Strength asymmetry of ductile dendrites reinforced Zr- and Ti-based composites

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We report on significant asymmetry phenomena, including failure mode, fracture strength, and plasticity under compression and tension, for Zr- and Ti-based composites containing ductile dendrites. The failure of the Zr-based composite always occurs in a shear mode with a small strength asymmetry and different plasticity under tension and compression. In contrast, the Ti-based composite exhibits a significant high strength asymmetry and zero tensile plasticity although its compressive plasticity is high. We propose that the ratio, $\alpha = \tau_0/\sigma_0$ (τ_0 and σ_0 are the intrinsic shear and cleavage strengths), is a substantial parameter controlling the strength asymmetry and the failure mode of various materials.

I. INTRODUCTION

The deformation, failure mode, and fracture strength of high-strength materials often strongly depend on the stress state.^{1–4} In nature, there exists a significant asymmetry phenomenon between compression and tension strengths for a variety of brittle materials, such as graphite, rock, ceramics, intermetallics, and so on.⁵⁻⁹ However, the strength asymmetry of conventional metallic materials can be neglected.¹⁰ In the past decade, several severe plastic deformation (SPD) techniques have been developed to refine the grain size for the production of ultrafine-grained or even nanostructured materials with extremely high strength.^{11,12} It is often found that the strength asymmetry becomes obvious for ultrafinegrained or nanostructured materials.^{13,14} In contrast to the grain refinement mechanism, creating dislocationfree materials, such as whisker or metallic glass, is another novel challenge for the purpose of achieving high strength. Since the 1990s, bulk metallic glasses (BMGs)

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have been successfully fabricated, and as expected, displayed extremely high strength.^{15,16} However, the final fracture of BMGs often happens catastrophically and the tensile fracture stress is also lower than that under compression.^{15–19} Consequently, this gives rise to the interesting question why those high-strength or brittle materials often display a significant strength asymmetry under compression and tension? However, there is so far no adequate theory or model available to explain this question.²⁰ Recently, in situ formed Zr- and Ti-based (glassy/nanostructured) composites containing ductile dendrites have been synthesized, which show very high compressive strength and an improved compressive plasticity.²¹⁻²⁴ In this article, we will elucidate the intrinsic difference in the asymmetries, including failure modes, fracture strength, or plasticity by means of two typical Zr_{56.2}Ti_{13.8}Nb_{5.0}Cu_{6.9}Ni_{5.6}Be_{12.5} and Ti₆₀Cu₁₄Ni₁₂Sn₄Nb₁₀ composites containing ductile dendrites to gain a better understanding of their strengthening and toughening mechanisms.

II. EXPERIMENTAL

The processing and microstructure information about the two kinds of composites, $Zr_{56.2}Ti_{13.8}Nb_{5.0}Cu_{6.9}Ni_{5.6}Be_{12.5}$ and $Ti_{60}Cu_{14}Ni_{12}Sn_4Nb_{10}$ have been reported by Hays et al.^{21,22} and He et al.^{23,24} The Zr-based composite is composed of homogeneously dispersed bcc β -dendrites

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(25% in volume) and a metallic glass matrix (75% in volume). The composition of the matrix and dendrites for the Zr-based composite is Zr₄₇Ti_{12.9}Nb_{2.8}Cu₁₁Ni_{19.6}Be_{16.7} and Zr₇₁Ti_{16,3}Nb₁₀Cu_{1.8}Ni_{0.9}, respectively. The dendrites are characterized by primary dendrite axes with a length of 20-60 µm and secondary dendrite arms with a spacing of 2–3 µm. The Ti-based composite consists of homogeneously dispersed bcc B-dendrites (30% in volume) and a mixed matrix (70% in volume) with nanoscale eutectic and CuTi₂ intermetallics.²⁴ The composition of the matrix and dendrites for the Ti-based composite is Ti54.3Ni19.8Cu19.2Sn1.6Nb5.1 and Ti_{62.1}Ni_{2.6}Cu_{4.8}Sn_{8.2}Nb_{22.3}, respectively. The primary dendrite axes are 30-80 µm in length and the secondary dendrite arm spacing is 2-3 µm. Uniaxial compressive and tensile tests were performed to the Zr- and Ti-based composite specimens with a MTS810 testing machine at room temperature. All compressive and tensile tests were conducted using a constant strain rate of about 1×10^{-4} s⁻¹. Each mechanical test was repeated at least three times. After mechanical tests, all the specimens were observed by a Cambridge S360 SEM to reveal deformation and fracture features.

III. RESULTS

Figure 1 shows the tensile and compressive true stressstrain curves of the Zr- and Ti- based composite specimens deformed at a constant strain rate of 10^{-4} s⁻¹. Under both tensile and compressive loading, the Zr-based BMG composite displays an initial elastic deformation behavior with an elastic strain of about 2.0%. The tensile specimen fails at a stress of 1486 MPa (σ_T^F) with a small plasticity of about 3–5% (measured from the fracture specimen). In contrast, the compressive specimen displays a very high plasticity of 24.8%, and finally fails at a stress of 1549 MPa (σ_C^F). These results are comparable



FIG. 1. Tensile and compressive true stress-strain curves of Zr- and Ti- based composites at a strain rate of 10^{-4} s⁻¹.

with the data in the literature.^{21,22} The plastic deformation of the Zr-based composite is a balance between the strain hardening of the dendrite and the strain softening of the metallic glass matrix. The dendrites deformed at first with a slight strain hardening, followed by the deformation of the metallic glass matrix with strain softening, which is widely observed in monolithic metallic glass.¹⁷ From Figs. 2(a) and 2(c), it can be seen that both the tensile and compressive specimens failed in a shear mode. The tensile shear fracture angle $\theta_{\rm T}$ is about 48° (i.e., slightly larger than 45°). The compressive shear fracture angle $\theta_{\rm C}$ is quite close to 45°, and the accurate angle is difficult to measure because the specimen became slightly bent upon compression. Besides, there is no obvious difference in the deformation morphologies on the fracture surfaces for the Zr-based composite under compression and tension, as shown in Figs. 2(b) and 2(d). Profuse fine slip bands in the dendrites and dense shear bands in the glassy matrix are activated on the Zr-based composite surfaces, and contribute to the plasticity.^{21,22} Due to the constraint of the dendrites, most shear bands generated from two major directions and then interact with each other. In contrast, monolithic metallic glasses only display very high strength with nearly zero plasticity due to a rapid propagation of the major shear band under tension and compression.^{4,18,19}

The tensile specimen of the Ti-based composite displays only an elastic deformation behavior and catastrophically fractures at a low stress of $\sigma_T^F = 670 \text{ MPa}$ with zero plasticity. However, the compressive specimen yields at 850 MPa, followed by an obvious strain hardening up to a plastic strain of 11.4% before fracture. Its compressive fracture stress, σ_{C}^{F} , is 1595 MPa, which is significantly higher than the tensile fracture stress (670 MPa). SEM observations show that the tensile specimen failed in a brittle mode and its tensile fracture plane is approximately perpendicular to the stress axis (i.e., $\theta_{\rm T} = 90^{\circ}$). No shear bands can be observed on the lateral surface even near the fracture surface, as shown in Figs. 3(a) and 3(b). Figures 3(c) and 3(d) reveal that the compressive specimen also failed in a shear mode with a fracture angle of $\theta_{\rm C}$ = 45°. Some fine slip bands and shear bands were formed within the dendrites and the glassy matrix on the specimen surfaces.^{23,24} However, the compressive shear deformation degree of the Tibased composite is relatively weak in comparison with that in the Zr-based composite. The low tensile strength and zero tensile plasticity are different from those of the Ti-based composite with more stable microstructure fabricated by slow cooling,²⁵ which will be discussed later.

IV. DISCUSSION

From the results discussed in Sec. III, it can be concluded that there is a pronounced difference in the



FIG. 2. SEM micrographs revealing deformation and fracture features of Zr-based composite under (a) and (b) tensile loading; (c) and (d) compression loading.

mechanical properties and fracture modes for the Zr- and Ti-based composites. The Zr-based BMG composite always fails in a shear fracture mode with only slightly different shear fracture angles ($\theta_C = 45^\circ$ and $\theta_T = 48^\circ$, respectively) and has a small strength asymmetry $\sigma_C^F/\sigma_T^F = 1.04$. However, the compressive and tensile fracture angles of the Ti-based composite are significantly different (i.e., equal to 45° and 90°), respectively. Moreover, it exhibits a distinctly higher strength asymmetry ($\sigma_C^F/\sigma_T^F = 2.38$) than the Zr-based composite

 $(\sigma_{\rm C}^{\rm F}/\sigma_{\rm T}^{\rm F} = 1.04)$. Besides, the ductility is also quite different under compression and tension for the two composites. This indicates that the Ti-based composite is more sensitive to the stress state (tension or compression) than the Zr-based composite. Similar phenomena were also widely observed for a variety of materials, such as graphite, rocks, ceramics, intermetallics, nanostructured materials, etc.^{5–9,13,14} This strongly suggests that a common mechanism controlling the failure of those materials with high strength asymmetry should exist. In the



FIG. 3. SEM micrographs revealing deformation and fracture features of Ti-based composite under (a) and (b) tensile loading; (c) and (d) compression loading.

following section, we will elucidate the intrinsic difference in the asymmetries occurring in the Zr- and Tibased composites for a better understanding of the strengthening and toughening mechanisms in a variety of materials.

As is well known, the compressive failure of brittle materials is either controlled by the Tresca criterion³ or by the Mohr-Coulomb criterion.^{1–4} For the compressive failure of the Zr- and Ti-based composites, their shear fracture occurs approximately along the maximum shear stress plane [see Figs. 2(c) and 3(c)]. For brevity, the Tresca criterion can be used to describe the critical failure condition, i.e. $\tau_{max}=\tau_0$ (τ_0 is the intrinsic shear strength of material). Therefore, the compressive fracture strength of the two composites should be equal to $\sigma_{\rm C}^{\rm F} = 2\tau_{\rm max} = 2\tau_0$. When the two composites are subjected to tensile loading, they fail either in a normal fracture mode ($\theta_{\rm T} = 90^{\circ}$ for the Ti-based composite) or in a shear fracture mode ($\theta_{\rm T}=48^\circ$ for the Zr-based composite). To explain the difference in the observed fracture modes, a unified tensile fracture criterion²⁶ (i.e., ellipse criterion) was proposed:

$$(\sigma_n/\sigma_0)^2 + (\tau_n/\tau_0)^2 = 1$$
 . (1)

Here, σ_0 is defined as the intrinsic cleavage strength of a material under the condition without shear stress τ_n . Accordingly, the tensile fracture strength σ_T^F can be derived in terms of the unified tensile fracture criterion²⁶. Otherwise stated:

$$\sigma_{\rm T}^{\rm F} = 2\tau_0 \sqrt{1-\alpha^2} \qquad (\alpha = \tau_0/\sigma_0 \le \sqrt{2}/2) \quad , \eqno(2a)$$

$$\sigma^F_T = \sigma_0 = \tau_0/\alpha \qquad \qquad (\alpha = \tau_0/\sigma_0 \ge \sqrt{2}/2) \quad . \eqno(2b)$$

Here, $\alpha = \tau_0/\sigma_0$ is the ratio of shear strength τ_0 to cleavage strength σ_0 of a material.²⁶ The details of the failure mode of a material are strongly controlled by the ratio $\alpha = \tau_0/\sigma_0$, which is a function of the tensile shear fracture angle θ_T .²⁶ Otherwise stated:

$$\alpha = \tau_0 / \sigma_0 = \sqrt{\cos(2\theta_{\rm T}) / (\cos(2\theta_{\rm T}) - 1)} \quad . \tag{3}$$

This indicates that the tensile fracture of a material is controlled by both shear and normal stresses (σ_n , τ_n) on the shear plane, and depends on the two intrinsic strengths (σ_0 , τ_0).

Based on the theoretical analysis above, the failure conditions of the Zr- and Ti-based composites under tension and compression can be schematically illustrated as in Figs. 4(a) and 4(b). According to the two failure



FIG. 4. Illustration of critical tensile and compressive failure conditions at (a) $0 < \alpha = \tau_0/\sigma_0 < \sqrt{2}/2$ for Zr-based composite and (b) $\alpha = \tau_0/\sigma_0 \ge \sqrt{2}/2$ for Ti-based composite.

	Tension			Compression						
	$\overline{\sigma_{T}^{F}(MPa)}$	$\epsilon_{\rm P}~(\%)$	$\theta_{\rm T}$	$\sigma^{\rm F}_{\rm C}~({\rm MPa})$	ϵ_{P} (%)	$\theta_{\rm C}$	τ_0 (MPa)	$\sigma_0 \; (MPA)$	α	$\sigma_{C}^{F}/\sigma_{T}^{F}$
Z	1486	5.2	48°	1549	24.8	45°	780	2538	0.31	1.04
Т	670	0	90°	1595	11.4	45°	798	670	1.19	2.38

TABLE I. Mechanical properties and failure modes of $Ti_{60}Cu_{14}Ni_{12}Sn_4Nb_{10}$ and $Zr_{56,2}Ti_{13,8}Nb_{5,0}Cu_{6,9}Ni_{5,6}Be_{12,5}$ composites containing ductile dendrites.

Note: $Z = Zr_{56.2}Ti_{13.8}Nb_{5.0}Cu_{6.9}Ni_{5.6}Be_{12.5}$ and $T = Ti_{60}Cu_{14}Ni_{12}Sn_4Nb_{10}$.

criteria,^{3,26} the strength asymmetry σ_C^F/σ_T^F can be expressed as:

$$\begin{split} \sigma^F_C/\sigma^F_T &= 1/\sqrt{1-\alpha^2} \qquad (\alpha &= \tau_0/\sigma_0 \leqslant \sqrt{2}/2) \quad, \eqno(4a) \\ \sigma^F_c/\sigma^F_T &= 2\alpha \qquad \qquad (\alpha &= \tau_0/\sigma_0 \geqslant \sqrt{2}/2) \quad. \eqno(4b) \end{split}$$

The strength asymmetry σ_C^F/σ_T^F increases when increasing the ratio $\alpha = \tau_0/\sigma_0$. From Eqs. (1)–(4) and the measured values of σ_{T}^{F} , σ_{C}^{F} , θ_{C} and θ_{T} , the constants τ_{0} , σ_0 , and α were calculated and are listed in Table I. Apparently, the two composites have nearly the same shear strength τ_0 and compressive fracture strength $\sigma_{\rm C}^{\rm F}$ when they fail in a shear failure mode. Therefore, shear bands were observed for both of the two composites under compression, as shown in Figs. 2(d) and 3(d). This indicates that the compressive strength $\sigma_{\rm C}^{\rm F}$ of a material is mainly controlled by its shear strength τ_0 , but is independent of the cleavage strength σ_0 . According to the unified criterion,²⁶ it is known that when $\alpha = \tau_0 / \sigma_0 \le \sqrt{2}/2$ or $\sigma_0 \ge \sqrt{2} \tau_0$, the specimen will fail in a shear mode under tension or compression loading, as schematically illustrated in Fig. 4(a). From Table I, it can be seen that the ratio $\alpha = \tau_0 / \sigma_0$ is equal to 0.31 for the Zr-based composite (i.e., is smaller than $\sqrt{2/2}$). Therefore, shear bands are easily activated under both compression and tension, resulting in a very small strength asymmetry and obvious shear plasticity, as shown in Figs. 2(b) and 2(d). In contrast, the ratio $\alpha = \tau_0/\sigma_0$ for the Ti-based composite is 1.19, obviously higher than the critical value of $\sqrt{2}/2$. Hence, shear bands can only be formed under compression, but are absent under tension in the case of the Ti-based composite, as shown in Figs. 3(b) and 3(d). Therefore, cleavage or normal fracture (i.e., $\theta_{\rm T} = 90^{\circ}$) becomes the more preferred failure mode than shear deformation for the Ti-based composite, as schematically illustrated in Fig. 4(b). This causes an early normal fracture prior to shear deformation under tension, leading to zero tensile plasticity. Because the tensile fracture strength σ_T^F depends on both shear strength τ_0 and cleavage strength σ_0 , it is deduced that the very low tensile fracture strength σ_{T}^{F} of the Ti-based composite should be attributed to a large decrease in its cleavage strength σ_0 . Therefore, materials with a ratio

higher than $\sqrt{2}/2$ will exhibit a substantial strength asymmetry under compression and tension. It is believed that the low cleavage strength σ_0 can be attributed to the casting flaws or pores in the Ti-based composite. Normally, it seems that there are no obvious flaws or pores in the microstructure of the Ti-based composite. However, it is often observed that there exists inhomogeneous microstructure or occasionally some coarse Nb particles in the Ti-based composite as reported previously.²⁷ Those inhomogeneous microstructures or occasional coarse Nb particles will affect the tensile properties to some extent. If the Ti-based composite is made into a specimen with a smaller size, it can display certain tensile plasticity due to the exclusion of the inhomogeneous microstructure or occasional coarse Nb particles.²⁴ Eliminating the inhomogeneous microstructure should be important for the increase in the cleavage strength, moreover to improve the tensile fracture strength, even the tensile plasticity for the Ti-based composite with high performance.²⁵ Moreover, it seems that the deformation mechanism of such kind of composites should be very interesting and we will further reveal the relationship between the mechanical property and the microscopic deformation mechanism in the future study.

V. CONCLUSIONS

In summary, the essence of strength improvement for traditional metallic materials is mainly due to the increase in the shear strength τ_0 , leading to a high $\alpha = \tau_0 / \sigma_0$ ratio. The compressive strength $\sigma_{\rm C}^{\rm F}$ is only controlled by its shear strength τ_0 ; however, the tensile strength σ_T^F is a function of shear strength τ_0 and cleavage strength σ_0 . With increasing the ratio $\alpha = \tau_0 / \sigma_0$, according to Eq. (4a), the strength asymmetry $(\sigma_{\rm C}^{\rm F}/\sigma_{\rm T}^{\rm F})$ becomes substantial, which results in the widely observed strength anisotropy in a variety of materials, such as ceramics, graphite, rock, intermetallics as well as ultra-fine grained and nanostructured materials or glasses.^{5-9,13,14} When the ratio $\alpha = \tau_0 / \sigma_0$ is larger than $\sqrt{2/2}$, increasing the shear strength τ_0 has no influence on the tensile strength $\sigma_T^{\rm F}$ according to Eq. (2b). In this case, shear bands will not be activated before fracture, resulting in zero tensile plasticity, as observed for the Ti-based composite. This indicates that one should control the ratio $\alpha = \tau_0 / \sigma_0$ to be below the critical value of $\sqrt{2}/2$. Otherwise, there should be no tensile plasticity. It is suggested that one should not only consider the shear strength τ_0 itself, but also the cleavage strength σ_0 when aiming for strengthening a material. Maintaining a higher shear strength τ_0 is substantially important for achieving both tensile and compressive strengths (σ_T^F and σ_C^F). However, for the first time, it is suggested that a good balance ($\alpha = \tau_0/\sigma_0$) between shear strength τ_0 and cleavage strength σ_0 is also necessary for the improvement of the tensile plasticity. This new strategy is important for the optimum design of high-performance materials, not only for the new BMG composites but also for nanostructured materials.

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