Flash lamp annealing of amorphous Al-Fe-Ni-La alloys

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Abstract. Multiphase nanocomposite gradient structure formed by Flashed Lamp Annealing (FLA) in aluminum amorphous alloys (Al=85 at. %) alloyed with transition (Ni=5÷7 at. %, Fe=4÷7 at. %) and rare-earth (La=3÷4 at. %) elements. FLA critical parameters when crystallization at subsurface layers of amorphous ribbons at the radiation treatment side starts are defined (fluence F=10 J/cm²; duration τ~0,5 sec). It is shown that alloy composition alteration within alloying elements content does not have significant influence on FLA critical parameters. Through-thickness crystallization of the amorphous ribbons (25-micron thickness) was observed after FLA with F≥20 J/cm². Hardness of alloys after FLA with different parameters was determined. The phase composition of alloys was analyzed after the different FLA treatment.

1. Introduction

The analysis of numerous modern works shows that a promising direction of the new aluminum-based alloys development is their alloying with transition and rare earth metals [1-3]. High-strength conditions formation in aluminum alloys due to the large number of multicomponent intermetallic phases of various types in the nanoscale size range formation is provided by such alloys fast crystallization and their subsequent processing under various technological schemes [4]. The amorphization of the alloys by high-speed quenching from the liquid state with subsequent photon treatment under the conditions of Flashed Lamp Annealing (FLA) is one of the production methods of multiphase nanocomposites with controlled phase composition and grain size.

2. Material and techniques

The melt spinning method was used to produce amorphous ribbons from aluminum-based (85 at. %) melt doped with transitional Ni=5÷7 at. %, Fe=4÷7 at. %) and rare-earth (La=3÷4 at. %) elements. Polycrystalline alloys of the same compositions are obtained by quenching from the melt under cooling conditions close to equilibrium (in air). The alloys structure in the initial condition and after the FLA under parameters - fluence F=10-30 J/cm², processing time τ=0,5…1,5 sec - was analyzed using transmission electron microscopy (TEM) with energy dispersive X-Ray spectroscopy (EDXS) and X-ray diffraction (XRD). The specimens were studied in a Titan 80-300 TEM/STEM (FEI, USA) electron microscope with spherical aberration probe, equipped with an EDXS (EDAX, USA); ARL XTRA X-ray diffractometer (radiation of copper). Polycrystalline specimens were subjected to shear deformation under the 8 GPa pressure (IPD) on Bridgman anvils before the FLA; deformation degree corresponded to one movable anvil turn φ=360°. Mechanical properties were estimated by the results.
of microhardness measurement on the 401/402 – HVD Wolpert Wilson Instruments hardness-testing machine at a load of 10 g, 10 s. Differential scanning calorimetry (DSC) of the alloy was carried out on the SETARAM calorimeter with a heating/cooling rate of 20°/min.

3. Results and discussion

3.1. The samples after formation

Alloys Al_{83}Ni_{7}Fe_{7}La_{3} and Al_{83}Ni_{7}Fe_{4}La_{4} in the initial state have an amorphous structure, which is characterized by an asymmetrical double-humped halo. The initial microhardness of alloys Al_{83}Ni_{7}Fe_{7}La_{3} and Al_{83}Ni_{7}Fe_{4}La_{4} is 456 HV and 406 HV respectively.

Table 1. Results of DSC analysis

|                  | Al_{83}Ni_{7}Fe_{7}La_{3} | Al_{83}Ni_{7}Fe_{4}La_{4} |
|------------------|----------------------------|----------------------------|
|                  | P_{1}                       | P_{2}                       | P_{3}                       | P_{1} | P_{2} |
| T_{xi}, °C       | 282                        | 286                        | 399                        | 295   | 381  |
| T_{pi}, °C       | 305                        | 364                        | 399                        | 295   | 381  |
| H, μV.s/mg       | 5.4                        | 14.0                       | 12.0                       | 15.0  |

H – thermal effect.
T_{x} – crystallization start temperature.
T_{p} – crystallization peak temperature.
1, 2, 3 – crystallization stages corresponding indexes.

The results of DSC studies of the initial amorphous alloy Al_{83}Ni_{7}Fe_{7}La_{3} are presented in the Table 1. Three exothermical peaks corresponding to multiphase crystallization of the amorphous matrix (P_{1}-P_{3} peaks) were observed during heating. The crystallization started form Al at a temperature of 282 °C, then intermetallic compounds were crystallized. Two complex DSC peaks are responsible for intermetallics crystallallization. Two asymmetrical peaks are associated with two-staged crystallization during continuous heating of an amorphous Al_{83}Ni_{7}Fe_{4}La_{4} alloy. The aluminum is crystallized together with intermetallic compounds from the alloy at the first crystallization stage.

Multistage crystallization development during heating of the alloys is associated with their multi-component doping. Al_{83}Ni_{7}Fe_{7}La_{3} and Al_{83}Ni_{7}Fe_{4}La_{4} alloys crystallization is completed at 454°C and 406°C respectively.

3.2. Al_{83}Ni_{7}Fe_{7}La_{3} alloy structure after FLA

Amorphous ribbons after the high-speed hardening undergo FLA in order to find optimal irradiation modes to obtain nanocrystalline composite with the enhanced properties. FLA was carried out at the scientific equipment shared utilization center, Voronezh State University.

Again, the XRD and TEM were used for structural analysis of the Al_{83}Ni_{7}Fe_{7}La_{3} alloy after FLA with different radiation doses. After irradiation at F=10 J/cm² any significant changes in XRD spectra were not found. The peaks from crystalline compound on XRD spectra were observed after the F=15 J/cm² annealing and they are associated with Al. The crystallization occurs in the area close to the surface. According to TEM data, the volume fraction of crystalline material is low and that explains the low intensity of the peaks of the XRD spectra. The halos presence on XRD and electron diffraction (ED) patterns indicate that the main structural component is a solid aluminum solution in the amorphous state. The crystallization penetration in the deep of the ribbon is low.

An amorphous nanocrystalline structure with a grain size of ~ 10 nm is formed in the surface layer of the alloy after the FLA with a dose of 15 J/cm². The phase composition of the amorphous-nanocrystalline composite includes amorphous, crystalline Al based solid solution and intermetallic compounds. TEM indicated the presence of nanocrystalline phases, which should have a significant
impact on the mechanical properties of the sample. At these conditions the crystallization is not complete. The intermetallic compounds as Al\textsubscript{11}La\textsubscript{3} and Al\textsubscript{9}Fe\textsubscript{2-x}Ni\textsubscript{x} [5] were detected by TEM and ED in the sample after the FLA with F=15 J/cm\textsuperscript{2}. The nanocrystallization undergoes on one side of the ribbon (facing the energy source) at low radiation doses allows to create nanocrystalline coatings on the surface of amorphous ribbons, to regulate the ratio of the volume fractions of the crystalline and amorphous components, which will probably determine the mechanical properties of the nanocomposite after processing.

Multiphase crystallization over the ribbon section was observed by TEM after the FLA at F=20 J/cm\textsuperscript{2}. The rest of an amorphous material along with the crystalline phases was found by XRD in the form of on diffraction patterns. Main crystal grains sizes is 130 nm. The grains adopted the faceted shape. The major fraction is crystaline Al together with Al\textsubscript{9}Fe\textsubscript{2-x}Ni\textsubscript{x} compound.

Figure 1. Al\textsubscript{85}Ni\textsubscript{5}Fe\textsubscript{7}La\textsubscript{3} alloy microhardness after Flashed Lamp Annealing.

The microstructure of the alloys changes significantly after FLA with F=25-30 J/cm\textsuperscript{2}. The average grain size increases and the morphology of particles changes. Both XRD and ED demonstrated patterns which are associated with crystalline Al, Al\textsubscript{11}La\textsubscript{3} and Al\textsubscript{9}Fe\textsubscript{2-x}Ni\textsubscript{x} compounds. The amorphous phase in the alloys were not found. The intensity redistribution from crystal phases was observed on XRD patterns in the samples after maximum FLA doses. The intensity of Al peaks decreases and Al\textsubscript{11}La\textsubscript{3} and Al\textsubscript{9}Fe\textsubscript{2-x}Ni\textsubscript{x} intermetallic compounds increases. TEM images demonstrated that major phase is the Al\textsubscript{9}Fe\textsubscript{2-x}Ni\textsubscript{x} adopted elongated morphology with the particle size of up to 1 \textmu m. The volume fraction of Al\textsubscript{11}La\textsubscript{3} compound are increased drastically. TEM images of these particles pointed that Al\textsubscript{11}La\textsubscript{3} particles exhibited elongated cylindrical morphology with the aspect ratio of 2:1. The rounded morphology was associated with annealing at energy close to 30 J/cm\textsuperscript{2}. The secondary equiaxed Al\textsubscript{11}La\textsubscript{3} particles were observed inside the volume the Al\textsubscript{9}Fe\textsubscript{2-x}Ni\textsubscript{x} particles. Their size is by an order of magnitude less than the grain size of the primary particles. Thus, transparent multi-phase crystallization in the amorphous ribbons with a thickness of 25 micron was observed after the FLA in the range of treatment parameters F 20…30 J/cm\textsuperscript{2} and \tau=1…1.5 sec [6].

The maximum ribbons microhardness was obtained after the FLA with F=10 J/cm\textsuperscript{2}, which exceeds the microhardness in the initial after the hardening amorphous state by ~30% (figure 1). The further
increase of F up to the 15-30 J/cm² leads to a gradual microhardness decrease. Microhardness maximum ratings are in consistence with the multiphase amorphous nanocrystalline structure incorporating amorphous and crystalline solid aluminum solutions along with the three-component intermetallic compounds with the grain sizes not exceeding ~130 nm. Fully crystallized structure with irregular grain morphology after the FLA with the 25 and 30 J/cm² is associated with the multiple phase presence.

Noticeable diffraction maxima due to the multiphase crystallization development during processing were observed the XRD spectra of the amorphous alloy Al₁₈Ni₁₅Fe₄La₄ after the FLA with F=25 J/cm². Along with the intense maxima (111) and (200) Al, peaks belonging to the intermetallic phases. Multiple supersaturation of the amorphous phase by Ni, Fe, La alloying elements leads to multiphase crystallization with the double and triple aluminides crystallization during FLA, and this significantly complicates the unambiguous phases identification by XRD.

Figure 2. The XRD Al₁₈Ni₁₅Fe₄La₄ spectra after the FLA with energy dose of 25 J/cm²:

a) initial condition – amorphous;

b) polycrystalline initial condition after the IPD P=8 GPa, φ=360°.

Notation: 1- Al, 2 - Al₁₁La₃, 3 - Al₁₁Fe₂₃Ni₈.

Halo indicating the presence of an amorphous component was not found by XRD (figure 2a). Transparent multiphase crystallization develops throughout the whole sample volume after the FLA with F=25 J/cm². Alloy phase composition includes crystalline aluminum and Al₁₁Fe₂₃Ni₈, Al₁₁La₃ intermetallic compounds. The microhardness of such a structure is 556 HV.

Polycrystalline alloy Al₁₈Ni₁₅Fe₄La₄ FLA with F=25 J/cm² after IPD 8 GPa with φ=360° leads to the phase transformations development with Al₁₁La₃ and Al₁₁Fe₂₃Ni₈ intermetallic compounds crystallization. Diffraction maxima formed by crystalline aluminum planes (111) and (200) reflection has the maximum intensity on X-ray diffractograms. The microhardness of the samples after the FLA with F=25 J/cm² (IPD 8 GPa with φ=360°) was 533 HV which is much lower than the microhardness of the amorphous-nanocrystalline alloys after the FLA.
4. Conclusions
Obtained results are important at utilization of high-strength aluminum alloys in the amorphous state to produce amorphous nanocrystalline multiphase composites with high strength characteristics FLA as a processing method is promising for the nanocrystalline coatings, gradient and nanocomposite structures creation in the products made of amorphous alloys.

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5. References
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