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# The Structure and Properties of Rapid Cooled Iron Based Alloy

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**Abstract.** In this paper we studied the structure of rapidly cooled alloy which composition was based on iron. Samples were prepared using arc melting under protective atmosphere of argon and then casted in the process of rapid cooling into water cooled copper mold. Samples of the same composition were also made using the liquid melt casting method on a spinning copper roll. The high purity samples in the form of rods and ribbons were obtained. As expected, the obtained samples were characterized by an amorphous structure as confirmed by Mössbauer spectroscopy and X-ray diffraction studies.

## 1. Introduction

Rapidly cooled alloys have been the focus of science and industry for decades. Rapidly cooled alloys are those which are produced in such a way as to ensure a fast cooling of the melted ingredients resulting in significant changes in the structure of the resulting material. Rapidly cooled alloys differ significantly from their classic crystalline counterparts, with identical chemical composition. Despite the identical chemical composition, the alloy obtained by the rapid cooling method is significantly different in comparison to the conventional alloy [5-8].

Rapidly cooled materials are usually obtained by cooling the molten material. Suitable high cooling rate results in changes in the structure of the resulting material. Depending on the chemical composition of alloy, cooling rate and alloy manufacturing methods, different structures can be obtained. When talking about fast-cooled alloys, amorphous alloys and alloys with only partial crystallization of the structure are considered. Such alloys depending on the size of the crystallized grains may be called nanocrystalline. Nanocrystalline alloys may also be in the form of an amorphous matrix containing crystalline phase grains of nanometer size. Amorphous and nanocrystalline materials are extremely



interesting due to their superior magnetic properties compared to classical counterparts [9-15].

The unique properties of fast-cooled alloys are directly derived from their structure. The high cooling speed prevents crystallization of the material, creating a structure characterized by the absence long range order of atoms. Production of alloys by rapid cooling causes disturbances in the structure of the material, resulting in structural defects in the form of free volumes and pseudodislocational dipoles [16-23].

The purpose of rapid cooling is to obtain an amorphous or only partially crystallized structure. The high speed of occurrence of the crystallization process greatly complicates the preparation of such materials. The need for a very fast cooling of the material reduces the size of the alloys produced, which is one of the biggest problems with rapidly cooled alloys. Rapidly cooled materials are often obtained on the base of iron. This type of material is characterized by very good magnetic properties such as low loss for demagnetization and high magnetization saturation. Rapidly quenched alloys are produced at various rates of cooling. The needed speed depends primarily on the chemical composition of the material. pending on the alloy produced, cooling rates of the order  $10^5$  K/s for classical amorphous materials and about  $10^2$  K/s for the production of massive amorphous alloys [24-28].

In this paper the results of structural studies of fast-cooled iron based alloys are presented. Samples were made of multi-component alloy  $\text{Fe}_{61}\text{Co}_{10}\text{Hf}_{2.5}\text{Zr}_{2.5}\text{Ti}_2\text{W}_2\text{B}_{20}$ .

## 2. Materials and experiments

The study used elements with a purity of 99.99%. The alloy components were weighed on the analytical balance. The accuracy of the weight and the purity of the elements ensure the desired alloy composition. The weighed alloy components were mixed together and then melt with a plasma arc on a water-cooled copper plate. Because of the high reactivity of oxygen with alloying elements, the batch melting process is carried out in a protective atmosphere of argon. Several times of ingot melting resulted in high homogeneity of the produced material, thus ensuring high reliability of the conducted investigations of the properties of the produced material. A fast-cooled alloy was obtained in the form of rod and ribbons. The used method of casting allows typical cooling rate of  $10^0 - 10^2$  K/s.

The melted material was injected under pressure into a water-cooled copper mold. Ribbons were made by fast cooling of liquid melt on a rotating copper roll. The structure of the obtained alloy was examined using X-ray diffractometer and structural studies using Mössbauer spectroscopy. X-ray diffraction was performed using Bruker's Advanced D8 automated x-ray equipment. The diffractometer was equipped with a semiconductor counter and CuK $\alpha$  X-ray tube. Measurements were made for powdered samples for  $2\Theta$  angle ranging from  $30^\circ$  to  $120^\circ$ . Samples were exposed for 7 seconds per measurement step at the resolution  $0.02^\circ$ . Mössbauer research was performed using the Polon Mössbauer spectrometer. The Mössbauer spectrometer was equipped with the source  $^{57}\text{Co}(\text{Rh})$  with 50 mCi activity. Mössbauer spectra were analyzed using NORMOS software developed by R.A. Brand gave the possibility of obtaining a distribution of hyperfine induction fields of on the  $^{57}\text{Fe}$  nucleus [17]. Structural studies were performed for samples in the post-solidified and post-heat-treated state. Samples were heated in a vacuum oven at 850K for 30 minutes. X-ray and Mössbauer studies were performed for low-energy powdered samples. The average grain size present in the sample volume in the form of a rod after the isothermal heating process at 850K / 30minutes was estimated based on the Scherrer's formula:

$$D = \frac{\lambda \cdot K}{2B_0 \cos \Theta}$$

K – Scherrer's coefficient (set to  $K = 0.91$ )

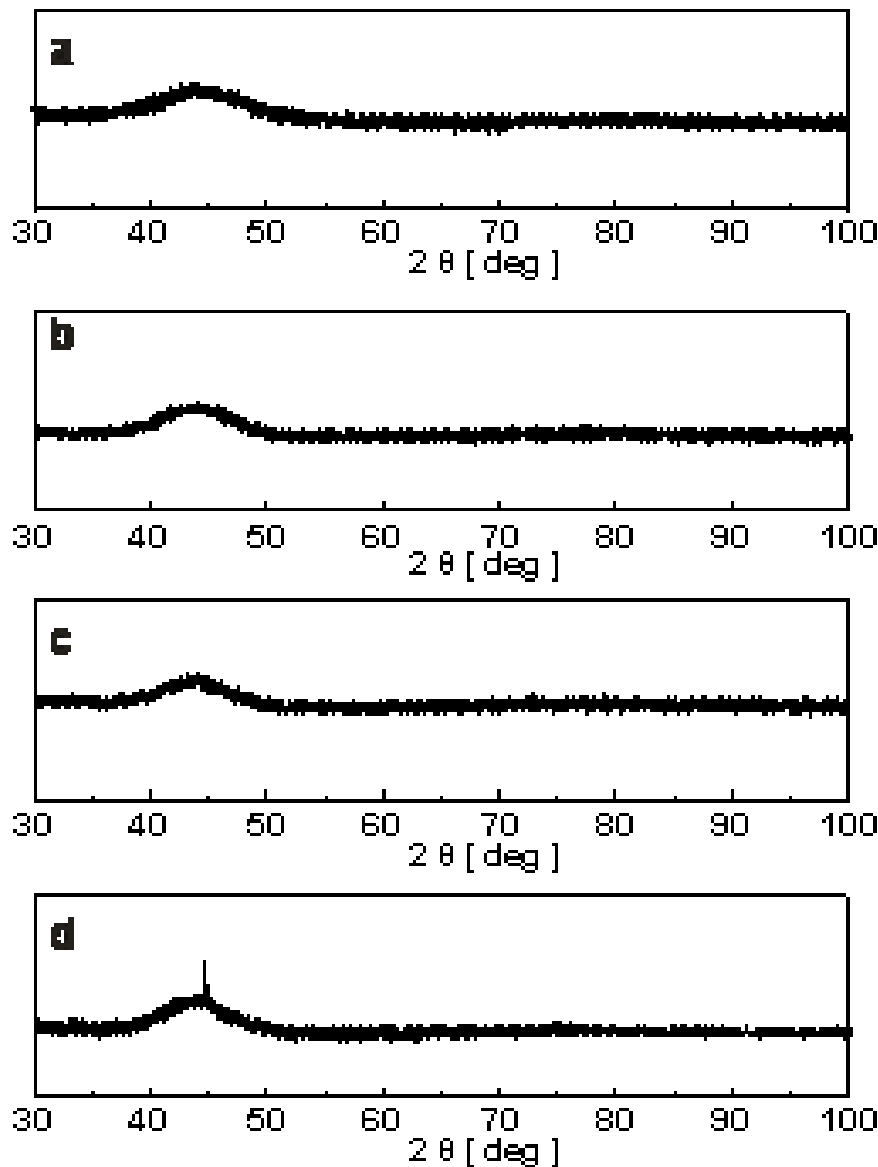
$\lambda$  – characteristic wavelength

$B_0$  – peak half width (also background was considered)

$\Theta$  – Bragg angle

### 3. Results

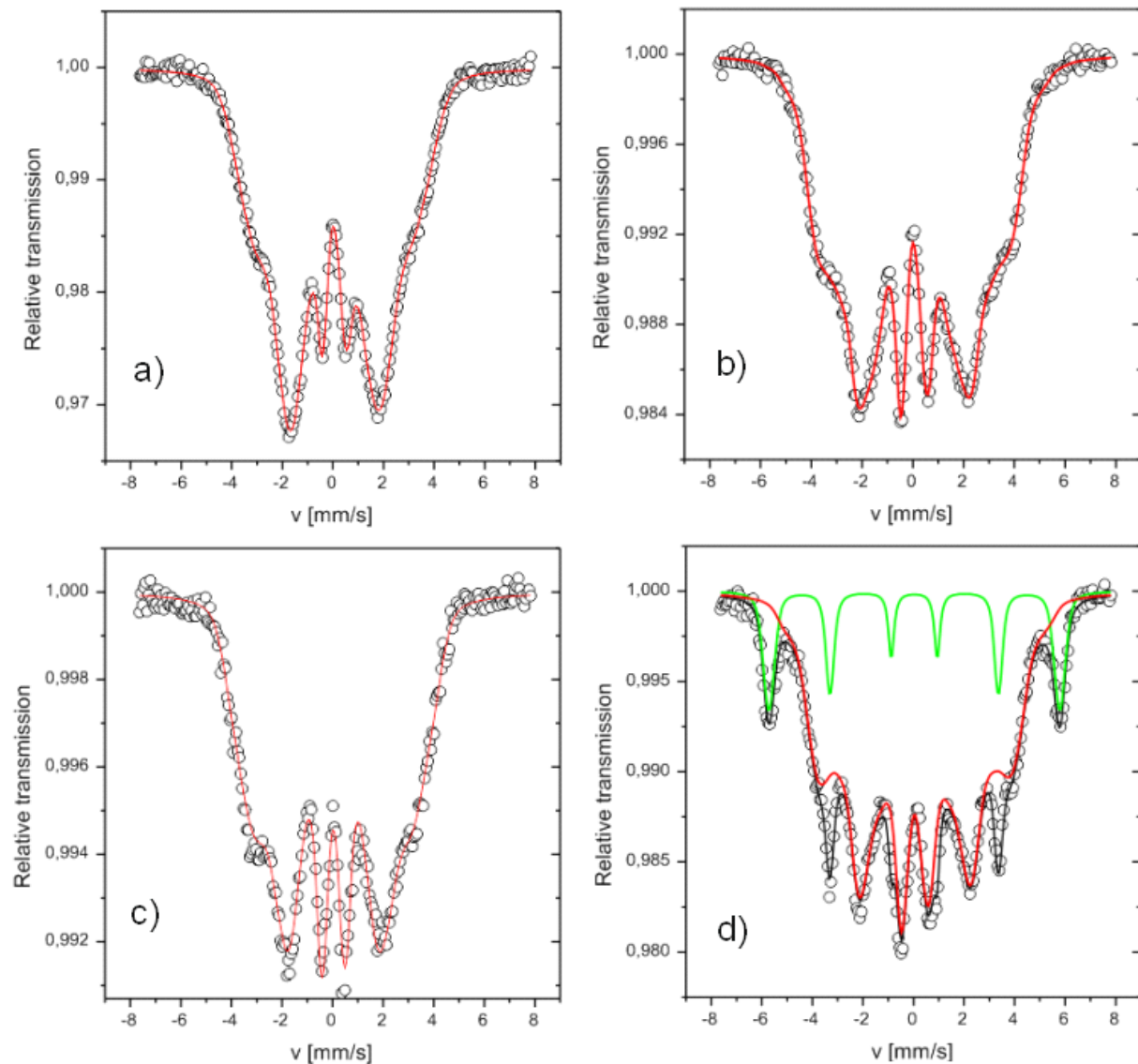
X-ray diffraction patterns from the analyzed samples are shown in figure 1.



**Figure 1.** The XRD images for sample  $\text{Fe}_{61}\text{Co}_{10}\text{Hf}_{2.5}\text{Zr}_{2.5}\text{Ti}_2\text{W}_2\text{B}_{20}$  in as cast state (a, c) and after isothermal annealing at 850K/30 minutes (b, d): a, b – ribbon 35  $\mu\text{m}$ , c, d – rod of 1 mm diameter.

Shown in figure 1 (a, b) diffraction images for the samples after solidification state are typical of materials with an amorphous structure. They consist only of a single broad maximum known as the amorphous halo. Thermal treatment temperature for the alloy samples  $\text{Fe}_{61}\text{Co}_{10}\text{Hf}_{2.5}\text{Zr}_{2.5}\text{Ti}_2\text{W}_2\text{B}_{20}$  in the form of ribbons and rods, was determined on the basis of differential scanning calorimetry (DSC). This temperature was determined slightly below the crystallization temperature for both two sample shapes. Therefore, it should be expected that the pretreatment of both rods and strips should not lead to the formation of privileged systems in the volume of alloys that meet the laws of spatial spacing and angular translations between atoms. In the case of a ribbon sample (figure 1b), the conducted isothermal

treatment did not result in a crystalline structure in the volume of the material. On the other hand, performing the same thermal treatment (850 K) for a rod sample (figure 1d) resulted in a decrease in the energy state of the material due to topological and chemical changes which resulted in the emergence of fine nuclei seed of the crystalline phase. X-rays penetrate into the sample just for a few interplanar distances. Therefore, to obtain reliable X-ray results, they should be done for powdered samples. Using the Scherrer equation, the mean crystal particle size present in the rod sample after the isothermal heating process was calculated.



**Figure 2.** Mössbauer transmission spectrum recorded for the investigated alloy  $\text{Fe}_{61}\text{Co}_{10}\text{Hf}_{2.5}\text{Zr}_{2.5}\text{Ti}_2\text{W}_2\text{B}_{20}$  in as cast state (a, c) and after isothermal annealing at 850K/30 minutes (b, d): a, b – ribbon 35 $\mu\text{m}$ , c, d – rod with 1 mm diameter.

The estimated grain size is 7 nm. Obtaining a finely granular structure at nanometric grains may be a factor in improving the properties of a given material. At present, metallic nanomaterials are heavily displacing their crystalline equivalents with the same chemical composition. The analysis of the sample structure was performed using a more sensitive test method, which is Mössbauer's spectroscopy. It is assumed that in the case of X-ray diffraction, samples can be analyzed with a minimum volume of

crystalline phase of 7% relative to the rest of the material. Mössbauer studies, on the other hand, provide the possibility to determine the volume fraction of the crystalline phase present in the sample even at less share.

Figure 2 shows the Mössbauer spectra recorded for the samples of the alloy  $\text{Fe}_{61}\text{Co}_{10}\text{Hf}_{2.5}\text{Zr}_{2.5}\text{Ti}_2\text{W}_2\text{B}_{20}$  in the state after solidification and after thermal treatment at 850K for 30 minutes. Mössbauer spectra obtained for samples in the as cast state in the form of a strip and rod are similar. Their shape is typical for samples with an amorphous structure. They consist of a broad asymmetric overlapping lines.

A similar shape of the Mössbauer spectra was observed for the strip sample after the annealing process. This means that the soaking process itself did not result in the formation in the sample volume of lower free energy areas corresponding to the crystalline phase.

It is known that raising the temperature promotes diffusion processes that take place in the sample volume. Amorphous systems are characterized by many states with different free energy values making them metastable. It follows that the amorphous state itself has many configurations of energy states which, with each successive jump through the potential barriers, reduce their free energy and eventually reach crystalline state. The initial phase of crystallization of amorphous alloys is manifested in the formation in the volume of alloys of clusters with similar arrangement of atoms from which crystalline seed is formed and then grains. It is assumed that such systems are evenly distributed in the volume of the material. This is the case with biphasic materials of an amorphous and crystalline structure. In the case of a rod sample subjected to isothermal heating at temperature 850K/30 min.

There is an additional line on the Mössbauer transmission spectrum associated with the presence of the crystalline phase in the sample (which is consistent with results of the X-ray examinations). Based on the Mössbauer spectra on  $^{57}\text{Fe}$  nuclei an analysis was made.

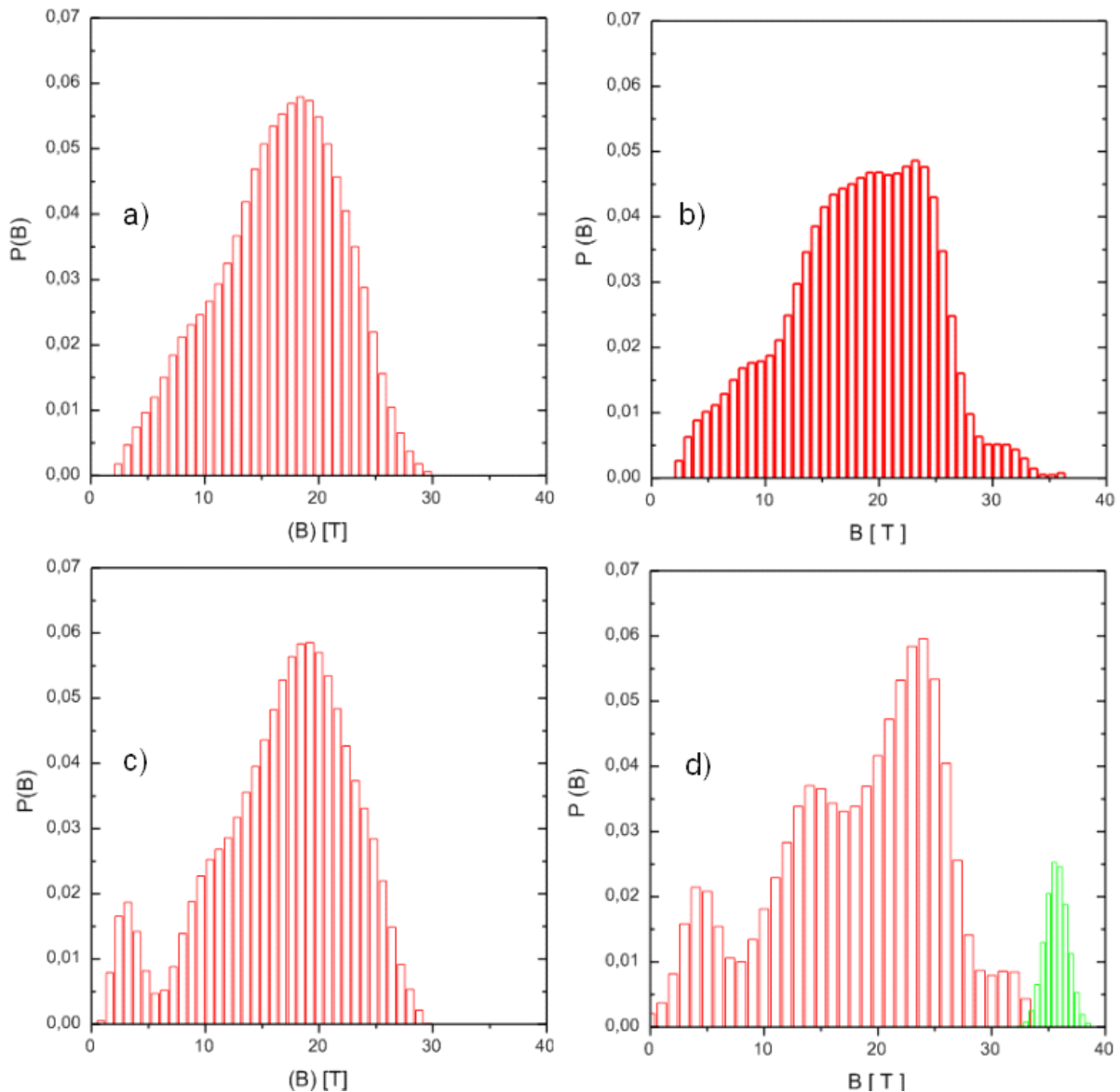
Figure 3 shows the distributions of the superfine field induction for the investigated samples. For the sample in the shape of ribbon in the as cast state and after isothermal annealing process the distributions of hyperfine induction fields are similar. There are clearly visible two lines low- and high-field components. The mean value of the low-field component for the ribbon sample in the as cast state is about 7 T whereas the value of this component for the same sample after heating is 10 T. The similar tendency is observed for second well seen high field component, where for sample in as cast state its value is about 16 T and after isothermal annealing is 20 T. In addition, it has to be stated that for the sample after heating in the distribution of induction of the hyperfine induction fields appeared the third component with the least probability occurring in the fields range from 25 to 35 T. Configuration changes in the environment of  $^{57}\text{Fe}$  are connected with nearest atoms. It is evident that the process of isothermal annealing influences the magnetic properties of the ribbon of  $\text{Fe}_{61}\text{Co}_{10}\text{Hf}_{2.5}\text{Zr}_{2.5}\text{Ti}_2\text{W}_2\text{B}_{20}$  alloy.

Based on a visual analysis of the distribution of the hyperfine induction field for a sample annealed at 850 K, it can be concluded that clusters of atoms begin to form in the volume of the melt. This is evidenced by the presence of the third field component and is consistent with the thermodynamics of amorphous systems.

A completely different shape of hyperfine induction is observed for rod shape sample. For the rod in as cast state in the distribution of hyperfine field there are three components, in which the low-field component is well separated. The mean values of induction of hyperfine fields are about 3 T, while for the components in the higher fields the values are 11 and 19 T.

After heating of rod samples, the shape of the induction of the hyperfine fields has changed considerably. It was possible to distinguish at least 5 components of which 4 were related to amorphous structure and one to crystalline structure. The mean values of induction of superfine fields associated with the amorphous structure were respectively: 5, 14, 23 and 30 T. The field component located at the highest magnetic induction is associated with the appearance of clusters with similar atomic configurations. The average value for this field was about 36 T.





**Figure 3.** Mössbauer transmission spectrum recorded for the investigated alloy  $\text{Fe}_{61}\text{Co}_{10}\text{Hf}_{2.5}\text{Zr}_{2.5}\text{Ti}_2\text{W}_2\text{B}_{20}$  in the as cast state (a, c) and after isothermal annealing at 850K for 30 min. (b, d): a, b – strip 35  $\mu\text{m}$ , c, d – rod of 1 mm diameter.

#### 4. Conclusions

Using suitable laboratory equipment an amorphous  $\text{Fe}_{61}\text{Co}_{10}\text{Hf}_{2.5}\text{Zr}_{2.5}\text{Ti}_2\text{W}_2\text{B}_{20}$  alloy samples be obtained in the form of strips of 35  $\mu\text{m}$  width, and rods of 1 mm diameter. Determining the right temperature and time for the isothermal annealing process is a decisive factor for the structure of the materials under study. It is known, the structure determines the properties of alloys. It has been shown in the present paper that the temperature of the heat treatment did not caused crystallization of the alloy in the form of a ribbon. However, as the Mössbauer studies indicate, changes in the topological and chemical arrangement of atoms in the amorphous state occurred in the volume of the sample. This is related to the reconfiguration to lower energy metastable states. Amorphous ribbons are produced at much higher cooling rates ( $10^4 - 10^6$  K/s) than massive amorphous rods ( $10^0 - 10^3$  K/s). The shape of the Mössbauer spectra obtained for rod samples was quite different from that of ribbon shaped samples. This means that the manufacturing process and the cooling speed of the liquid alloy itself have the

greatest influence on the forming of the structure. For larger specimens where the solidification time is longer, volume diffusion occurs at further distances, which promotes the formation of energy-prone systems such as clusters of atoms or nuclei.

### References:

- [1] McHenry M E, Willard M A and Laughlin D E 1999 *Prog. Mater. Sci.* **44** 291
- [2] Lesz S, Szewczyk R, Szewieczek D and Bienkowski A 2004 *J. Mater. Process. Tech.* **743** 157-158
- [3] Inoue A, Shen B L and Chang C T 2006 *Intermetallics* **14** 936-944
- [4] Shen B and Inoue A 2004 *Applied Physics Letters* **85** (21) 4911-4913
- [5] Hasiak M, Sobczyk K, Zbroszczyk J, Ciurzyńska W, Olszewski J, Nabiałek M, Kaleta J, Świerczek J and Łukiewska A 2008 *IEEE Trans. Magn.* **11** 3879-3882
- [6] Sobczyk K, Świerczek J, Gondro J, Zbroszczyk J, Ciurzyńska W, Olszewski J, Brągiel P, Łukiewska A, Rzącki J and Nabiałek M 2012 *J. Magn. Magn. Mater.* **324** 540-549
- [7] Pawlik P, Nabiałek M, Żak E, Zbroszczyk J, Wysłocki J J, Olszewski J and Pawlik K 2004 *Arch. Mat. Sci.* **177** 25/3
- [8] Inoue A, Yano N and Masumoto T 1984 *J. Mater. Science* **19** 3786-3795
- [9] Wang W H, Dong C and Shek CH 2004 *Materials Science and Engineering R* **44** 45-89
- [10] Li W, Yang Y Z and Xu J 2017 *Journal of Non-Crystalline Solids* **461** 93-97
- [11] Willard M A and Daniil M 2013 in: *Handbook of Magnetic Materials*, Vol. 21, ed Buschow K H J (London: North Holland) p 173
- [12] Hasiak M, Sobczyk K, Zbroszczyk J, Ciurzyńska W, Olszewski J, Nabiałek M, Kaleta K, Świerczek J and Łukiewska A 2008 *IEEE Trans. Magn.* **44** 3879
- [13] Gondro J, Świerczek J, Rzącki J, Ciurzyńska W, Olszewski J, Zbroszczyk J, Bloch K, Osyra M and Łukiewska A 2013 *J. Magn. Magn. Mater.* **341** 100-107
- [14] Chiriac H and Lupu N 2001 *Physica B: Condensed Matter* **299** (3-4) 293-301
- [15] Sun H J, Man Q, Dong Y Q, Shen B, Kimura H, Makino A and Inoue A 2010 *J. Alloys Comp.* **504** 31
- [16] Gruszka K, Nabiałek M, Szota M, Bloch K, Gondro J, Pietrusiewicz P, Sandu A V, Mustafa Al Bakri A M, Walters S, Walters K, Garus S, Dośpiał M and Mizera J 2016 *Arch. Metall. Mater.* **61** 641-644
- [17] Makarov V A, Belenkii A Ya and Kozlova O S 1993 *Phys. Stat. Sol. (A)* **139** 173-179
- [18] Nabiałek M, Pietrusiewicz P, Błoch K and Szota M 2015, *Int. J. Mater. Res.* **106** 682-688
- [19] Pietrusiewicz P, Błoch K, Nabiałek M and Walters S 2015 *Acta Phys. Pol. A* **127** 397-399
- [20] Gondro J, Błoch K, Brągiel P, Nabiałek M and Szota M 2016 *Arch. Metall. Mater.* **61** 451-456
- [21] Kronmüller H 1981 *Journal of Applied Physics* **52** (3) 1859-1864
- [22] Lange H and Kronmüller H 1986 *Phys. Stat. sol. (a)* **95** 621-633
- [23] Kronmüller H 1984 *J. Magn. Magn. Mater.* **41** 366
- [24] Nabiałek M 2016 *Arch. Metall. Mater.* **61** 439-444
- [25] Inoue A, Shen B and Ohsuna T 2002 *Mater. Trans.* **43** (9) 2337-2341
- [26] Inoue A 1998 *Mater. Sci. Foundations* 6, Trans Tech Publications, 1-116
- [27] Sanchez L I J L, Bustamante S R, Barthem V M T S and de Miranda P E V 2005 *Journal of Magnetism and Magnetic Materials* **294** 131-135
- [28] Inoue A 1997 *Materials Science and Engineering A* **226-228** 357-363.
- [29] Brand R A 1987 *Nuclear Instruments and Methods in Physics Research B* **28** 398-416
- [30] Gu B X, Xue D S, Shen B G and Li F S 1997 *J. Magn. Magn. Mater.* **167** 105
- [31] Szewieczek D and Lesz S 2005 *J. Mater. Process. Tech.* **162-163** 254