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Structural relaxations in the bulk amorphous alloy Fe₆₁Co₁₀Ti₃Y₆B₂₀



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ABSTRACT

The paper presents studies of annealing effect on the disaccommodation phenomenon in bulk amorphous alloy $Fe_{61}Co_{10}Ti_3Y_6B_{20}$. The investigated sample was prepared by suction-casting method in the form of rod. The annealing process has been performed at temperature well below the crystallisation temperature. The amorphous structure has been confirmed using X-ray diffractometer. The susceptibility and its disaccommodation were determined using completely automated set up. The disaccommodation curve was decomposed into three elementary processes, each of them was described by Gaussian distribution of relaxation times. The obtained results indicate that the disaccommodation phenomenon in studied alloy is related with directional ordering of atom pairs near the free volumes; this is in agreement with H. Kronmüller's theorem.

1. Introduction

One of the characteristics of amorphous materials is the presence of short-range atomic order and a non-uniform distribution of so-called "free volumes" [1-3]. These materials are thermodynamically unstable. During annealing processes at temperatures well-below the glass transition temperature, these alloys endeavour to reach a more thermally-stable state [4]. Due to fact that bulk amorphous alloys are produced at a relatively low cooling speed, some structural relaxations already occur during the manufacturing process - leading to the stabilisation of the structure. The intensity of the structural relaxations depends on the chemical composition of the alloy and the thermal annealing conditions. The structural relaxations influence the shortrange atomic order and the resulting changes are both topological and chemical. The topological changes in the short-range atomic order lead to an increase in the atomic packing density. This is due to the relocation and diminution of the free volumes in such an amorphous alloy. This (topological) type of change is irreversible [5]. On the other hand, changes in the chemical order of the atoms can be either reversible or irreversible, and such changes are connected with the ordering of a certain assembly of atoms. The structural relaxations could be connected to the relocation of a single atom or several atoms, but also possibly with the collective movement of a large volume of the atoms [6,7]. The creation of an activation energy spectrum has been facilitated through empirical examination. The activation of one relaxation process has been found to cause the activation of additional processes. This is because each relaxation process introduces changes into the local structure of the material. From this point of view, treating

these processes as occurring in two-level isolated systems with a continuous energy spectrum is incorrect. The processes of structural relaxations lead to many changes in the microstructure of amorphous alloys, as well as in their chemical, mechanical and magnetic properties [8–10]

The aim of this work was to investigate the influence of isothermal annealing on the magnetic properties of the amorphous alloy $Fe_{61}Co_{10}Ti_3Y_6B_{20}$.

2. Experimental procedure

Ingots of the investigated alloy were prepared by arc-melting high-purity components under a protective argon atmosphere (Fe - 99,98%, Co - 99,99%, Ti - 99,99%, Y - 99,98%). The element boron was added in the form of an alloy of known composition: Fe $_{\rm 45.4}B_{\rm 54.6.}$

The samples of the alloy were prepared in the form of rods with the following approximate dimensions: diameter 2 mm and length 20 mm; a casting method was used in which molten alloy was sucked into a water-cooled, copper mould. The calorimetric measurements were performed using a NETZSCH differential scanning calorimeter (DSC). The DSC curves were recorded using a heating rate of 10 K/min under a protective argon atmosphere. From analysis of the DSC curves, values for the optimal annealing temperature were found. The structure of the alloy was investigated in the as-cast state, as well as after a process of isothermal annealing at a temperature of 800 K for 20 min. A BRUKER "ADVANCE D8" X-ray diffractometer was used to facilitate these structural investigations, which were carried out over the 2Θ range of $30-120^{\circ}$ using a measurement step of 0.02° and a measurement time

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of 5 s per step. Measurements of the magnetic susceptibility within low magnetic fields were measured using the transformer method. The amplitude and frequency of the magnetising field were 0.26~A/m and 2 kHz, respectively. The measurements were taken on samples, following exposure to a demagnetising field with a frequency of 100~Hz and amplitude that decreased to zero over a time-span of 1.1~s. A flow thermostat was used to facilitate measurements of magnetic susceptibility at temperatures lower than room temperature; at higher temperatures, a vacuum furnace was used with a bifilar-wound heating element.

The disaccommodation of the magnetic susceptibility was calculated from the measurements of magnetic susceptibility as a function of time-elapsed, following demagnetising of the sample, at a specific temperature. The results were presented in the form of isochronal disaccommodation curves [11]:

$$\Delta\left(\frac{1}{\chi}\right) = \frac{1}{\chi_{120}} - \frac{1}{\chi_2} = f(T),\tag{1}$$

where: χ_2 and χ_{120} are the values of the magnetic susceptibility measured after 2 s and 120 s after sample demagnetisation, respectively. The Gaussian distribution of the relaxation times was used for analysis of the isochronal disaccommodation curves [12]:

$$P(\ln \tau) = \beta^{-1} \pi^{-\frac{1}{2}} \exp \left[-\left(\frac{\ln \frac{\tau}{\tau_{m}}}{\beta} \right)^{2} \right]$$
 (2)

where: τ_m is the average value of the relaxation times τ

 β - width of the Gaussian distribution at the height of $\frac{1}{\alpha}$.

An isochronal disaccommodation curve was presented as a sum of several elementary processes in the form of [13]:

$$\Delta\left(\frac{1}{\chi}\right) = \sum_{i=1}^{l} \int_{-3\beta_i}^{+3\beta_i} \beta_i^{-1} \pi^{-1/2} \frac{I_{pi} T_{pi}}{T} (e^{-t_l/\tau_{mi}e^z} - e^{-t_2/\tau_{mi}e^z}) e^{-(z/\beta_i)^2} dz, \tag{3}$$

where: $z=\ln\frac{\tau_i}{\tau_{mi}},~I_{pi}\text{-}$ the intensity of the process at the peak temperature $T_{pi}.$

Eq. (2) was used to enable the numerical analysis of the isochronal disaccommodation curves. The following fitting parameters were determined, using the least squares method: the peak temperature T_{pi} , at which the maximum intensity I_{pi} occurs, the average activation energy E_{mi} and the distribution width β_i .

3. Results and discussion

Fig. 1 shows the isochronal DSC curves, recorded for the investigated alloy.

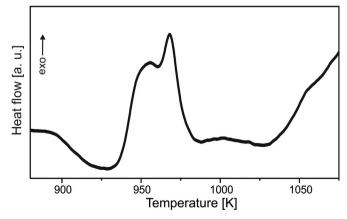


Fig. 1. DSC curves measured at the heating rate of 10 K/min for the bulk amorphous alloy $Fe_{61}Co_{10}Ti_3Y_6B_{20}$, produced in the form of a rod.

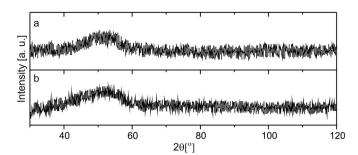


Fig. 2. X-ray diffraction patterns for powdered samples of the bulk amorphous alloy $Fe_{61}Co_{10}Ti_3Y_6B_{20}$: (a) in the as-cast state; (b) in the state following annealing at 800 K for 20 min.

Two collocated exothermic peaks can be seen on the DSC curves. The thermal annealing of the alloy was performed at a temperature of 800 K; i.e. well below the crystallisation temperature for this alloy. The annealing of the sample was supposed to stimulate the relaxation processes in the structure of the material and not cause crystallisation of the material.

In Fig. 2, X-ray diffraction patterns are shown for the investigated alloy samples in the form of rods of approximate dimensions: diameter 1 mm and length 2 cm.

On the obtained X-ray diffraction patterns, a broad maximum can be seen at the angle $2\Theta{\approx}~50^{\circ},$ which is typical for such results obtained from the amorphous alloys. This type of pattern indicates a lack of both translational symmetry and angular correlation in the atomic distribution.

In contrast to crystalline alloys, it is very difficult to observe directly the structure of amorphous alloys. Therefore, an indirect method was used for measuring parameters that are sensitive to structural changes. Two of these parameters are the magnetic susceptibility and the disaccommodation thereof. Measurements of these parameters were performed in low magnetic fields, in which only reversible magnetisation processes occur (the so-called Rayleigh region). The relationship of magnetic susceptibility with temperature, for the bulk amorphous alloy in the as-cast state, is shown in Fig. 3.

The magnetic susceptibility (χ) increases with temperature, reaching a maximum close to the Curie (T_C) temperature of the alloy. For the alloy in the as-cast state, this temperature is about 540 K. For higher temperatures (> Tc) χ decreases sharply. As can be seen from Fig. 3, the magnetic susceptibility for the investigated alloy increases rapidly with temperature. This behaviour can be explained by taking into account two competing processes: the decreases in the values of the magnetic anisotropy and the magnetisation with temperature. In the

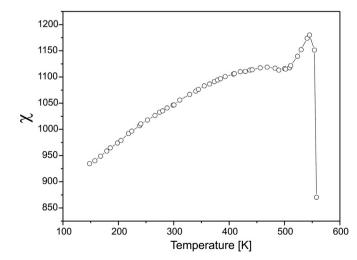


Fig. 3. The magnetic susceptibility of the bulk amorphous alloy $Fe_{61}Co_{10}Ti_3Y_6B_{20}$ in the as-cast state.

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