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Influence of structure on soft magnetic properties of $Co_{70}Fe_5Si_{15}B_{10}$ metallic glass ribbons

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ABSTRACT

Purpose: The paper presents a structural, thermal and magnetic characterization of Co-based amorphous samples in as-cast state and after annealing process.

Design/methodology/approach: The studies were performed on $Co_{70}Fe_5Si_{15}B_{10}$ metallic glass in form of ribbons with different thickness. The amorphous structure and phase analysis of studied samples after annealing process was examined by X-ray diffraction (XRD). The crystallization behaviour of the studied ribbons was also examined by differential scanning calorimetry (DSC). The fracture morphology of the ribbons after heat treatment was analysed using the scanning electron microscopy (SEM). The soft magnetic properties examination contained relative initial magnetic permeability and magnetic permeability relaxation measurements.

Findings: The X-ray diffraction investigations revealed that the tested ribbons with different thickness were amorphous. The two exothermic peaks describing crystallization process of $Co_{70}Fe_5Si_{15}B_{10}$ alloy were observed for all studied samples with different thickness. The heat treatment process of ribbon samples involved crystallization of α-Co(Si), Co_2Si phases and cobalt borides at temperature above 773 K. The study of fracture morphology of samples after annealing at 623 K shows mixed fractures with "river" patterns, which are characteristic for glassy materials and some areas of scaly morphology. The initial magnetic permeability decreases in function of the increase of annealing temperature, but a local maximum could be determine at 673 K.

Practical implications: The soft magnetic properties of studied metallic glasses can be formed by different sample thickness and applying the appropriate conditions of annealing process.

Originality/value: The applied investigation methods are suitable to determine the changes of structure and soft magnetic properties of examined Co-based alloy with function of sample thickness and heat treatment conditions.

Keywords: Amorphous materials; Co-based metallic glasses; Crystallization; Soft magnetic properties

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PROPERTIES

1. Introduction

Co-based amorphous alloys have been assumed as very important engineering materials due to their soft magnetic properties. Almost zero magnetostriction and low coercivity, high initial and maximal permeability allowed to attract much interest from the view point of their potential application in different elements like measuring systems, magnetic wires, magnetic sensors, band-pass filters, magnetic shielding, energy-saving electric power transformers and other applications [1-3].

In addition to the excellent soft magnetic properties of Co-based metallic glasses (Table 1), their properties could be formed by a heat treatment process. Structural changes in relation with magnetic properties of amorphous alloys can be often studied [4, 5].

Table 1.
Magnetic properties of the selected Co-based metallic glasses [13]

Glassy alloy	B_s, T	H_c, A/m	μ_r	λ_s
$Co_{70.3}Fe_{4.7}Si_{15}B_{10}$	0.8	0.48	8500	0
$Co_{75.3}Fe_{4.7}Si_4B_{16}$	1.1	1.2	20000	0
$Co_{65.7}Fe_{4.3}Si_{17}B_{13}$	0.53	0.48	55000	0
$Co_{61.6}Fe_{4.2}Ni_{4.2}Si_{10}B_{20}$	0.54	0.16	120000	0
$Co_{69.6}Fe_{4.6}Mo_{1.8}Si_8B_{16}$	0.63	-	10000	0
$Co_{72}Mn_4B_{24}$	0.95	0.4	26000	0
$Co_{81.5}Mn_{9.5}Zr_9$	0.73	0.24	21000	0

B_s – saturation induction, H_c – coercive force, μ_r – magnetic permeability, λ_s – magnetostriction

The soft magnetic properties of amorphous ferromagnetic alloys are generally improved by low-temperature annealing, which indicates relaxation of the atomic structure and a reduction of the internal stresses. The development of a crystalline structure by using high-temperature thermal treatment has generally caused the decrease of soft magnetic properties of amorphous alloys [5].

Annealing process of metallic glasses under the temperature higher than the Curie point is commonly used to improve their soft magnetic properties. Usually, the alloys with high saturation induction exhibit the possibilities to improve their magnetic properties by the simple heat treatment [2].

Crystallization process of metallic glasses, which exhibit a metastable structure, is associated with the change of some physical properties, such as heat capacity, electrical resistivity, free volume and magnetic properties [6].

Crystallization kinetics have been studied by a variety of research methods including X-ray diffraction, transmission electron microscopy, resistance measurements or magnetic properties measurements. These techniques are typical to determine the volume fraction of the crystalline phases and their influence on physical parameters [7].

Differential scanning calorimetry (DSC) is the most useful method to study the crystallization behaviour of metallic glasses. DSC method needs a relatively large heat of crystallization and is not very useful when the reaction rate is small. Therefore, electrical resistivity measurements are better, because they are not only related to the phase type and can give more detailed information about the crystallization [8].

The crystallization behaviour of metallic glasses has been studied for many times. It seems to be a very important process for amorphous materials due to ability of achieving information of the influence of nanocrystalline or crystalline phases on the improve of their soft magnetic properties [8-12].

2. Material and research methodology

The aim of the paper is the structural, thermal and magnetic properties analysis of $Co_{70}Fe_5Si_{15}B_{10}$ metallic glass ribbons in as-cast state and after annealing process by using of XRD, SEM, DTA, DSC and magnetic examination methods.

The investigated materials were cast as ribbon shaped metallic glasses with thickness 0.03, 0.04 and 0.06 mm and width 3 mm. The ribbons were manufactured by the "chill-block melt spinning" (CBMS) technique, which is a method of continuous casting of the liquid alloy on the surface of a turning copper based wheel [14-22].

The casting conditions include linear speed of copper wheel of 10-20 m/s and ejection over-pressure of molten alloy under argon atmosphere of 0.02-0.04 MPa.

In order to study structural relaxation and crystallization process, the studied samples in the "as-cast" state were annealed at the temperature range from 373 to 923 K with the step of 50 K. Tested ribbons were annealed in electric chamber furnace under protective argon atmosphere. The annealing time was constant and equalled to 1 hour.

Structure analysis of the samples in as-cast state was carried out by using of X-ray diffractometer (XRD) with $Co_{K\alpha}$ radiation. In addition, phase analysis of samples after annealing process was carried out by using of X-ray diffractometer with $Cu_{K\alpha}$ radiation The data of diffraction lines were recorded by "step-scanning" method in 2θ range from 30° to 90°.

The fracture morphology of the ribbons with thickness of 0.06 mm after annealing at 623, 723 and 823 K was analysed by using of scanning electron microscopy (SEM).

The solidus temperature of studied Co-based master alloy was measured by using of differential thermal analysis (DTA) at a constant heating rate of 6 K/s under an argon protective atmosphere.

Thermal properties associated with onset (T_x) and peak (T_p) crystallization temperature of first and second stage of crystallization of studied ribbons was examined by differential scanning calorimetry (DSC). The heating rate of calorimetry measurements, under an argon protective atmosphere, was 20 K/min.

Magnetic measurements of annealed samples (determined at room temperature) included following properties:

(1) relative initial and maximum magnetic permeability – determined by E4980A Agilent LCR Meter at a frequency of 1030 Hz, (magnetic permeability measurements were carried out for ribbons of length of 100 mm);
(2) disaccommodation of magnetic permeability $\Delta\mu/\mu$ (magnetic after-effects) - determined by measuring changes of magnetic permeability as a function of time after demagnetization, where $\Delta\mu$ is difference between magnetic permeability determined at t_1 = 30 s and t_2 = 1800 s after demagnetization and μ at t_1 [23-27].

3. Results and discussion

The samples of $Co_{70}Fe_5Si_{15}B_{10}$ metallic glass in as-cast state were examined by XRD methods to check their amorphous state before annealing process. The X-ray diffraction investigations revealed that the tested ribbons with different thickness were amorphous. The diffraction patterns of studied samples have shown the broad diffraction halo characteristic for the amorphous structure (Fig. 1).

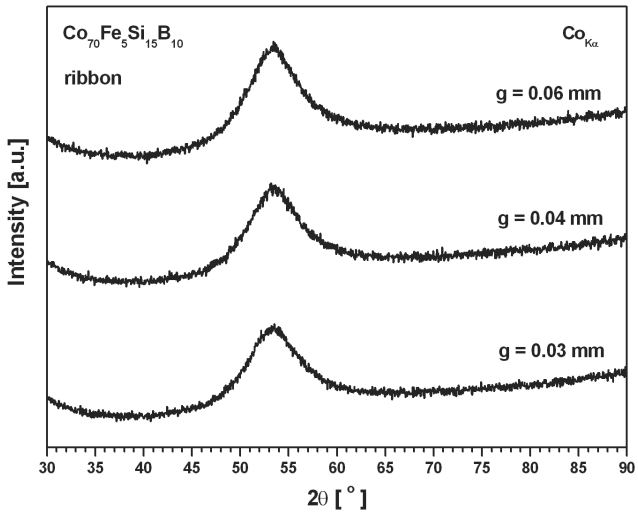

Fig. 1. X-ray diffraction patterns of $Co_{70}Fe_5Si_{15}B_{10}$ metallic glass in as-cast state in form of ribbons with thickness of 0.03, 0.04 and 0.06 mm

The solidus temperature (T_m) and temperature of the end of melting process (T_l) assumed to be the onset and end temperature of the melting peak on the DTA curve is presented in Figure 2. The endothermic peak observed on DTA curve of master alloy of studied metallic glass allowed to determine the solidus temperature (T_m), which has a value of 1271 K and temperature of the end of melting process (T_l = 1352 K).

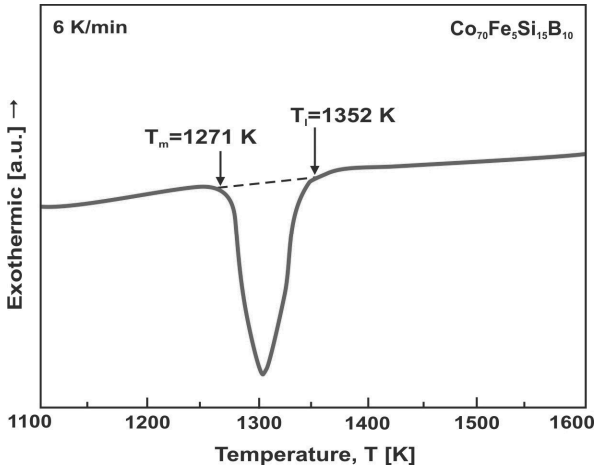

Fig. 2. DTA curve of $Co_{70}Fe_5Si_{15}B_{10}$ alloy as master-alloy

The DSC curves at heating rate of 20 K/min measured on amorphous ribbons of $Co_{70}Fe_5Si_{15}B_{10}$ alloy with thickness of 0.03, 0.04 and 0.06 mm in as-cast state are shown in Figure 3.

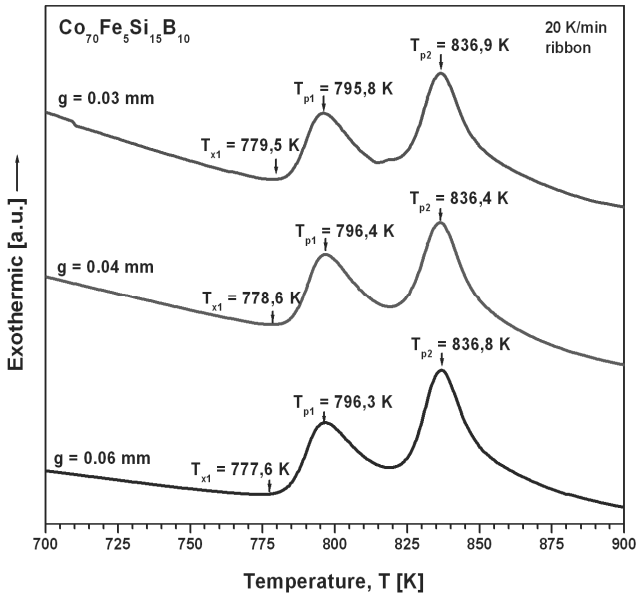

Fig. 3. DSC curves of $Co_{70}Fe_5Si_{15}B_{10}$ metallic glass in as-cast state in form of ribbons with thickness of 0.03, 0.04 and 0.06 mm (heating rate 20 K/min)

The two exothermic peaks describing crystallization process of $Co_{70}Fe_5Si_{15}B_{10}$ alloy were observed for all studied samples with different thickness. The crystallization effect for the ribbon sample with thickness of 0.03 mm includes onset crystallization temperature of T_{x1} = 779.5 K and peak crystallization temperature of T_{p1} = 795.8 K and T_{p2} = 836.9 K. For sample with thickness of 0.04 mm onset crystallization temperature reached a value of T_{x1} = 778.6 K and peak crystallization temperature has a value of T_{p1} = 796.4 K. The second crystallization stage of studied ribbon is described by peak with temperature of T_{p2} = 836.4 K. The crystallization temperature for the ribbon with thickness of 0.06 mm included onset crystallization temperature of T_{x1} = 777.6 K and peak crystallization temperature of T_{p1} = 796.3 K and T_{p2} = 836.8 K.

The thermal stability parameters of studied glasses – onset crystallization temperature (T_x) and peak crystallization temperature (T_p) are presented in Table 2. Comparison of crystallization peaks of studied samples with different thickness shows only the slightly change of crystallization temperature.

Table 2.
Thermal properties of the studied alloy in forms of glassy ribbons, in as-cast state

Glassy alloy	Sample thickness, mm	T_{x1}, K	T_{p1}, K	T_{p2}, K
$Co_{70}Fe_5Si_{15}B_{10}$	0.03	779.5	795.8	836.9
	0.04	778.6	796.4	836.4
	0.06	777.6	796.3	836.8

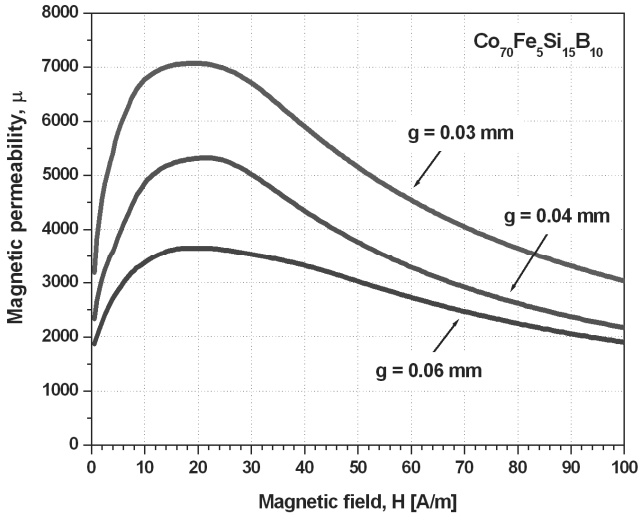

Fig. 4. Relative magnetic permeability of $Co_{70}Fe_5Si_{15}B_{10}$ glassy ribbon in as-cast state with different sample thickness

Figure 4 presents relative magnetic permeability determined at applied magnetic field up to 100 A/m. The maximum magnetic permeability (μ_{rmax}) for glassy ribbon with thickness of 0.06 mm has a value of 3650, however the value of (μ_{rmax}) is much higher for sample with thickness of 0.04 and 0.03 mm and reached a value of 5350 and 7100, adequately. This is a very good results, which allow to classify the studied Co-based glassy alloy for suitable material for soft magnetic applications.

Figure 5 shows X-ray diffraction patterns obtained for studied alloy in form of ribbon with thickness of 0.03 mm after annealing at 673, 723 and 773 K for 1 hour. It is noticed, that annealing in studied temperature range leads to a formation of crystalline phases from amorphous matrix. Figure 5 informs that three phases: hexagonal α-Co(Si) and orthorhombic Co_2Si, Co_3B are formed at lower temperature of heat treatment.

Comparison of diffraction patterns of studied alloy obtained after annealing at higher temperatures from 823 K to 923 K shows the narrowing of diffraction lines and increase of their intensity (Fig. 6). That result informs of further crystallization process of studied samples. Annealing at higher temperatures obviously caused a formation of mentioned crystalline phases. Qualitative phase analysis also enables the identification of tetragonal Co_2B phase.

The appearance of the fracture surface of studied samples in form of ribbon with thickness of 0.06 mm was investigated by SEM at different magnifications. Figures 7, 8 and 9 present micrographs of tested ribbons after annealing process at 623, 723 and 823 K, adequately.

The study of fracture morphology of samples after annealing at 623 K shows a mixed fractures with "river" patterns, which are characteristic for glassy materials and some areas of scaly morphology (Fig. 7). With the increasing of annealing temperature up to 723 K, it could be observed some areas with a greater density of good formed "veins" (Fig. 8). These patterns may inform of existing of the structural relaxation mechanisms and probably the beginning of nanocrystallization process.

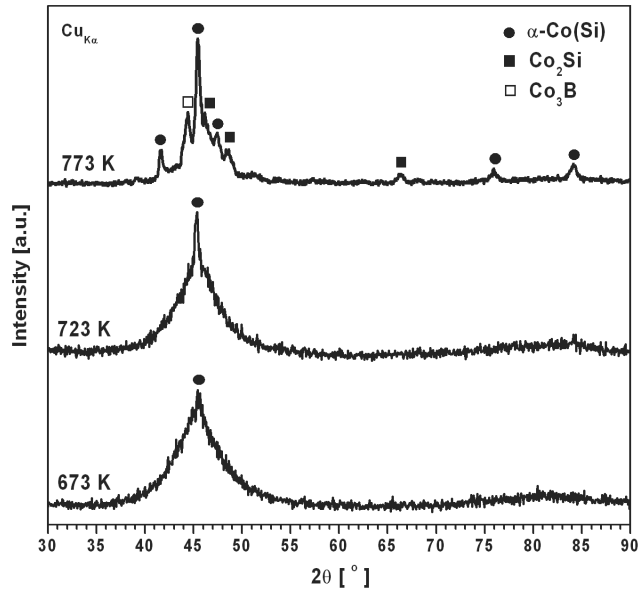

Fig. 5. X-ray diffraction patterns of $Co_{70}Fe_5Si_{15}B_{10}$ alloy after annealing at 673, 723 and 773 K for 1 hour

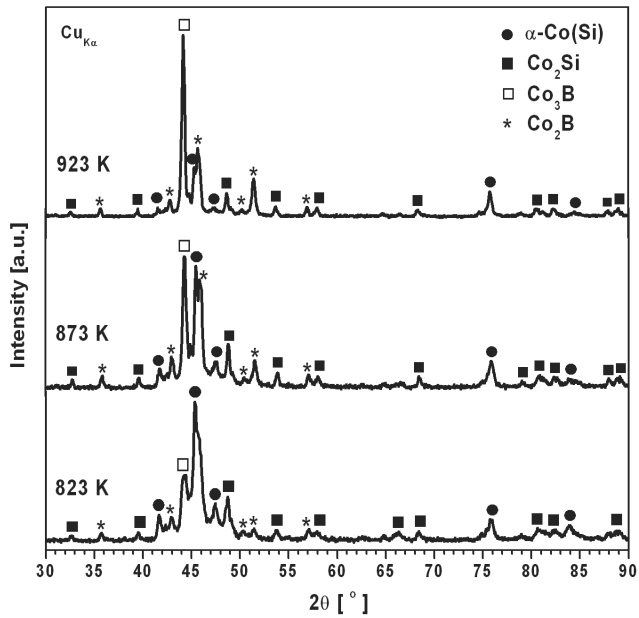

Fig. 6. X-ray diffraction patterns of $Co_{70}Fe_5Si_{15}B_{10}$ alloy after annealing at 823, 873 and 923 K for 1 hour

The fracture morphology analysis of ribbons after annealing at 823 K revealed the occurrence of scaly fractures, which are typical for metallic glasses with reduced ductility. Additionally, the scaly morphology is related with crystallization process of metallic glasses, which was also confirmed by XRD method.

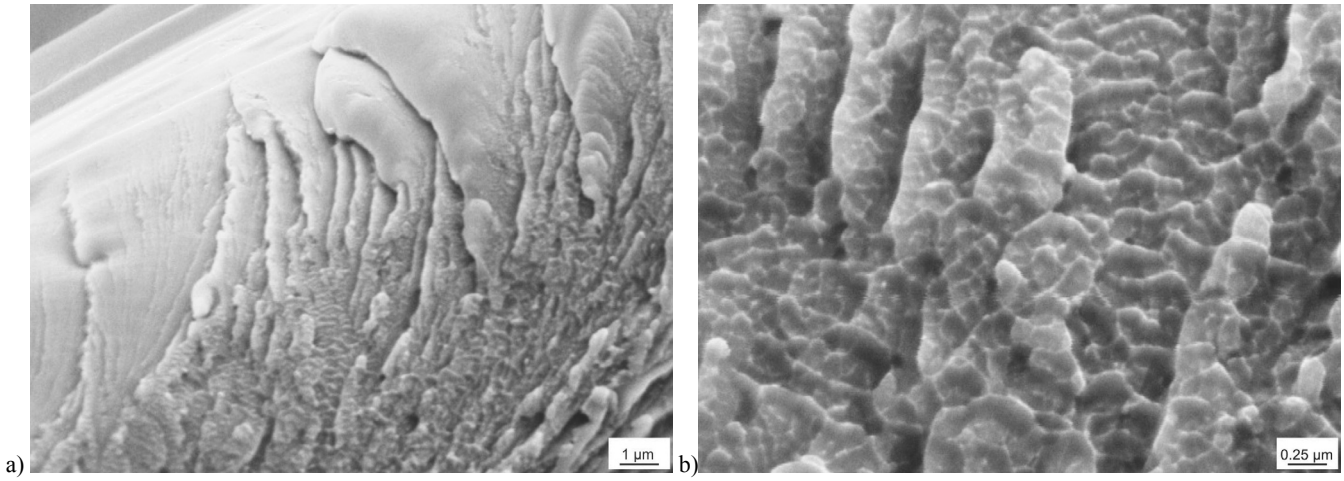

Fig. 7. SEM micrographs of the fracture morphology of $Co_{70}Fe_5Si_{15}B_{10}$ alloy after annealing at 623 K/1 h: a) magn. 10 000x, b) magn. 40 000x

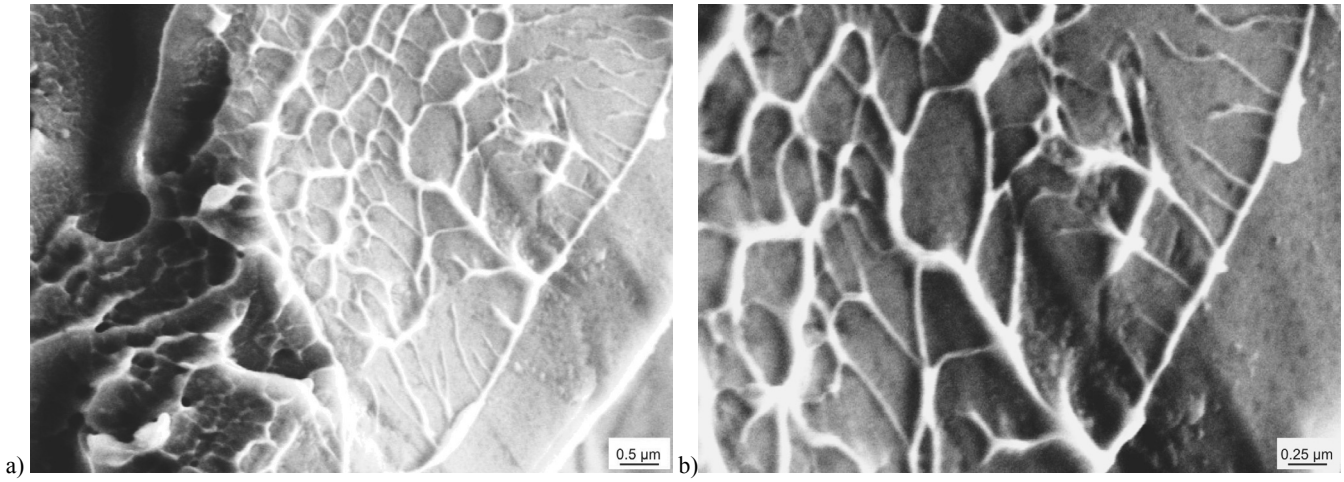

Fig. 8. SEM micrographs of the fracture morphology of $Co_{70}Fe_5Si_{15}B_{10}$ alloy after annealing at 723 K/1 h: a) magn. 20 000x, b) magn. 40 000x

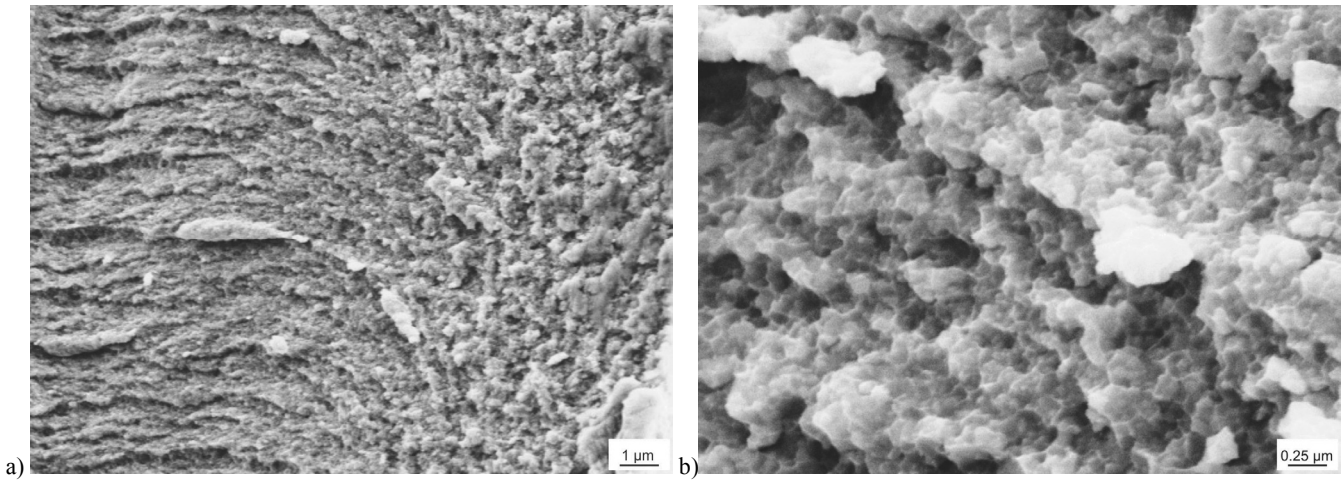

Fig. 9. SEM micrographs of the fracture morphology of $Co_{70}Fe_5Si_{15}B_{10}$ alloy after annealing at 823 K/1 h: a) magn. 10 000x, b) magn. 40 000x

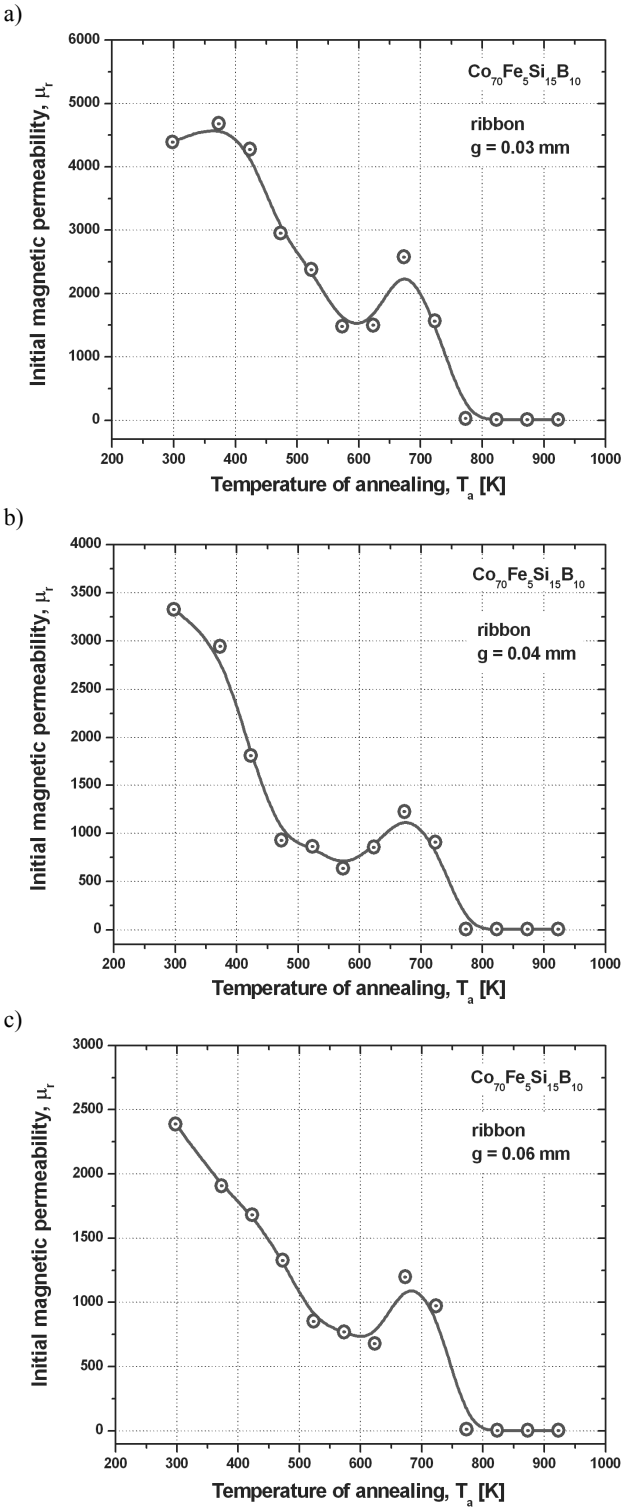

Fig. 10. Initial magnetic permeability of $Co_{70}Fe_5Si_{15}B_{10}$ metallic glass in form of ribbons with thickness of: a) 0.03 mm, b) 0.04 mm and c) 0.06 mm determined at room temperature versus annealing temperature

The initial magnetic permeability (μ_r) determined at room temperature versus annealing temperature (T_a) for three studied ribbon samples is shown in Figure 10.

It could be noticed that initial magnetic permeability of studied samples decreases in function of the increase of annealing temperature. However, a local maximum could be determine at 673 K for all studied ribbons. That temperature of annealing process corresponding to the local maximum of initial magnetic permeability and could be defined as the local optimization annealing temperature (T_{op}).

The study of initial magnetic permeability curves in function of annealing temperature allowed to appoint the local maximum of that property. For sample with thickness of 0.03 mm the initial magnetic permeability at the local optimization temperature (μ_{rop}) reached a value of 2600, the ribbon with thickness of 0.04 mm has μ_{rop} = 1250 and last sample reached μ_{rmax} = 1200.

Tables 3 and 4 give information about magnetic properties – initial magnetic permeability (μ_r) and disaccommodation magnetic permeability ($\Delta\mu/\mu$) of studied alloy in as-cast state and at local optimization temperature, adequately. Basing on literature, the intensity of disaccommodation of magnetic permeability (magnetic after-effects) is directly proportional to the concentration of defects in amorphous materials – free volume caused by microvoids concentration [24].

Table 3.
Magnetic properties of studied $Co_{70}Fe_5Si_{15}B_{10}$ alloy in as-cast state

Glassy alloy	Sample thickness, mm	State	μ_r	$\Delta\mu/\mu$, %
$Co_{70}Fe_5Si_{15}B_{10}$	0.03	as-cast	4400	13.5
	0.04		3350	10.6
	0.06		2400	9.3

As could be observed, a value of μ_r and $\Delta\mu/\mu$ decreases with the thickness of samples. That instability of magnetic permeability is probably due to changing of amorphous structure of glassy materials by different microvoids concentration [25-27]. The successive increase of annealing temperature also caused that $\Delta\mu/\mu$ decreases. This result means that the optimization annealing temperature reduces the time instabilities of magnetic permeability, but the values of initial magnetic permeability, even existing of the local optimization temperature, also decreased.

Table 4.
Magnetic properties of studied $Co_{70}Fe_5Si_{15}B_{10}$ alloy at optimization annealing temperature (T_{op})

Glassy alloy	Sample thickness, mm	T_{op}, K	μ_r	$\Delta\mu/\mu$, %
$Co_{70}Fe_5Si_{15}B_{10}$	0.03	673	2600	6.5
	0.04		1250	1.4
	0.06		1200	3.8

It is also shown that in studied alloy, the optimization effect takes place in amorphous state, probably with very small nano-crystalline grains from α-Co(Si) phase. However, the increasing of annealing temperature leads to determine the local optimization

temperature (T_{op}), but obtained improvement of soft magnetic properties does not give such good results like in as-cast state of tested materials.

4. Conclusions

The investigations performed on the samples of $Co_{70}Fe_5Si_{15}B_{10}$ alloy after annealing process allowed to formulate the following statements:

- the X-ray diffraction investigations revealed that the tested ribbons with different thickness were amorphous,
- the endothermic peak observed on DTA curve of master alloy allowed to determine the solidus temperature (T_m), which has a value of 1271 K and temperature of the end of melting process (T_l = 1352 K),
- the two stage crystallization process was observed for studied glassy ribbons with different thickness,
- comparison of crystallization peaks of studied samples with different thickness showed only the slightly change of crystallization temperature,
- the heat treatment process of ribbon samples involved crystallization of α-Co(Si), Co_2Si phases and cobalt borides at temperature above 773 K,
- the fracture morphology of samples after annealing at 623 K showed a mixed fractures with "river" patterns and some scaly areas,
- the initial magnetic permeability of studied samples decreased in function of the increase of annealing temperature,
- the temperature of annealing process corresponding to the local maximum of initial magnetic permeability could be defined as the local optimization temperature (T_{op}) and reached the same value of 673 K for all studied ribbons,
- the optimization annealing temperature reduced time instabilities of magnetic permeability.

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