. To eliminate this disadvantage, it is necessary to disperse the reinforcing phases in the alloy. The efficiency of using such alloys is achieved by optimal combination of alloying and processing.

The objective of this study was to optimize the processing modes of cast aluminum alloy with a high content of transition and rare earth metals to obtain the structure of a multiphase nanocomposite with a high complex of mechanical properties.

2. Material and techniques
Polycrystalline hardened samples of Al$_{85}$Ni$_7$Fe$_4$La$_4$ alloy were subjected to severe plastic deformation (SPD) by shear under pressure $P=8$ GPA on Bridgman anvils. The movable anvil rotation angle was $\varphi = 360^\circ$. The samples obtained after IPD were subjected to flash lamp annealing (FLA) with fluence $E = 10$, $20$ and $25$ J/cm$^2$ under the conditions of gas-discharge lamps emission.

Samples for metallographic studies were prepared by grinding-and-polishing machine MECAPOL (MODEL P 230, manufacturer - PRESI S. A., France) in accordance with the guidelines of the company PRESI S.A. for surface treatment of metallic materials.

For metallographic tests, polished samples were etched in reagents: nitric acid (4 ml) + hydrochloric acid (3 ml) + hydrofluoric acid (3 ml) + water (90 ml), etching time up to 20 sec. This reagent causes staining of the phases present in the alloy in different colors, which enables their identification.

The structure of the alloy Al$_{85}$Ni$_7$Fe$_4$La$_4$ after various types of processing and "color" etching was studied by metallography on an inverted light microscope company "Carl Zeiss" Axiovert 40 Mat with
digital high resolution camera Canon PowerShot A640. The images of the structure were obtained with magnifications from 200 to 1000 times in the modes of light and dark fields.

The phase composition of the alloy was studied by X-ray diffraction: DRON-3M diffractometer (Russia) using Cu-Kα radiation (shooting mode: U = 38 kV, I = 20 mA, slit 1×2×0.5 + Soller slit system); ARL XTRA diffractometer, copper radiation, parallel beam geometry (parabolic mirror on the primary beam, thin - film collimator on the secondary beam). Phase analysis of the X-ray diffraction patterns was performed using PDF-2 data. The microhardness of the samples was measured on a 402 MVD – Wolpert Wilson Instruments microhardometer (Germany) at a load of 10 g and a loading time of 10 sec.

3. Results and its discussion

The literature review was held on the presence and types of intermetallic compounds, the allocation of which is possible in alloys based on aluminum with its content of more than 70 at. % alloyed with transition and rare earth metals. Such compounds - two, three and four – component were detected 16 [1].

Typical images of the alloy structure obtained by metallography are shown in Figure 1. Different color, contrast, morphology (shape, size) indicate the presence of four phases in the alloy, differing in composition. Light-field metallographic images of the tempered alloy structure (Figure 1a, b) supplemented with dark-field image (Figure 1c), which are formed by light scattered on the inhomogeneities of the structure, which gives an increase in contrast at the boundaries of phases, grains and twins. Dendritic crystallization of hypereutectic composition alloy begins with the selection of primary quaternary phase Al₈Fe₂ₓNiₓLa with the formation of a large number of defects such as twins (Figure 1). When solid aluminum solution crystallizes in the form of round cells, excess alloying elements are pushed to the cell boundaries, where the eutectic structure, which is characterized by the simultaneous growth of two or more phases, is formed. By methods of metallography and X-ray diffraction it was found, that the structure of the alloy is four- component. It is shown that in the alloy, along with a crystalline solid solution of aluminum, there are three types of intermetallic compounds: Al₄La (rhombic system), Al₈Fe₂ₓNiₓLa (rhombic system), Al₉Ni₂ₓFeₓ (monoclinic system) [2]. Since the investigated alloy contains a significant amount of iron (7 at.%), isolating of iron aluminate Al₁₁Fe₄ during crystallization is possible. On X-ray pictures the intensity peaks obtained by reflection from the specified phase are coincident with the maxima of reflection from other phases, which does not allow to state unambiguously that this phase is present in the alloy. Most intermetallic compounds have a size of more than one micron, some have a rack morphology (eg, lanthanum aluminate), which leads to embrittlement of the alloy after crystallization. The initial microhardness of the alloy is 216 HV.
One of the known ways to optimize such a structure is deformation processing, which is used in the work on the scheme of intense plastic deformation by shear under pressure (IPD). After IPD $\phi = 360^\circ$, a "vortex" structure is formed (Fig. 2), accompanied by dispersion, spheroidization and partial dissolution of intermetallic compounds [3].

The nanocrystalline structure, formed after IPD, characterized by a high concentration of defects, is thermodynamically unstable. Under high-energy influences, such as FLA, this structure undergoes changes: it develops a return and static recrystallization, and it is also possible to disintegrate the solid solution of aluminum with the precipitation of secondary intermetallides (Figure 3-5). The short-term effect of such a heat shock leads to increased dispersion of secondary precipitation without changing the phase composition, which is confirmed by the X-ray (Figure 6).
The broadening of peaks on diffractograms decreases, and their intensity increases. Secondary highly dispersed intermetallic discharges on dark-field images of the sample structure after complex exposure to IPD ($\varphi = 360^\circ$) and FLA with a fluence of 20 J/cm$^2$ are particularly pronounced (Figure 4). By coloring on light-field metallographic images, these secretions are identified as intermetallic phase Al$_{11}$La$_3$. Thus, the development of two competing processes - return, leading to softening, and dispersion hardening - provides a non-monotonic dependence of the microhardness of the deformed polycrystalline alloy on the FLA fluence. The microhardness growth in the range of values $E=10 \ldots 20$ J/cm$^2$ indicates the predominant role of dispersion hardening. The maximum value of microhardness 569 HV was obtained on samples that underwent complex treatment of IPD ($P = 8$ GPa, $\varphi = 360^\circ$) and FLA ($E = 20$ J/cm$^2$). The fluence increasing to 25 J/cm$^2$ stimulates the development of static processes of softening and coagulation of secondary precipitations of dispersed phases, which leads to a decrease of the hardness of the alloy (Table 1).

**Figure 3.** Alloy microstructure after IPD 8 GPA, $\varphi = 360^\circ$ followed by FLA 10 J/cm$^2$: light-field (a, b) and dark-field (c) images.

**Figure 4.** Alloy microstructure after IPD 8 GPA, $\varphi = 360^\circ$ followed by FLA 20 J/cm$^2$: light-field (a) and dark-field (b) images.

**Figure 5.** Alloy microstructure after IPD 8 GPA, $\varphi = 360^\circ$ followed by FLA 25 J/cm$^2$: light-field (a, b) and dark-field (c) images.
Figure 6. X-ray analysis of the alloy after IPD 8 GPa, φ = 360° + FLA: 10 J/cm² (a), 20 J/cm² (b), 25 J/cm² (c); 1 - Al, 2 - Al₃La₃, 3 - Al₆(Fe,Ni)₂, * - Al₃Ni, o - Al₃Fe₂Ni₃La.

Table 1. Microhardness of Al₈₅Ni₇Fe₄La₄ alloy in polycrystalline state after IPD of 8 GPa and subsequent FLA with energy doses of 10, 20 and 25 J/cm²

|         | without FLA | 10 J/cm² | 20 J/cm² | 25 J/cm² |
|---------|-------------|----------|----------|----------|
| HV      | 492         | 522      | 569      | 533      |

4. Conclusions

In order to improve the strength characteristics, the complex and separate effects of deformation (IPD 8 GPa and the full angle of the Bridgeman anvil twist 360°) and pulsed photon irradiation using xenon lamps (FLA with a fluence from 10 to 25 J/cm²) as an effective method of processing polycrystalline aluminum alloy (85 at.% Al) alloyed with Ni (7 at.%), Fe (4 at.%), and La (4 at.%). It was found that the complex treatment, including IPD + FLA (20 J/cm²), provides a maximum increase in microhardness to 569 HV and introduces additional hardening compared to the microhardness of the hardened alloy (216 HV) and its microhardness after IPD without IFO (492 HV).

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