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Structure and properties of Fe-Cr-Mo-C bulk metallic glasses obtained by die casting method

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Materials

ABSTRACT

Purpose: The goal of this work is to investigate structure and properties of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy rods with different diameters obtained by the pressure die casting method.
Design/methodology/approach: Master alloy ingot with compositions of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ was prepared by induction melting of pure Fe, Cr, Mo, C elements in argon atmosphere. The investigated material were cast in form of rods with different diameters. Glassy and crystalline structures were examined by X-ray diffraction. The microscopic observation of the fracture morphology was carried out by the SEM with different magnification. The thermal properties of the studied alloy were examined by DTA and DSC method.
Findings: These materials exhibit high glass-forming ability, excellent mechanical properties and corrosion resistance.
Research limitations/implications: It is difficult to obtain a metallic glass of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy. The investigations carried out on the different samples of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ bulk metallic alloy allowed to state that the studied ribbon was amorphous whereas rods were amorphous – crystalline.
Originality/value: The formation and investigation of the casted Fe-Cr-Mo-C bulk materials and the study of glass-forming ability of this alloy.
Keywords: Metallic glasses; Bulk Metallic Glasses; Glass-forming ability; Fe-based alloys; Thermal properties

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1. Introduction

The glass forming ability (GFA) is the ability of an alloy melt to form a glassy phase on solidification. The GFA of an alloy indicates whether the alloy is a candidate for glass formation in bulk forms using conventional casting processes. It is very important parameter in designing and developing new BMG with unique properties [1-5]. The GFA is the frequently determined by measuring the maximum section thickness (t_{max}) or by estimating the lowest critical cooling rates (R_c) to produce the bulk metallic glasses (BMGs). Glass formation requires prevention of formation of crystalline phase when the melt is quenched. This involves suppression of the crystal nuclei formation or suppression of the growth of crystalline phase. When the melt is significantly under cooled to enough low temperatures so that the viscosity of the under cooled melt is high and consequently it gets frozen into the glassy state [1, 2, 6, 7].

Various empirical parameters have been proposed to specify the GFA of BMGs. A lot of GFA indicators have been determined by measuring the characteristic thermal parameters. A few simple criteria were developed to explain the GFA of alloys [1, 6, 8, 9]:

1) The T_{rg} criterion – the alloy should have a high reduced glass transition temperature. T_{rg} is defined as the ratio of the glass transition temperature T_g to the liquidus temperature T_l of the alloy, $T_{rg} = T_g / T_l$. When the liquid alloy is cooled from the molten state to the glass transition temperature, the viscosity of the melt increases to a high value and the glass is formed. The T_{rg} value of the known metallic glasses varies between 0.4 and 0.7;
2) The ΔT_x criterion – the supercooled liquid region ΔT_x is described as the temperature difference between T_g and T_x ($\Delta T_x = T_x - T_g$). T_x is the onset temperature of the first crystallization peak in the DSC curve. The width of the supercooled liquid region is a good measure of the glassy phase stability. The GFA should increase with ΔT_x increasing;
3) The γ parameter is defined as $\gamma = T_x / (T_g + T_l)$. This parameter could be used to most satisfactorily explain the GFA of metallic glasses;
4) The δ parameter is defined by $\delta = T_x / (T_l - T_g)$. Q. Shen et al. [2] claim that δ is a better GFA indicator than T_{rg} and γ;
5) Critical cooling rate (R_c) is the minimum cooling rate required to allow the alloy melted to be cast into a bulk glass. It is very difficult to obtain the R_c value on an alloy. This value has to be determined by time consuming experimentation or heat transfer calculations;
6) The maximum section thickness or diameter (D_{max}) of a plate or rod like BMG sample are quantifiable parameters.

All GFA indicators exhibit a proportional relationship with respect to the maximum thickness D_{max} indicating that all these parameters can reflect the GFA of the alloys.

Inoue [6, 8, 10-13] has formulated three empirical rules for achieving high GFA:

1) The alloy system should contain at least three components;
2) The atomic size difference between the constituent elements must be at least 12%;
3) The alloying elements must have a large negative of mixing.

According to Inoue these criteria are useful in selecting alloy compositions that could be easily formed into glasses.

For the last decade, there has been intense activity to synthesize and characteristic bulk metallic glasses. A lot of problems demand explanation or extension, for example [6, 14, 15]:

1) The reasons why some alloys can be easily formed into glasses while others can't are still not clearly known;
2) If reasons for the stability and formation of the glassy phase are known, then it would be possible to design new alloy compositions to increase the GFA and to obtain BMG alloys of larger diameters for commercial applications;
3) The real reasons for the improved GFA of many alloys are still not clear and ability to design alloy compositions to enable synthesis of larger diameter rods has not improved;
4) The limitations of size and shape as well as low thermal stability have prevented a further extension of their applications fields.

The bulk glassy alloys in Mg-, Ln- and Zr- based systems have a large supercooled liquid region before crystallization and high resistance against crystallization. They exhibit good corrosion resistance too. Preparation of BMG on light elements based is very difficult and the materials are very expensive. So, for the last few years, Fe-, Co-, Ni- based systems have been significantly extended [16-31].

S. J. Pang et al. have found that melt spun Fe-Cr-Mo-C-B alloys have a high GFA and good corrosion resistance in aggressive HCl solutions [32]. Fe- based bulk glassy alloys exhibit high mechanical strength, soft magnetic properties and high corrosion resistance. They have produced and tested the $Fe_{50-x}Cr_{16}Mo_{16}C_{18}$ system alloy. The ΔT_x is about 54-60 K and the T_g / T_m is as high as 0.62-0.63 at this system. The corrosion rates of the obtained alloys with a diameter of 1.2 mm are in the range of 10^{-3}-10^{-2} year^{-1} in 1, 6, 12 N HCl solution at room temperature. The high corrosion resistance is due to the formation of chromium – rich passive film during immersion in HCl solutions [32].

Q. Chen et al. [1] investigated the GFA of the $Fe_{48-x}Co_xCr_{15}Mo_{14}Y_2C_{15}B_6$ system alloys. They found that these alloys had an extraordinary GFA, enabling for motion of a large glassy ingot with a critical casting size of at least 16 mm. The thermal parameters, the maximum sample diameters and calculated GFA parameters of the Fe-Cr-Mo-C system alloys are listed in Table 1. In the work [33] the origins for high GFA and good corrosion resistance for the Fe-Cr-Mo-C-B-P metallic glasses were discussed. The $Fe_{43}Cr_{16}Mo_{16}(C,B,P)_{25}$ alloys can be formed to a glassy state over a wide composition range. Bulk metallic glasses with a maximum thickness (t_{max}) of 1.0-2.7 mm were synthesized in this system alloy. The obtained glasses exhibit a large ΔT_x of 40-90 K, high T_g / T_l of 0.54-0.60 K, which indicating a high GFA and high thermal stability.

This paper reports the examine of the glass forming ability of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ metallic glasses.

Table 1.
Thermal stability of the selected bulk glassy Fe-Cr-Mo-C system alloys [1, 32, 34]

Glassy alloy	Diameter [mm]	T_g [K]	T_x [K]	ΔT_x [K]	T_m [K]	T_g / T_m	γ	δ
$Fe_{46}Cr_{16}Mo_{16}C_{18}B_4$	1.2	862	915	53	1389	0.62		
$Fe_{44}Cr_{16}Mo_{16}C_{18}B_6$	1.2	870	932	62	1414	0.62		
$Fe_{42}Cr_{16}Mo_{16}C_{18}B_8$	1.2	887	947	60	1405	0.63		
$Fe_{65}Cr_9Mo_8C_{10}B_6Er_2$	≥ 5	796	823	27	1430	0.55	0.373	
$Fe_{64}Cr_{10}Mo_9C_{15}Er_2$	≥ 5	803	850	47	1429	0.56	0.381	
$Fe_{48}Cr_{15}Mo_{14}C_{15}B_6Y_2$	7	839	886	47	1464	0.57	0.385	1.418
$Fe_{45}Co_3Cr_{15}Mo_{14}C_{15}B_6Y_2$	8	834	880	46	1446	0.57	0.386	1.438

2. Materials and research methodology

The aim of the presented work is the examination of the GFA of Fe-Cr-Mo-C system alloy. The studies were carried out on bulk metallic materials as rods and ribbons too. Multicomponent alloy with nominal composition of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ were investigated. Fe- based ingot with specified composition were prepared by induction melting of the pure Fe, Cr, Mo, C elements in argon atmosphere (Table 2). The ingot was melted double. From the master alloy, ribbon samples of 0.015 mm thick and 0.5 mm wide were prepared by a single – roller melt – spinning technique in an argon atmosphere. The investigated materials were cast in form of rods with diameters of ø = 1.5 mm, ø = 2 mm, ø = 3 mm, ø = 4 mm. The rods were prepared by the pressure die casting method. The master alloy was melted in a quartz crucible using an induction coil and pushed in a copper mould by applying an ejection pressure.

Table 2.
Chemical composition of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy

No	Elements	mass. [%]	at. [%]
1	Fe	57.82	54.00
2	Cr	15.95	16.00
3	Mo	22.07	12.00
4	C	4.14	18.00

The structure of rapidly solidified ribbon and rod specimens was examined by X-ray diffraction using a Seifert – FPM XRD diffractometer with Co Kα radiation at 35 kV. The data of diffraction pattern lines were recorded by means of the stepwise method within the angular range of 30º to 80º (or 30º to 100º). The counting time in the measuring point was 3s.

The thermal stability associated with glass transition temperature, supercooled liquid and crystallization temperature was investigated by differential scanning calorimetry and differential thermal analysis at a constant heating rate of 10 K/min.

The microscopic observation of the fracture morphology of studied glassy and crystalline materials in form of ribbon and rods with different diameter was carried out by means of the Supra 25 made by Zeiss factory scanning electron microscope, within different magnification.

3. Results

The ribbons and bulk alloy samples in rod form were produced and investigated. The structure of cast $Fe_{54}Cr_{16}Mo_{12}C_{18}$ ribbons were examined by X-ray diffraction, DSC, DTA and SEM method. The samples of determined chemical composition with a thickness of 0.015 mm consist of a single glassy phase as was evidenced from a main halo peak without crystalline peaks in their X-ray diffraction patterns. One of the obtained halo peak of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ ribbon is presented in Fig. 1. The analysis of fractures of tested ribbon (Fig. 2.) gives information of the existence of smooth areas and zones contained fluvial patterns.

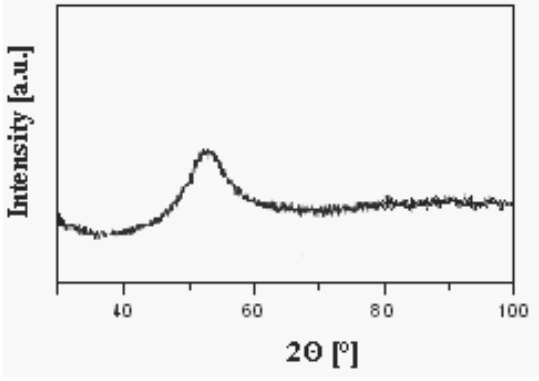

Fig. 1. X-ray diffraction pattern of the $Fe_{54}Cr_{16}Mo_{12}C_{18}$ amorphous melt – spun ribbon

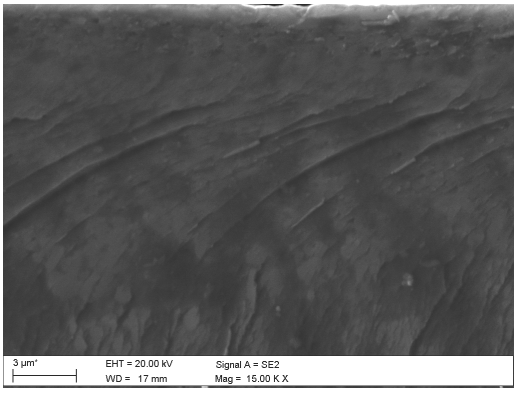

Fig. 2. SEM micrographs of the fracture morphology of the $Fe_{54}Cr_{16}Mo_{12}C_{18}$ ribbon

The ribbon has a smooth surface and metallic luster. DSC curve of the metallic glasses in a ribbon form exhibit glass transition, supercooled liquid region and crystallization. The glassy ribbon exhibit the ΔT_x is as large as about 84 K, implying a high thermal stability of the supercooled liquid.

In this work, a series of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ metallic glasses were prepared by copper mold casting method in rod form. Figure 3 shows the DSC curves of the bulk metallic rods with a diameter of 1.5 mm; 2 mm; 3 mm; 4 mm. For the metallic glasses of the same composition with different diameters, no obvious difference in their DSC curves was recognized. The values of T_g, T_x and ΔT_x are close to each other. The thermal parameters, the maximum sample diameters and calculated GFA parameters of the $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy in rod form are listed in Table 3. These metallic alloys exhibit the sequential transition of the glass transition temperature, supercooled liquid region and crystallization temperature. It noticed that the ΔT_x value is in the range from 24 to 84 K. The largest ΔT_x value was obtained for $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy in rod with a diameter of 1.5 mm.

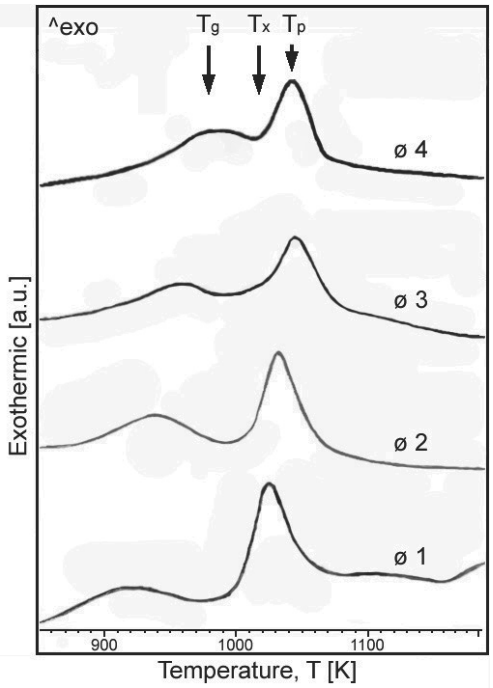

Fig. 3. DSC curve of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy in as cast state in different diameter

In Figs. 4 and 5 the pictures of fracture surface as a 2 mm diameter rod obtained in different magnification (1000x, 5000x) were shown. The analysis of fractures of tested rods gives information of the existence of smooth areas and zones contained fluvial and shell patterns. The fracture surface of two zones probably informed about different amorphous and crystalline structure of the tested materials. Veining fractures form at the place of the direct contact of metallic liquid with walls of mould.

Total different morphology of fracture surface of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy in different diameter (middle of samples) is presented in Figs. 6 and 7. These pictures illustrate the fractures of teeming lap morphology.

Smooth area of the fracture and veining exist at the edges of rods, whereas the teeming laps in the middle part of the samples. Similar phenomenon was observed for residual rods.

The alloys were also checked with EDS attachment to identify chemical composition of chosen areas. Chemical analysis of this areas show the presence of Fe, Cr, Mo and C elements. The choosen curve of the X-ray dispersive of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy is presented in Fig. 9.

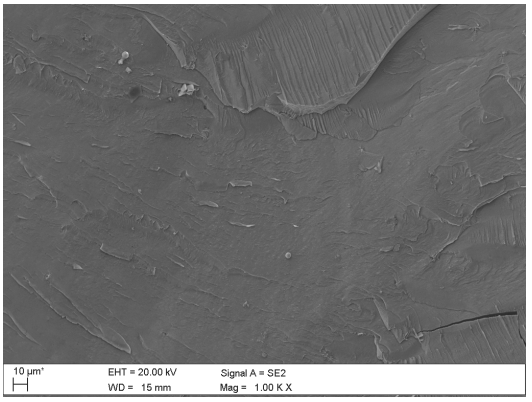

Fig. 4. SEM micrographs of the fracture morphology of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ amorphous rod in as – cast state with diameter of 2 mm (edge of sample)

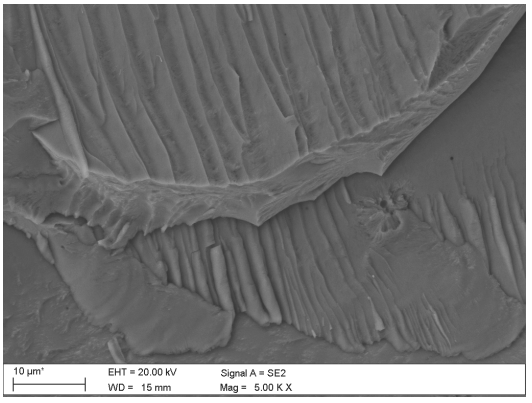

Fig. 5. SEM micrographs of the fracture morphology of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ amorphous rod in as – cast state with diameter of 2 mm (edge of sample)

Table 3.
Thermal properties of the studied $Fe_{54}Cr_{16}Mo_{12}C_{18}$ samples in a form of ribbon and rods with different diameter

sample	T_g [K]	T_x [K]	ΔT_x [K]	T_p [K]	Amorphous zone size [mm]
$Fe_{54}Cr_{16}Mo_{12}C_{18}$, ribbon, D = 0.015	926	1010	84	1031	
$Fe_{54}Cr_{16}Mo_{12}C_{18}$, rod, ø = 1,5 mm	937	1016	79	1034	0.30
$Fe_{54}Cr_{16}Mo_{12}C_{18}$, rod, ø = 2 mm	946	1011	65	1034	0.67
$Fe_{54}Cr_{16}Mo_{12}C_{18}$, rod, ø = 3 mm	950	1012	62	1032	0.85
$Fe_{54}Cr_{16}Mo_{12}C_{18}$, rod, ø = 4 mm	992	1016	24	1038	0.67

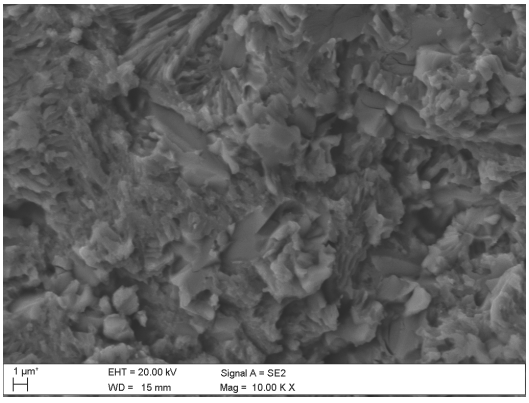

Fig. 6. SEM micrographs of the fracture morphology of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ rod in as – cast state with diameter of 1.5 mm (middle of sample)

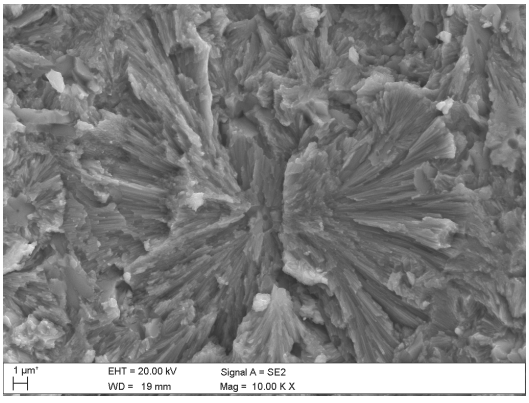

Fig. 7. SEM micrographs of the fracture morphology of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ rod in as – cast state with diameter of 4 mm (middle of sample)

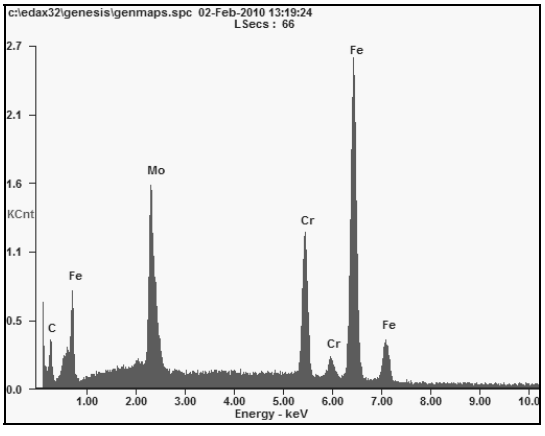

Fig. 8. Plot of the X-ray dispersive energy spectrometer measurement from the $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy in as-cast state with diameter of 2 mm (area in Fig. 4).

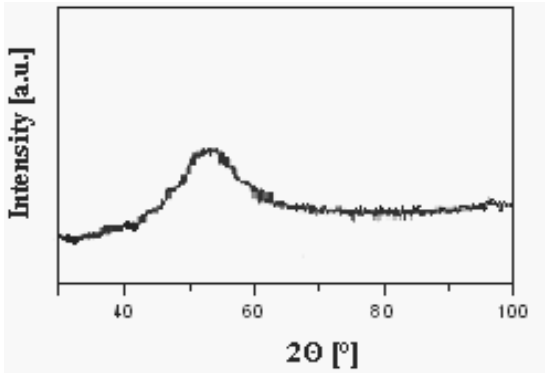

Fig. 9. X-ray diffraction pattern of the $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy rod with diameter 3 mm (amorphous, external zone)

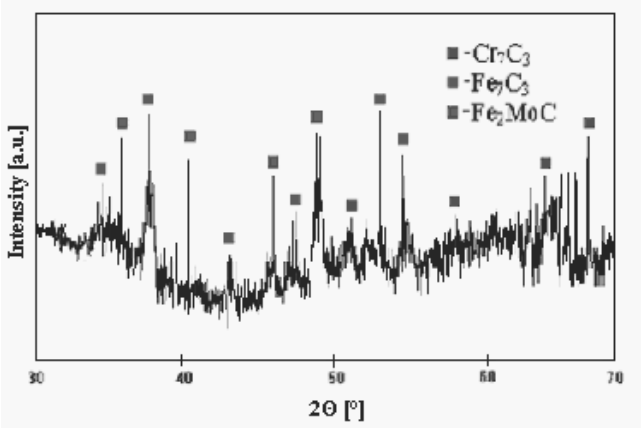

Fig. 10. X-ray diffaction pattern of the $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy rod with diameter 3 mm (crystalline, internal zone)

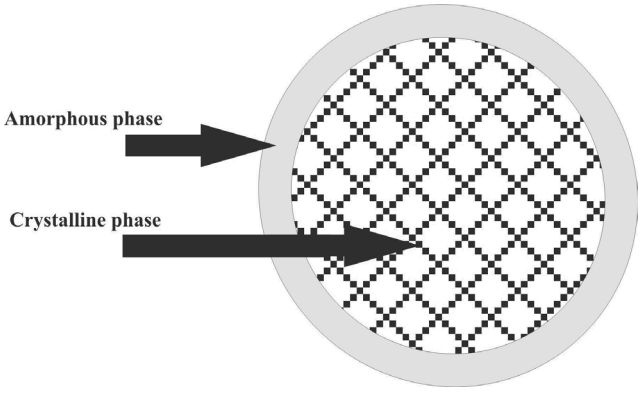

Fig. 11. Schematic illustration of sample

A production attempt of selected metallic glasses with Fe- based was carried out. Fig. 9 and Fig. 10 show the XRD patterns set of the $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy rod with diameter 3 mm. As a result, the amorphous – crystalline alloy rods with diameter ranking up to 4 mm were synthesized. Broad peaks without crystalline peaks can be seen for all obtained rods

of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy like for the rod with diameter 3 mm. Broad peaks are characteristic for external edges of 1.5 mm, 2 mm, 3 mm and 4 mm. The diffraction pattern recorded for cores of 3 mm rod shows the peaks characteristic for Cr_7C_3, Fe_7C_3 and Fe_2MoC phase.

Schematic illustration of samples are presented in Fig. 11. The provided investigations show that obtained rods are included of different areas. All samples were tested. In this paper only chosen results and pictures are exhibited.

4. Conclusions

To obtain high GFA two aspect should be taken into consideration: stability of the liquid phase and resistance of the glassy phase to crystallization. It is difficult to obtain a metallic glass of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy. Probably, the $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy composition was not exactly in eutectic point. Results of DSC investigations for rods confirmed that the peak crystallization temperature (T_p) increased while increasing the sample diameter. This may be the reason for the changes in the amorphous structure and crystalline structure at the sample diameter increase.

In this work, the glass forming stability of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy system has been specified by measuring thickness of ribbon and diameter of rods (maximum thickness of amorphous zone). Thin ribbon with thickness 0.015 mm were formed at $Fe_{54}Cr_{16}Mo_{12}C_{18}$ alloy by single copper roller melt spinning method. The rods exhibit a supercooled liquid region of 24-79 K indicating rather high glass forming ability and high thermal stability of the supercooled liquid. The amorphous zone size is equal 0.30 mm, 0.67 mm, 0.85 mm and 0.67 mm for rods with diameter of 1.5 mm, 2 mm, 3 mm, 4 mm, adequately.

In particular, the investigations carried out on the different samples of $Fe_{54}Cr_{16}Mo_{12}C_{18}$ bulk metallic alloy allowed to state that the studied ribbon was amorphous whereas rods were amorphous – crystalline. The XRD and SEM analysis showed that studied fractures of rods in – as cast state indicated structurally different zones. The characteristics of the fracture surface showed zones, that could by classified as a mixed types with fluvial, smooth areas and scaly structure.

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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