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Influence of pulse laser irradiation on structure and mechanical properties of amorphous $Fe_{73.1}Nb_3Cu_{1.0}Si_{15.5}B_{7.4}$ alloy

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ABSTRACT

Purpose: The aim of this work was to study structure changes in Fe-based amorphous ribbon under laser radiation, determine it dependence from laser treatment parameters and establish the correlation between structure and microhardness.
Design/methodology/approach: Amorphous ribbons of $Fe_{73.1}Nb_3Cu_{1.0}Si_{15.5}B_{7.4}$ alloy, obtained by rapid cooling from the melt, has been treated by pulsed laser radiation with wavelength $\lambda = 1.06$ μm and pulse duration $\tau = 130$ ns. Structure transformation has been studied by means of X-ray diffraction method, which allowed us to determine the phase composition, volume fraction and grain size of crystalline phases has been determined.
Findings: It has been shown, that laser treatment method allows forming an amorphous-nanocrystalline composite. It was found that microhardness of ribbon increases after irradiation and linearly depends on percent of crystalline phase.
Practical implications: Laser treatment can be used as an substitute of isothermal heat treatment to produce amorphous-nanocrystalline materials with improved properties.
Originality/value: The originality of this work is based on applying of pulse laser irradiation for modifying structure of amorphous $Fe_{73.1}Nb_3Cu_{1.0}Si_{15.5}B_{7.4}$ alloy.
Keywords: Amorphous alloy; Surface treatment; X-ray phase analysis; Mechanical properties

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1. Introduction

Laser treatment is a method which allows achieving improvement of the chemical, mechanical and other properties of the material surface [1-3]. There are several laser surface treatments, namely surface hardening, alloying, cladding and etc. Heating of amorphous materials using laser radiation leads to transformation of metastable amorphous phase into more stable crystalline phases. At real conditions the heating process deviates from equilibrium one but in most cases of treatment such deviation is assumed to be unsignificant [4]. On that reason it is of interest to study the such treatment as local heating on structure of amorphous alloys. Taking into account that at rapid local heating the diffusion processes, needed for creation of crystalline phase

are damped, one can suppose that new phases, formed in this way inherit the amorphous like short range order [5]. Such heating can be realized by means of laser irradiation. Using powerful focused laser radiation it is possible to obtaine the heating rates greater than 10^6 K/sec. Hence, the heating is local and essentially nonequilibrium that allowed us to suggest the formation of thermal waves, propagating over sample. In other words the temperature in any place of sample depends on time and position. On that reason, the influence of pulse laser irradiation on the structure and mechanical properties of amorphous ribbons $Fe_{73.1}Nb_{3.0}Cu_{1.0}Si_{15.5}B_{7.4}$ has studied in this work.

2. Experimental methodology

Amorphous ribbons of $Fe_{73.1}Nb_3Cu_{1.0}Si_{15.5}B_{7.4}$ alloy has obtained by rapid cooling from the melt [6]. The ribbon thickness was about 30 μm and width about 15 mm. To irradiate ribbons the laser equipment Laser Graver (produced by ALPHA R&M, Russia) has used. This laser radiated periodic pulses at the mode of a single pulse per microzone. Number of irradiated microzones per unit area was $N \sim 2000$ mm^{-2}, wavelength $\lambda = 1.06$ μm, laser pulse duration $\tau = 130$ ns and pulse generation frequency $f = 50$ kHz. During treatment radiation was focused on the ribbon surface in spot with radius $r \approx 15$ μm. The pulse energy W was varied within interval 0.13-0.25 mJ. Laser treatment has been carried out on the surface, which was opposite to contact one during amorphization process. Irradiation of initial amorphous sample was carried out for every value of pulse energy.

Structure of irradiated samples have been studied by means of X-ray diffraction method (XRD) using DRON-3 diffractometer (Fe-Kα radiation, $\lambda = 1.9373$ Å). By analyzing of diffraction patterns the phase composition, fraction of each phase (X) and the grain size (d) of crystalline phase has been determined.

Microhardness (H_V) of initial and irradiated samples has been determined using hardness measuring unit PMT-3 (LOMO, Russia). Measurements has been carried out at loading on sample 165 g and its duration 15 sec. Determination of microhardness was carried out from both sides of the ribbon based on 5–7 indenter prints with the following results averaging.

3. Results and discussions

3.1. Structure investigation

Results of X-ray analysis showed that crystallization process in amorphous ribbons after irradiation occurs and the degree of crystallinity depends on the pulse energy. Fig. 1 shows the diffraction curves for samples irradiated at different pulse energies.

Phase composition of products of crystallization for the sample irradiated at $W = 0.24$ mJ is presented in Table 1.

Comparing the peak's position ($d = 2.018691$ and 1.423980 Å) with diffraction patterns for the various phases that may exist upon crystallization, peak positions for the phase $Fe_{1.72}Si_{0.28}$ ht (Fe-Si, 14 at.% Si, $d_{(101)} = 2.017$ Å, $d_{(020)} = 1.426$ Å [7]) were found to be in best accordance than bbc-Fe or Fe_3Si that forms during isothermal annealing and heat treatment [8,9].

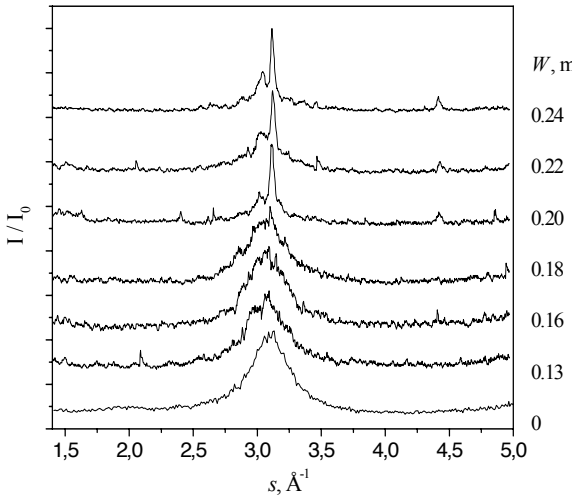


Fig. 1. Diffraction patterns of irradiated samples

Table 1.
Phase composition of crystallized phases for the sample irradiated at $W = 0.24$ mJ

No	s, Å^{-1}	d, Å	I/Io	hkl	Phase
1	2.550	2.463883	9.0	331	$Fe_{23}B_6$
2	2.630	2.388541	11.2	420	$Fe_{23}B_6$
3	2.663	2.359569	9.2	301	Fe_3B
4	2.877	2.184289	13.5	422	$Fe_{23}B_6$
5	3.011	2.087090	25.6	231	Fe_3B
6	3.051	2.059054	45.2	333	$Fe_{23}B_6$
7	3.113	2.018691	100.0	220	$Fe_{1.72}Si_{0.28}$
8	3.206	1.959556	13.5	112	Fe_3B
9	3.269	1.922068	12.1	440	$Fe_{23}B_6$
10	3.370	1.864513	9.0	411	Fe_3B
11	3.464	1.814007	9.0	531	$Fe_{23}B_6$
12	4.412	1.423980	19.2	400	$Fe_{1.72}Si_{0.28}$

Volume fractions of crystalline phases and grain sizes have been determined by analysis of diffraction curves, and they are shown in Figs. 2a and 2b.

As follows from X-ray analysis data, laser processing with pulse energy of about $W = 0.13$ mJ results in the formation of nanocrystalline phase in the amorphous matrix, the grain size and percent are $d = 7$ nm and $X = 17$ % respectively.

Structure of samples treated at $W = 0.16$ mJ, due to X-ray data, can be characterized as amorphous or quasi-amorphous. Irradiation with pulsed energy $W = 0.18$ mJ, led to the formation of nanocrystalline phase $X = 1 – 2$ % only. At the time, irradiation with lager pulse energy, $W = 0.2$ mJ, leads to complete

crystallization of the sample with forming of phase $Fe_{1.72}Si_{0.28}$ with average grain size about d = 60 nm, (X = 54%), Fe_3B with d = 40 nm (X = 7 %) and with $Fe_{23}B_6$ d = 9 nm (X = 39 %).

a)

b)

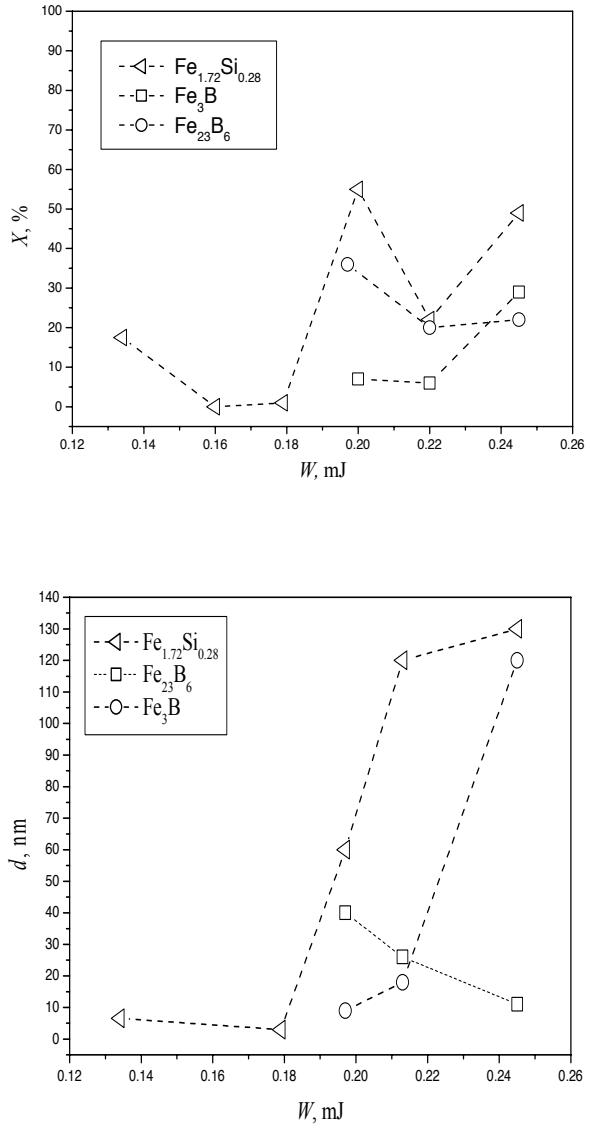


Fig. 2. Fraction of crystalline phases (a) and grain size (b) versus pulse energy

Irradiation at higher pulse energy W = 0.22 mJ resulted to smaller percentage of crystalline phases: phase $Fe_{1.72}Si_{0.28}$ with the grain size about d = 120 nm, and X = 22 % only, phase $Fe_{23}B_6$ with grain size about d = 18 nm, X = 20 %, and phase Fe_3B with grain size about d = 26 nm, X = 20 %. Irradiation with energy W = 0.24 mJ leads to complete crystallization of ribbon, but unlike the mode of W = 0.2 mJ observed slightly different phase fraction and size of grains. Phase composition in this case is 49% $Fe_{1.72}Si_{0.28}$ +22% $Fe_{23}B_6$ + 29% Fe_3B. Therefore, we can see two minimums in the percent of crystalline phases versus pulse energy. Reducing of the percent of crystalline phase with increasing of energy radiation also has observed after laser irradiation of Co-based amorphous ribbons [10]. We suppose, it can be due to next cause: at a certain level of power at the edge of irradiated area conditions may arise for the crystallization of the amorphous phase. As is state in [11,12], the increase of power range is followed by the increase in the size of the remelted area. So, at higher power level, subjected to short scanning step next pulse results in melting of zone that could be crystallized by previous pulse.

Due to the unusual nature of the crystallization of this sample, we further studied the opposite surface to irradiated one by X-ray analysis, (see Fig. 3 and Table 2).

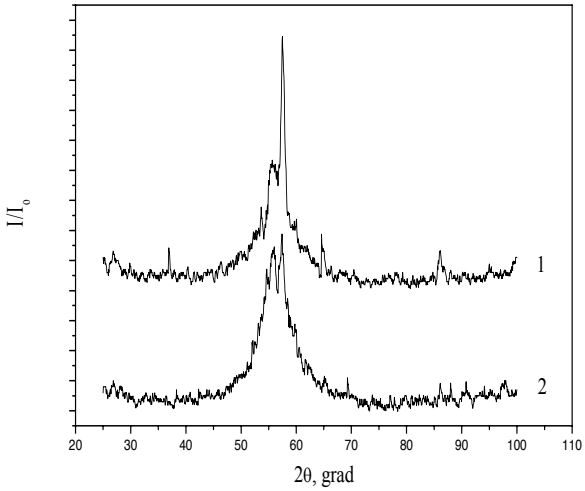


Fig. 3. Diffraction patterns for irradiated surface (1) and opposite surface (2), W = 0.22 mJ

Table 2.
Phase composition after crystallization for for the sample irradiated at W = 0.24 mJ

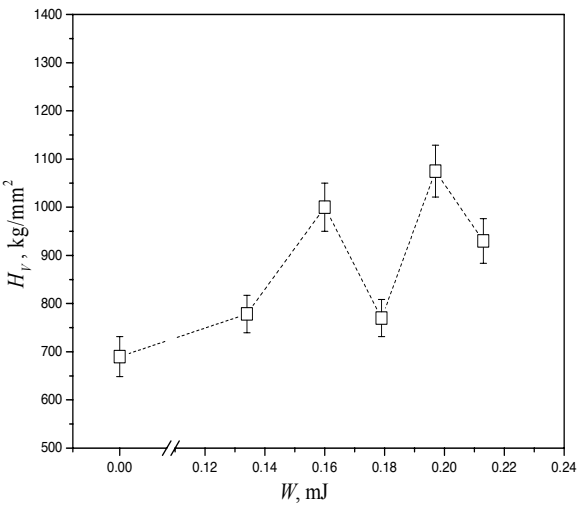
Surface	$Fe_{1.72}Si_{0.28}$		Fe_3B		$Fe_{23}B_6$	
	d, nm	X, %	d, nm	X, %	d, nm	X, %
Irradiated	120	22	26	6	18	20
Opposite	8.5	10.7	-	-	63	4.3

Degree of crystallinity of irradiated side is significantly higher than opposite one. There are also differences in the phase composition and their grain size. All this testify about formation of amorphous-nanocrystalline composite material.

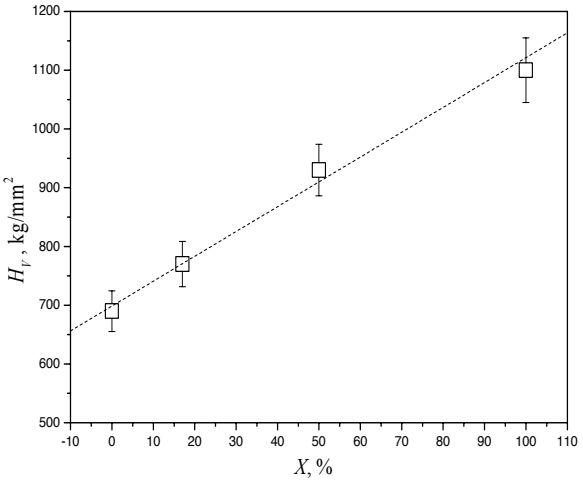
3.2. Microhardness measurements

Results of microhardness measurements of irradiated samples are presented on Fig. 4, where the microhardness versus pulse energy of irradiated surface (a) and microhardness versus crystalline phase's percent (b) shown.

a)



b)



b)

Fig. 4. Microhardness versus pulse energy (a), and microhardness versus crystalline phase's percent (b)

As can be seen on the Fig. 4a, microhardness nonlinearly increased after laser annealing. The comparison of diffraction data with microhardness behavior shows, that at slight differences in diffraction patterns for samples irradiated at W= 0.13, 0.16 and 0.18 mJ, the H_V values are more significantly different. Namely for the sample irradiated at W = 0.16 mJ, it is observed growth of microhardness up to H ~ 1000 kg/mm^2. The same nonlinear character with a peak of H_V value at 0.16 mJ has been observed for alloy $Fe_{75}Mo_{2,5}Co_{2,5}Si_6B_{14}$ that was treated at the same mode of laser radiation [13]. This may indicate a general phenomenon. On the other hand, according to data, presented in the literature [14], for similar material $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$, whose grain size is about d ~ 6 nm, microhardness is about H ~1400 kg/mm^2. Therefore, one can assume that irradiation with pulse energy 0.16 mJ results in nanocrystallisation process with size of grains about ~ 5 nm and they are difficalt to indicate due to special feature of the X-ray method.

Microhardness of the sample irradiated at W = 0.20 mJ significantly increased after exposure and is H = 1075 kg/mm^2, that, apparently, caused by complete crystallization of amorphous alloy. According to X-ray analysis (Fig. 2a), crystal phase content here is 100%, average grain size is about d ~ 38 nm. Thus, the main contribution to the H_V value makes crystal phase and grain boundaries. The next decrease of microhardness at W = 0.22 mJ is probably due to decrease of crystalline phases percent. The measurement of H_V of samples, irradiated at W= 0.24 mJ, failed due to their fragility. Notably, the ribbons, that were irradiated at W = 0.20 mJ and W = 0.22 mJ also characterized by increased fragility, as evidenced in the predisposition to the formation of cracks.

So, as shows experimental results, microhardness versus pulse energy, like crystalline phase percent versus pulse energy, has nonlinear character, that demonstrate microhardness dependence on structure state. On the Fig. 4b is presented microhardness versus total crystalline phase percent. As one can see, microhardness linearly increases with increasing of total percentage of crystalline phases.

4. Conclusions

Results, obtained using the method of X-ray diffraction analysis, showed that in initially amorphous Fe-based ribbons the crystallization processes occur after its laser irradiation. It has observed the formation of primary nanocrystals of $Fe_{1.72}Si_{0.28}$ phase. The concentration of crystalline phases $Fe_{1.72}Si_{0.28}$, Fe_3B and $Fe_{23}B_6$ nonlinearly depends on applied pulse energy. This can be due to that increasing of pulse energy leads to changes in spatial and temporal temperature distribution on the sample surface, and this resultes in uneven distribution of phases, their concentration and grain size.

It has been shown, that laser processing in mentioned mode leads to formation of amorphous-nanocrystalline composite (ANC). Combining spatial temporal parameters of the laser beam it is possible create two or more layers with different structure state.

It was shown also that microhardness depends nonlinearly on pulse energy, like fraction of crystalline phases, but linear dependence of microhardness from total percentage of crystalline phases was observed.

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