

Magnetic properties of Co-based amorphous ribbon under cyclic heating and cooling

J. Konieczny ^{a,*}, A. Borisjuk ^b, M. Pashechko ^c, L.A. Dobrzański ^a

^a Division of Materials Processing Technology, Management and Computer Techniques in Materials Science, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland

^b Institute of Applied Mathematics and Basic Sciences, Lviv Polytechnic National University, Banderi 12, Lviv, Ukraine

^c Education Technical and Information Department, Lublin University, ul. Nadbystrzycka 38, 20-618 Lublin, Poland

* Corresponding author: E-mail address: jaroslaw.konieczny@polsl.pl

Received 01.06.2010; published in revised form 01.09.2010

Materials

ABSTRACT

Purpose: The aim of the work is to investigate the changes of magnetic properties of the cobalt based $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ alloy under cycling heating and cooling.

Design/methodology/approach: The amorphous metallic ribbons were manufactured by planar-flow-casting method. Investigations of the magnetic properties were observed under permanent heating amorphous and partially crystallized alloy. Observations of the structure were made on the JOEL transmission electron microscope (TEM). Using the HFQS program the distributions of the magnetic hyperfine P(H) fields were determined for spectra smoothed in this way, employing the Hesse-Rübartsch method.

Findings: The analysis of the magnetic properties under permanent heating and structure of the Co-based amorphous ribbons obtained in the by planar-flow-casting process proved that the permanent heating caused the crystallization of second magnetic phase after transition near to paramagnetic state.

Research limitations/implications: The appropriate cyclic heating and cooling significantly decreasing soft magnetic properties of examined amorphous alloy. The cyclic heating beginning of elementary crystallization processes and the end of crystallization alloy.

Practical implications: According to the results presented in the paper the examined Co-based glassy alloys as a soft ferromagnetic material may be utilized in construction of magnetic cores such as choke coils, common mode and noise filter and is of great technological interest.

Originality/value: The paper presents influence of permanent heating on structural changes of metallic ribbons. Results and discussion of the influence of permanent heating on magnetic properties of metallic ribbon are presented.

Keywords: Amorphous materials; Structural relaxation; Crystallization; Soft magnetic properties

Reference to this paper should be given in the following way:

J. Konieczny, A. Borisjuk, M. Pashechko, L.A. Dobrzański, Magnetic properties of Co-based amorphous ribbon under cyclic heating and cooling, Journal of Achievements in Materials and Manufacturing Engineering 42/1-2 (2010) 42-49.

1. Introduction

Amorphous metallic materials based on iron (FINEMET), cobalt (HITPERM) or nickel alloys received method melt spinning with attention on characteristic, unique mechanical [1] and magnetical properties [2-5] are on all world the object of intensive investigations of research centres [6-12]. The materials obtained in this way are characteristic of the very high initial magnetic permeability μ , high magnetic saturation B_s , very low coercion field H_c and very low remagnetising losses. These properties may be improved by isothermal heat treatment or during heat treatment with continuously increased temperature or by heat treatment in the magnetic field [13,14].

Most of the researches have focused on the nanocrystalline ribbons and wires or bulk metallic glasses [15-19] which are appropriate for a wide range of applications, like sensors and pulse generators [20,21].

The nanocrystalline materials display a very low magnetic anisotropy. This is caused by the fact that the originated grains are significantly smaller from the correlation length for the ferromagnetic exchange interactions [22].

In CoSiB amorphous alloys Fe additions plays the same role as the Cu addition in the iron based amorphous alloys [23,24]. Additions of Fe and Nb have the advantageous effect on the CoSiB alloy structure. Addition of Nb improves the thermal stability of the amorphous phase and the Fe and Nb additions make it possible to develop the nanocrystalline structure with one FeCo-A2 crystalline phase in the CoSiB amorphous alloy. The commutable Nb or Hf elements used in the 5% concentration have the equally effect on the structure [25,26].

With regard on a receiving (developing) method amorphous alloys (metallic glasses) have metastable structure. When they are thermal activated they undergo structural transformation and crystallize according to their chemical composition, heating temperature and time. The crystallization of amorphous materials leads to change of mechanical, physical and chemical properties [27].

The degree of growth and range of atoms settlement in forming crystallization nucleus are the consequence of thermal activation.

The materials the nanocrystalline in shape of tape received on them by crystallization of metallic glasses characterize with good magnetic proprieties however considerable fragility defect them, making difficult the practical use of them [28].

The aim of the work is to investigate the properties of crystallization temperature and magnetic properties amorphous alloy based on cobalt $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ during heating and cooling with different rate.

2. Material and methods

The investigations were carried out on a $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ metallic glass in form of 0.025 mm thick and 10.2 mm wide ribbons.

Microscope examinations were made on the JEOL JEM 200CX transmission electron microscope. Specimens in the form

of bars were made for magnetic properties tests on the Vibrating Sample Magnetometer (VSM).

Because of the low iron content in the examined specimens and, in addition, due to the low probability of the resonance absorption of the ^{57}Fe γ isotope radiation for the investigated materials, measurement of each Mössbauer spectrum was carried out for about one week. The resonance effect turned out to be very low in spite of the significant extension of the measurement time period and big measurement statistics. Therefore, each measured Mössbauer spectrum was smoothed, which consisted in the decomposition of the experimental spectra to the convergent Fourier series and in discarding those harmonic components that have very small input to the total dispersion, by using the Parseval relationship [29,30]. Distributions of the magnetic hyperfine P(H) fields were determined for spectra smoothed in this way, by using the HFQS [31] program, employing the Hesse-Rübartsch method [32]. The P(H) distributions obtained are characterised by the following set of parameters:

- $\langle H \rangle$ - average value of the magnetic hyperfine field,
- $\langle IS \rangle$ - average value of the isomeric shift,
- D_H - dispersion of the P(H) hyperfine fields distribution.

The D_H parameter characterizes the hyperfine P(H) magnetic fields' distribution breadth and changes of its value are connected with fading or growth of the particular configurations of the vicinity of the 57-Fe isotope that occur in the thermal or mechanical treatment of the amorphous phase. The average values of the isomeric shift $\langle IS \rangle$ (in a smaller range) and the average values of the hyperfine magnetic field $\langle H \rangle$ are sensitive to the closest atomic neighbourhood of the 57-Fe isotope and depend on the kind of atoms surrounding the Mössbauer isotope. Analysing changes of the D_H and $\langle H \rangle$ parameters gives grounds to draw conclusions pertaining to changes occurring in the closest neighbourhood of the Mössbauer isotope, featured by ^{57}Fe .

3. Results and discussion

The investigated $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ alloy was delivered in the as quenched state and had the amorphous structure. In the electron diffraction patterns (Fig. 1a) the broad blurred rings are visible coming from the amorphous phase. No spot reflexes in the diffraction pattern for a test piece in the as quenched state attests to the absence of the crystalline phase in the structure. No crystalline phase was revealed in the as quenched state and the X-ray diffraction (Fig. 2) displays the evident wide-angled, diffused spectrum, characteristic for the amorphous state. The collection of all diffraction patterns obtained for the $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ alloy, subjected to heat treatment at the temperature 623 and 773 K, is presented in Figure 1b and 1c.

The Mössbauer spectrum of the $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ amorphous ribbon obtained in the melt spinning process is shown in Fig. 3, whereas the hyperfine fields distribution P(H) calculated for this test piece is shown in Fig. 4.

The evaluated Mössbauer spectrum is characteristic for the amorphous alloys and has a typical form of the Zeeman spectrum, consisting of 6 broadened asymmetric absorption lines. The average value of the magnetic field $\langle H \rangle = 21.3$ T, much lower than the relevant value of the metallic iron (33.0 T), suggests that the iron atoms have atoms like Si and B in their closest neighbourhood, strongly lowering the internal magnetic field.

As it turns out from the research [33], the presence of Co atoms increases the value of the internal magnetic field for the Fe atoms. Therefore, the small, characteristic peak occurring in the zone of the magnetic fields of about 35.0 T may be attributed to the Fe-Co type precipitations. The big value of the proportion of lines' No (2 and 4) intensities to the (3 and 4) ones' intensities, equal to 3.65 indicates that the magnetization vector M is parallel to the test piece plane.

In order to examine processes which are in material of the ribbon and their influence on magnetic properties, research of magnetic properties in the function of the temperature was conducted.

Alloy after pouring out in the amorphous state is characterized by a narrow hysteresis loop, which is characteristic for soft magnetic materials. Maximum magnetic permeability of the ribbon in the initial state is $\mu_{\max}=131900$, value of the coercive force $H_c=15.1$ A/m and magnetic remains $B_r=0.25$ T.

Material of the ribbon in the state "as quenched" is magnetic in the room temperature (Fig. 5). From the course of two curves of primary magnetization amorphous alloy (1) and after heating to temperature 890K with rate 30 K/s (2) is seen, that bigger magnetization of saturation got for amorphous alloy, almost $B_s=0.8$ T and after heating only $B_s=0.5$ T. It has been also noticed that the ribbon in the amorphous state is getting the saturation state at the much smaller magnetic field strength than the annealed ribbon.

In the Figure 6 have been presented the isochoric curves characterized the changes of magnetization saturation in the function of the temperature. The ribbon of metallic glass was heated to the temperature 720 K with rate 30 K/s and then chilled with the same rate. At the beginning of experiment in temperature 299K magnetization was $B_s=0.78$ T and along with the rise of temperature of glass this value is diminishing monotonic achieving the minimum in temperature 694K and is equal $B_s=0.1$ T, and then grows achieving the B_s value in the final phase of heating 0.25 T. Cooling of alloy with the same rate causes the height of magnetization saturation, which at temperature 299 K is $B_s=0.72$ T, which is the proof (evidence) of the crystallization of the phase with magnetic properties.

Next heating the amorphous ribbon was conducted increasing the maximum temperature to which amorphous alloy was being heated. In the Figure 7 presented the curves characterized the changes of magnetization saturation in the function of the temperature. Metallic glass was heated to the temperature 905K with rate 30 K/s and then cooled with the same rate. With the beginning of the heating process in temperature 296 K magnetization saturation of amorphous alloy $\text{Co}_{66}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ is $B_s=0.78$ T and along with the rise in temperature this value is dropping monotonic achieving the minimum at temperature 715 K which is $B_s=0.07$ T.

This hardly proves the it is the crystallization temperature T_K of amorphous glasses. With further increase of temperature the magnetic saturation increasing too, receiving the final phase with the value of $B_s=0.28$ T at temperature 904 K. The increase in value of magnetization saturation is proving the fact that the crystallizing new, other phase is characterized by magnetic properties. At the temperature 715–904 K is appearing distinct local minimum $B_s=0.26$ for $T=810$ K and the maximum $B_s=0.3$ T for $T=866$ K.

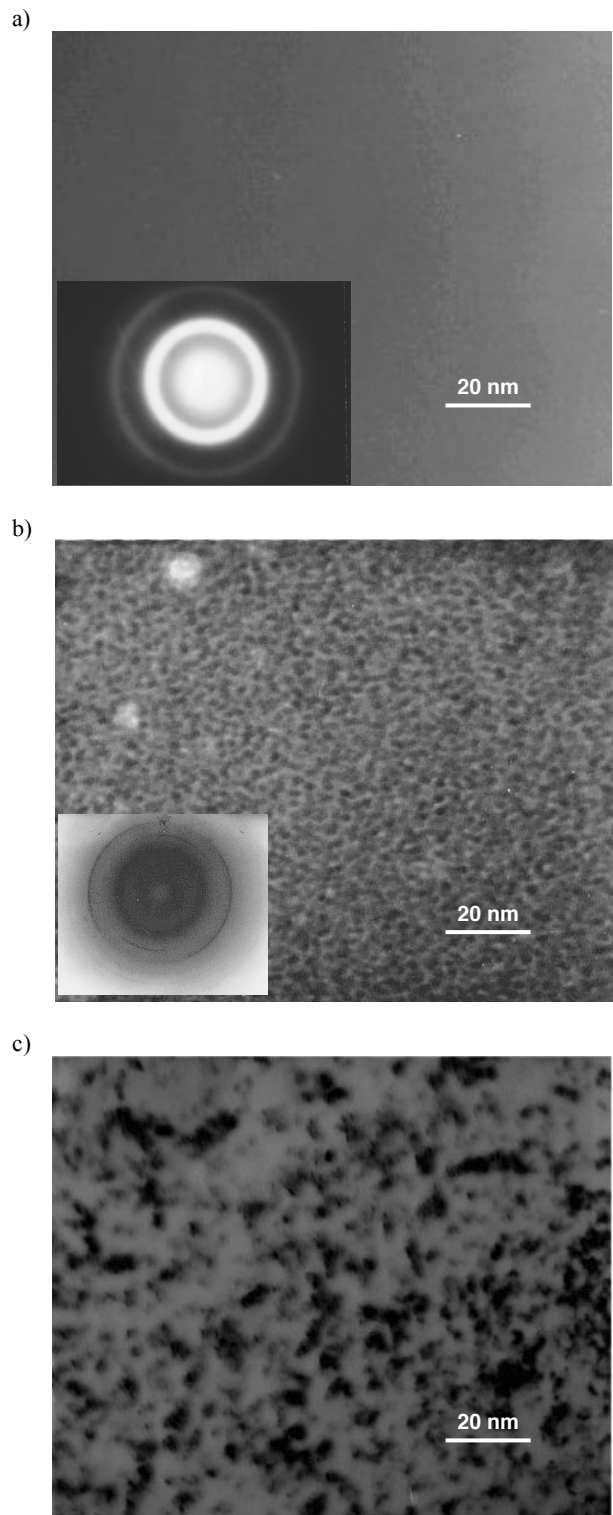


Fig. 1. Structure of the $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ alloy, a) in "as quenched" state, b) annealed at 623 K/1 hour, c) annealed at 773 K/1 hour; TEM

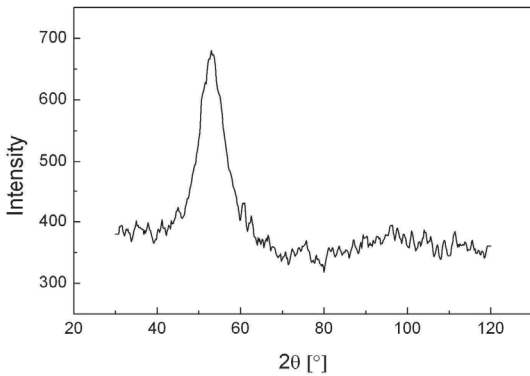


Fig. 2. X-ray diffraction pattern of the $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ alloy in "as quenched" state

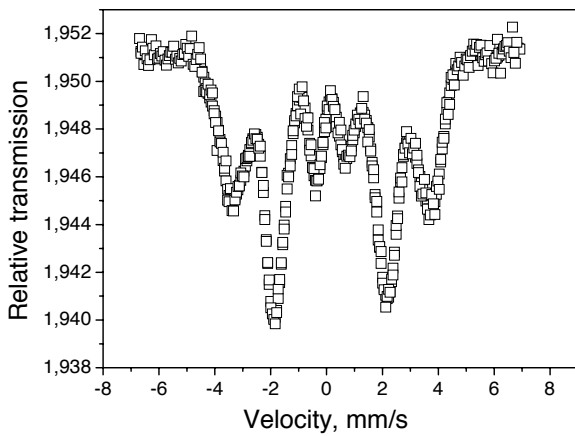


Fig. 3. The Mössbauer spectrum of the as quenched $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ amorphous ribbon

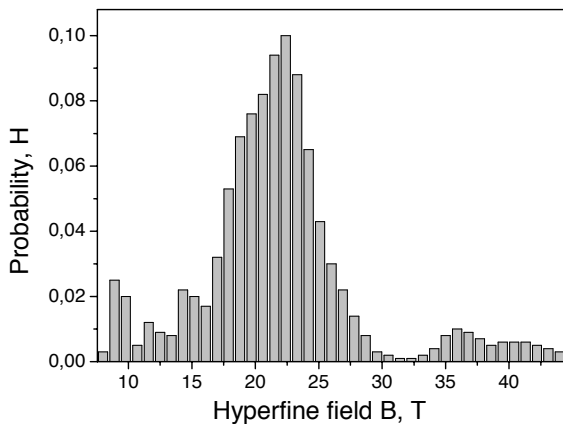


Fig. 4. Distribution of the hyperfine fields $P(H)$ of the as quenched $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ amorphous ribbon

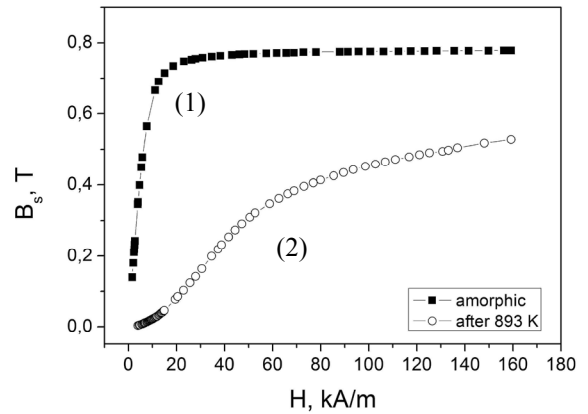


Fig. 5. Curves of primary magnetization of amorphous alloy $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ (1) and after heating to 890 K (2); maximum magnetic field strength 160 kA/m

Considering the influence of the temperature on magnetic properties it should be thought that initial increase of temperature does not cause significant changes in relaxation processes. This indicates that relaxation processes do not occur in this range of temperature. Observed changes can be associated with initiating the structural relaxation, particularly little changes in areas of close ordered atoms. However changes of magnetic properties of studied material above 400 K are still associated with the development of the relaxation process of the amorphous structure.

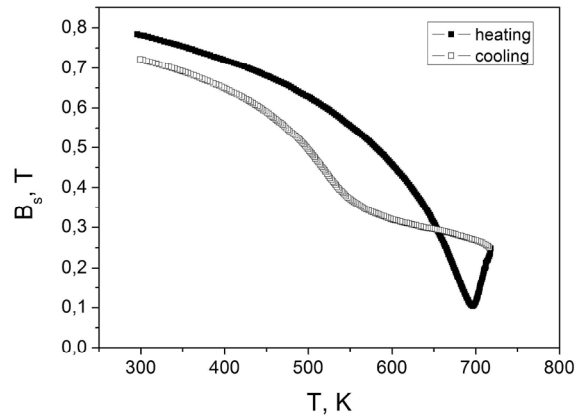


Fig. 6. Temperature relation of magnetic saturation of amorphous alloy $\text{Co}_{66}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$, measured at the heating to 720 K and next cooling down; Initial state – amorphous, heating rate 30K/min., cooling rate 30 K/min

The Mössbauer spectrum annealed at the temperature of 773 K is very similar to the initial test piece's spectrum in the as quenched state. The values of the $\langle H \rangle$ and $\langle IS \rangle$ parameters, increased compared to the initial test piece indicate to increase of

a number of Co atoms in the closest neighbourhood of Fe atoms in the amorphous matrix.

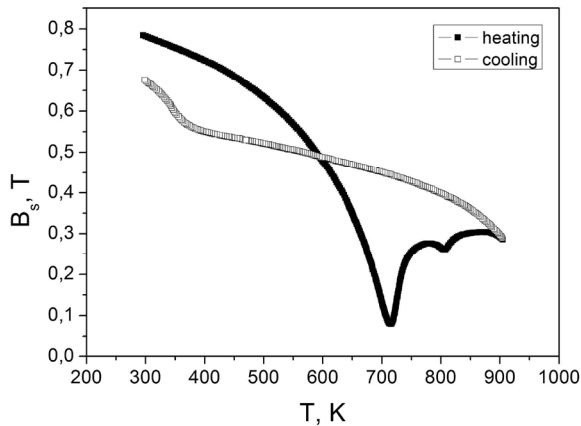


Fig. 7. Temperature relation of magnetization saturation amorphous alloy $\text{Co}_{66}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$. Initial state amorphous; heating with rate 30 K/min., cooling with rate 30 K/min

A significant reduction of the Fe-Co type precipitations, occurring in the initial test piece, is also visible. The changes observed are undoubtedly the result of the structural relaxation and holding of the redundant voids at an elevated temperature.

Fig. 8 shows the Mössbauer spectrum of the initial test piece annealed at 773K and also distribution of the magnetic hyperfine $P(H)$ fields (Fig. 9).

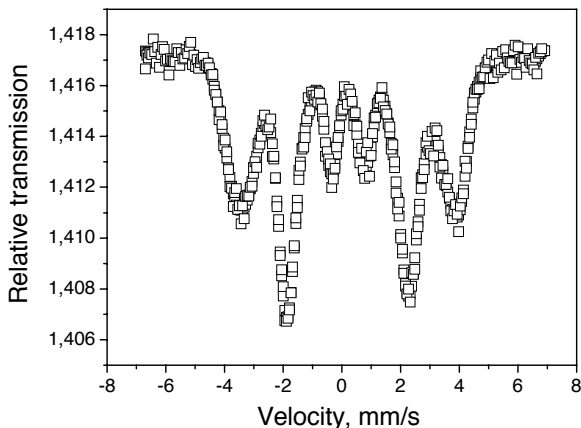


Fig. 8. The Mössbauer spectrum of the $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ amorphous ribbon annealed at the temperature of 773 K

Along with the more height of temperature fall in the soft magnetic properties can be caused by the relaxation of the amorphous structure. Irreversible changes of inner stresses and disappearance of the volume of structure defects, as well as changes of chemical and topological character are accompanying it within the close atoms packing [34].

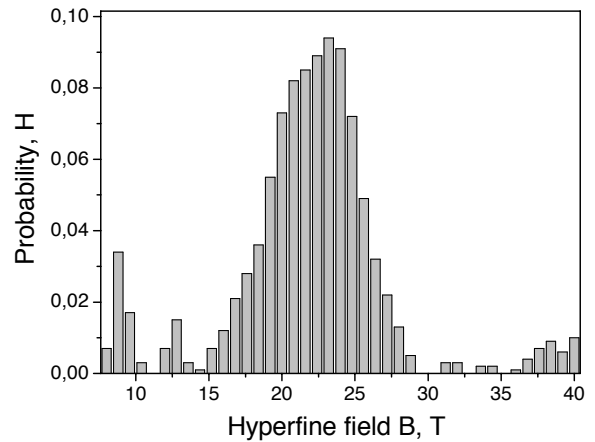


Fig. 9. Distribution of the hyperfine fields $P(H)$ of the $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ amorphous ribbon annealed at the temperature of 773 K

In the temperature above 623K a process of the crystallization is starting in few areas of alloy and creating in amorphous warp small spherical precipitates of the crystal structure. The starting crystallization process of glass is also in the form of the violent reduction magnetization saturation associated with increasing with the free move of electrons. Similar results were received at the work [35].

At the temperature above 673K is taking place blocking magnetic domain through border interfacial areas and magnetic properties of alloy are undergoing the further deterioration [36]. Huge magnetic saturation takes place at temperature above 720 K with proves that crystallization of an alloy occurs [37].

The curve of cooling from this temperature is presenting the thermal magnetization relation which is characterized by multi-phase nanocrystalline material. Opposite to the variant of cooling from 840K, the curve of cooling is typical for material being characterized by the only one crystalline phase which is showing magnetic properties in the room temperature and demonstrating Curie temperature above the crystallization temperature. Curves of similar character were received at the work [38].

It seems that under the influence of the temperature a permanent confusion of ferromagnetic domain in the structure of alloy was observed [39].

In the following stage of experiment the changes of physical properties were examined under the influence of the temperature of metallic glass $\text{Co}_{66}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$, heated earlier (previously) to temperature 720 K with rate 30 K/s. In the Figure 5 were presented the curves being characteristic of changes magnetization saturation in the function of the temperature. The ribbon of metallic glass was heated to temperature 880 K with rate 30 K/s and then cooled with the same rate. In the initial state at temperature 299K alloy magnetization is $B_s=0.72$ T (it is worth stating that earlier heating caused, that magnetization saturation in the room temperature is lower than in the initial state – amorphous) and along with the rise of temperature the value of magnetization saturation are being lowered achieving the minimum in temperature 708K, which is $B_s=0.26$ T and similarly

in previous cases is taking place height of magnetization saturation however in this case slight and in temperature 883 K, is $B_s=0.28$ T.

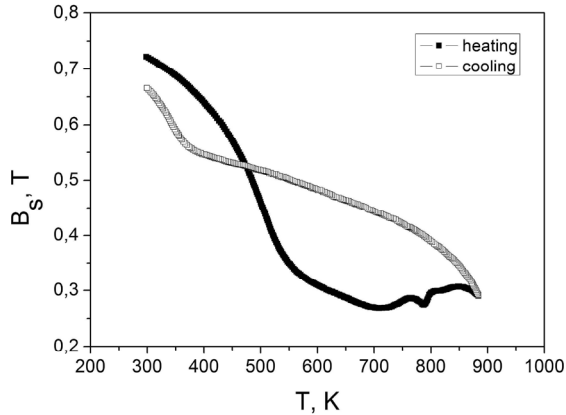


Fig. 10. Temperature relation of primary magnetization saturation amorphous alloy $\text{Co}_{66}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$. Initial state – after heating to 720 K (Fig. 8); heating rate 30 K/min., cooling rate 30 K/min

It clearly states about the crystallization of the phase which is characterized by ferromagnetic properties. Cooling such hot alloy with the same rate causes the height magnetization saturation and in temperature 299K is achieving $B_s=0.66$. So this value is lower than the initial state about 0.8 T. It has been noticed that B_s change under the influence of the temperature in this case isn't proceeding monotonic, but in the range of temperature 299–550 K is being reduced violently from the value 0.72 T to the circa 0.35 T that is the half value.

After achieving the minimum (following) in higher temperatures (about 900 K) a fall of magnetization was observed which is associated with the thermal relation of magnetization saturation, with primary crystallization and disintegration of material associated with formation of the more compound nanocrystalline structure.

Important factor causing the change in the temperature above 500K in comparing to the state after pouring out can be caused mainly with disappearance of the free volume at unchanged atoms packing in the areas of the close reach and with effects causing changes of inner stresses preceding the crystallization [40].

In the last stage of experiment were examined the changes of physical properties occurred under the influence of the temperature of metallic glass $\text{Co}_{66}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ heated earlier (previously) to the temperature 720 K, cooling to the room temperature but later reheating to the temperature 890 K with speed 30 K/s. In the Figure 6 were presented the curves of changes magnetization saturation in the function of the temperature.

In this case ribbon of $\text{Co}_{68}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ alloy was heated to the temperature 880 K with speed 30 K/s and then cooled with the same rate. In the initial state magnetization alloy is $B_s = 0.67$ T and along with the rise in temperature to 350 K is taking place sudden fall of magnetization saturation but later this fall is already

monotonic and less intense achieving the minimum in the temperature 880 K, which is $B_s = 0.31$ T.

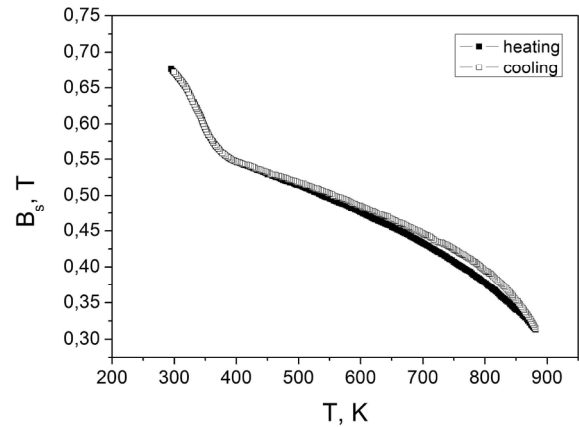


Fig. 11. Temperature relation of primary magnetization saturation amorphous alloy $\text{Co}_{66}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$. Initial state - after double heating to 720 K (Fig. 8) and 890 K (Fig. 9); heating rate 30 K/min., cooling rate 30 K/min

Performed investigations of $\text{Co}_{66}\text{Fe}_4\text{Mo}_1\text{Si}_{13.5}\text{B}_{13.5}$ alloy at critical heating operations (i.e. heating in amorphous state to the temperature 720 K (Fig. 7), reheating to 890 K (Fig. 10) and another heating to 890 K (Fig. 11)) let formulate the following conclusion, irreversible transformations in structure of the alloy were completed and observed relations upon heating and cooling have exchange character.

On the Fig. was presented a relation magnetization saturation in the function of the temperature in the range 293–900 K (higher than the temperature Curie of amorphous matrix). Along with a rise in temperature, magnetization of alloy is decreasing and temperatures of heating rise, what is associated with the rise in the content of ferromagnetic phase.

Conducted research show that in the alloy are occurring in the range of temperatures to 350 K complex effects of the structural relaxation and initiating the crystallization [40]. At the further heating it was stated that the crystallization of alloy was occurring in the temperature 715 K. Further cyclical heating with following cooling causes total crystallization of alloy what is visible in the relation of curves heating-cooling, which are demonstrating no occurring transformations of the structure (Fig. 11).

4. Conclusions

Coercive force (H_c) and magnetization (B_s) changes progressing along with a rise in temperature of heating are the result of the reduction of stresses in glass, and reducing of magnetoelastic anisotropy. Because amorphous alloys in the state after pouring out are instable, each their heating causes the structural relaxation, and hence lowering mechanical properties. In literature [41] authors state that the relaxation of the structure of amorphous metallic alloys influences on changes of physical properties. It constitutes the very disadvantageous feature,

because in many cases applying them is possible only in the narrow range of temperatures. On the other hand, susceptibility to relatively simple changes of physical properties of amorphous alloys as a result of thermal treatments enables using this effect for controlling alloy properties.

Additional information

Selected issues related to this paper are planned to be presented at the 16th International Scientific Conference on Contemporary Achievements in Mechanics, Manufacturing and Materials Science CAM3S'2010 celebrating 65 years of the tradition of Materials Engineering in Silesia, Poland and the 13th International Symposium Materials IMSP'2010, Denizli, Turkey.

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