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Effect of treatment conditions on parameters of nanocrystalline structure in Al-based alloys

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ABSTRACT

Formation of aluminum nanocrystals in light $Al_{90}Y_{10}$ and $Al_{87}Ni_8Gd_5$ amorphous alloys at heating and deformation was studied by X-ray diffraction and electron microscopy methods. High pressure torsion and multiply rolling were used for the deformation influences. The average size of the nanocrystals induced by deformation was found to be lower than that formed during the heat treatment. Combined treatment (deformation + annealing) leads to the formation of nanocrystals of intermediate size. Increasing the deformation degree leads to an increase in the fraction of the nanocrystalline phase. The dependence of the microhardness of amorphous - nanocrystalline structure on the deformation degree was measured. Increasing the deformation degree (and thus rising fraction of the nanocrystalline phase) increases the microhardness of the alloys. The value of the microhardness, close to a record for light Al-based alloys is obtained for amorphous-nanocrystalline $Al_{87}Ni_8Gd_5$ alloy.

Amorphous aluminum-based alloys have a high strength with a relatively low specific weight. The amorphous structure is known to be unstable. When heated, the transition to a more stable state takes place and nanocrystals form and grow. The formation and growth of nanocrystals occurs by primary crystallization reaction leading to Al nanocrystal formation. The amorphous phase changes chemical composition, it becomes enriched with components that are not soluble in aluminum. Nanocrystallization process in light amorphous alloys was studied in a lot of papers [1–5]. The nanocrystal formation leads to increased strength characteristics. Record values of microhardness [2] and hence the yield strength (1.6 GPa) were obtained for Al-based alloy. A specific weight of this alloy is only 3.3 g/cm^3 [2,6]. The values of strength and microhardness are more typical for of structural steel, but its density is greater than about 2.5 times. An increase of nanocrystalline phase fraction leads to enhancing strength [7], however the dependence of the strength on the nanocrystals size is non-monotonic [8]. It reaches a maximum at a certain critical size of the nanocrystals. Therefore it would be very tempting to find out how, firstly, increase the proportion of the nanocrystalline phase, and secondly, to create nanocrystals close to the critical size.

Traditionally, formation and growth of nanocrystals in these alloys occurs at heating or annealing. The maximum proportion of the nanocrystals is determined by the alloy composition and it is about 20–30% [9]. Recently an alternative way to create a nanocrystalline phase in amorphous alloys was developed: deformation of amorphous phase was used instead of thermal treatment. In this case, the nanocrystals induced by deformation are formed [10–12]. The nanostructures formed by the deformation of amorphous alloys, have other characteristics compared with nanostructures formed at heat treatments [13–17]. Firstly, the proportion of the nanocrystalline phase induced by deformation may exceed the maximum share of the nanocrystals formed during heating. Secondly, more dispersed nanostructure form at the deformation [16,17]. Currently, there are no data on strength property measurements and their dependence on the proportion of the nanocrystalline phase in amorphous-nanocrystalline light alloys. Therefore, it is important to identify ways of creating materials with different sized nanocrystals (with the same volume fraction of them), as well as of increasing the maximum share of the nanocrystals. This paper is devoted to comparative study of the structure of amorphous-nanocrystalline $Al_{90}Y_{10}$ and $Al_{87}Ni_8Gd_5$ alloys, formed by deformation, by heat treatment and by combinations thereof, as well as to the study of strength characteristics.

The amorphous alloys as ribbons were prepared by melt spinning; the ribbon thickness was 50–70 μm. As-prepared samples were amorphous; x-ray diffraction patterns and electron microscopic images contain no signs of crystalline phases. Nanocrystallization was carried out in several ways. The samples were heated in the calorimeter with a constant rate (20 K/min) to the temperatures corresponding to crystallization finishing (510 K for $Al_{90}Y_{10}$ alloy and 560 K for $Al_{87}Ni_8Gd_5$ alloys). The temperatures of the nanocrystallization start were determined by differential scanning calorimetry (Perkin-Elmer system). Deformation induced

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nanocrystallization was carried out by high pressure torsion (HPT) method with a pressure of 4 GPa. Deformation was carried out at room temperature, value of the deformation was 0.1–5 revs. The diameter of the samples for HPT was 8 mm; foils for the electron microscopy investigations were prepared from areas in the mid-range of the sample. Structure of the samples was studied by X-ray diffraction (Siemens D500) and transmission electron microscopy (JEM 2100 и JEM-100CX). Microhardness was measured with a Micro-Vickers hardness tester with a load of 10 g. A print size was less than 10 μm.

The nanocrystal size was determined by X-ray diffraction data as well as by dark field electron microscopy images. The nanocrystal size was estimated by the halfwidth of the diffraction line using the known Scherrer formula [18]:

$$L = \lambda(1/\cos\theta)/\Delta(2\theta) \quad (1)$$

where L is the nanocrystal size, λ the radiation wavelength, θ the angle of reflection, Δ(2θ) the halfwidth of the corresponding reflection. Since the x-ray diffraction patterns of nanocrystalline materials exhibit a small halfwidth of diffraction reflection, instrumental broadening may be neglected. The fraction of the nanocrystalline phase was estimated by the ratio of the integral intensities of the reflections in the x-ray diffraction patterns [19].

Fig. 1a shows the structure of the sample $Al_{87}Ni_8Gd_5$ after HPT. The structure consists of Al nanocrystals dispersed in an amorphous matrix. Fig. 1b display the dark field TEM image and electron diffraction pattern of amorphous-nanocrystalline structure of $Al_{90}Y_{10}$ alloy after HPT. The nanocrystals are seen to be uniformly distributed in the amorphous matrix. Fig. 1c shows the structure of $Al_{90}Y_{10}$ alloy after nanocrystallization by heating to the temperature of the crystallization end (513 K). According to preliminary DSC studies, this temperature corresponds to the end of the stage of Al nanocrystal formation. In accordance with the results obtained **t**he volume fraction of nanocrystals produced by HPT is larger than the nanocrystalline structure fraction produced by heating. The nanocrystals formed upon heating are larger than that formed by deformation. The data on nanocrystal size and nanocrystal volume fraction after different treatments are given in Table 1.

Note the characteristic features of the structure of the samples.

– The nanocrystal size is much smaller in the nanostructures formed by HPT than that in the nanostructures formed during heating. Ultimate volume fraction of nanocrystals formed upon the deformation is larger than that formed upon heating. This fact is particularly pronounces in $Al_{90}Y_{10}$ alloy.

– The average size of nanocrystals induced by deformation does not change with increasing degree of deformation. This result applies to both the investigated alloys. It agrees well with previous data for alloys of different chemical composition [4,16,20].

Since the size of the nanocrystals after the deformation is substantially less than after heat treatment, a complex processing was performed to obtain an intermediate nanocrystals size. It consisted of 2 parts. First deformation was performed (cold rolling) followed by isothermal annealing. The degree of deformation at the rolling deformation was calculated using the formula:

$$\varepsilon = (h_0 - h_1)\cdot 100/h_0 \quad (2)$$

where h_0, h_1 are the thicknesses of as-prepared and deformed ribbons. The degree of deformation was 65%. The choice of the annealing temperature (450 K) was based on preliminary DSC

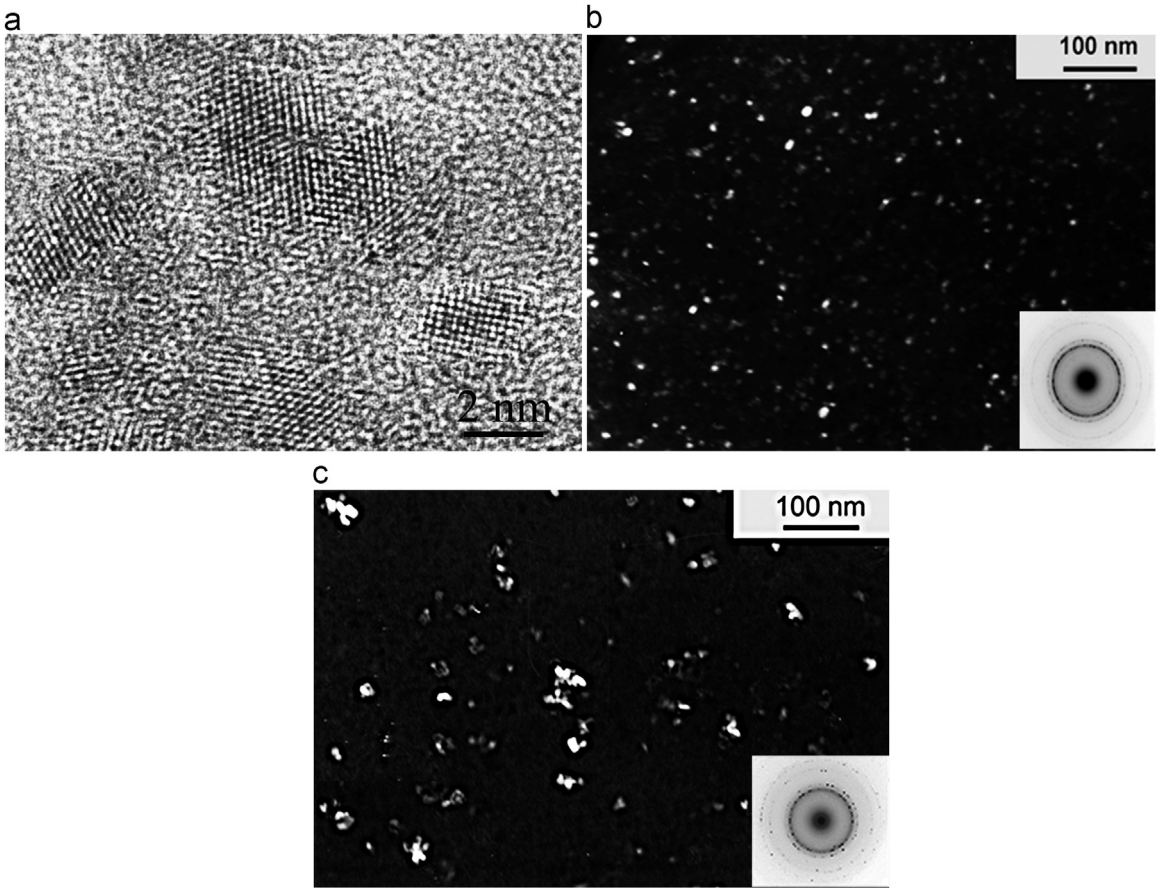

Fig. 1. Nanostructures in Al-based alloys: HREM image of amorphous $Al_{87}Ni_8Gd_5$ alloys after HPT for 5 rev. (a); dark field TEM image and electron diffraction pattern of amorphous $Al_{90}Y_{10}$ alloy after HPT for 2 rev, (b); dark field TEM image and electron diffraction pattern of amorphous $Al_{90}Y_{10}$ alloy after heating up to 510 K (c).

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