



Glass forming ability of Zr-based Zr–Cu–Ni–Al–(Ag) alloys

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Abstract

A series of Zr-based glasses has been obtained by rapid melt quenching and their thermal characteristics (T_g , T_x , T_l) were determined by DSC/DTA. Some of the most recognized glass forming ability (GFA) criteria were applied to predict the glass forming ability of the alloys and to study its dependence on the alloy composition. As a result, the best glass forming compositions among the studied alloys could be selected. It was found that as a general trend an increase of the Zr content results in T_x and $\Delta T_x (= T_x - T_g)$ decrease. The determined liquidus temperatures of chosen compositions with substantially different ΔT_x were found to vary relatively little. Therefore, it was concluded that the variation in the GFA of the studied alloys is mainly due to difficulties in the crystallization process, rather than to different stability of the melts.

Keywords Zr-based glasses · Glass transition · Crystallization · Glass forming ability

Introduction

Zr-based alloys are among the intensively studied multicomponent metallic glasses [1–3]. The reasons for this interest are their good mechanical properties included high tensile strength, high bending strength, high fracture toughness, high hardness and fatigue strength, high impact fracture energy, good castability and cutting machinability [3–5] as well as high corrosion stability [6–8]. They are also known as easy glass formers and appropriate alloys to be prepared in a bulk amorphous form [9]. Thus, having the size in the centimeter-scale, bulk metallic glasses (BMG) allow being developed as good structural and functional materials [3, 10–13].

The first Pd-based BMG, which belongs to such a group, was prepared at the beginning of 1980s by Turnbull. Later large number of BMG on the base of La, Mg, and Zr were developed at the end of the decade by the group of Inoue [14–16] and Johnson [2]. Zr–Al–TM system was also studied,

showing a large supercooled liquid region. The multicomponent alloys offer significantly larger glass forming ability due to their relatively lower critical cooling rate and bigger critical glass formation size in comparison to the binary systems investigated. The additional elements improve both, stabilize the liquid phase and prevent the crystallization process. The improvement of GFA in Zr–Al–Ni alloys could be related to an optimum atomic configuration achieved with the dissolution of Al and increased short-range packing density of the amorphous phase [17–19].

The glass forming ability is usually expressed by the maximum obtainable diameter of a fully amorphous ingot, called critical diameter [20]. However, the methods for the critical diameter determination are time and materials exhausting. Therefore, many papers were devoted to attempts to correlate the GFA with the thermal characteristics of the alloys and already accepted criteria, such as temperature interval of supercooled liquid region $\Delta T_x = T_x - T_g$ [1], reduced glass transition temperature $T_{rg} = T_g/T_l$ [21], $\alpha = T_x/T_l$ [22], $\gamma_m = (2T_x - T_g)/T_l$ [23], $\gamma = T_x/(T_l + T_g)$ [24], $K = (T_x - T_g)/(T_l - T_x)$ [25], $\delta = T_x/(T_l - T_g)$ [26], and $\omega = (T_g/T_x - 2T_g)/(T_g + T_l)$ [27]. Some of these criteria take into account the difficulties in the crystallization process of the glasses and others the stability (thermodynamic and rheological properties) of their melts [28–33].

Some of the amorphous alloys included in the present work have already been the subject of our previous studies, which mainly traced their crystallization behavior,

The article is dedicated to Prof. Uwe Koester (Technical University Dortmund), with whom all the authors of this study were closely related.

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crystalline products (quasicrystalline and equilibrium crystalline phases) at different annealing conditions [34]. For the purpose of the present work a large series of similar in composition Zr-based amorphous alloys has been selected, all obtained under well-controlled and uniform quenching conditions, and on the base of determined thermal characteristics T_g , T_x , T_l using known criteria their GFA has been predicted. Dependence of GFA on the composition of the glasses was sought, as well as the specific heat change ΔC_p at glass transition temperature was determined for the whole series of zirconium glasses studied.

Experimental part

The alloy ingots were prepared by arc-melting the mixture of Zr (99.5%), Cu (99.9%), Ni (99.9%), Al (99.99%) and Ag (99.95%) in a pure argon atmosphere approx. 1.2 bar on a water-cooled copper substrate. Homogeneous alloys composition was achieved by 3 times remelting the master alloys. The alloys were then rapidly solidified by melt spinning in a He atmosphere of 300 mbar to obtain ribbons with a thickness of 40 μm and width of 3 mm. The temperature of the melt was between 1100 and 1200 $^\circ\text{C}$. Since oxygen contamination significantly affects the thermal stability [34], special care was taken to ensure comparable conditions for all ribbons produced.

The microstructure of the as-cast samples was characterized by X-ray diffraction (XRD) with $\text{Cu-K}\alpha$ radiation (diffractometer Siemens D 500, at a step of 0.05° and counting time 2 s/step). Thermal analysis was carried out by differential scanning calorimetry (Perkin-Elmer DSC7) and differential thermal analysis (Perkin-Elmer DTA). Scanning rate of $10 \text{ K}\cdot\text{min}^{-1}$, sample mass of 15–20 mg and purified nitrogen purging gas were used for all thermal experiments.

Results and discussion

X-ray diffraction analysis reveals the amorphous character of all studied Zr-based alloys. The diffraction patterns of the amorphous alloys, shown in Fig. 1, are ordered according to their composition. Figure 1c shows XRD patterns of Zr alloys containing Ag and Au in their composition. For all studied alloys two clearly observed diffraction halloes with little difference in the position of their maximums could be detected. The differences in the positions of the diffraction maxima are minor due to the small differences in the overall glasses' composition. Therefore, it can be concluded that the most frequently observed interatomic distances in the studied glasses are comparable ($2.45 \pm 0.04 \text{ \AA}$). Nevertheless, some minor differences in the interatomic distance could be detected, e.g., $\text{Zr}_{70}\text{Cu}_{12}\text{Ni}_{10.5}\text{Al}_{7.5}$ glass is characterized with the smallest mean interatomic distance (2.41 \AA), whereas $\text{Zr}_{63.5}\text{Cu}_{16}\text{Ni}_{13}\text{Al}_{7.5}$ with the largest one (2.48 \AA). The observed minor variations can be explained with the difference in the alloy compositions and with the atomic radii of the metals constituting the alloys.

The DSC scans of the Zr-based amorphous alloys are presented in Fig. 2, as the glasses were again grouped in the same order as in Fig. 1. It is noteworthy that despite the relatively similar compositions of the glasses, there are significant differences in their thermal behavior. However, on all thermal curves, the glass transition is clearly detected, as well as the temperature range of the supercooled liquid ($T_x \div T_g$) is well determined. The last observation allowed relatively accurate definition of the region of supercooled liquid, as well as of the specific heat difference due to the glass transition, ΔC_p . The enthalpies of crystallization were also reliably determined. All the above thermal characteristics of the glasses are presented in Table 1.

Comparing the thermal characteristics of the studied Zr-based glasses and applying the simplest GFA criterion

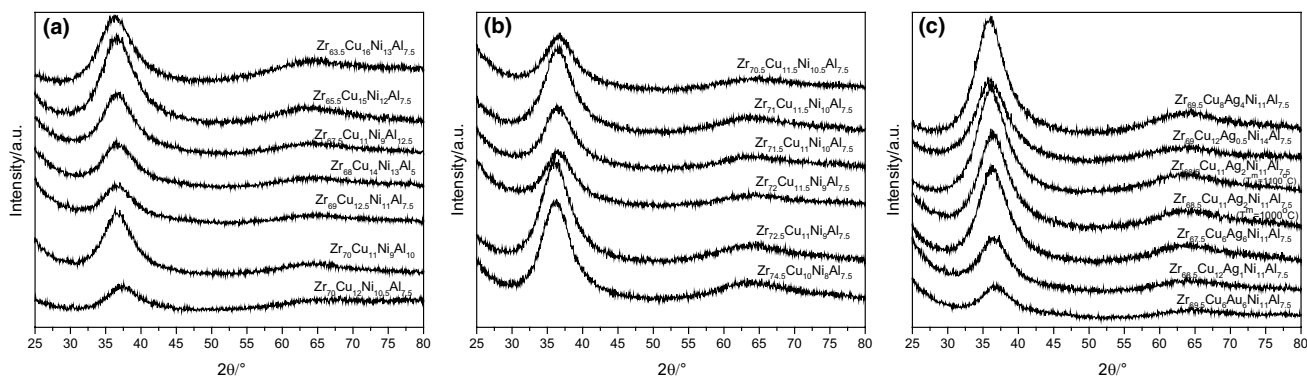


Fig. 1 XRD patterns of Zr-based glasses, obtained by melt-spinning

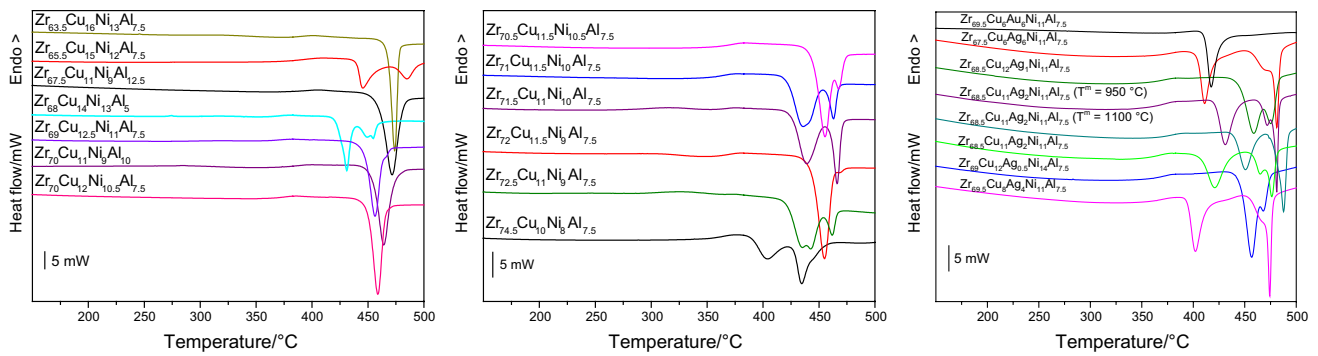


Fig. 2 DSC curves of the Zr-based glasses studied

Table 1 Glass transition (T_g) and crystallization (T_x) temperatures, liquidus temperatures (T_l) and specific heat change (ΔC_p) at the glass transition, determined by DSC/DTA

Composition	T_g , °C	T_x , °C onset	T_l , °C (liquidus)	ΔC_p $J g^{-1} °C^{-1}$	$T_x - T_g$	$(T_x - T_g)/T_l$	$(2T_x - T_g)/T_l$	$T_x/(T_g + T_l)$	T_g/T_l
Zr _{74.5} Cu ₁₀ Ni ₈ Al _{7.5}	358	389	–	0.380	31	–	–	–	–
Zr _{72.5} Cu ₁₁ Ni ₉ Al _{7.5}	370	420	–	0.315	50	–	–	–	–
Zr ₇₂ Cu _{11.5} Ni ₉ Al _{7.5}	365	441	–	0.42	76	–	–	–	–
Zr _{71.5} Cu ₁₁ Ni ₁₀ Al _{7.5}	365	425	–	0.444	60	–	–	–	–
Zr ₇₁ Cu _{11.5} Ni ₁₀ Al _{7.5}	355.6	420.5	–	0.395	64.9	–	–	–	–
Zr _{70.5} Cu _{11.5} Ni _{10.5} Al _{7.5}	369	440	–	0.352	71	–	–	–	–
Zr ₇₀ Cu ₁₂ Ni _{10.5} Al _{7.5}	371	446	–	0.367	75	–	–	–	–
Zr ₆₉ Cu _{12.5} Ni ₁₁ Al _{7.5}	367.7	443	–	0.401	75.3	–	–	–	–
Zr _{65.5} Cu ₁₅ Ni ₁₂ Al _{7.5}	391.6	440	–	0.38	48.4	–	–	–	–
Zr _{63.5} Cu ₁₆ Ni ₁₃ Al _{7.5}	394	464	–	0.59	70	–	–	–	–
Zr _{67.5} Cu ₁₁ Ni ₉ Al _{12.5}	386.5	458	–	0.567	71.5	–	–	–	–
Zr ₇₀ Cu ₁₁ Ni ₉ Al ₁₀	377	450	–	0.51	73	–	–	–	–
Zr ₆₈ Cu ₁₄ Ni ₁₃ Al ₅	369	421	–	0.35	52	–	–	–	–
Zr _{67.5} Cu ₆ Ag ₆ Ni ₁₁ Al _{7.5}	375	401	–	0.28	26	–	–	–	–
Zr ₆₉ Cu ₁₂ Ag _{0.5} Ni ₁₄ Al _{7.5}	367	440	–	0.385	73	–	–	–	–
Zr _{68.5} Cu ₁₂ Ag ₁ Ni ₁₁ Al _{7.5}	367	439	965.2	0.353	72	0.0582	0.633	0.379	0.517
Zr _{69.5} Cu ₈ Ag ₄ Ni ₁₁ Al _{7.5}	370	395	995.8	0.22	25	0.0197	0.546	0.349	0.507
Zr _{68.5} Cu ₁₁ Ag ₂ Ni ₁₁ Al _{7.5} ($T^m = 1000$ °C)	369	422	1004.8	0.414	53	0.0415	0.585	0.362	0.502
Zr _{68.5} Cu ₁₁ Ag ₂ Ni ₁₁ Al _{7.5} ($T^m = 1100$ °C)	377	437	1004.8	0.465	60	0.0470	0.602	0.368	0.509
Zr _{68.5} Cu ₁₁ Ag ₂ Ni ₁₁ Al _{7.5}	361.6	408	–	0.36	46.4	–	–	–	–
Zr _{69.5} Cu ₆ Au ₆ Ni ₁₁ Al _{7.5}	394	410	–	0.150	16	–	–	–	–

($\Delta T_x = T_x - T_g$) [1, 35, 36] some important conclusions could be formulated.

In the first place, the general trend shows crystallization temperature, T_x , increase with the decrease in the Zr content of the glasses (with all the conditionality that the concentration of other metals also slightly changes), Fig. 3. Depending on the alloy composition the glass transition temperature, T_g , varies within a range of only about 30 °C (360–390 °C), while T_x differs significantly more (from 390 to 465 °C). Therefore, the different temperature range of the supercooled

liquid ($\Delta T_x = T_x - T_g$) of the glasses is mainly due to their different crystallization temperatures, and to lesser extend to variations in their glass transition temperatures.

Another notable compositional dependence is related to the Zr glasses containing silver/gold. These amorphous alloys generally show higher T_g and lower T_x , i.e., smaller ΔT_x , compared to the glasses without noble metal, Table 1. Figure 4 reveals the dependence of T_g and T_x on the Ag/Au content of the studied glasses. While T_g does not vary with the Ag concentration, the crystallization temperature of the

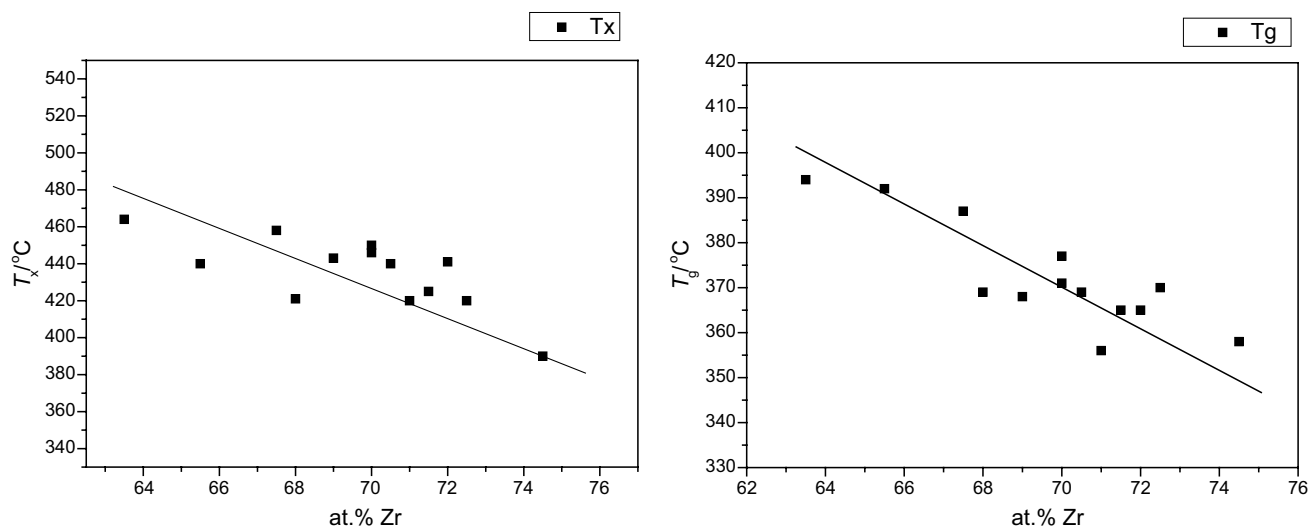


Fig. 3 Glass transition and crystallization temperatures vs. Zr content of the studied glasses

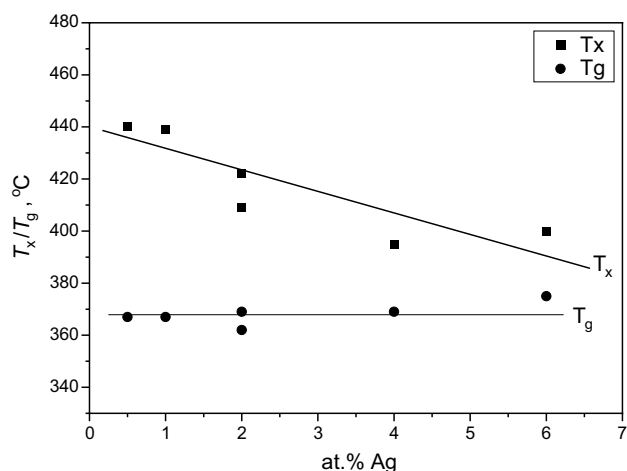


Fig. 4 Glass transition and crystallization temperatures vs. Ag/Au content of Zr-based glasses from the present study

glasses decreases visibly with increasing the silver content. The last leads to the conclusion that the GFA of the studied Zr-based glasses deteriorates when the silver concentration is increased. The only glass with Au in its composition also reveals lower ΔT_x , compared to the Zr glasses that do not contain precious metals. Thus, it can be summarized that the presence of precious metals in the Zr–Cu–Ni–Al glasses results in a GFA decrease. The comparison between the two glasses with the same composition ($Zr_{68.5}Cu_{11}Ag_2Ni_{11}Al_{7.5}$) but vitrified by different melt temperatures (1000 and 1100 °C) is also interesting. The melt, cooled from a higher temperature, leads to a glass with higher T_g and T_x compared to that vitrified from a lower temperature, the difference in T_x being greater. This reasonably results in a larger ΔT_x

for the glass obtained from the melt annealed at 1100 °C, which result can be explained by a better homogenization of the melt at the higher temperature and hence to a impeded nucleation of the crystalline phase(s).

Other dependence that can also be established from the thermal behavior of the studied Zr glasses (without Ag/Au) is related to alloys with the same or very similar Zr and Al content, but with varying Ni and Cu content. These glasses showed similar T_g and T_x values, and therefore comparable ΔT_x value, Table 1.

Although the glass transition is a kinetic phenomenon strongly dependent on the heating rate, and ΔC_p is a value at a particular temperature (T_g) it is worth noting the observation that glasses with large ΔT_x are generally characterized with larger ΔC_p ($> 0.35\text{--}0.50\text{ J g}^{-1}\text{ K}^{-1}$) and the opposite, glasses with smaller ΔT_x show also reduced ΔC_p ($< 0.3\text{ J g}^{-1}\text{ K}^{-1}$). To confirm and clarify this fact, a separate systematic measurement of the absolute value of the specific heat at different temperatures, both in the alloys' glassy state and as supercooled liquid, is necessary.

DTA analysis to higher temperatures of Zr-based amorphous alloys, showing very different ΔT_x , was also carried out, Fig. 5. Besides, two glasses with the same composition ($Zr_{68.5}Cu_{11}Ag_2Ni_{11}Al_{7.5}$) but solidified from different temperature of the melt were also analyzed. This study revealed the liquidus temperature of the alloys, T_l (Table 1), and thus allowed the GFA criteria $\Delta T_x/T_l$ or $(2T_x - T_g)/T_l$, T_g/T_l , $T_x/(T_g + T_l)$ related to the overall liquid phase stability (both at undercooled state and at the equilibrium), to be also applied [23, 37]. There is an agreement between the result of the applied GFA criteria: glasses with a larger ΔT_x follow a larger $\Delta T_x/T_l$, $(2T_x - T_g)/T_l$ and $T_x/(T_g + T_l)$, as well (Table 1). Some exception is observed only when

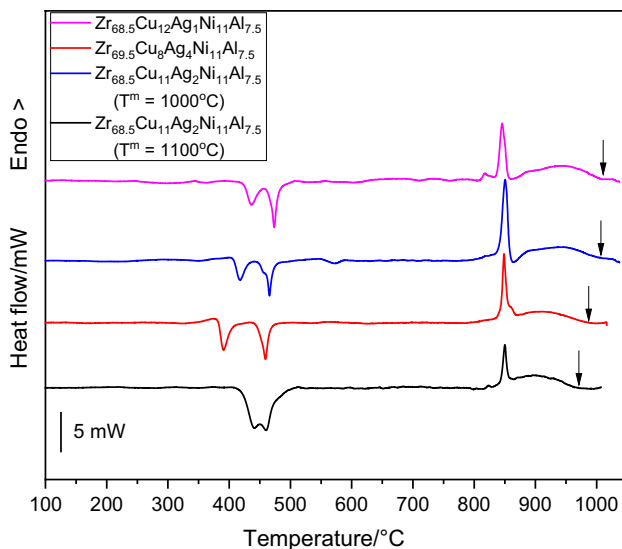


Fig. 5 DTA curves for selected Zr-based glasses

we apply the T_g/T_l criterion for glasses with similar T_g and T_l , but with a significant difference in T_x . Thus, $Zr_{69.5}Cu_8Ag_4Ni_{11}Al_{7.5}$ alloy shows a better GFA compared to $Zr_{68.5}Cu_{11}Ag_2Ni_{11}Al_{7.5}$ ($T^m = 1000^\circ C$), although ΔT_x is significantly larger for the latter alloy.

Based on the thermal characteristics of the studied Zr-based glasses, it can be concluded that the evaluated higher GFA of certain alloy compositions should be attributed mainly to their higher crystallization temperatures T_x , reflecting the crystallization resistance of the glass, than to the increased liquid phase stability.

Conclusions

The thermal characteristics (T_g , T_x , T_l) of a series of Zr-based amorphous alloys, obtained by rapid quenching (melt spinning), were determined. The specific heat change at glass transition ΔC_p was also determined. The alloys were obtained under the same well-controlled conditions of quenching (temperature of the melt, quenching disk size and velocity, He atmosphere), which fact allowed to search for a relationship between the compositional variations and thermal behavior of the glasses. From the obtained thermal parameters and using well accepted glass forming ability (GFA) criteria the compositions with the higher GFA were determined. An answer was also given to the question of what determines the high GFA for these glasses: the difficulties in crystallization or the stability of the melt.

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Author contributions Tony Spassov: Conceptualization, Methodology, Investigation, Writing—original draft, Writing—review & editing. Lyudmila Lyubenova: Investigation, Sample preparation and characterization, Data curation. Vesselina Rangelova: Sample preparation and characterization, Data curation. Maya Spassova: Investigation, Characterization.

Declarations

Conflict of interest The authors have no competing interests to declare that are relevant to the content of this article.

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