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Investigations of crystallization behaviour of $Co_{80}Si_9B_{11}$ amorphous alloy

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ABSTRACT

Purpose: This paper describes crystallization kinetics and changes of magnetic properties involved by process of crystallization of the amorphous $Co_{80}Si_9B_{11}$ alloy.

Design/methodology/approach: The following experimental techniques were used: X-ray diffraction (XRD), electrical resistivity in situ measurements (four-point probe), static and dynamic measurements of magnetic properties (magnetic balance, fluxmeter, Maxwell-Wien bridge).

Findings: In this work has been performed influence of thermal annealing on crystallization kinetics and magnetic properties amorphous $Co_{80}Si_9B_{11}$ alloy.

Practical implications: The attractive properties of Co-Si-B alloy are of special interest for basic research on the materials as well as for their potential applications, like magnetic sensors. The Co soft magnetic material is used in noise filters, saturable reactors, miniature inductance elements for abating spike noise, mains transformers, choke coils, zero-phase current transformers, and magnetic heads etc., i.e., devices which are expected to exhibit high levels of permeability at high frequencies.

Originality/value: It has been shown that thermal annealing at temperature close to the crystallization temperature leads to a significant increase of the initial magnetic permeability. The maximum permeability for examined alloy in as quenched state is about 11300.

Keywords: Materials; Amorphous Materials; Nanomaterials; Heat treatment; Crystallization behavior; Magnetic properties

PROPERTIES

1. Introduction

Amorphous alloys can be prepared by various techniques, including melt spinning [1-4], mechanical alloying [5-8] vapor deposition, chemical alloying [9], etc.

The melt spinning technique allows the rapid solidification of metallic alloys to produce amorphous or nanocrystalline material [3,4].

Alloys of the binary Co-B system can easily be amorphized by melt spinning. More complex alloys with specific properties can be obtained by adding other elements to this system and various chemical compounds can be crystallized from the amorphous phase at specific temperatures [10].

Both Fe-based as well as Co-based amorphous materials have good soft magnetic properties, such as high saturation magnetization, high permeability, low coercivity and loss, which find their applications in antitheft security system, power

electronics, telecommunication devices and automotive magnetics [1-3,9,11,12]. Permeability as well as core-losses can be developed by a further heat treatment. In the heat treatment, the material is typically heated to a specific temperature in an inert atmosphere for a certain time [13-15]. The heat treatment can be realized by impulse methods, too [16,17]. The method most often used is isothermal heating in constant time, for instance 0.5, 1.0, 1.5, 4.0, 8.0 hour [4,18÷21].

For this reasons they have been the subject of much scientific research over the past few decades. In addition to the excellent magnetic properties, the magnetic amorphous alloys present a new system through which crystallization process and corresponding changes in magnetic properties can be systematically studied [13-15]. The kinetics of crystallization of metallic glasses is often described by the well-known phenomenological Johnson-Mehl-Avrami equation for isothermal experiments [2,20,21]. The activation energy of the crystallization process can be obtained from the temperature dependence of the reaction-rate constant, which is known as Kissinger's method [14,15,20÷22].

Our recent researches have focused on crystallization behavior of $Co_{80}Si_9B_{11}$ amorphous alloy induced by thermal treatment. In this paper, the results obtained on amorphous $Co_{80}Si_9B_{11}$ alloy heat treated at different temperatures are presented and discussed.

2. Experiments

Amorphous $Co_{80}Si_9B_{11}$ alloy investigated in the present paper was obtained by melt spinning technique as the form of strip 7 mm wide and 14 μm thickness. In order to verify the chemical composition the X-ray fluorescence (XRF) method was used (SPEUPERPROBE 733 JEOL). The samples in the as quenched state were preliminary annealed by 1h and 0,5 h in the temperature range 373-873 K with step of 50 K. Structural investigations were carried out by applying the X-ray diffraction (XRD) method (XRD7, SEIFERTFPM with filtered Co-K_α radiation) for samples in the as quenched state and after annealing. The parameters of diffractometer are listed in Table 1.

For samples in the as quenched state, the relative magnetic permeability (Maxwell-Wien bridge, frequency 1 kHz, magnetic field 0.5 A/m), saturation magnetization and maximum of the permeability (fluxmeter) at room temperature were obtained. Relative magnetic permeability in weak magnetic field was also determined at room temperature for samples after annealing at T_a temperatures.

Kinetics of the crystallization process was examined by applying electrical resistivity $\rho(T)$ (four point probe) and saturation magnetization $M(T)$ (magnetic balance) measurements in situ with heating rates from 0.5K/min to 10.0 K/min.

The crystallization temperature T_{x1}, and the activation energy of the crystallization process E_c from the isochronal resistivity curves were determined. The temperature T_{x1} can be obtained from the condition $d\rho/dT=0$. The effective activation energy E_c was evaluated by the Kissinger method [20,22], which can be described:

$$\ln\frac{V_l}{T_h^2} + \ln const = -\frac{E_c}{k_B}\cdot\frac{1}{T_h} \tag{1}$$

where: E_c, is the effective activation energy for the crystallization processes, V_l - is linear heating rate, T_h – is the so-called temperature of an homological point determined for the heating rate V_l, i.e. temperature which the rate of crystallization process is maximum [20,23], and k_B is the Boltzman constant.

Results obtained from $M(T)$ measurements were presented as normalized $M(T)/M$(300 K) curves.

Table 1.
The diffractometer's parameters used in XRD method for samples of $Co_{80}Si_9B_{11}$ alloy

Diffractometer's parameters	Diffractometer XRD 7, SEIFERT - FPM firm	
	a	**b**
Current intensity of X – ray tube	40 mA	40 mA
Voltage of X – ray tube	35 kV	35 kV
The time of counting in one measurement's point	7s	20s
Step between measurement's points	0.05°Θ	0.01°Θ

3. Results and discussion

The examinations of structure performed by X-ray diffraction (XRD) technique show that in as quenched state $Co_{80}Si_9B_{11}$ alloy has amorphous structure (Fig. 1).

Only a broad diffraction peak at about $2\Theta\cong52°$ can be observed from Fig. 1, indicating that obtained ribbon had amorphous structure.

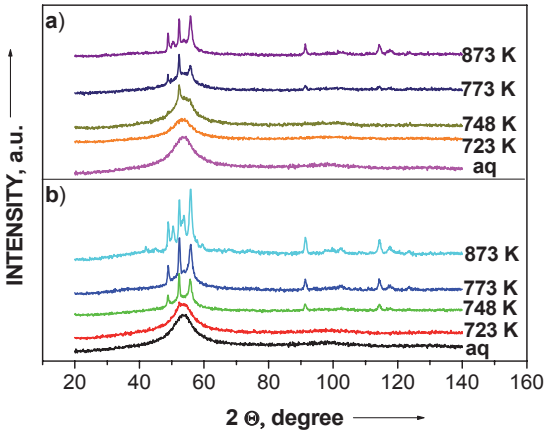

Fig. 1. X-ray diffraction pattern of the $Co_{80}Si_9B_{11}$ alloy in as quenched state and after annealing in temperature range T_a=723÷873K for 0.5 h (a) and 1.0 h (b). The diffractometer's parameters – see Table 1 (a)

The investigated $Co_{80}Si_9B_{11}$ alloy in as quenched state has a following properties: high value of resistivity ρ equal 1.15 μΩm (Fig. 2), saturation magnetization M= 0.9 T (Fig. 3), initial relative magnetic permeability μ_r=1090 (Table 2) and μ_{max}=11300 (Fig. 4).

The obtained physical properties, i.e. ρ, M, μ_r and μ_{max} allow to classify the $Co_{80}Si_9B_{11}$ alloy in as quenched state as a soft magnetic material.

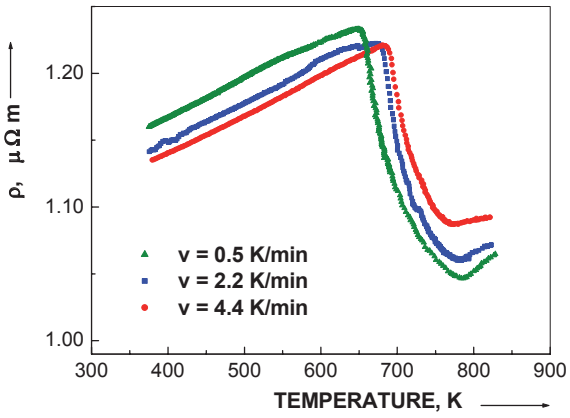

Fig. 2. The in situ isochronal resistivity curves for $Co_{80}Si_9B_{11}$ alloy obtained with heating rate 0.5, 2.2 and 4.4 K/min

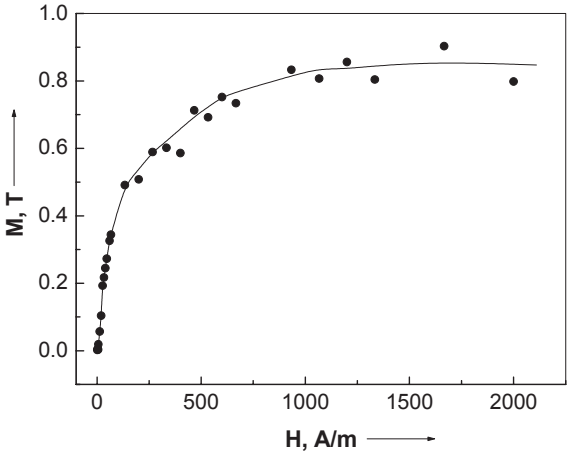

Fig. 3. Magnetization M versus magnetic field for amorphous $Co_{80}Si_9B_{11}$ alloy in as quenched state obtained by magnetic balance

There are three types of crystallization transformations for amorphous alloys: primary, polymorphous and eutectic [4].

The different methods (both isothermal and non-isothermal) were used for determining of crystallization temperatures (primary crystallization temperature - T_{x1} and secondary crystallization temperature - T_{x2}) of $Co_{80}Si_9B_{11}$ alloy.

In order to determine the characteristic temperature of the first (T_{x1}) and second (T_{x2}) stage of crystallization process electrical resistivity measurements with continuous heating rate in the range from 0.5÷10 K/min were used.

From Fig. 2 and 5 shows that value of the crystallization temperature T_{x1} is dependent of the heating rate and is in the range of 655÷681 K for the heating rate 0.5÷4.4 K/min, respectively.

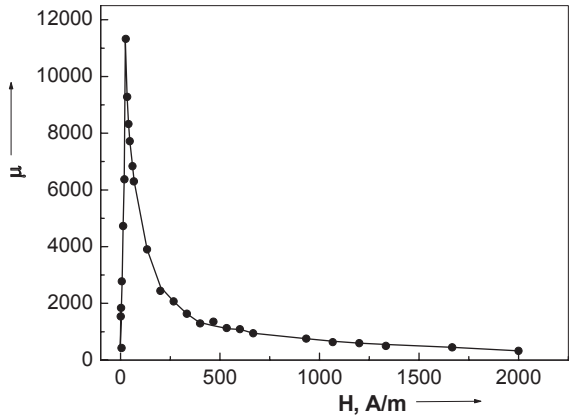

Fig. 4. The maximum permeability μ_{max} for amorphous $Co_{80}Si_9B_{11}$ alloy in as quenched state obtained by magnetic balance

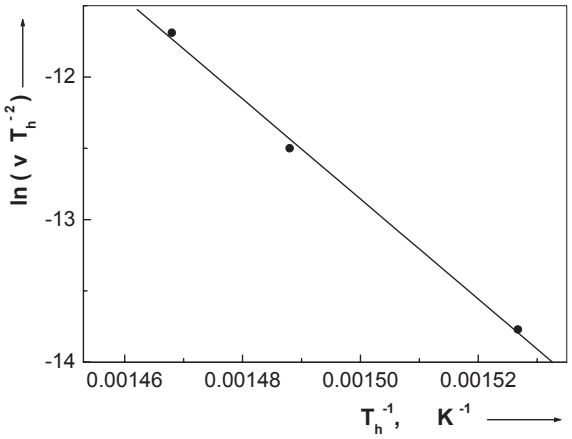

Fig. 5. Plot of $\ln(v/T_h^2)$ versus T_h^{-1} for $Co_{80}Si_9B_{11}$ alloy (see Fig. 2)

Fig 5 shows the plot $\ln(v/T_h^2)$ versus T_h^{-1}, which according to Eq. (1) allows determining the activation energy E_c.

The activation energy E_c for crystallization of $Co_{80}Si_9B_{11}$ is calculated to be 3.0±0.2 eV [14,24]. As an example in Fig. 6 a and 7 a we present $\rho(T)$ curves obtained for $Co_{80}Si_9B_{11}$ alloy with heating rate 5 and 10 K/min, respectively. Fig. 6 b and 7 b represent $d\rho(T)/dT$ curves from which the temperatures of the first (T_{x1}) and second (T_{x2}) stage of crystallization process can be determined from the condition $d\rho(T)/dT=0$. From Fig. 6 b and 7 b shows that characteristic temperatures T_{x1} and T_{x2} for the heating rate 5 and 10K/min are: T_{x1}=680 and 692 K and T_{x2}=819 and 824 K, respectively.

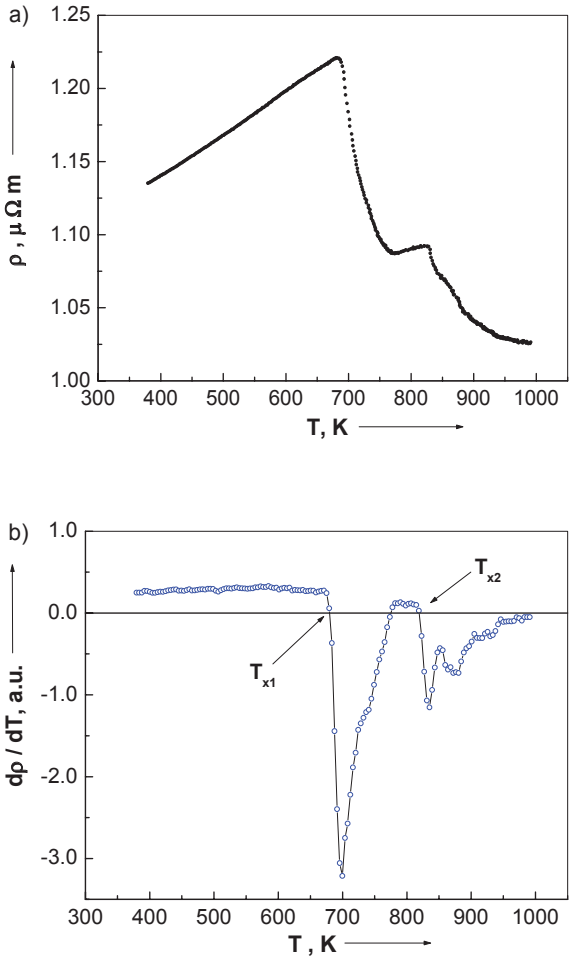

Fig. 6. Electrical resistivity ρ (T) measured with heating rate 5 K/min for the $Co_{80}Si_9B_{11}$ alloy (a) and (b) the dρ/dT curve for the data presented in (a)

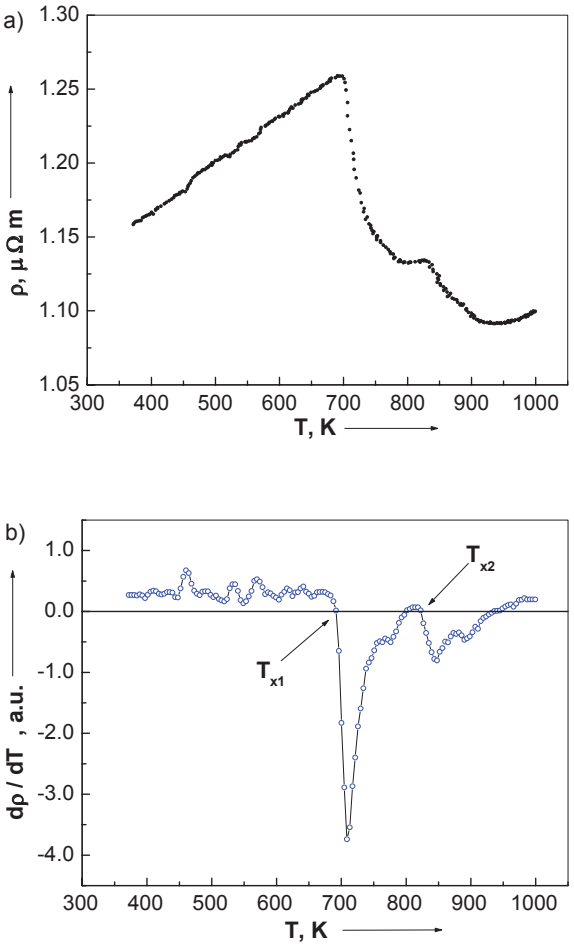

Fig. 7. Electrical resistivity ρ (T) measured with heating rate 10 K/min for the $Co_{80}Si_9B_{11}$ alloy (a) and (b) the dρ/dT curve for the data presented in (a)

Fig 8, 9 a and Fig. 8, 9 b presents a family of magnetization curves $M(T)$ normalized to the value at 300 K (heating rate v=5 and 10 K/min) and the corresponding dM/dT curves, respectively. The characteristic temperature T_{x1} is determined from the condition dM/dT maximum and is about 725 and 730 K for the heating rate 5 and 10 K/min, respectively. The value of T_{x1} deduced in this way corresponds to the highest formation rate of the new ferromagnetic phase. With increasing heating rate the maxima of dM/dT shift towards higher temperatures which is characteristic feature of thermally activated (diffusion controlled) process [25].

From the results presented in Fig. 8 and 9 it can be seen that the formation of Co_2B phase is quite noticeable in $Co_{80}Si_9B_{11}$ alloy as indicated by the arrow [26].

First stage of crystallization was found in 723 K for the $Co_{80}Si_9B_{11}$ alloy heat – treated for 0.5 and 1.0 h (Fig. 1, Table 3). The phases formed in different heat treatment conditions were identified using XRD, as in Fig. 1 an in Table 3.

Further increase of annealing temperature leads to changes in X – ray diffraction (Fig. 1) and at annealing temperature 773 K the α-Co, Co_2Si and Co_3B phases was identified. Samples heat – treated at 748 and 773 K (Fig. 1) for 0.5 and 1.0 h has similar phases. As can be seen from Figure 1 at 748 K the crystallization of the amorphous alloy proceeds trough nucleation of the hexagonal (h.c.p.) α-Co phase in the amorphous matrix. For samples heat – treated for 0.5 and 1.0 h at temperature 873 K Co_2B phase appear (Fig. 1, table 3). There are sharp changes in the intensity of the phases formed at higher temperature and longer time.

Additionally for the samples heat-treated at 873 K for 1 h the structure investigation performed by XRD method using diffractometer's parameters – Table 1 (b). The aim of this investigation was found existence/non-existence the body-centered cubic (bcc) β-Co phase. The diffraction pattern (Fig. 10) did not indicate (bcc) β-Co phase in the samples annealed at 873 K for 1 h.

Table 2.
The parameters of heat treatment (temperature and time) and initial relative magnetic permeability μ_r of the $Co_{80}Si_9B_{11}$ alloy

Annealing temperature	The initial relative magnetic permeability μ_r of $Co_{80}Si_9B_{11}$ alloy after	
T_a, K	0.5 h annealing	1.0 h anneling
aq[1)]	1090	1090
373	1049	1056
423	916	1361
473	850	942
523	875	611
573	528	1073
623	531	445
673	361	581
723	589	798
748	120	145
773	65	69
823	58	56
873	57	56

[1)] - as quenched state

The obtained of experimental data shows that the values of crystallization temperature T_{x1} obtained from magnetization measurements and from resistivity curves are not the same [27,29].This difference cannot be explained neither by the unavoidable temperature gradient existing in both apparatuses nor by the natural dependence of magnetization on temperature [25].

Based on investigation results the value of the crystallization temperature T_{x1} of an amorphous $Co_{80}Si_9B_{11}$ is strongly depend on the heat treatment conditions (linear heating or isothermal heating) and on the using methods. Using different methods the compare the value of the crystallization temperature T_{x1} can take place only under a constant heating rate.

The changes of structure and magnetic properties have been observed with increasing the temperature annealing of investigated alloy in range 373÷873 K by 0.5 and 1.0 h (Fig. 1, 2, 6-10, Table 2, 3) [14].

Initial magnetic permeability μ_r measured as a function of annealing temperature T_a. From Table 2 it can be recognized that the investigated alloy annealed for 0.5 and 1.0 h in temperature range 373÷723 K the μ_r has high value, i.e. 1049÷589 and 1056÷798, respectively.

It can be recognized that the heat treatment of $Co_{80}Si_9B_{11}$ alloy (at temperature $T_a > T_{x1}$) initial relative magnetic permeability μ_r passes by a distinet maximum related to formation of nanocrystalline phase α-Co, Co_3B and Co_2Si. Increase of annealing temperature leads to further decrease of initial relative magnetic permeability μ_r (Table 2) which can be related to the formation of boride Co_2B besides mentioned phases [14,24].

The structural instability alloy in the amorphous and nanocrystlline phases leads to anomalies in the dependence of the magnetic properties on temperature [27]. In amorphous ribbons, a number of physical properties change due to structural relaxations. It is known that amorphous alloy annealing below the crystallization temperature relaxes the residual internal stresses induced during the preparation process, improving the magnetic response of the material. Higher temperatures of annealing initiate the crystallization process in the amorphous alloy [17,29].

Table 3.
The phase analysis results for the $Co_{80}Si_9B_{11}$ alloy annealed at T_a=773 and 873 K by 0.5 and 1.0 h (see Fig. 1)

d, Å	α-Co	d_α (hkl) Co_3B	for Co_2B	phases: Co_2Si
2.500			2.510 (200)	
2.340		2.363 (112)		
2.165*	2.165 (100)			
2.130		2.128 (121)		2.130 (310)
2.100			2.113 (002)	
2.030*	2.023 (002)	2.031 (210)		2.020 (220)
2.000				2.000 (301)
1.980		1.975 (103)	1.983 (211)	1.970 (121)
1.940*		1.942 (211)		
1.910*	1.910 (101)			
1.870		1.860 (122)		1.870 (002)
1.850		1.848 (113)		1.850 (311)
1.800			1.815 (112)	
1.740		1.732 (212)		
1.700				1.700 (112)
1.660*		1.657 (004)		1.670 (410)
1.616		1.620 (130)	1.616 (202)	
1.590			1.588 (310)	1.600 (130)
1.485	1.480 (102)			
1.250*	1.252 (110)			1.250 (412)
1.187			1.192 (213)	1.190 (113)
1.179			1.183 (330)	
1.167			1.169 (411)	
1.149*	1.149 (103)			
1.105				1.110 (023)
1.101				1.100 (512)
1.083	1.083 (200)			
1.065*	1.066 (112)			
1.046*	1.047 (201)			1.050 (341)
1.031				1.032 (332)
1.016*	1.015 (004)			
0.987			0.973 (204)	

d – lattice parameters calculated from Fig. 1
d_α – lattice parameters of identified phases [28]
* - the peak appearing in $Co_{80}Si_9B_{11}$ alloy annealed at T_a=773 K by 0.5 and 1.0 h, too

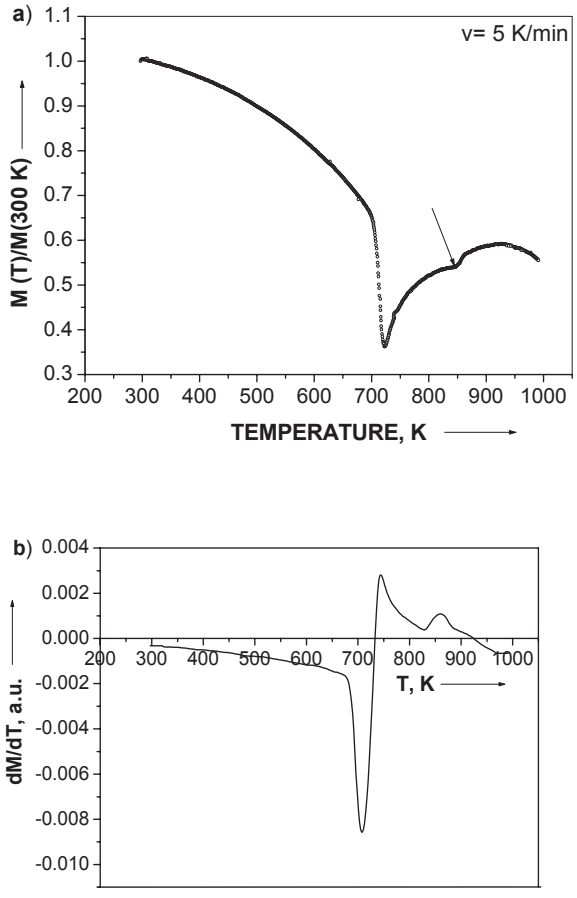

Fig. 8. Normalized magnetization versus temperature T of $Co_{80}Si_9B_{11}$ alloy (a) and (b) - dM/dT curves for the data presented in (a)

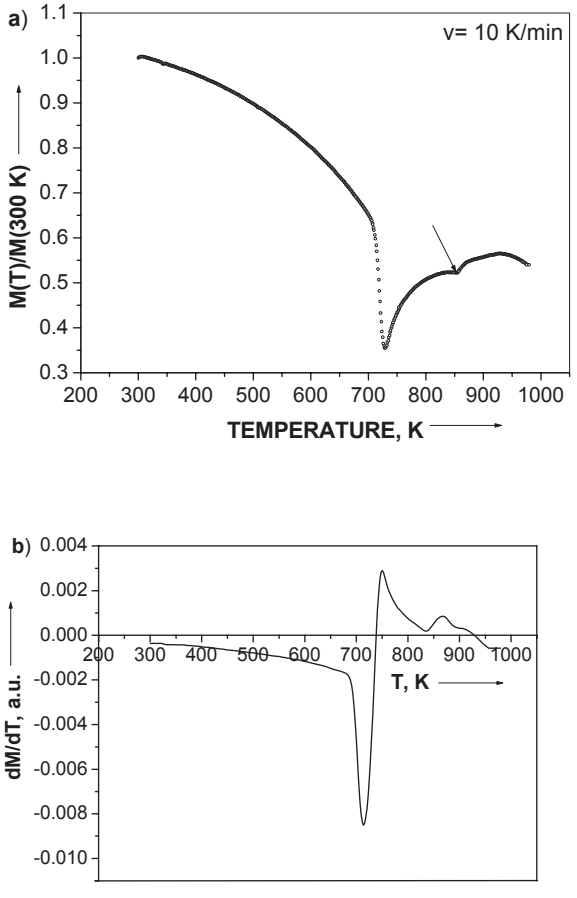

Fig. 9. Normalized magnetization versus temperature T of $Co_{80}Si_9B_{11}$ alloy (a) and (b) - dM/dT curves for the data presented in (a)

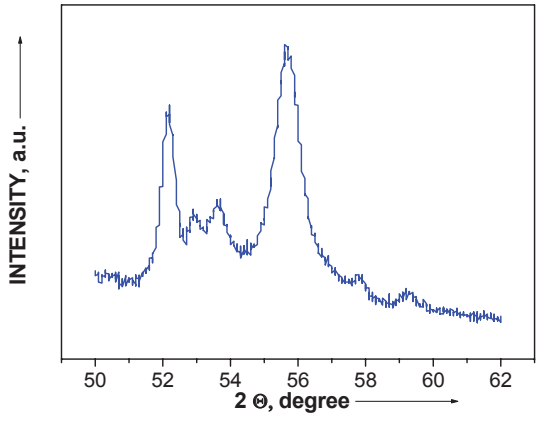

Fig. 10. X-ray diffraction pattern of the $Co_{80}Si_9B_{11}$ alloy after annealing at temperature T_a=873K for 1.0 h. The parameters of diffractometer – see Table 1 (b)

4. Conslusions

The crystallization behaviour of the $Co_{80}Si_{11}B_9$ metallic glasses is studied using different methods.The main conclusion of the paper can be summarized as follows: Amorphous $Co_{80}Si_9B_{11}$ type alloy is not in thermodynamic equilibrium state as a consequence of rapid cooling from the liquid phase. Production process causes the time and thermal instabilities of physical (magnetic and electric) properties of this material. These instabilities are reduced by thermal annealing. It can be explained by structural relaxation connected with annealing out of free volume frozen during rapid cooling and crystallization of material. After annealing at elevated temperatures, the α-Co phase is formation. For higher annealing temperatures the Co_3B and Co_2Si phases are formed, it causes a strongly decrease of relative magnetic permeability.

The activation energy of crystallization process obtained by Kissinger method is 3.0±0.2 eV. The value is similar for the high cobalt alloys in all cases.

Additional information

The presentation connected with the subject matter of the paper was presented by the authors during the 14th International Scientific Conference on Achievements in Mechanical and Materials Engineering AMME'2006 in Gliwice-Wisła, Poland on 4th-8th June 2006.

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