



Effect of oxygen content on the microstructure, compression properties and work-hardening behaviors of ZrCuAlNi glassy composites



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ABSTRACT

Zr₅₅Cu₃₀Al₁₀Ni₅ bulk metallic glass (BMG) and its composites with different oxygen contents from 0.1 at% to 1.0 at% were prepared by copper-mold-casting. Primary crystallizing phases with different microstructures and volume fractions were produced in these alloys due to the incorporation of different oxygen contents. The X0.1 monolithic BMG has no plasticity. The X0.2, X0.3 and X0.4 composites contained the tiny crystallites or crystalline phases show some plasticity. The X0.2 composites exhibits the largest fracture strain of 8.10%. It is believed that the presence of the randomly distributed nanometer-sized crystallites in these alloys is effective to seed the formation and propagation of multiple shear bands. As the oxygen content increases from 0.5 to 1.0 at%, volume fraction of the crystalline phases in the composites increases remarkably, and the yield strength, fracture strength and fracture strain decreased, while θ , H_c , K and n increased. The X0.5, X0.6, X0.8 and X1.0 composites display work-hardening and plastic deformation. The work-hardening effect and the plasticity result from the continuous martensitic transformation, the dislocation multiplication in the crystalline phase and the interactions of shear bands during deformation.

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1. Introduction

Bulk metallic glasses (BMGs) exhibit very limited plastic strain before fracture [1–3]. In order to overcome this critical disadvantage, the efforts to improve the macroscopic plasticity have been made for decades through controlling microstructural heterogeneity within the amorphous matrix [4–9]. In these BMG composites, the second phase hinders single shear band to extend through the whole sample at the onset of plastic deformation and seeds the initiation of multiple shear bands. Therefore, plasticity is distributed more homogeneously in the shear band patterns, which results in local high strains to the failure of the composites. On the other hand, the size, geometry and volume fraction of the primary crystallizing phases are also crucial for efficiently controlling the shear band propagation and improving the ductility [10–12].

Since the end of 1980s, the multi-component Zr-based alloy system has been one of the most successful and promising systems in the formation of BMGs or BMG-based composites due to its excellent glass forming ability (GFA), physical and chemical properties [13–21]. The GFA of the Zr-based alloys strongly depends on the degree of cleanliness in the molten alloy which is governed

by impurity-induced nucleation of crystalline phases during cooling [22]. Many efforts have been made to investigate the effect of oxygen addition on the microstructure and volume fraction of the primary crystallizing phase [22–31]. Gebert et al. [22] studied the effect of small amounts of oxygen on the phase formation and the thermal stability of BMG samples. With increasing oxygen content a drastic reduction of the supercooled liquid region is observed, resulting mainly from a change of the crystallization sequence from a single- to a double-step process which is attributed to the oxygen-triggered formation and the sub-sequent transformation of the metastable f.c.c. phase. Lin et al. [24] found that the oxygen additions strongly affect the critical cooling rate for glass formation in a Zr-based alloy. Eckert et al. [25] reported that oxygen impurity in the Zr-based BMGs changed the crystallization processes from a single to a double stage, and thereby reducing the supercooled liquid region [defined as the difference between the crystallization temperature (T_x) and the glass transition temperature (T_g)]. Murty et al. [26] investigated the influence of oxygen on the crystallization behavior of ZrCuAlO and ZrCuO metallic glasses. In the case of the Zr–Cu metallic glass, the crystallization product (Zr₂Cu) is not influenced by the oxygen content, while in Zr–Cu–Al alloys the oxygen level has a strong influence on the crystallization sequence. At low oxygen level, the ternary glass crystallizes polymorphously to Zr₂(Cu,Al). At higher oxygen content, the ternary amorphous alloy crystallizes in two stages by primary crystallization into an icosahedral phase and subsequently to the

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stable $Zr_2(Cu,Al)$ phase. All of these studies indicate that the oxygen impurity dramatically reduces the GFA and influences the crystallization behaviors of glasses. However, there is a little research dealing with the effect of the different oxygen contents on the variable microstructures, and the effect of the primary crystallizing phases induced by oxygen on the mechanical properties of the $ZrCuAlNi$ BMG composites. In this paper, the $Zr_{55}Cu_{30}Al_{10}Ni_5$ BMG and its composites with different oxygen contents were prepared by copper-mold-casting. The different oxygen contents lead to variable microstructures under solidification. The effect of the oxygen content on the microstructures and volume fraction of the primary crystallizing phase, and further on the mechanical properties, were systematically investigated.

2. Experimental details

$Zr_{55}Cu_{30}Al_{10}Ni_5$ alloys with the addition of different oxygen contents (at%) (0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.8 and 1.0) are denoted by X0.1, X0.2, X0.3, X0.4, X0.5, X0.6, X0.8 and X1.0, respectively. They were prepared by arc-melting of mixtures of alloying components (oxygen was added in the form of CuO) in the Ti-gettered argon (99.999 vol%) atmosphere. These alloys were melted and turned in the water-cooled copper crucible for four times using electromagnetism stirring to ensure chemical homogeneity, and then the master alloy was remelted in a quartz crucible and casted into a copper mold with a cylindrical cavity of 3.5 mm in diameter using a spurt casting method. The real oxygen contents in these alloys were analyzed by the pulse heating inert gas fusion-infrared thermal conductivity method, [32–35] and the results are listed in Table 1.

The spurt-cast alloys were characterized by X-ray diffraction (XRD, D8 Discover with GADDS, Bruker AXS, Germany) using $Cu-K_{\alpha}$ radiation, laser confocal scanning microscope (LCSM, Olympus LEXT OLS3000, Japan), TEM (JEM-2000FX, Japan) and HRTEM (JEM-2100F, Japan). The TEM samples were first sliced and grinded, and then electrochemically polished by the twin-jet method.

The volume fractions of the crystalline phases in the alloys were evaluated by calculating the ratio of the heat released during annealing to the total crystallization enthalpy of the fully glassy alloy from differential scanning calorimeter (DSC) curves measured using DSC (DSC-7, USA) at a heating rate of 20 K/min: $V_{cry} = (\Delta H_{max} - \Delta H) / \Delta H_{max}$, where ΔH_{max} is the total enthalpy of transformation from the fully amorphous alloy to the completely crystallized alloy and ΔH from the tested samples [36,37]. The same method was also used by Bian et al. to evaluate the volume fractions of the crystalline phases in the Cu–Hf–Ti–Ag–Ta alloy [38].

The cylindrical samples with a diameter of 3.5 mm and a height of 7 mm were used for uniaxial compression test performed at a

constant strain rate of $1 \times 10^{-4} s^{-1}$ using a servo-hydraulic materials testing system (MTS, MTS 810, USA) at room temperature. The fracture surface morphologies were observed using a SEM (JSM-5310, Japan).

3. Results and discussion

3.1. Microstructural characteristics of $ZrCuAlNi$ glassy composites

Fig. 1 shows the XRD patterns recorded from the cross-section of the as-cast $ZrCuAlNiO$ alloys with different oxygen contents. For X0.1, X0.2 and X0.3 composites, the patterns consist of only a series of broad diffraction maxima without any detectable sharp peaks. With the increase in the oxygen content, some crystalline peaks appear in the X-ray patterns. The crystalline phase in the X0.4 composites is $ZrCu$ (B2) austenite, and that in the X0.5 composites should be $ZrCu$ (B2) austenite and Zr_2Cu phases. For X0.6, X0.8 and X1.0 composites, several small crystalline diffraction peaks superimposed on the broad amorphous diffraction peak were identified as $ZrCu$ (B2) austenite, Zr_2Cu and $ZrCu$ martensite phases [39–42]. The Al and Ni atoms may situate in the Zr–Cu crystals to form solid solutions. It can be deduced that oxygen impurity more than 0.3 at% in the $ZrCuAlNi$ alloy deteriorates the GFA and triggers the formation of the crystalline phases, and the crystalline phases in the bulk samples increase with increasing oxygen content. These results agree with the experimental results in references [22] and [27].

The DSC profiles of these $ZrCuAlNiO$ composites with different oxygen contents are shown in Fig. 2. The dependence of T_g , T_x and ΔT [$\Delta T = T_x - T_g$ (temperature interval between T_g and T_x)] on the oxygen content and the volume fractions of the crystalline phases calculated from the DSC results for these composites are listed in Table 1. It is obvious that the heat of crystallization decreases with the increasing oxygen content, which causes an increase in the crystalline volume fraction in the as-cast samples. T_g shifts first slightly to a higher temperature, and then decreases gradually. With the increasing oxygen content, T_x shifts to a lower value at a higher oxygen content. It results in a remarkable reduction in the

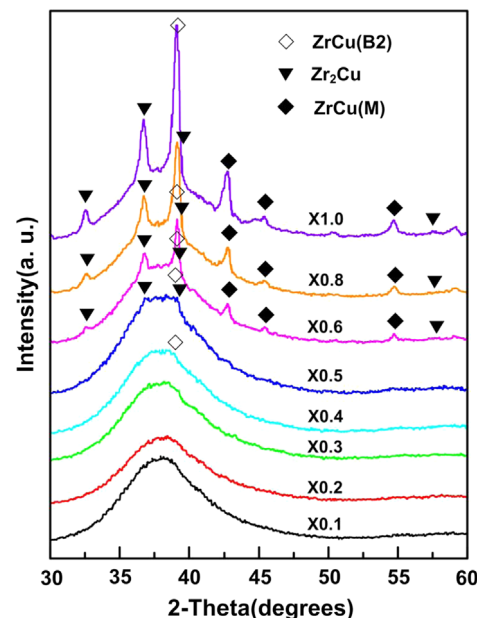


Fig. 1. The XRD patterns recorded from the cross-section of the as-cast $ZrCuAlNiO$ composites with the different oxygen contents.

Table 1
Dependence of T_g , T_x , ΔT and volume fractions of the crystalline phases on oxygen content.

Composites	Nominal oxygen content (at%)	Real oxygen content (at%)	V_{cry} (%)	T_g (K)	T_x (K)	ΔT (K)
X0.1	0.1	0.094	0.00	666.8	754.8	88.0
X0.2	0.2	0.187	0.05	668.4	757.4	89.0
X0.3	0.3	0.336	2.80	677.8	750.5	72.7
X0.4	0.4	0.411	7.35	687.9	757.3	69.4
X0.5	0.5	0.560	14.63	692.6	752.4	59.8
X0.6	0.6	0.652	28.84	688.7	747.4	58.7
X0.8	0.8	0.883	48.25	688.1	745.9	57.8
X1.0	1.0	1.024	61.22	687.5	744.4	56.9

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