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Triple-yieldable multiphase reinforced bulk metallic glass matrix composites under tension

Wei Guo *, Junji Saida

Frontier Research Institute for Interdisciplinary Sciences, Tohoku University, Sendai 980-8578, Japan

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ABSTRACT

Multiphase (Ta-rich phase, B2 and B19′-CuZr) reinforced bulk metallic glass matrix composites (BMGMCs) were successfully developed. The Ta-rich phase appeared not only to hinder the propagation of shear bands, but also to act as a nucleant for the B2 and B19′ phases to homogenize their distribution. The composites showed superior tensile plasticity, work-hardening, and a unique triple yielding behavior. The martensitic transformation of B2-CuZr, the deformation of the Ta-rich phase, and the twinning and detwinning of B19′-CuZr all contributed to these mechanical properties. The present novel BMGMC is expected to lead to a breakthrough in designing work-hardenable and ductile BMGMCs.

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1. Introduction

Recently, the *in-situ* precipitation of B2-CuZr shape-memory phase has been reported, opening a new path for designing bulk metallic glass matrix composites (BMGMCs) [1]. Such a shape-memory phase can suppress the rapid propagation of shear bands through a stress-induced martensitic transformation during deformation, which enables a composite with work-hardening behavior to be produced [1]. However, owing to the temperature gradient and cooling rate variation in the sample, the nucleation and growth of B2-CuZr during casting are not homogenous. Such an inhomogeneous strengthening limits the further improvement of mechanical properties [2]. Recently, Song et al. have proposed a feasible method using element doping [3]. At only 0.75 at.% Sn doping, B2-CuZr became uniformly dispersed. The mechanism of this phenomenon is considered to be the primary homogenous nucleation of Zr_5Sn_3, acting as a nucleant for the B2-CuZr phase.

In the present study, we fabricated a multiphase reinforced BMGMC by introducing a Ta-rich phase, which also plays a role as an effective nucleant for B2-CuZr and B19′-CuZr. The microstructure and properties of the novel multiphase reinforced BMGMC were investigated in detail.

2. Experimental

The compositions of the monolithic glassy base and the Ta-doped composite were $Cu_{47.5}Zr_{47.5}Al_5$ and $(Cu_{0.475}Zr_{0.475}Al_{0.05})_{95}Ta_5$ (at.%), denoted as Ta0 and Ta5, respectively. The master alloys were prepared by arc-melting under a Ti-gettered argon atmosphere. The dog-bone-shaped samples (cross-section of $1 \times 2.5\ mm^2$, gauge length of 8 mm) were fabricated by injection copper mold casting. The structures were examined by X-ray diffraction (XRD; Bruker D8 Advance) with Cu Kα radiation, and scanning electron microscopy with energy-dispersive X-ray spectrometry (SEM-EDX; Carl Zeiss Ultra 55 with Bruker AXS). The glass transition temperature (T_g) and onset crystalline temperature (T_x) were measured with a differential scanning calorimeter (DSC; Perkin Elmer DSC8000) at a heating rate of 20 K/min. The transition temperature of the B2-CuZr phase transformation was measured by differential thermal analysis (DTA; TA Instruments SDT-Q600) at a heating rate of 20 K/min. The samples used for hardness tests were mechanically polished to a mirror surface. Tensile tests were performed at a strain rate of $5 \times 10^{-4}\ s^{-1}$ at room temperature using an EZ-Graph (Shimadzu Co. Ltd.) table-top type precision universal tester.

3. Results and discussion

Fig. 1(a) shows XRD patterns taken from cross-sections of Ta0 and Ta5. A single broad peak of monolithic glassy phase was observed for Ta0, while several crystalline peaks were superimposed on the broad peak in the pattern of Ta5. These crystalline peaks were assigned to three phases: a Ta-rich phase, B2-CuZr,

* Corresponding author.
E-mail address: weiguo@fris.tohoku.ac.jp (W. Guo).

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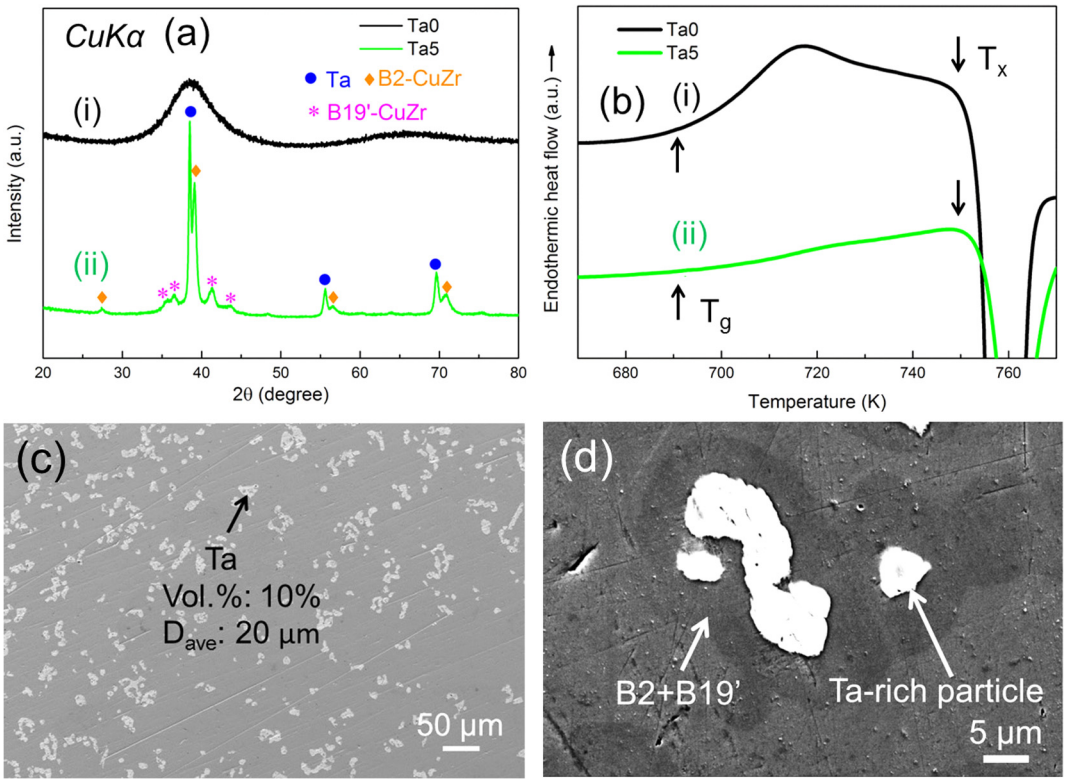

Fig. 1. (a) XRD patterns and (b) DSC curves for (i) Ta0 and (ii) Ta5; (c) SE image and (d) BSE images of Ta5.

and B19′-CuZr. Fig. 1(b) shows the DSC curves of Ta0 and Ta5, which exhibited similar T_g and T_x, 691 and 754 K for Ta0, and 693 and 753 K for Ta5, respectively, indicating a similar thermal stability of the glassy phase in both alloys. The volume fraction of crystalline phase can be estimated as [4]:

$$Vol_{crystal} = \frac{\Delta H_{BMGMC}}{\Delta H_{BMG}} \quad (1)$$

where $Vol_{crystal}$ is the volume fraction of crystalline phase, ΔH_{BMGMC} is the heat of crystallization or the area of the exothermic peak for the BMGMC, and ΔH_{BMG} is the heat of crystallization for the monolithic glassy counterpart. For the present Ta0 and Ta5, ΔH was ~55.6 J/g and 10.6 J/g, respectively. Thus, the $Vol_{crystal}$ (Ta + B2 + B19′) was calculated to be ~80% for Ta5.

Fig. 1(c) shows a SE (second electron) image of Ta5, which exhibited a very homogenous distribution of Ta-rich particles (gray region) as confirmed by EDX (not shown here). By image analysis of Fig. 1(c), the average size and volume fraction of Ta-rich particles in the sample was estimated to be ~20 μm and 10%, respectively. As stated above, the total volume fraction of crystalline phases was ~80%; thus the volume fraction of B2 and B19′-CuZr phases was ~70%, which is similar to the previously reported value [5]. Fig. 1(d) shows BSE (backscattered electron) images of Ta5. As expected, the dark region appeared to have formed around the gray regions (Ta-rich phase), a similar behavior to that observed in the case of Zr_5Sn_3 [3]. These results indicate that the Ta-rich precipitates acted as effective nucleants that promoted copious nucleation of the shape-memory phase. However, unlike the case of Zr_5Sn_3 [3], B19′ phase also formed for Ta5. The reason may be the difference in the size of the primary nucleant phase; that is, nano-sized particles for Zr_5Sn_3 while micro-sized ones for Ta. Significant residual stress and hot areas around Ta during quenching, resulting from the mismatch in thermal expansion and thermal conductively between the Ta-rich phase and the matrix, triggered B2 to B19′ phase transformation [6].

Fig. 2(a) shows true tensile stress-strain curves for both Ta0 and Ta5. The monolithic glassy Ta0 sample fractured at ~1848 MPa after ~1.8% elastic deformation without any plasticity. In contrast, the multiphase reinforced Ta5 showed a superior plasticity of ~8.4% plastic strain. The detailed data are summarized in Table 1. Additionally, not only large plastic deformation, but also obvious work-hardening and a unique triple yielding phenomenon were observed for Ta5. After a first yielding at ~400 MPa, a second and third yielding occurred at ~750 MPa and ~1400 MPa, respectively. For B19′ phase already existed in the as-cast state, it is difficult to prove that the martensitic transformation happened during deformation. Therefore, we conducted DTA experiments to trace the B2 phase transformation during heating, as shown in Fig. 2(b). An endothermic heat peak corresponding to B19′ to B2 phase transformation and the exothermic heat peak corresponding to the crystallization of the glassy matrix were clearly observed for both the as-cast and as-fractured states of Ta5. By calculating the peak area of the B2 phase transformation, the heat of B2 transformation for the as-cast and as-fractured states were found to be ~0.56 and 2.12 J/g, respectively. These results confirmed that more martensites exist after deformation and the martensitic transformation did occur during deformation. Meanwhile, the hardness of the Ta-rich particles before and after deformation was measured to be 321 ± 8 and 357 ± 9, an increase of ~11% during deformation. Therefore, not only the B2 and B19′ phases but also the Ta-rich particles, which also played a role of an effective nucleant, contributed to the overall mechanical properties of the composite.

The work-hardening rate (Θ) can be calculated using the equation ($\Theta = d\sigma_{true}/d\varepsilon_{true}$) [7], where σ_{true} and ε_{true} represent the true stress and true strain, respectively. Using the data shown in Fig. 2(a), the work-hardening rate of Ta5 is calculated and plotted as a function of the true strain in Fig. 3, together with the true stress-strain curves. The total mechanical behavior of Ta5 can be approximately separated into 4 stages: In stage I, the amorphous phase and the multiple reinforcing crystalline phases all deform

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