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Investigations of temperature distribution in metallic glasses fabrication process

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ABSTRACT

Purpose: The goal of paper is investigations of temperature distribution which is appearance during fabrication process of metallic glasses. In present work particular attention focused on system for registration of temperature distribution.

Design/methodology/approach: Bulk metallic glasses in the composition as the following: $Fe_{36}Co_{36}B_{19.2}Si_{4.8}Nb_4$ were fabricated by the die casting method. Distribution of temperature was carried out by a prototype measure system. Investigations were realized for casting process of the samples in form of rods with diameter 2, 3 and 4 mm. Temperature distributions were executed for series of samples. Moreover, investigations also enclosed structure characterization tested by X-ray diffraction and SEM.

Findings: On the base of temperature distribution curves it can be observed that during casting of metallic glasses a temperature gradient have been occur. It should be note that prototype system allows to measure temperature only in cooper mould not inside of sample. Diffraction patterns confirmed that structure of tested samples was amorphous. Electron microscope observations revealed fracture morphology which is characteristic fore glassy structure.

Practical implications: Analysis of temperature during casting process plays an important role in effective fabrication of metallic glasses. Cooling rate can be estimated on the base of results these analyse. Knowing the cooling rate, it could be possible to determine the glass forming ability of studied alloy.

Originality/value: Investigations which have been taken in present work are novelty for the sake of optimization of casting process not only for metallic glasses, but also for nanocrystalline engineering materials.

Keywords: Bulk Metallic Glasses; Cooling rate; Temperature distribution; Temperature measurement; Casting

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1. Introduction

Metallic glass is a metal or alloys which exhibit amorphous structure. There is no one precisely definition of "amorphous structure". Generally, we could say that material have amorphous structure when size of arrangement area is no bigger than 1 nm. But for obtaining an amorphous structure molten alloy (or metal) must be cooling with high rate [1-4]. Those conditions caused that nucleation and growth of crystallite are not occur and structure of material remains structure of liquid. Assurance of demand cooling rate depends on few factors e.g: chemical composition of alloy and its temperature of melting point or technology of production of metallic glass. Among fabrication methods of metallic glasses it could be distinguished melt spinning, centrifugal casting, pressure die casting (into copper mould), suction casting [3, 5-9]. Cooling conditions are different in each method, however, in melt spinning method alloy have contact with copper barrel on one side. In this method it could be possible obtain metallic glass in form of thin ribbons. In centrifugal casting method thanks to centrifugal force the samples in the form of ring could be formed. Suction casting method using system of vacuum pump for introducing molten alloy into copper mould. Often, for fabrication bulk metallic glasses in the form of rods or plates pressure die casting method is using. This technique was described in next chapter. In every fabrication method it is very difficult to set directly the cooling rate. That is why there is necessary to realized many tests of casting and optimization of casting conditions [2,3,6].

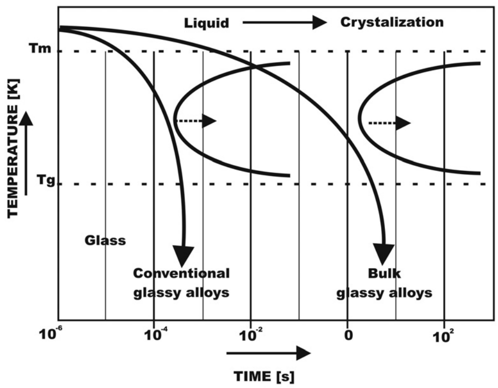

Fig. 1. Critical cooling rate for conventional and bulk metallic glasses [3,5]

Fe based metallic glasses are attractive for many engineering applications because of their ultrahigh strength, excellence corrosion resistance and relatively low cost of material [10-14]. Fe-based metallic glasses could be produced as conventional or bulk form. It should be known that preparation of conventional metallic glass required cooling rates of about 10^4-10^6 K/s but for bulk metallic glasses cooling rate may be equal only 10^3 K/s [1, 3-5, 14]. However, it is also dependent on chemical composition of alloy. For multicomponent alloys cooling rate may be lower.
In this work authors successfully cast the $Fe_{36}Co_{36}B_{19,2}Si_{4,8}Nb_4$ bulk glassy rods and realized the temperature measurements which could be helpful to determination cooling rate of bulk metallic glasses.

2. Experimental technique

2.1. Casting

In order to realization of casting process, first the master alloy with chemical composition $Fe_{36}Co_{36}B_{19,2}Si_{4,8}Nb_4$ (Table 1) was prepared. Pure elements were mixed and melted by induction technique. To better homogeneity process of melting was made few times. Moreover, it was realized in protective atmosphere (Fig. 2).

Table 1.
Shape, purity and composition of alloying elements

Elements	Shape	Purity [%]	Composition	
			at. [%]	mass. [%]
Fe	solid particles	99.75	36	41.53
Co	flat solid particles	99.89	36	43.83
B	pieces	99.9	19.2	4.29
Si	pieces	99.9	4.8	2.67
Nb	powder	99.85	4	7.68

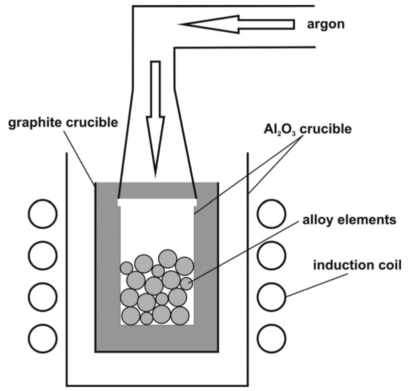

Fig. 2. Scheme of master alloy preparation

Next, alloy ingot was crushed and put into quartz crucible in order to sample casting. Samples were performed by pressure die casting. Alloy which was put into crucible was melted and thanks to gravitation force and argon pressure, injected into mould (Fig. 3).

2.2. Temperature measurement

In order to measure change of temperature during bulk metallic glasses fabrication the prototype system was created. (Fig. 4). A basic component of system were termoelements type K standardized in Poland, which could be used for temperature measurement range up to 1100°C. The other components of system are:

- digital temperature gauges,
- digital-analog card,
- PC with software.

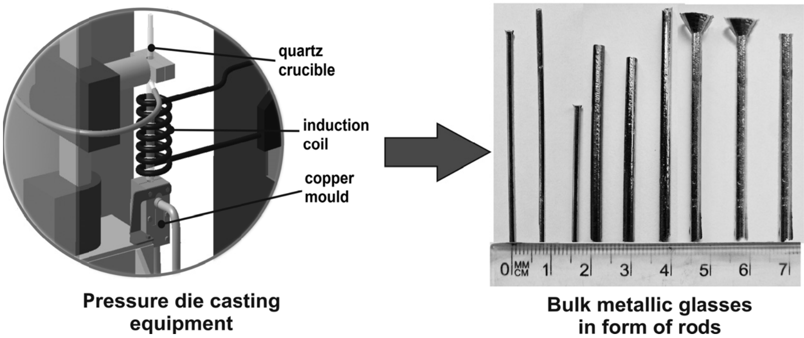

Fig. 3. Schematic illustration of the pressure die casting method used for fabrication of bulk metallic glasses

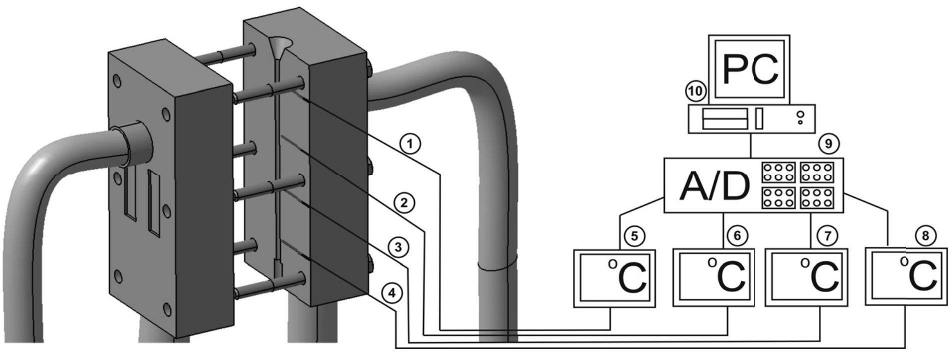

Fig. 4. Schematic illustration of system to measuring temperature distribution: 1, 2, 3, 4 - termoelements type K; 5, 6, 7, 8 - digital temperature meters; 9 - analog-digital card; 10 - PC with software

Investigations were realized for samples with diameter 2, 3 and 4 mm. Measurements were done in four points of copper mould. Points were arranged along mould, within the distance of 16, 39, 62 and 85 mm from top of the mould. Temperature was read at a distance equal to 4 mm from sample core. This means that temperature was measure not into the sample but only in mould. Measurement method follows from fact, that if the termoelement will be located inside molten alloy, it will be destroyed during casting. Additionally, a presence of termoelement inside alloy could disturb solidification process. Before measurement, termoelements were put into grooves which were milled on internal side of one half of mould. Next the casting process was started. Simultaneously, computer software which enables measurements was activating. The registration of data was started when temperature on first termoelement (on distance 16 mm from top of the mould) reach specified value. Time of measurement equals 3s. After registration the copper mould was disassembled in order to remove sample. For next casting, installation of termoelements was necessary again.

2.3. Structure characterization

Structure analysis of the sample in as-cast state was carried out using X-ray diffractometer (XRD) with $Co_{K\alpha}$ radiation. The data of diffraction lines was recorded by “step-scanning” method in 2θ range from 40° to 90°. The fracture morphology of casting samples in the form of rods with diameter 2, 3 and 4 mm were analysed using scanning electron microscope (SEM).

3. Results and data analysis

On the base of temperature measurement the cooling curves were draw. Each curve was assign by average of temperature for three measurements which is realized in repeatable conditions. Figures 5-7 present results of temperature measurements. For each diameter four curves (for all measuring points within the distance of 16, 39, 62 and 85 mm from top of the mould) are shown. It could be observed that for sample with diameter 2 mm the highest temperature occurs on the distance 16 mm (Fig. 5a). Because of near to inlet and the influence of molten alloy which was in crucible the temperature may increase in this point. Additionally, there is no influence of water chamber which are in mould in order to better cooling. The curves were stabilized after 0,75s, when temperature reaches about 50°C. Only for point on the distance 16 mm temperature was still higher and decreased much more lower.

Fig. 6 shows the cooling curves for sample with diameter 3 mm. As could be seen for this sample temperature on the measuring start for the first point (for a distance 16 mm) is higher than for 2 mm. In last point (for a distance 85 mm) temperature reaches about 350°C on the start.

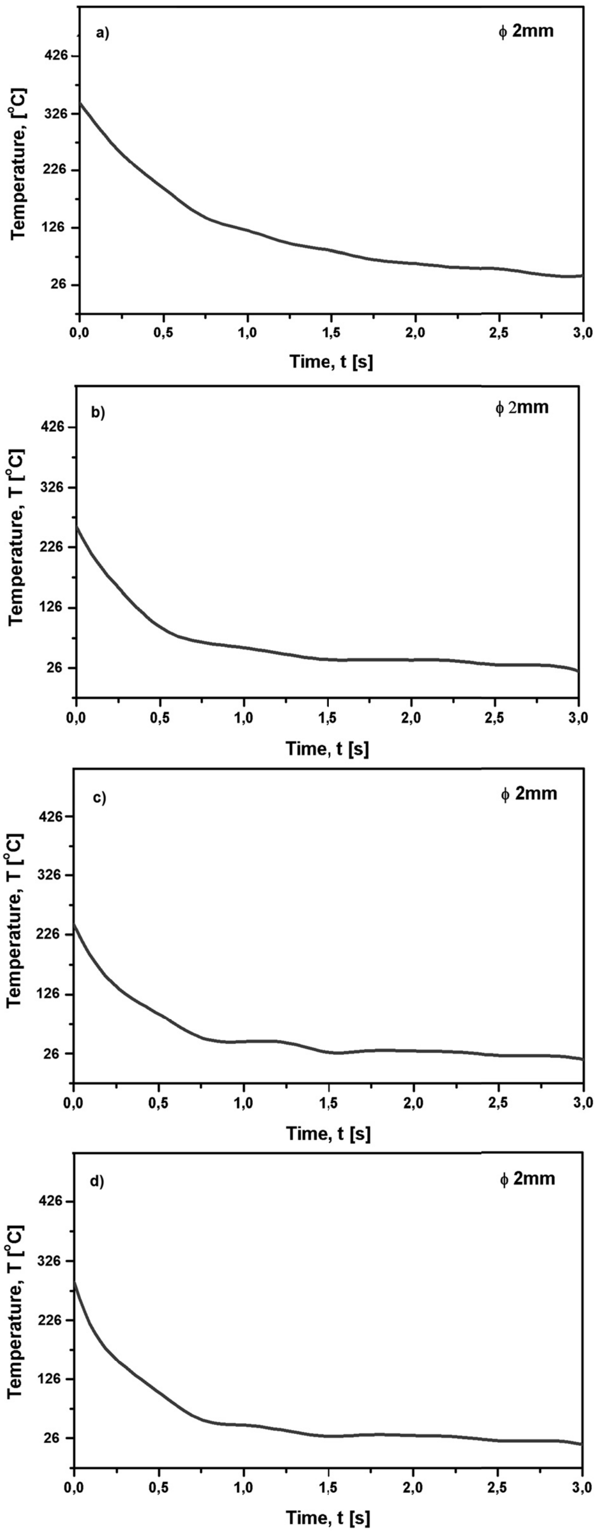

Fig. 5. Cooling curves determinated in four measuring points for sample with diameter 2 mm: a) distance 16 mm, b) distance 39 mm, c) distance 62 mm, d) distance 85 mm

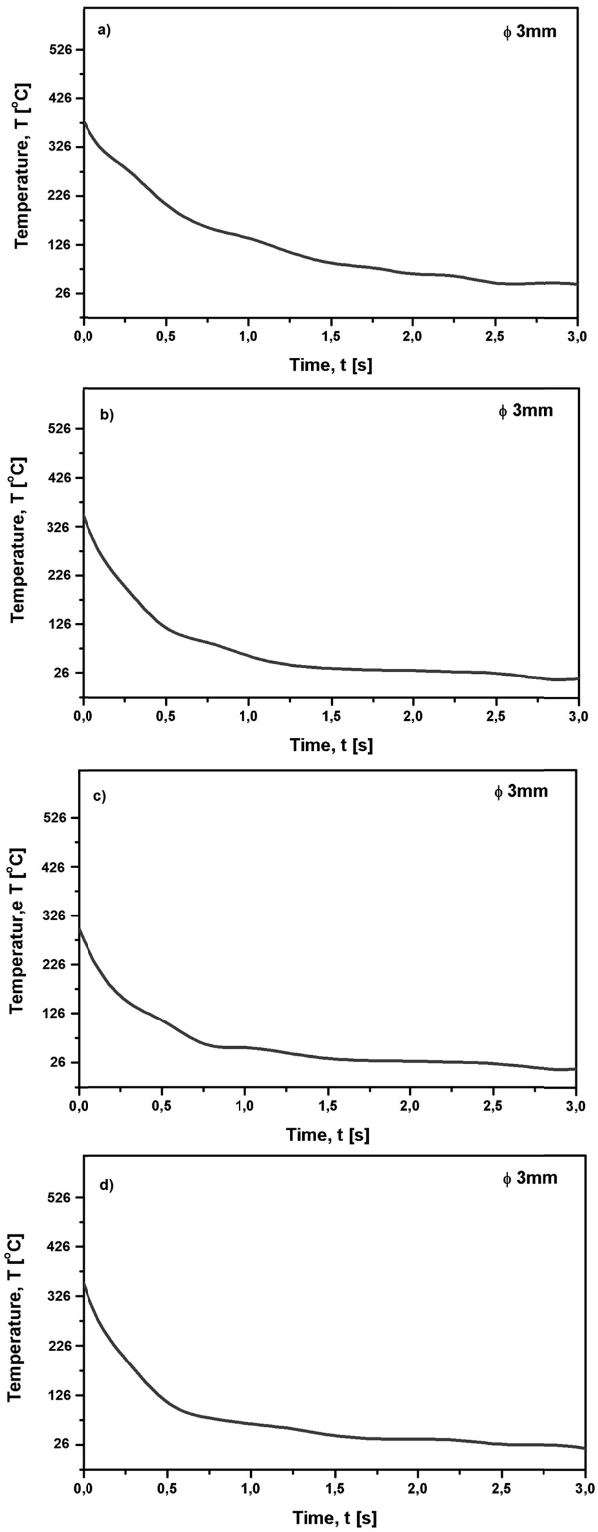

Fig. 6. Cooling curves determinated in four measuring points for sample with diameter 3 mm: a) distance 16 mm, b) distance 39 mm, c) distance 62 mm, d) distance 85 mm

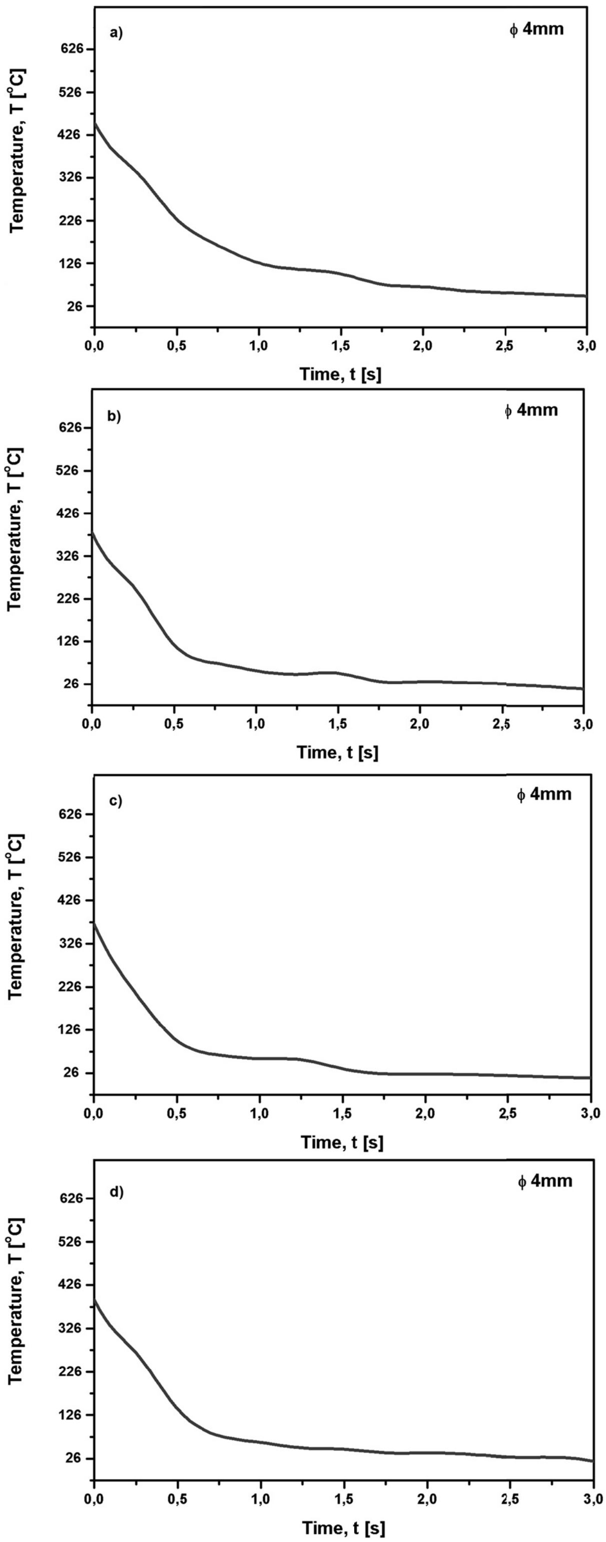

Fig. 7. Cooling curves determinated in four measuring points for sample with diameter 4 mm: a) distance 16 mm, b) distance 39 mm, c) distance 62 mm, d) distance 85 mm

Temperature measurement was carried out also for sample with diameter 4 mm. Fig. 7 presents cooling curves which were drawn on the base of the results for those test. On the measurements start temperature obtain highest value of tree samples diameters and it is equaled about 440°C for measuring point for a distance 16 mm and about 380°C for last measuring point (for a distance 85 mm).

It is clearly should be note that with change of diameter of sample the temperature distribution is different. The significant role plays in this case amount of molten alloy, of course, which heats the copper mould. An important aspect of temperature distribution results is also a geometry of casting mould. The presence of cooling chamber in which flow the water during casting process, cause that temperature of molten alloy is different in different parts of mould (points of measurement). A water caused the better cooling conditions in places which are near the water chambers.

The X-ray diffraction investigations reveal that the studied as-cast $Fe_{36}Co_{36}B_{19,2}Si_{4,8}Nb_4$ samples were amorphous. The diffraction patterns (Fig. 8) of tested rods with diameter of 2, 3 and 4 mm show the broad diffraction halo characteristic for the amorphous structure

The surface fracture morphology of glassy rods was investigated by SEM method at different magnifications. A present work shows only selected micrographs. Figures 9-11 show micrographs of as-cast glassy rods. Tests reveal a fracture that is regular for amorphous structure. It could be classified as mixed fracture with two regions: "vein" patterns and "smooth" areas [15]. Different morphology could be a result of different cooling conditions during fabrication process or maybe different state of amorphous structure in this part of sample.

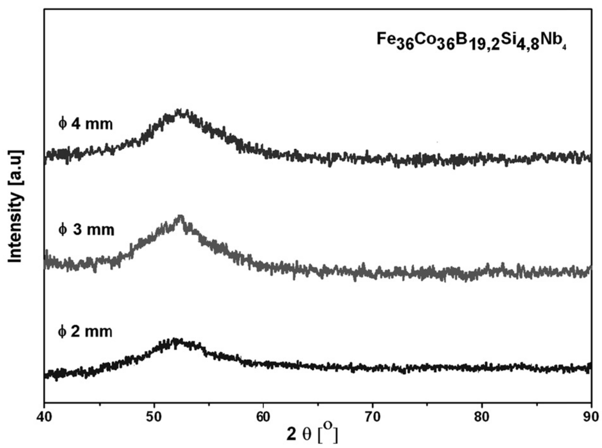

Fig. 8. X-ray diffraction patterns of $Fe_{36}Co_{36}B_{19,2}Si_{4,8}Nb_4$ glassy rods in as-cast state with diameter 2, 3 and 4 mm

4. Conclusions

The goal of investigation in a present work was measuring of temperature distribution during casting process of metallic glasses.

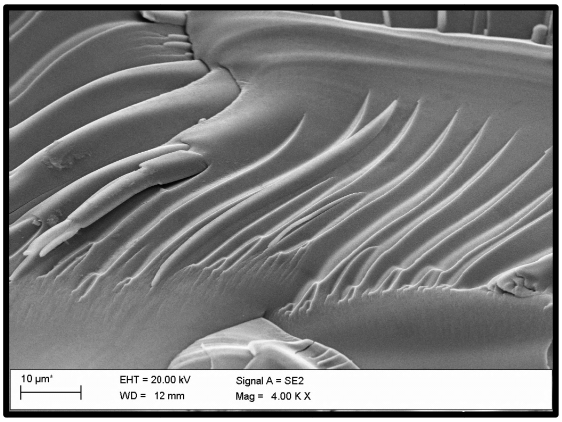

Fig. 9. SEM micrograph of the fracture morphology of $Fe_{36}Co_{36}B_{19,2}Si_{4,8}Nb_4$ amorphous rod in as-cast state with diameter 2 mm, magn. 4000x

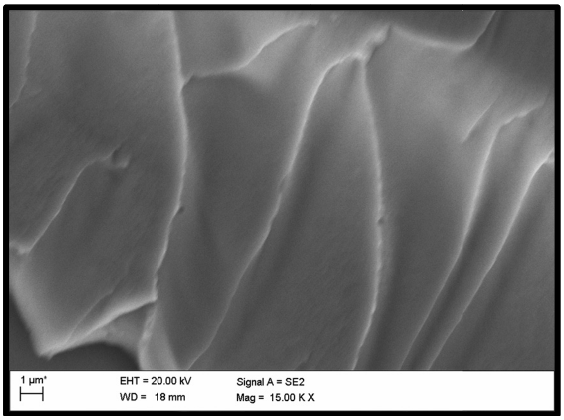

Fig. 10. SEM micrograph of the fracture morphology of $Fe_{36}Co_{36}B_{19,2}Si_{4,8}Nb_4$ amorphous rod in as-cast state with diameter 3 mm, magn. 15000x

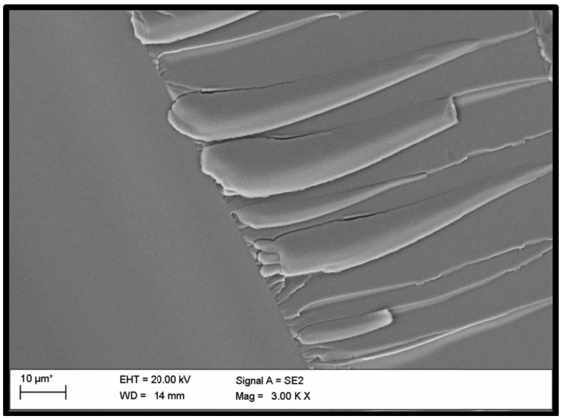

Fig. 11. SEM micrograph of the fracture morphology of $Fe_{36}Co_{36}B_{19,2}Si_{4,8}Nb_4$ amorphous rod in as-cast state with diameter 4 mm, magn. 3000x

The present results have shown that temperature during fabrication of $Fe_{36}Co_{36}B_{19,2}Si_{4,8}Nb_4$ bulk metallic glasses depended on diameter of casting sample. Moreover, the measuring points were located for different distances from top of the mould, and in every point the distribution of temperature was also diversified. Measurements which were realized within a framework of present investigations could be good beginning to knowing a cooling rate for analyzed alloy and also for another alloys systems.

The structure characterization of samples in as-cast state in the form of rods with diameter 2, 3 and 4 mm reveal that all samples exhibit amorphous structure. It should be noted the XRD investigations included only surface examination. However, a SEM observation shows fracture morphology typically for glassy structure. On the base SEM results it could be assumed that in cross section of sample structure is also amorphous. On the micrographs, it could be seen bands which may testify about different fracture mechanism in particular regions of sample.

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