The Influence of the Manufacturing Method on the Structure and Magnetic Properties of Rapid Cooled Iron Based Alloys

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The studies include the production of an amorphous alloy with the chemical composition $Fe_{62}Co_9Y_8W_1B_{20}$ by two methods: injection and suction casting while maintaining similar production parameters (such as: chamber pressure, protective gas) and comparison of their structure and magnetic properties. The alloy was examined for structure by X-ray diffraction. The magnetic properties were determined on the basis of the VSM and the Faraday magnetic balance measurements. Solid amorphous iron-based materials are characterized by good magnetic properties. The influence of the method of production on these properties turns out to be significant, especially in the case of coercive field. The obtained results lead to further research on massive iron-based amorphous materials. Future research will address the effect of changes in the chemical composition of the alloy on magnetic properties and the continuation of studies on the impact of the manufacturing method on the structure and properties of alloys. The obtained alloy is characterized by good soft magnetic properties. It is possible to use it in electronics and electrotechnics, especially in devices where easy remagnetization of the material at specific frequencies is required.

Keywords: amorphous materials, magnetic properties, suction casting, injection casting

Amorphous materials have been known for over 50 years [1]. Such alloys have a structure reminding of glass, from where the name metallic glass is coming. Describing amorphous materials, it is commonly referred to as freezing the liquid structure in solid form. Amorphous material, although undoubtedly behaves like a solid body, is not characterized by long range ordering of atoms, which is appropriate for liquids. The amorphous structure of materials determines completely different properties compared to their crystalline counterparts with similiar chemical composition [2-7]. It concerns both the strength properties such as hardness, toughness and tensile strength and magnetic properties like coercivity or the value of the initial permeability [7-10]. A particularly interesting group of amorphous materials are magnetic materials, in particular, exhibiting the so called soft magnetic properties. Such a material is characterized by a low coercive field value and a relatively high saturation magnetization value. Based on amorphous materials, nanocrystalline materials can be produced whose magnetic properties can be significantly better than amorphous precursors [11-14]. Amorphous materials with magnetically soft properties are often used in electronics and electrotechnics, and the reason for this is their unique properties. Due to the lack of crystalline structure and disturbed system of atoms, very low losses can be obtained in them for remagnetization, which makes them an attractive material for the construction of transformer cores [15]. In the 1960s, amorphous materials were produced in the form of layers, coatings and thin ribbons, what significantly reduced the possibilities of their application. With time, the necessity to obtain amorphous materials of greater thicknesses arose. Therefore, several new scientific methods for the production of these materials have been developed in many scientific and industrial centers. As the date of the creation of a completely new group of amorphous materials is considered 1989 when A. Inoue from University of Tohoku proposed Tyree empirical criteria giving the possibility of repeatability in the production of amorphous alloys with thicknesses far beyond the capabilities of earlier methods. Such materials are called massive amorphous materials [16-28].

The purpose of this work is to produce an amorphous alloy with chemical composition $Fe_{62}Co_9Y_8W_1B_{20}$ by two methods and to examine and compare their structures and selected magnetic properties.

Experimental part

An alloy with chemical composition $Fe_{62}Co_9Y_8W_1B_{20}$ was designated for research. The alloy components were weighed with an accuracy of 0.001 g. Batch materials with a high degree of purity were used. All elements they were characterized by a purity of over 99.99% of which Cobalt were at the level of purity above 99.995%. The batch material was weighed in the form of a 10 gram weight. The prepared batch was melted using an electric arc in properly prepared conditions. The melting process was carried out on a copper, water cooled plate. The disc has a recess for the batch and titanium. As the research shows, the remelting of titanium has a significant impact on the purity and quality of the material obtained. Melting of the batch takes place after the high vacuum has been produced in the working chamber by means of a pump system. After the vacuum has been created, argon is introduced into the chamber in the atmosphere in which the arc melting is carried out. The arc temperature is regulated by the current supplied to the electrode. Particular attention should be paid to the first remelting of the batch. Too fast arcing on the batch causes scattering of the alloy components along the working chamber. From the point of view of the desire to obtain high accuracy of the chemical composition of the alloy obtained, this is a very undesirable phenomenon. Therefore, the first remelting should be carried out with great care. Proper preparation of the batch will significantly increase the chance of obtaining an amorphous structure in the final stage. Subsequent

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remelting takes place after the inversion is reversed to the other side using the manipulator. Five time remelting of the ingot at a sufficiently high current ensures good mixing of the alloy components. The high homogeneity of the ingot generates a greater chance of reproducing the properties of the produced amorphous alloy.

Figure 1 presents a scheme for the production of amorphous materials by pressing. The purified polycrystalline ingot is crushed into smaller pieces and placed in a quartz capillary. The process of producing the amorphous material is carried out in the atmosphere of argon after earlier production of a high vacuum in the working chamber. The batch is melted using eddy currents. After reaching the right consistency, the liquid melt is injected under pressure into the copper mold. The mold is placed in a copper block cooled by a stream of water. In addition, the induction furnace is cooled during the generation of current flowing through the copper inductor. It is advisable to keep constant parameters during the production of amorphous materials, i.e. constant pressure prevailing in the chamber, constant injection pressure and constant current in the induction furnace. It is also important to maintain a constant flow of water stream through the copper block, which ensures reaching a similar cooling rate.

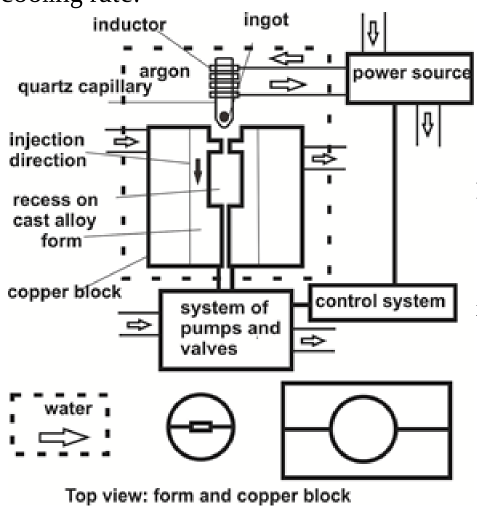

Fig.1. Scheme of making amorphous materials by injection casting

The cooling rate achieved with this method is in the range of 10^1 - 10^3 K/s. The mold to which the melt is injected has the shape of a cylinder. The round cross-section of the mold enables good cooling of the alloy. It is possible to cast alloys in various shapes such as tiles, cores or rods. As a standard, 0.5mm thick tiles are cast. The length and width of the tiles are different, most often the plate has a length of 20mm and a width of 10mm. Figure 2 presents a schematic of producing amorphous materials using the suction method. The idea of the method is very similar to the method of injection. The alloy casting process is carried out in an argon atmosphere after the operating chamber has been purged from the air. The method makes it possible to cast the alloy into three molds during one melting cycle in a protective atmosphere of argon. The batch material is melted with an electric arc whose temperature is regulated by the current flowing through the electrode. Before the batch is processed, pure titanium is melted in order to capture residual impurities remaining in the working chamber. The plates on witch batch is placed have holes through which the melts are sucked into the copper molds by opening the valves connecting the working chamber with the rotary pump. As in the injection method, the molds are placed in a water-cooled copper block. The suction method uses identical forms as in the injection method. During the production of amorphous materials, this method

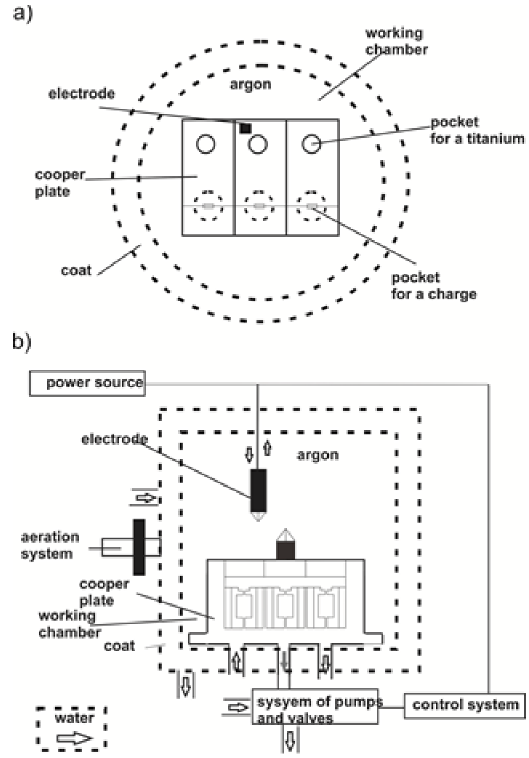

Fig. 2. Scheme of making amorphous materials by suction casting: a) top view, b) side view [11]

is used to cool the working chamber and the electrode used to melt the polycrystalline ingot.

The suction method makes it possible to achieve a similar cooling rate as the injection method.

The alloy with the $Fe_{62}Co_9Y_8W_1B_{20}$ atomic composition was produced by two methods while maintaining similar production parameters. The same vacuum level was used as well as the constant argon pressure prevailing during the casting of the alloy. The alloy was cast into identical forms.

The structure of the obtained material was examined using X-ray diffraction.

The magnetic properties of the alloy were tested using a Faraday magnetic balance and a vibration magnetometer.

Results and disscussions

Figure 3 shows X-ray diffractograms measured for a high-temperature melt produced by suction and injection. The study was carried out with the Brucker model Advanced 8 (CuKα X-ray tube). Diffraction measurements were made from the angle of 2Θ from 30° to 100° by irradiating samples for 7 s per measuring step (0.02°).

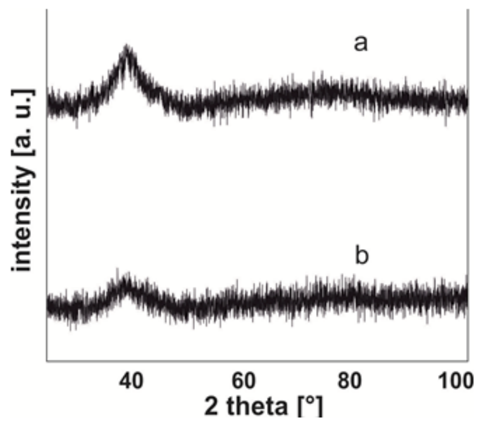

Fig. 3. XRD patterns for $Fe_{62}Co_9Y_8W_1B_{20}$ in powdered form plater of 0.5mm thickness made by: a) injection b) suction casting

The investigations were carried out for the alloy in powder form. The measured diffractograms are similar. The wide maximum called the amorphous halo is clearly visible. This suggests obtaining a chaotic arrangement of atoms in the whole volume of the alloy, which together with the lack of distinct other peaks indicates obtaining an amorphous structure in the produced material. The diffractograms obtained differ in the intensity of the broad maximum, which may indicate a different degree of ordering the amorphous structure. Amorphous, for the powder from the alloy produced by pressing, the peak is much more pronounced.

Figure 4 shows the reduced magnetic saturation polarization curves for the test alloy. The measurement of magnetic saturation polarization was carried out with the help of the Faraday magnetic balance for the temperature range from 350K to 850K. The measured saturation polarization curves as a function of temperature in the positive direction are quite similar to each other. The m0 curves obtained as a result of cooling indicate small differences in the magnetic variation of the saturation polarization. Based on the analysis of the curves shown in figure 4, it can be concluded that no crystalline or magnetically hard phase occurs in the temperature range up to 850K. One can only see a gentle transition in the temperature range of approximately 580K, typical for the transition from ferro or paramagnetic. Only one transition from ferro to paramagnetic from the amorphous structure is visible. The measured return curves are similar to the original curves, which suggests high temperature stability of the alloy produced [22].

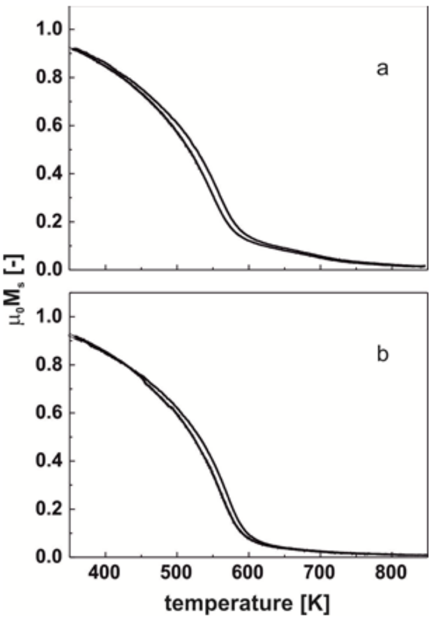

Fig. 4. Thermomagnetic curves for sample in as cast state for alloy $Fe_{62}Co_9Y_8W_1B_{20}$ made by: a) injection, b) suction casting

However, it should be noted that for the injection alloy the separation of the positive curve with the negative curve takes place at a temperature of about 800 K and for the sample produced by the method of injection, practically at the transformation temperature of the ferro - para. This means that during the temperature measurement, the structure of the alloy was relaxed in the homogeneous magnetic field. The result is the rearrangement of atoms and the release of free volumes to the surface of the sample.

The obtained curves in figure 4 were subjected to numerical analysis assuming for the tested alloys that the Heisenberg conditions were met and the critical factor β

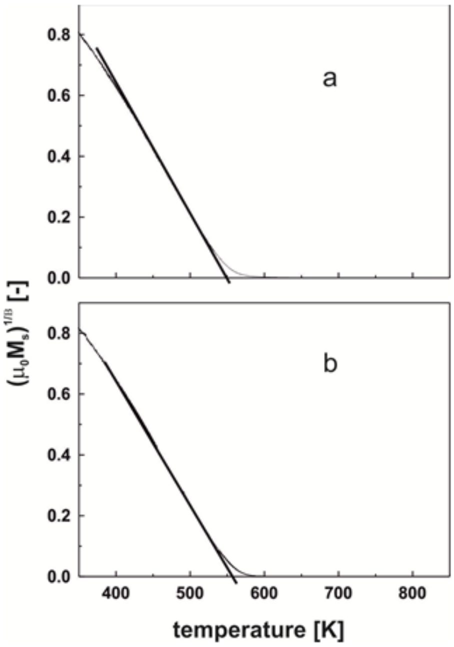

Fig. 5. Curie temperature for $Fe_{62}Co_9Y_8W_1B_{20}$ made by: a)injection, b) suction casting

was accepted as 0.36. Using this analysis, it was possible to determine the Curie temperature of the samples, which is consistent with figure 5.

For the amorphous alloys, accurate determination of the Curie temperature is very difficult. The reason for this is the state itself describing the amorphous structure. From the very definition of the amorphous material, it is impossible to determine the discrete Curie temperature value. In this type of alloys, chaos in the arrangement of atoms and various energy configurations is mentioned, which excludes the passage of the second type at a strictly defined temperature. In figure 6 there are the primary magnetization curves for the alloy produced. Powder samples were tested in the magnetic field up to 1.7T. The original magnetization curves for the samples shown in figure 6 are similar to each other. Based on the analysis of these curves, it can be concluded that the alloy was produced by suction casting, more easily magnetized to saturation. This is indicated by a lower value of effective anisotropy.

Figure 7 contains static magnetic hysteresis loops. The obtained magnetic hysteresis loops have a typical shape

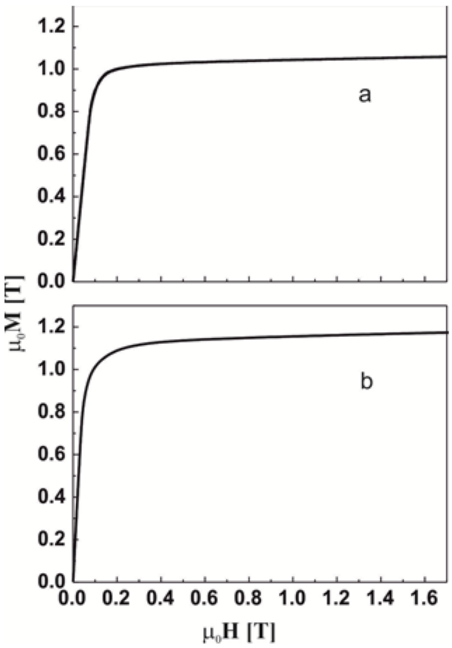

Fig. 6. Primary magnetization curves for $Fe_{62}Co_9Y_8W_1B_{20}$ made by: a) injection, b) suction casting

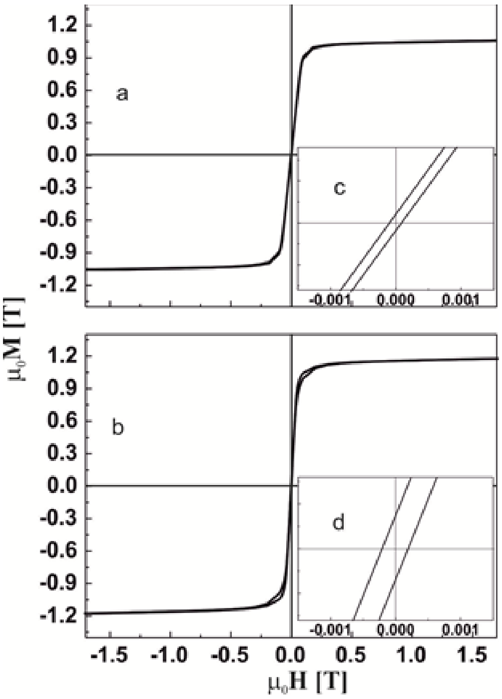

Fig.7. Coercive field for $Fe_{62}Co_9Y_8W_1B_{20}$ made by: a) injection, b) suction casting

as for magnetic materials with soft magnetic properties. Saturation magnetization for the prepared samples is over 1T. For the sample generated by suction, the value of saturation is slightly higher, which is indirectly explained by changes in the magnetic saturation polarization in the positive and negative directions. On the basis of the analysis of the static hysteresis loop in the middle of the M-H system, the value of restraint intensity can be calculated (fig. 7c, d). From figure 7c, d it follows that the magnetization process in the areas 1-3 describing the magnetization curve of soft magnetic materials occurs for the sample produced by suction.

Data in table 1 summarizes the calculated parameters based on the analysis of magnetic saturation polarization curves and static hysteresis loops. The magnetic properties of the $Fe_{62}Co_9Y_8W_1B_{20}$ alloy noticeably differ from each other depending on the manufacturing method.

The Curie temperature determined for the studied alloy differs by 8K depending on the method of its production. This value is relatively small in the case of amorphous alloys, for which the Curie temperature is not described as a discrete value, it may lie within the limit of the change in the position of magnetic atoms in the volume of the alloy as a result of their diffusion. The difference in magnetization of saturation for the method of producing the alloy is 0.11T. It would be expected that the coercive field value for the same alloy produced by different methods should be similar.

As can be seen from the data in table 1, the coercive field value for the alloy produced by the suction method is more than five times higher than for the alloy produced by the injection method.

Table 1

MAGNETIC PROPERTIES OF $Fe_{62}Co_9Y_8W_1B_{20}$ MADE BY INJECTION AND SUCTION CASTING

$Fe_{62}Co_9Y_8W_1B_{20}$	Injection	Suction
Curie temp. [K]	549	557
Saturation [T]	1.06	1.17
Coercive field [A/m]	41	159

Conclusions

Paper presents study results for $Fe_{62}Co_9Y_8W_1B_{20}$ alloy made by two methods: injection and suction of molten alloy to copper water cooled mold. It should be noted that the parameters for the production of rapid cooled alloys for both methods were the same. The only difference in their preparation was the method of introducing a liquid alloy into the copper mold. In the above mentioned methods, the cooling rate of the alloy is the same and is about 10^1 - 10^3K/s. On the basis of the analysis of X-ray diffractograms, it can only be stated that there are no well developed crystallographic systems present in the volume of the alloy, regardless of the method of its production. At this point, it must be added that the description of the amorphous structure is very difficult. The reason for this fact is the lack of a pattern for its characterization. Therefore, it should be assumed that the properties of such materials should be the same. However, this is not the case as can be seen.

During the process of fast solidification of the alloy, diffusion processes occur between atoms, which affects the chemical and topological order. It is from this that the values of the obtained magnetic parameters depend mainly. The configuration of magnetic pairs Fe - Fe, Fe - Co and the closest surrounding of central atoms play a major role in the formation of the magnetic structure. Changes in magnetic parameters in the tested alloy, taking into account the method of its production, are visible in the shapes of magnetic saturation polarization curves and static hysteresis loops. These differences, however, are insignificant what to expect. However, with a thorough analysis and in particular one of the main parameters for soft magnetic materials, that is for the coercive field values, these differences are clear. The change in the value of restraint to the sample produced by the suction method in relation to the injection method is 358%. This means that the method of manufacturing itself can affect the application capabilities of the tested alloy. It is obvious that the re-magnetization of the alloy produced by the suction method requires four times more energy. From an economic and environmental point of view, these types of alloys should not be produced by suction. Additionally, it should be taken into account that the cost of fabrication of the material by the method of injection is lower and the time of its production is shorter. These economic factors clearly indicate that in the case of the massive amorphous alloy in the form of a plate and taking into account only the soft magnetic properties, a better variant is the production of an alloy by injection casting.

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