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Preparation and glass-forming ability of Mg-based bulk amorphous alloys

R. Babilas [a,*], A. Zajączkowski [b], W. Głuchowski [b], R. Nowosielski [a]

[a] Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-200 Gliwice, Poland

[b] Institute of Non-Ferrous Metals, ul. Sowińskiego 5, 44-100 Gliwice, Poland

* Corresponding e-mail address: rafal.babilas@polsl.pl

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ABSTRACT

Purpose: The work presents preparation methods, structure characterization and glass-forming analysis of Mg-based bulk metallic glasses in as-cast state.

Design/methodology/approach: The studies were performed on $Mg_{60}Cu_{30}Y_{10}$ and $Mg_{60}Cu_{29}Y_{10}Si_1$ glassy alloys in the form of plates and rods. The amorphous structure of tested samples was examined by X-ray diffraction (XRD). The thermal properties associated with solidus and liquidus temperature of master alloys were measured using the differential thermal analysis (DTA). The crystallization behavior of the studied plates and rods was also examined by differential scanning calorimetry (DSC). The fracture morphology of the rods in as-cast state was analyzed using the scanning electron microscopy (SEM).

Findings: The X-ray diffraction investigations revealed that the tested samples with different thickness and shape were amorphous. The single exothermic peaks describing crystallization process of studied alloys were observed for all examined samples with different thickness. The endothermic and exothermic peaks observed on DTA curves of master alloys allowed to determine the solidus and liquidus temperatures. The characteristics of the fractured surfaces showed different zones, which might correspond with different amorphous structures. The changes of glass transition and crystallization temperatures as a function of sample thickness were stated.

Practical implications: The pressure die casting method are useful technique to fabricate bulk amorphous materials in the form of plates and rods. Proposed casting technology could open new possibilities to easier preparation of Mg-based nanostructured materials and forming their properties that is essential for further applications.

Originality/value: The Mg-based bulk amorphous alloys are regarded as promising engineering materials with high strength, low density and good corrosion resistance in contrast to the crystalline alloys, due to their different atomic configurations

Keywords: Amorphous materials; Bulk metallic glasses; Mg-based alloys; Glass-forming ability

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MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

It is well known that metallic amorphous alloys also called as metallic glasses exhibit excellent properties such as high strength, large elastic elongation, low Young's modulus, high corrosion resistance, smooth surface and viscous deformability due to their unique structures of dense random atomic configurations, without lattice defects and existing of the free volume [1-3].

Many systems of bulk metallic glasses such as La-, Mg-, Zr-, Pd-, Fe-, Ni-, Cu- and Co-based alloys have been extensively studied because of their potential applications. Therefore the finding of new bulk amorphous alloys enables to apply them in industrial products with very high performance [4,5].

The search for bulk metallic glasses with high glass-forming ability and excellent properties is of great importance in the research field of amorphous engineering materials [6].

The high glass-forming ability allows to obtain bulk metallic glasses by copper mold casting with the good mechanical properties. These properties are fundamental for final applications as engineering materials [7].

The glass forming ability of metallic alloys is very important to the preparation of glassy samples. Moreover, there are some empirical parameters which can be used to estimate the glass-forming ability of metallic glasses [8]. The first of all is parameter called as the reduced glass transition temperature (T_{rg}). The reduced glass temperature is ratio between the glass transition temperature (T_g) and liquidus temperature (T_l) [9]. The temperature interval (ΔT_x) between the glass transition temperature (T_g) and the onset crystallization temperature (T_x) is another glass-forming ability parameter. It is also called as the supercooled liquid region [10].

The high glass-forming ability of Mg-based bulk metallic glasses together with good mechanical properties, low density, relatively low cost and easy recycling ability makes the Mg-based amorphous alloys attractive for engineering applications [11].

Mg-based alloys are often prepared in Mg-TM-RE system, where TM is transition metal element and RE is rare earth element. These amorphous alloys exhibit a large undercooled liquid region and high glass forming ability [12].

In 1991, Inoue et al. succeeded in preparing a bulk glassy alloy with chemical composition of $Mg_{65}Cu_{25}Y_{10}$ (at.%) by conventional copper mold casting method. The critical cooling rate for amorphous samples with diameter of 4 mm is about 10^2 K/s for this alloy system. Moreover, the Mg-based alloy exhibited high tensile fracture strength over 800 MPa which is higher than strength of conventional Mg-based crystalline alloys [13-20]. Unfortunately, the Mg-based bulk metallic glasses with high glass-forming ability have a lacking of ductility [7,21].

Generally, Mg-based glassy alloys can be classified into three groups: Mg-Cu based alloys, Mg-Ni based alloys and recently proposed Mg-Zn based alloys [22]. In addition, there has been reported another various alloy systems such as Mg-Cu-Ag-Y [21,23], Mg-Cu-Zn-Y [24], Mg-Cu-Si-Y [25] and Mg-Cu-Nb-Y [11], Mg-Cu-Gd [26,27], Mg-Cu-Tb [12,28], Mg-Cu-Y-Nd [29], Mg-Cu-Y-Ti [30] and Mg-Y-Ni [31]. Despite of this, most studies were done on Mg-Cu-Y alloys with various complementary element additions like Ag, Ni or Zn [26].

Unfortunately, Mg-based bulk metallic glasses have been found to be macroscopically brittle. The addition of a second phase to obtained composite material is an alternative way to improve their mechanical properties [32]. Mg-based amorphous alloys exhibit a very brittle manner with almost no plastic deformation. The brittleness of these alloys limits their engineering applications as constructional materials. This is why, it is important to achieve new Mg-based glassy alloys with improved strength and ductility. Detailed knowledge about the structure of these bulk metallic glasses could open new possibilities to better control or modify their properties that is essential for further applications [22].

In literature [11] it was reported that minor replacement of one of the elements in alloy system with an element having positive heat of mixing caused the improvement of properties of glassy materials. What is more, the addition of Nb or Si can also optimize the distribution of atomic sizes. The atomic radius of magnesium is 0.160 nm, copper - 0.128 nm, yttrium - 0.180 nm, niobium - 0.143 nm and silicon - 0.118 nm.

In this paper, the minor addition of Si into Mg-Cu-Y alloy is expected to increase the glass-forming ability of studied material.

2. Material and research methodology

The aim of the paper is the characterization of preparation method, microstructure, thermal and glass-forming properties of Mg-based alloys in as-cast state. Investigations were done by using of XRD, SEM, DTA and DSC measurements methods.

The studies were performed on $Mg_{60}Cu_{30}Y_{10}$ and $Mg_{60}Cu_{29}Y_{10}Si_1$ (at.%) master alloys and bulk metallic glasses in the form of rods and plates.

The main problem associated with the preparation of amorphous materials based on magnesium is to apply the appropriate cooling rate of the liquid alloy to avoid the crystallization process. An additional difficulty is the rapid oxidation of alloying elements. These difficulties could be minimized by choosing the appropriate method of preparation and using of appropriate equipment and taking into account of all the casting parameters.

To obtain Mg-based amorphous alloys the special technology has been developed by using pressure casting of molten alloy into the copper mould. The developed technology allowed to produce glassy materials with including the stage of preparation of master alloys and casting of bulk amorphous samples.

Fig. 1 presents the schematic way of preparing Mg-Cu-Y based master alloys. The studied alloy was prepared by two steps. The first stage included the preparing of binary alloy with pure elements of Cu (99.9%) and Y (99.9%) by induction melting under an argon atmosphere in a ceramic crucible. This alloy was then melted with Mg (99.9%) pieces in an electric furnace under an argon atmosphere to obtain a master alloy with the nominal composition (in atomic percentage).

The master alloy was re-melted in a protective atmosphere using induction melting, then injected into the copper mold by the pressure casting method to obtain glassy samples in the form of rods and plates. Moreover, Fig. 2 presents the pressure casting method which has been used to fabricate the samples of bulk metallic glasses in the form of rods [33-42]. The scheme includes main elements of the casting equipment which are installed in a protective atmosphere chamber. The proposed casting technology solved the problem of melting alloys with elements of different melting temperature.

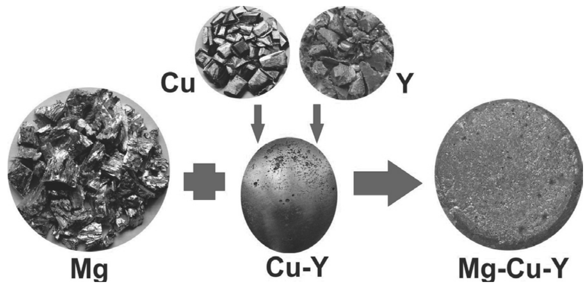

Fig. 1. Schematic illustration of the preparing master alloy with chemical composition of Mg-Cu-Y for casting bulk amorphous samples

Structure analysis of the samples in as-cast state was carried out by using of X-ray diffractometer with $Cu_{K\alpha}$ radiation. The data of diffraction lines were recorded by "step-scanning" method in 2θ range from 20° to 90°.

The fracture morphology of studied master alloys in the form of ingots and glassy samples in the form of rods in as-cast state was analyzed by using of scanning electron microscopy (SEM).

The solidus (T_S) and liquidus (T_L) temperature of studied Mg-based master alloys was measured by using of differential thermal analysis (DTA) at a constant heating rate of 6 K/s under an argon protective atmosphere.

Thermal and glass-forming properties associated with glass transition temperature (T_g), onset (T_x) and peak (T_p) crystallization temperature of first stage of crystallization of studied rods and plates was examined by differential scanning calorimetry (DSC). The heating rate of calorimetry measurements, under an argon protective atmosphere, was 20 K/min.

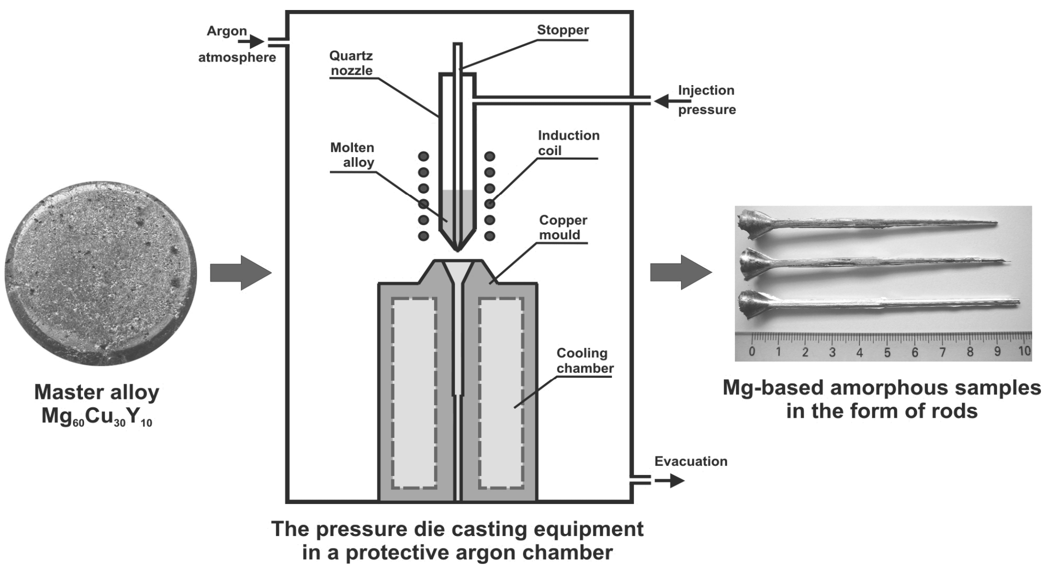

Fig. 2. Schematic illustration of casting Mg-based amorphous materials by using the pressure die casting method in a protective chamber

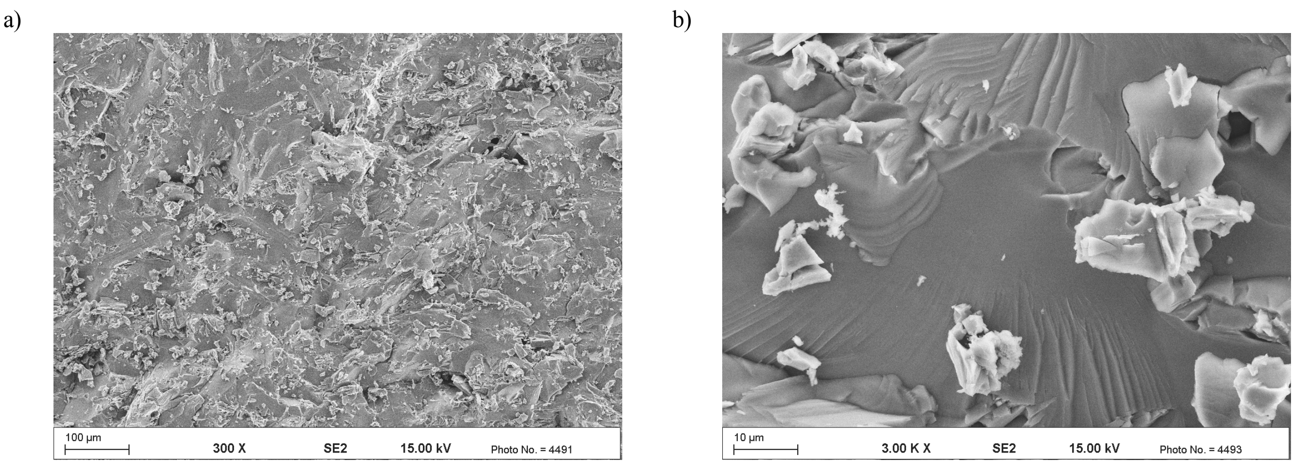

Fig. 3. SEM micrographs of the surface morphology of $Mg_{60}Cu_{30}Y_{10}$ master alloy: a) overview b) detail from Fig. a

a) b)

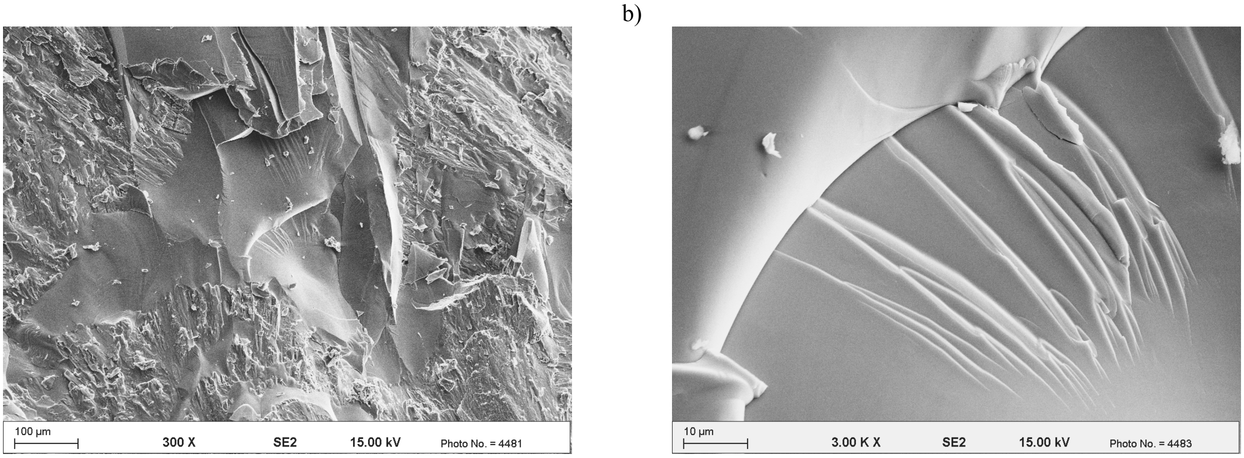

Fig. 4. SEM micrographs of the surface morphology of $Mg_{60}Cu_{29}Y_{10}Si_1$ master alloy: a) overview b) detail from Fig. a

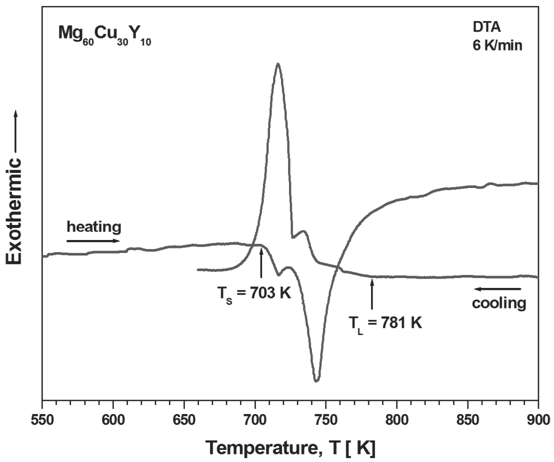

Fig. 5. DTA curves of $Mg_{60}Cu_{30}Y_{10}$ alloy as master alloy

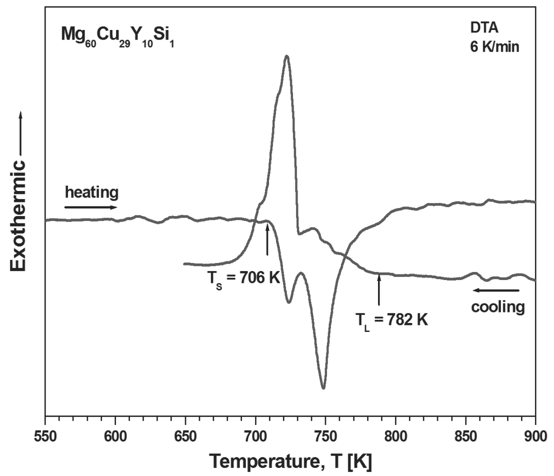

Fig. 6. DTA curves of $Mg_{60}Cu_{29}Y_{10}Si_1$ alloy as master alloy

3. Results and discussion

The ingots of $Mg_{60}Cu_{30}Y_{10}$ and $Mg_{60}Cu_{29}Y_{10}Si_1$ master alloys in as-prepared state were examined by SEM and DTA methods.

Micrographs of surface morphology obtained by using SEM for studied Mg-based master alloys in the form of ingots presented transcrystalline and brittle fractures (Figs. 3, 4).

The solidus temperature (T_S) assumed to be the onset of the heating isotherm on the DTA curves. Consequently, the liquidus temperature (T_L) was measured by cooling the molten alloy samples with differential thermal analyzer.

The endothermic peak observed on DTA curves (Fig. 5) of master alloy of $Mg_{60}Cu_{30}Y_{10}$ metallic glass allowed to determine the solidus temperature (T_S), which has a value of 703 K. In the similar way the endothermic effect was also observed for master alloy of second studied material ($Mg_{60}Cu_{29}Y_{10}Si_1$) - Fig. 6.

In addition to DTA analysis of master alloys, Table 1 showed T_S and T_L temperatures of studied materials. The addition of Si to $Mg_{60}Cu_{30}Y_{10}$ alloy insensibly increased the solidus and liquidus temperature in comparison to the studied alloy without Si.

Table 1.
Solidus and liquidus temperature of the studied master alloys in as-prepared state

Master alloy	T_S [K]	T_L [K]
$Mg_{60}Cu_{30}Y_{10}$	703	781
$Mg_{60}Cu_{29}Y_{10}Si_1$	706	782

The T_S reached a value of 706 K for alloy with Si addition. Meanwhile, the T_L in cooling curve is located around 781 K for $Mg_{60}Cu_{30}Y_{10}$ master alloy and the liquidus temperature reached a value of 782 K for second studied alloy.

In addition, the two endothermic peaks in heating curve were observed for both examined master alloys, but for alloy with Si addition the first endothermic effect is more distinct. The heating and cooling behaviors of studied alloys informed that the alloy compositions are approached to an eutectic point.

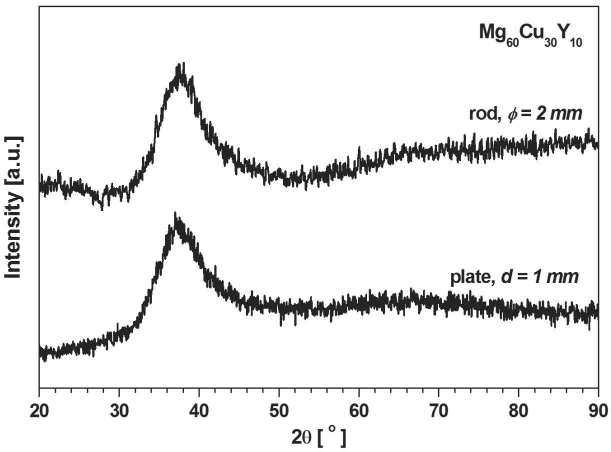

Fig. 7. X-ray diffraction patterns of $Mg_{60}Cu_{30}Y_{10}$ metallic glass in as-cast state in form of rod and plate

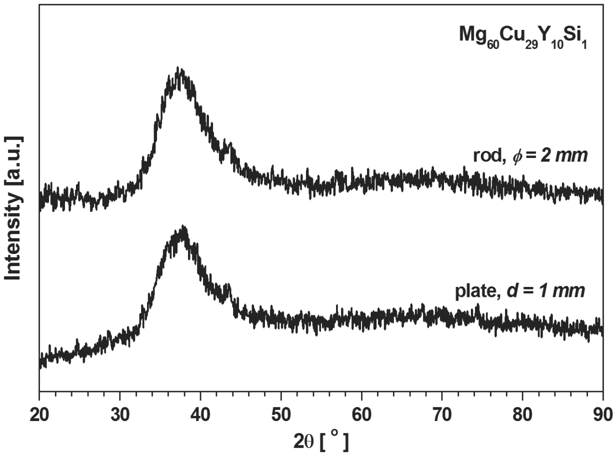

Fig. 8. X-ray diffraction patterns of $Mg_{60}Cu_{29}Y_{10}Si_1$ metallic glass in as-cast state in form of rod and plate

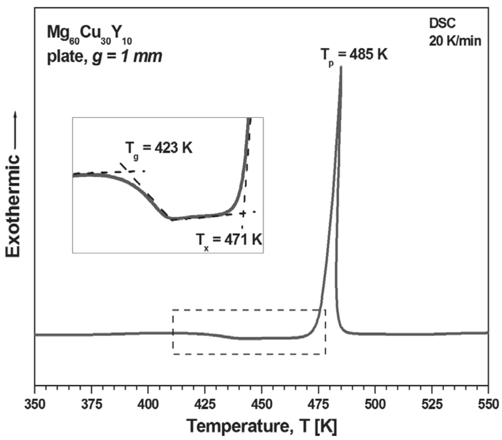

Fig. 9. DSC curves of $Mg_{60}Cu_{30}Y_{10}$ metallic glass in as-cast state in form of plate with thickness of 1 mm (heating rate 20 K/min)

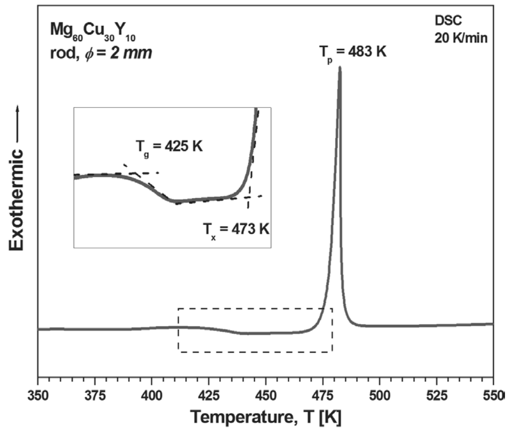

Fig. 10. DSC curves of $Mg_{60}Cu_{30}Y_{10}$ metallic glass in as-cast state in form of rod with diameter 2 mm (heating rate 20 K/min)

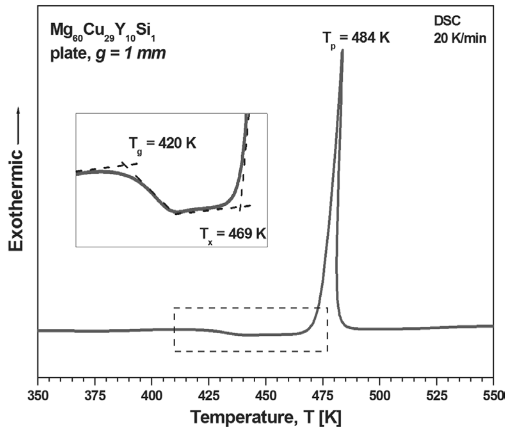

Fig. 11. DSC curves of $Mg_{60}Cu_{29}Y_{10}Si_1$ metallic glass in as-cast state in form of plate with thickness of 1 mm (heating rate 20 K/min)

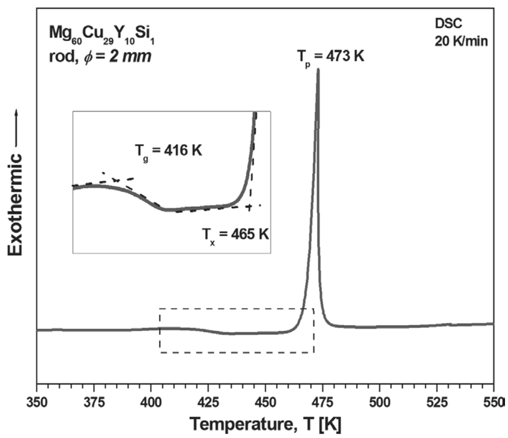

Fig. 12. DSC curves of $Mg_{60}Cu_{29}Y_{10}Si_1$ metallic glass in as-cast state in form of rod with diameter of 1 mm (heating rate 20 K/min)

Table 2.
Glass-forming properties of the studied alloys in the forms of glassy plates and rods, in as-cast state

Glassy alloy	Sample thickness [mm]	T_g [K]	T_x [K]	T_p [K]	ΔT_x	T_{rg}
$Mg_{60}Cu_{30}Y_{10}$	1.0	423	471	485	48	0.542
	2.0	425	473	483	48	0.544
$Mg_{60}Cu_{29}Y_{10}Si_1$	1.0	420	469	484	49	0.537
	2.0	416	465	473	49	0.532

a) b)

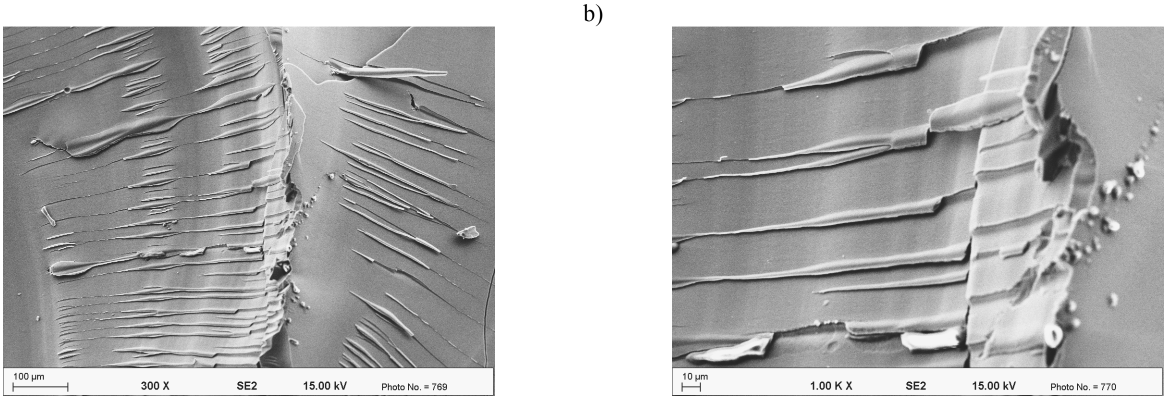

Fig. 13. SEM micrographs of the fracture morphology of $Mg_{60}Cu_{30}Y_{10}$ glassy rod: a) overview b) detail from Fig. a

a) b)

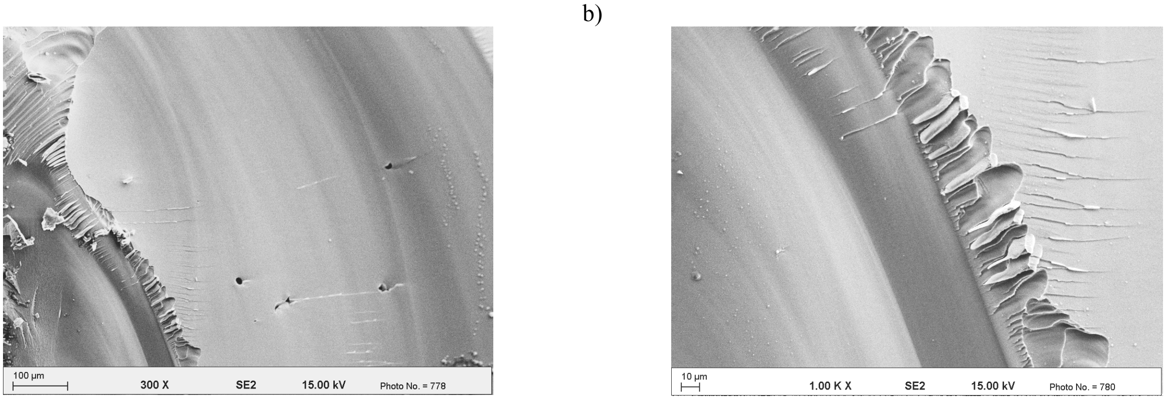

Fig. 14. SEM micrographs of the fracture morphology of $Mg_{60}Cu_{29}Y_{10}Si_1$ glassy rod: a) overview b) detail from Fig. a

The achieving of an eutectic composition of master alloy is very important for casting process of metallic glasses. The proper chemical composition of master alloys for casting glassy materials should be deep eutectic [1].

The X-ray diffraction investigations revealed that the examined samples in the form of rods and plates were amorphous. The diffraction patterns of studied Mg-based alloys have shown the broad diffraction halo characteristic for the amorphous structure. Figs. 7 and 8 show XRD results of studied bulk metallic glass $Mg_{60}Cu_{30}Y_{10}$ and $Mg_{60}Cu_{29}Y_{10}Si_1$ in as-cast state, adequately.

The DSC curves measured on amorphous rods and plates of Mg-based alloys in as-cast state are also shown in Fig. 9-12. The examined alloys exhibit the sequence of the glass transition temperature (T_g), onset (T_x) and peak (T_p) crystallization temperature. The shapes of DSC curves for all tested samples are very similar. The exothermic peaks describing a single stage of crystallization for all samples.

The crystallization effect for $Mg_{60}Cu_{30}Y_{10}$ alloy cast in the form of plate (with thickness of 1 mm) includes onset crystallization temperature T_x = 471 K and peak crystallization temperature T_p = 485 K (Fig. 9). In the case of the rod (diameter of 2 mm), the exothermic effect includes onset of the crystallization temperature at T_x = 473 K and peak crystallization temperature at T_p = 483 K (Fig. 10).

DSC results for the samples of Mg-based alloy with Si addition informed that the onset crystallization temperature reached a value of 469 K (for plate) and 465 K (for rod).

Similarly, the peak crystallization temperature was 484 K for plate and 473 K for rod.

It is important to noticed that a minor addition of 1 at.% Si into $Mg_{60}Cu_{30}Y_{10}$ alloy caused the distinct decreased of both onset and peak crystallization temperature. For comparison the temperature difference of T_p for samples in the form of rods (with diameter of 2 mm) is 10 K.

The DSC analysis also allowed to determine glass transition temperatures of examined glassy samples, which was 423 K for plate and 425 K for rod ($Mg_{60}Cu_{30}Y_{10}$), 420 K for plate and 416 K for rod ($Mg_{60}Cu_{29}Y_{10}Si_1$), respectively. The glass transition temperature difference between rod of alloy with Si addition and alloy free of silicon is 9 K.

Obtained results of DSC investigations confirmed that the crystallization temperatures: (T_x), (T_p) and the glass transition temperature (T_g) decreased by silicon minor addition.

The temperature interval of the supercooled liquid region (ΔT_x) defined by the difference between T_g and T_x, reached the highest value of 49 K for the plate and rod of $Mg_{60}Cu_{29}Y_{10}Si_1$ alloy.

The reduced glass transition temperature (T_{rg}) for samples of $Mg_{60}Cu_{30}Y_{10}$ alloy had a value about 0.54. The slightly decreasing of T_{rg} parameter was noticed for samples in the form of rod for studied Mg-based amorphous alloy with silicon addition. The T_{rg} parameter reached the lowest value (0.532) for $Mg_{60}Cu_{29}Y_{10}Si_1$ cast in the form of rod.

The parameters of glass-forming properties of studied alloys - T_g, T_x, T_p, ΔT_x and T_{rg} are presented in Table 2. Comparison of glass-transition effects and crystallization peaks of studied samples with different chemical compositions shows the distinct change of glass transition and crystallization temperature. These results confirmed the fact that a minor addition of Si into Mg-Cu-Y alloy caused an improvement of glass-forming ability which is necessary for casting glassy samples with higher dimensions.

The study of fracture morphology of samples in the form of rods with diameter of 2 mm for both examined alloys in as-cast state shows a mixed fractures with “river” and “mirror” patterns, which are characteristic for glassy materials and some “smooth” areas (Fig. 13a, 14a).

High magnification observations reveal that the fracture surface of studied metallic glasses is covered by typical and good formed “river” and “shell” patterns (Fig. 13b, 14b). Moreover, for sample of Mg-based alloy with Si addition, the “smooth” area is more significant in comparison with Mg-Cu-Y alloy. The fracture surface of two zones probably informed about different amorphous structures of the tested glassy materials. The microscopic observation of studied samples allows to state that examined fractures presented the classical fracture mode of macroscopically brittle materials.

The empirical rules which have been suggested by Inoue to achieve a high glass-forming ability for metallic alloys have been done in this paper. These rules have proposed that alloy should consist of more than three components. Moreover, the alloy with high glass-forming ability should contain metallic elements with different atomic sizes and a large negative heat of mixing in the liquid state of molten alloy [1].

The increase of the glass- forming ability of studied materials was achieved by the partial replacement of Cu by Si element in $Mg_{60}Cu_{30-x}Y_{10}Si_x$ alloy system, where the atomic share of silicon is 0 or 1 at.%.

The higher glass forming ability which was confirmed by GFA parameters calculation of the alloys with silicon addition may be explained by the increased the number of elements (multi-component system) and a large difference in atomic size between Si and main elements. A large atomic size difference between Si and the basic Mg, Cu, Y elements of the alloy is suitable to increase the atomic packing density of the amorphous structure achieved by cooling of molten alloy during casting process.

The light metal-based bulk metallic glasses are regarded as promising engineering materials with high strength and good corrosion resistance in contrast to the crystalline alloys, due to the different atomic configuration. In spite of the promising practical interest, little is known about the atomic structure of Mg-based amorphous alloys. Detailed knowledge about the structure of that bulk metallic glasses is important for the future research and could open new possibilities to better control the properties of these materials.

4. Conclusions

The investigations performed on the samples of studied Mg-based alloys allowed to formulate the following statements:

- the XRD investigations revealed that the studied alloys $Mg_{60}Cu_{30}Y_{10}$ and $Mg_{60}Cu_{29}Y_{10}Si_1$ (in the form of rods and plates) were amorphous in as-cast state.
- the surface morphology for studied Mg-based master alloys in the form of ingots presented transcrystalline and brittle fracture,
- the solidus temperature reached a value of 703 K for $Mg_{60}Cu_{30}Y_{10}$ alloy and increased to 706 K for alloy with Si addition, meanwhile, the liquidus temperature is located around 781 - 782 K for both studied alloys, respectively,
- the DSC curves obtained for samples in the form of plate and rod inform about the single stage of crystallization process,
- DSC investigations confirmed that the crystallization temperatures: (T_x), (T_p) and glass transition temperature (T_g) decreased by Si addition,
- the difference of peak crystallization temperature between rod samples for alloy with Si addition and alloy free of silicon is 9 K.
- the minor addition of Si (1 at.%) into Mg-Cu-Y alloy caused an improvement of glass-forming ability which is necessary for casting glassy samples with higher dimensions,
- the temperature interval of the supercooled liquid region reached the highest value of 49 K for the glassy plate and rod of $Mg_{60}Cu_{29}Y_{10}Si_1$ alloy,
- the highest reduced glass transition temperature (T_{rg}=0.544) has been achieved for $Mg_{60}Cu_{30}Y_{10}$ alloy cast in the form of rod,
- the presented fractures could be classified as mixed fracture with indicated mirror-like and river-like patterns and smooth fractures, which are characteristic for metallic glassy alloys,
- the success in preparation of the studied Mg-based bulk metallic glass in the form of rods and plates is important for the future progress in research and many practical applications as light-weight structural materials.

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