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# Investigation of amorphous alloys nanostructure and phase composition formed during integrated and split exposure of deformation and flash lamp annealing

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**Abstract.** Microstructure and phase composition of the Al<sub>85</sub>Ni<sub>7</sub>Fe<sub>4</sub>La<sub>4</sub> amorphous alloy after separate and complex impacts of severe plastic deformation by shift under the pressure (SPD) with P=8 GPa, n=1-3 revolutions and flash lamp annealing (FLA) with the fluence F=10-25 J/cm<sup>2</sup> were investigated. By X-ray structural analysis and transmission electronic microscopy (TEM) was shown, that in initially amorphous structure partial multiphase crystallization with formation of amorphousnanocrystalline composite (solid solution of crystalline Al<sub>cr</sub> and amorphous Al<sub>am</sub> and Al<sub>11</sub>La<sub>3</sub>, Al<sub>9</sub>(Fe, Ni)<sub>2</sub> in aluminum) is developing during the SPD. FLA leads to partial nanocrystallization at F=10 J/cm<sup>2</sup> and full crystallization after the FLA at 20-25 J/cm<sup>2</sup>. Phase transformation and static recrystallization processes are developed along with the crystallization in amorphous structural component at SPD and FLA integrated exposure.

## 1. Introduction

Aluminum alloys obtained in amorphous condition demonstrate increased levels of strength, hardness, corrosion characteristics, high levels of tribological properties at low specific weight preservation – thus define interest in new compositions development and in treatment technologies. Research groups in Russia, USA, Japan and PRC are engaged in development of new technologies of creation of nanocrystalline materials and multiphase amorphous-nanocrystalline composites on the basis of amorphous alloys. The attention of researchers is focused on the problems of multicomponent aluminum alloys optimization – multicomponent aluminum alloys of “Al - transition metal - rare-earth element” system able to amorphize under conditions of high-speed quenching from the liquid state [1, 2]. Multicomponent amorphous alloys of the Al-Ni-Fe-La system are included in this class of materials and are investigated in the scopes of this article. Scientific achievement results of interrelationship between amorphous alloys structure and properties study are widely presented in the monograph [3]. Phase transformations and nanocrystalline structure formation trends in amorphous alloys of the Al-Ni-La system with silicon additives at flash lamp annealing (FLA) are investigated in scientific papers of academician Ievlev V.M. and his school [4, 5]. FLA of gas-discharge lamps (IFO) method allows energy localizing in the material near-surface region and reducing the heat treatment time. Researchers in Russian Federation have accumulated wide experimental material on the structural transformations study, mechanical properties changes; light exposure mechanisms on the material, the treatment effect dependence on the emission wavelength, pulse duration and strength were discussed [6]. In open sources there are no publications about systematic studies of aluminum-



based amorphous alloys nanocrystallization during simultaneous deformation and radiation treatment. Such studies are required to optimize amorphous alloys technological processing to obtain nanocomposite structure with advanced mechanical properties.

## 2. Material and techniques

Amorphous structure in  $\text{Al}_{85}\text{Ni}_7\text{Fe}_4\text{La}_4$  alloy in the samples in the form of the ribbons was obtained after the melt spinning method with the speed of  $10^6$  K/s. Amorphous ribbons were exposed to severe plastic deformation (SPD) by the shift under the 8 GPa pressure on the Bridgeman anvils. Rotation angle of the moveable anvil was equal to one or three full revolutions ( $\varphi=360^\circ$  and  $\varphi=360^\circ \times 3$ ). Amorphous ribbons after the high-speed quenching and SPD were exposed to flashed lamp annealing with the  $F=10, 20$  and  $25 \text{ J/cm}^2$  fluence in conditions of gas-discharge lamp radiation.

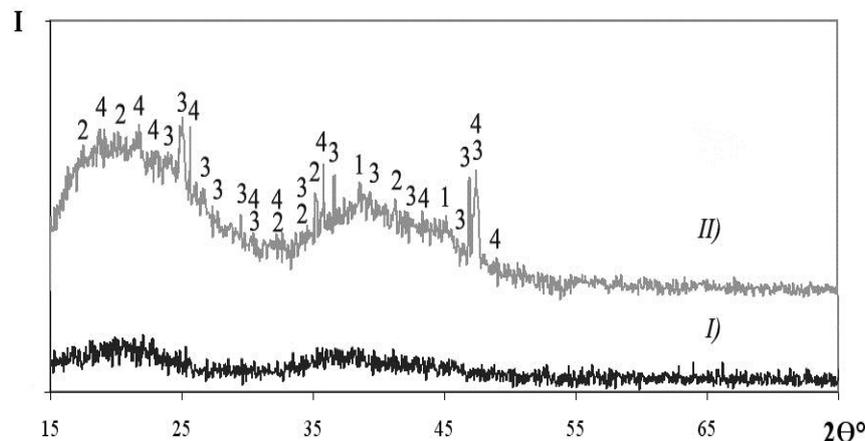
Alloy structure was investigated by X-Ray spectroscopy, DRON-3M diffractometer (Russia) with Cu-K $\alpha$  radiation (exposure mode:  $U=38 \text{ kV}$ ,  $I=20 \text{ mA}$ ,  $1 \times 2 \times 0.5$  slit + Sollers slit system) and ARL X'TRA diffractometer, copper radiation, geometry of a parallel beam (parabolic mirror on the primary beam, thin film collimator – on the secondary beam) were used. X-Ray pattern phase analysis was performed using PDF-2 data. Mechanical tests were performed on an Instron 5848 MicroTester machine at room temperature in pneumatic grippers with 0.01 and 10 mm/min tensile rates. Pneumatic grippers allow quick and continuous adjusting of the ribbons clamping forces and ensure reliable clamping of the samples. Working area of the test specimens length was 20 mm, the width of the ribbon is 1.1 mm. Samples microhardness was measured on 402 MVD Wolpert Wilson Instruments hardness-testing machine (Germany) at a load of 10 g, 10 s.

## 3. Results and its discussion

Structure, phase transformations and microhardness in the amorphous state and after the severe plastic deformation by a shift under the pressure 8 GPa were previously investigated by the authors [7] in amorphous alloys of the Al-Ni-Fe-La system. It was shown that nanocrystallization develops during plastic deformation in the structure of aluminum alloys with the formation of a multiphase amorphous-nanocrystalline structure including nanoscale grains of intermetallic compounds of various types and aluminum.

$\text{Al}_{85}\text{Ni}_7\text{Fe}_4\text{La}_4$  alloy diffraction pattern obtained by the X-ray diffraction method after the high-speed quenching and subsequent shift under the 8 GPa pressure with  $360^\circ$  torsion angle is shown in Figure 1. The  $\text{Al}_{85}\text{Ni}_7\text{Fe}_4\text{La}_4$  alloy after spinning has an X-Ray amorphous structure with a typical halo on the diffractogram (Figure 1, curve I) according to the data of X-ray structural analysis. SPD shift under the 8 GPa pressure with the deformation degree matching to one movable anvil full revolution ( $\varphi=360^\circ$ ) leads to partial crystallization development with formation of small amount of crystal aluminum grains and  $\text{Al}_{11}\text{La}_3$ ,  $\text{Al}_9(\text{Fe,Ni})_2$ ,  $\text{Al}_8\text{Ni}_x\text{Fe}_{1-x}\text{La}$  intermetallic compounds. Intensity peaks corresponding to these phases appear on the diffractogram (Figure 1, curve II). An amorphous-nanocrystalline structure with a small volume fraction of crystalline phases is formed in the alloy after the SPD with  $\varphi=360^\circ$ .

Quenched and deformed ribbons were subjected to pulsed photon processing with a fluence of 10, 20, 25  $\text{J/cm}^2$ . Multi-phase crystallization develops in the entire fluence parameters range of the FLA process both in quenched (initially amorphous) and in deformed (with  $\varphi=360^\circ$  and  $\varphi=360^\circ \times 3$ ) ribbons. Multiple intensity peaks are formed on diffractograms, corresponding to the phase composition  $\text{Al}_{kp} + \text{Al}_{11}\text{La}_3 + \text{Al}_9(\text{Fe,Ni})_2$ . Diffraction maximum presence at  $2\Theta=36,09^\circ$  with an intensity of 100% does not exclude the presence of the  $\text{Al}_8\text{Fe}_{2-x}\text{Ni}_x\text{La}$  phase in the structure. Lesser intensity lines for the indicated intermetallide coincide with the diffraction maxima of the other phases. The intermetallic compound  $\text{Al}_8\text{Fe}_{2-x}\text{Ni}_x\text{La}$  ( $\text{Al}_8\text{Fe}_2\text{Eu}$  prototype) was first discovered and certified by the authors in [8]. This phase refers to a rhombic system (*Pbam* space group) with lattice parameters  $a=1.258(3) \text{ nm}$ ,  $b=1.448(3) \text{ nm}$ ,  $c=0.405(8)$ .



**Figure 1.** Alloy  $Al_{85}Ni_7Fe_4La_4$  diffraction pattern: I) amorphous condition, II) SPD 8 GPa,  $\varphi=360^\circ$ . Symbols: 1 – Al, 2 -  $Al_{11}La_3$ , 3 -  $Al_9(Fe,Ni)_2$ , 4 -  $Al_8Ni_xFe_{2-x}La$ .

SPD and FLA integrated exposure does not lead to noticeable changes in diffraction patterns. Halo is partially retained after the SPD with  $\varphi=360^\circ$  and FLA with a fluence of  $10 J/cm^2$  on the diffractogram which indicates that crystallization is incomplete. Volume ratio of uncrystallized amorphous matrix decreases with fluence magnitude growth; the halo on the diffractograms disappears completely after the  $F=25 J/cm^2$  fluence. Alloy phase composition is a mixture of crystalline phases: an aluminum based solid solution and  $Al_{11}La_3$ ,  $Al_9(Fe,Ni)_2$  intermetallic compounds. Deformation degree increase to  $\varphi=360^\circ \times 3$  under the integrated exposure of SPD and FLA does not significantly affect the phase transformations regular pattern. Reflections intensity from the crystalline aluminum decreases and reflexes intensity from intermetallic phases increases as the fluence increases at FLA. The effect of intensity redistribution between the different phases reflexes is associated with volume ratio of aluminum based crystalline solid solution decrease and intermetallic phases volume increase.

Mechanical tensile testing results of amorphous ribbons in the initial state at room temperature with tensile rates 0.01-10 mm/min are presented in Table 1. For comparison the mechanical properties of industrial cast aluminum alloys are shown in Table 2.

**Table 1.**  $Al_{85}Ni_7Fe_4La_4$  amorphous alloy tensioning mechanical tests results

Alloy	Speed of the test, mm/min	Tensile strength, MPa
$Al_{85}Ni_7Fe_4La_4$	0.1	384
	10	553

In all test modes the amorphous alloys yield strength is equal to the tensile strength which is stipulated by the strain hardening absence during the strain process. It was established that with deformation rate increase from 0.1 to 10 mm/min the tensile strength increases from 384 to 553 MPa (Table 1). Similar results concerning the effect of deformation rate on the amorphous ribbons tensile strength were obtained and described earlier in [9]. As noted by the author this dependence is typical for the homogeneous deformation realization in amorphous alloys, which, apparently, is also observed in the ribbons studied after uniaxial tension. The obtained mechanical characteristics are close to the tensile strength values of Al-Cu based industrial high-strength alloys utilized in aircraft manufacturing (VAL10, VAL12, Table 2).

The tensile test method applied by the authors like most of the modern mechanical testing methods does not allow complete mechanical properties characterization of the new amorphous high-strength materials due to the small sizes of ribbon samples and especially of the samples exposed to shift under

the pressure. Various indentation methods are widely used to determine and characterize mechanical properties of such materials.

**Table 2.** Results of industrial casting alloys mechanical tests [10]

Alloys and their assignment	Grade	Tensile strength, MPa
Al (~85 %)-Si based high-technology alloys doped with Mg, Ti, Cu, Mn, Fe. For production of modular castings	AL9-1	320
	V 124	400
Al-Cu based high-strength alloys doped with Cu, Mn, Ti, Zn, Mg, Cd, Zr, Fe, Si. For castings production (aircraft industry)	VAL10	420
	VAL12	550
Al-Cu-Ni based high-temperature alloys doped with Cu, Ni, Mn, Ti, Zr, Ce, Mg, Fe, Si, works at 200-250 °C	AL33	280
	VAL18	300

Original amorphous structure hardness of the  $Al_{85}Ni_7Fe_4La_4$  alloy of the amorphous ribbon is 406 HV. The maximum hardness values 659 and 651 HV were obtained on an amorphous  $Al_{85}Ni_7Fe_4La_4$  alloy after the FLA with a fluence of 10 and 20 J/cm<sup>2</sup> respectively.

The results of  $Al_{85}Ni_7Fe_4La_4$  amorphous alloy samples microhardness measuring after the SPD and FLA integrated exposure depending on the radiation dose demonstrated that the sample hardness after deformation increased with twist angle rotation increasing from 406 HV in the amorphous state to 580 HV and 619 HV after the SPD with  $\varphi=360^\circ$  and  $\varphi=360^\circ \times 3$  respectively. However, after irradiation this indicator decreased in samples with a large twist angle ( $\varphi=360^\circ \times 3$ ) reaching the minimum value of 495 HV after treatment with 20 J/cm<sup>2</sup> fluence. The most probable cause of hardness decrease is the static recrystallization development at thermal exposure on a sample during FLA as a result of FLA. Microhardness gradually increased and reached 653 HV value at energy dose increase to 20 J/cm<sup>2</sup> for the samples subjected to SPD exposure with  $P=8\text{GPa}$ ,  $\varphi=360^\circ$ . The microhardness values of both series of the samples turned out to be approximately the same (within the measurement error limits) after FLA with the maximum energy dose of 25 J/cm<sup>2</sup>.

#### 4. Conclusions

Separate and complex SPD and FLA exposure of amorphous ribbons processing methods leads to solid-phase crystallization processes development and phase transformations in the  $Al_{85}Ni_7Fe_4La_4$  amorphous alloy with the multiphase composite structure formation. Volumetric fraction of crystallization development and the grain size depend on the FLA (fluence 10...25 J/cm<sup>2</sup>) and SPD ( $\varphi=360^\circ \dots 360^\circ \times 3$ ) parameters. With these characteristics increase the crystallization volume fraction increases. Gradient structure is formed over the ribbon cross section at FLA as against to the deformation processing; this allows creating of high-strength coatings of the given thickness on the processed products surface.

The maximum microhardness of 659 and 651 HV was obtained after FLA with fluences 10-20 J/cm<sup>2</sup> resulted in multiphase nanocrystalline structure formation at the alloy which included aluminum-based solid solution ( $Al_{cr}$ ) and  $Al_{11}La_3 + Al_9(Fe,Ni)_2$  intermetallic compounds.

Integrated exposure including SPD (8 GPa with  $\varphi=360^\circ$ ) + FLA (with 20 J/cm<sup>2</sup> fluence) provides 653 HV microhardness and does not introduce additional hardening as compared to an amorphous alloy after FLA.

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