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STUDY OF SELECTED PHYSICAL AND STRUCTURAL PROPERTIES IN THE PROCESS OF PRIMARY CRYSTALLIZATION IN THE Fe-Cu-Si-B AMORPHOUS ALLOY

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K e y w o r d s: amorphous alloys, nanocrystalline alloys, structural parameters.

A b s t r a c t

According to literature data, the physical and mechanical properties of amorphous alloys depend on their chemical constitution. Further modification of the physical properties of these alloys may be achieved by proper heat treatment. The paper presents the results of experimental investigations of the amorphous alloy $Fe_{79,62}Cu_{0,38}Si_6B_{14}$ crystallized by isothermal annealing. The objective of the experiment was to determine changes in selected structural and mechanical parameters during primary crystallization of the alloy examined. Root-mean square (RMS) microstrains, sizes of coherent scattering blocks D, and the lattice constant of the crystallizing phase were used as parameters to evaluate structural changes in the material. The research was based mainly on X-ray diffraction measuring techniques, supported with an appropriate mathematical apparatus.

BADANIE ZMIAN WYBRANYCH WŁAŚCIWOŚCI FIZYKOSTRUKTURALNYCH W PROCESIE KRYSTALIZACJI PIERWOTNEJ STOPU AMORFICZNEGO TYPU FE-Cu-Si-B

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S ł o w a k l u c z o w e: stopy amorficzne, stopy nanokrystaliczne, parametry strukturalne.

Streszczenie

Z danych literaturowych wynika, że unikatowe właściwości fizykomechaniczne stopów amorficznych ściśle są związane z ich składem chemicznym. Dalsza ich modyfikacja jest możliwa przez odpowiednią obróbkę cieplną. W pracy przedstawiono rezultaty badań eksperymentalnych przeprowadzonych na stopie amorficznym $Fe_{79,62}Cu_{0,38}Si_6B_{14}$ poddanym obróbce cieplnej polegającej na izotermicznym wygrzewaniu w określonym przedziale czasowym i temperaturowym. Eksperyment miał na celu określenie zmian wybranych parametrów strukturalnych zachodzących w procesie krystalizacji pierwotnej badanego stopu amorficznego. Do oceny zmian struktury materiału wybrano parametry w postaci średniokwadratowych mikroodkształceń sieci (mikronaprężeń), wielkości bloków D rozpraszania koherentnego promieni rentgenowskich (krystalitów) oraz stałej sieciowej krystalizującej fazy. Badania głównie oparto na dyfrakcyjnych technikach pomiarowych wspartych odpowiednim aparatem matematycznym.

Introduction

Amorphous alloys belong to the group of modern engineering materials with unique mechanical and magnetic properties, including high resistance, hardness, magnetic permeability, and low coercive force. Materials characterized by desirable physical and mechanical properties can be obtained by proper selection of chemical components of the alloy. Further modification of the above properties, especially as regards magnetic applications, may be achieved by proper heat treatment. The structural changes taking place in amorphous alloys during annealing, and especially the crystallization process, have a significant effect on the physical and mechanical properties of the material.

Literature data indicate that amorphous alloy optimization is aimed first of all at obtaining possibly the best soft magnetic materials. This is connected with the fact that industrial amorphous alloys have the form of a thin film (to ~0.03 mm), which considerably limits their construction applications. The optimum magnetic properties can be obtained in the case of metallic glass, partly or fully crystallized, whose grain size does not exceed 100 nm. Such materials are known as nanocrystalline alloys.

Therefore, further investigations into the phenomena taking place in the structure of amorphous alloys subjected to crystallization may contribute to further optimization of the physical and mechanical properties of nanocrystalline alloys.

Subject of the study

The subject of the study was the amorphous alloy $Fe_{79,62}Cu_{0,38}Si_6B_{14}$ crystallized by isothermal annealing. The range of heat treatment temperatures was 693÷803 K (420–530°C), and time of annealing – 30, 60 and 120

min. The objective of the experiment was to determine changes in selected structural parameters in the form of root-mean square (RMS) microstrains $(<\varepsilon^2>)^{1/2}$, sizes of coherent scattering blocks D and the lattice constant of the crystallizing phase. These parameters were determined on the basis of X-ray diffraction measurements and analysis of changes in the intensity distribution of a diffracted beam of radiation, employing the method of harmonic analysis described by BRECZKO (1989).

Results

Preliminary studies, conducted on alloy samples, were aimed at determining its crystallization temperature. Figure 1 presents a diffraction pattern of the alloy sample after isothermal annealing at a temperature T_a=653 K (380°C) in time t=30 min. The visible reflection indicates the beginning of alloy crystallization. In order to identify and control the phase composition of material samples, diffraction patterns were made for each of them in an angle range of 2Θ = 35÷105° – Fig. 2. The annealing temperature range was a little wider this time – T_a= 693÷903 K (420–630°C).

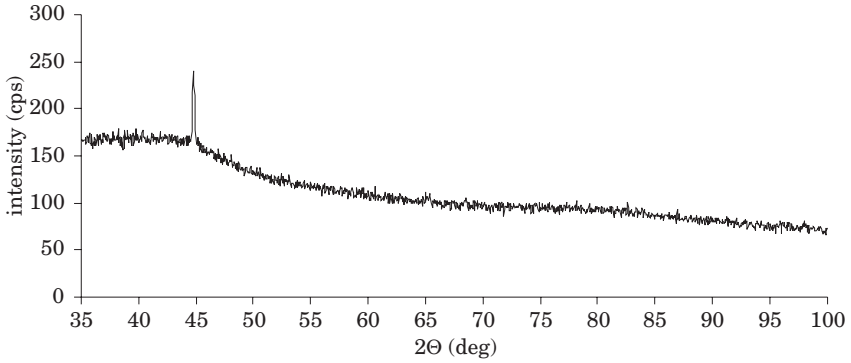

Fig. 1. Diffraction pattern of amorphous alloy
(annealing temperature T_a= 653 K (380°C), time of annealing t=30 min, CuKα)

The reflections in the diffraction pattern were identified as representing the ∝-Fe phase. Figure 3 shows changes in the values of the lattice constant a of the crystallizing phase as a function of annealing temperature Ta. An analysis of the above pattern suggests that in the case of lower annealing temperatures (693 and 753 K – 420–480°C) the value of the parameter a of the crystalline phase is lower than the lattice constant α-Fe (a=2.8663 Å). This is probably connected with the fact that at lower annealing temperatures (693 and 753 K – 420–480°C) the α-Fe(Si) phase is obtained. No other crystalline phases were found within the temperature range examined, so it

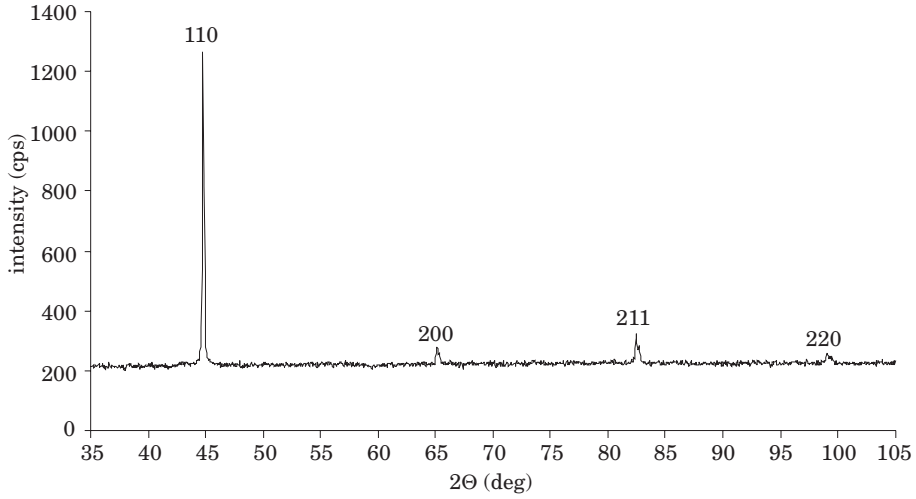

Fig. 2. Diffraction pattern of amorphous alloy (annealing temperature T_a= 803 K (530°C), time of annealing 120 min, *CuKα*)

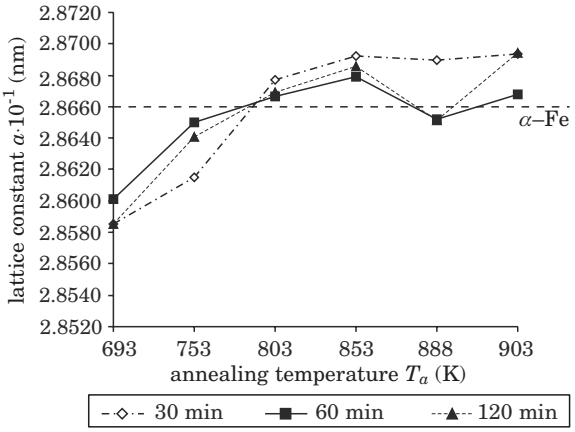

Fig. 3. Changes in the lattice constant a for the crystalline phase vs. annealing temperature T_a

can be assumed that the crystallization observed was of primary nature. As reported by ZOLOTUCHIN (1986), amorphous alloys containing less than 25% of non-metals crystallize primarily, i.e. in two stages. The first stage of crystallization was observed in the present experiment.

The next parameters determined in experimental investigations were microstrains of the crystal lattice and sizes of coherent scattering blocks D, estimated on the basis of selected diffraction lines (110) and (211). Figures 4 and 5 illustrate examples of changes in root mean-square microstrains of the lattice as a function of multiplicity of the base length L.

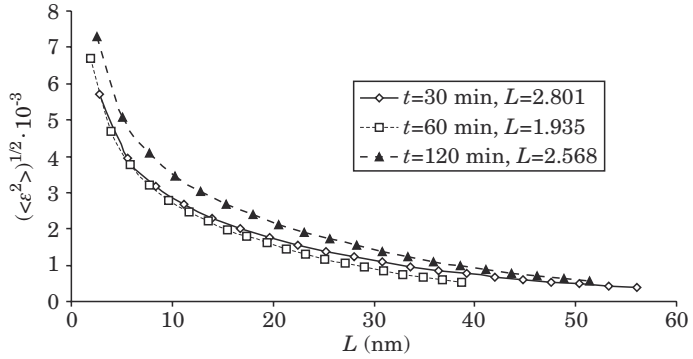

Fig. 4. Changes in RMS microstrains as a function of base length L after heat treatment (annealing) at a temperature 693 K (420°C). Time of annealing t = 30, 60 and 120 min

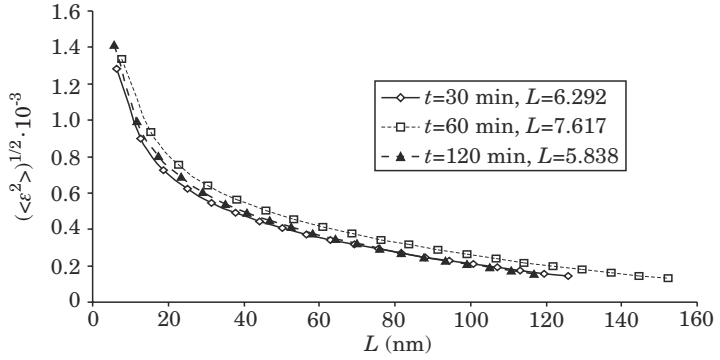

Fig. 5. Changes in RMS microstrains as a function of base length L after heat treatment (annealing) at a temperature 803 K (530°C). Time of annealing t = 30, 60 and 120 min

An analysis of these Figures shows that the highest level of microstrains was recorded in alloy samples annealed at a temperature T_a=693 K (420°C), and the lowest – in those annealed at T_a=803 K (530°C). The formation of microstrains in crystallizing amorphous alloys probably results from anisotropy of crystal growth. According to KULIK (1998), addition of Si to Fe should lead to growth anisotropy. Since measurements of the lattice constant a indicate the presence of the -Fe(Si) phase at annealing temperatures T_a=693 and 753 K (420–480°C), the highest microstrains can be observed at these temperatures.

Figure 6 shows changes in the sizes of coherent scattering blocks D as dependent upon the temperature of isothermal annealing T_a. Crystallite diameters of the order of 21÷29 nm were obtained after annealing at a temperature T_a=653 K (380°C). Based on the data presented by KULIK (1998), such a size of crystallites is adequate for magnetic applications of the nanocrystalline alloy obtained.

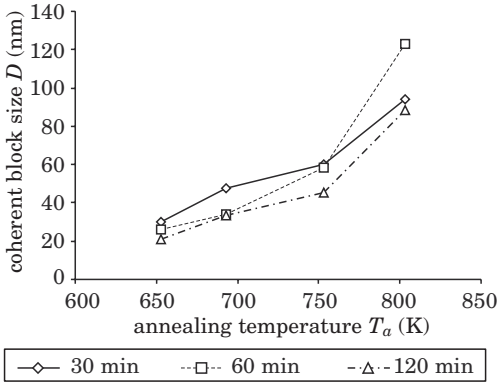

Fig. 6. Changes in the sizes of coherent scattering blocks D vs. annealing temperature T_a

Conclusions

The temperature of primary crystallization of the amorphous alloy $Fe_{79,62}Cu_{0,38}Si_6B_{14}$ during isothermal annealing at a time t=30 min is Tx = 653 K (380°C). In the present experiment the time of heat treatment t=30, 60 and 120 min. had no significant effect on changes in the structural parameters examined. It may be assumed that isothermal annealing at a temperature close to T_a=653 K (380°C) results in the formation of a nanocrystalline structure in an amorphous matrix, with the average size of coherent scattering blocks varying from 21 to 29 nm, depending on the time of annealing. The development of the nanocrystalline structure is accompanied by the formation of lattice microstrains, diminishing with an increase in the annealing temperature.

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