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Outstanding shear resistance in a low-density refractory Ti₃NbVAl high-entropy alloy subjected to dynamic loading

ABSTRACT

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strate that the Ti₃NbVAl HEA exhibits great toughness under dynamic-loading conditions. An excellent loadbearing capacity under high-strain rate loading conditions leads to its promising applications in aerospace, transportation, and energy. High-entropy alloys (HEAs), as an emerging group of highperformance metallic materials, have attracted great attention since its origin at 2004 [1–6]. Refractory HEAs, especially low-density refractory HEAs (LDRHEAs) are developing due to their unique mechanical properties, such as high specific strength, good fracture toughness, excellent fatigue resistance and corrosion resistance at elevated temperatures, as well as their promising application emerges in aviation, transportation,

and energy-saving fields [7-10]. Among all the lightweight metallic elements, titanium can obtain remarkable strength and replace some ceramic components and highdensity superalloys. The titanium-containing LDRHEAs as a broader concept of titanium alloys can achieve the long-standing goal of surpassing the high-temperature structural performance of conventional alloys. Stepanov et al. [11,12] have reported a series of Ti-based LDRHEAs with different elemental additions for improving mechanical properties and weight reduction. The coarse-grained single-phase BCC-structured AlNbTiV alloy with a density of 5.59 g cm⁻³ has a compressive yield strength of 1,020 MPa at room temperature and 685 MPa at 800 °C. Its specific yield strength is comparable to the that of the

multiphase-refractory HEA [9]. However, it does not exhibit significant plastic deformation and is brittle at room temperature. Later, its BCC-structured single phase is proved to be an ordered B2 compound with a considerable yield strength of 1,000 MPa and plasticity of 6.0 % at room temperature [12].

The mechanical property of a low-density refractory Ti₃NbVAl high-entropy alloy (HEA) with an ordered B₂

structure is investigated in the dynamic regime. The yield strength increased from 751 MPa at a strain rate of

 10^{-3} s⁻¹ to 1,400 MPa at a strain rate of $\sim 10^3$ s⁻¹, indicating an extraordinary strain-rate-hardening capability.

Dynamic compression of the hat-shaped sample suggests that it also exhibits excellent shear resistance under a

deviatoric strain rate of 10⁵ s⁻¹, which resists the adiabatic shear-band formation at a large imposed shear strain of \sim 16. The microstructure inside the shear bands shows that a severe grain refinement combined with a B₂ to body-centered-cubic (BCC) crystal structure transition occurred during shear localization. Our results demon-

> Due to the excellent performance of the LDRHEAs and their outstanding potential as candidates for aerospace applications, it is important to establish the mechanical behavior of the alloys especially under harsh environments-dynamic conditions [13–17]. Take the CrMnFeCoNi (Cantor) HEA an example, it shows the most remarkable resistance to shear localization with a critical shear strain up to ~ 7 [18, 19], which is attributed to its excellent strain-hardening ability as well as weak thermal-softening effect [19]. The extraordinary strain-hardening ability of the annealed CrMnFeCoNi HEA under a dynamic condition has also been attributed to dynamically formed nanoscale twins [20]. It has been reported that a variety of deformation mechanisms, including stacking faults, twins, transformation from the face-centered-cubic (FCC) to the hexagonal-close-packed (HCP) structure, and the amorphous-structured area were found in a

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severely-deformed equiatomic CrMnFeCoNi HEA, which provide the outstanding mechanical properties with additional strengthening and/or toughening mechanisms of the HEA alloys under extreme loading conditions [21]. Meanwhile, both the tensile strength and plasticity of the CrMnFeCoNi HEA were enhanced significantly at high strain rates under dynamic tension due to significant deformation nano-twins. However, due to the single deformation mechanism, such as dislocation planar slip, and the inadequate dislocation interaction, the strain-hardening capacity in RHEAs with a single BCC structure is relatively weak [22]. As for ordered B2 intermetallic compound RHEAs, the intrinsic brittleness bottlenecks the application of the materials, whose dynamic-mechanical behavior is rarely addressed.

In this investigation, we carried out dynamic tests on the newlydesigned ordered B2 intermetallic compound in a Ti_3NbVAl LDRHEA by the Split Hopkinson Pressure Bar to investigate its dynamic behavior. Special attention was paid to the formation of adiabatic shear bands and their microstructural evolution characteristics.

The as-received Ti₃NbVAl LDRHEA was fabricated by arc-melting its constituent elements of Ti, Al, V, and Nb (purity > 99.9 weight percent, wt. %) elements and was then subjected to hot-isostatic-pressing (HIP) at 1,204 °C and 103 MPa for 4 h. The quasi-static compression tests were performed in a Universal Testing Machine Instron 3367, using a cylinder specimen with 4 mm in diameter and 6 mm in length. The Split Hop-kinson Pressure Bar (SHPB) was employed to test the normal- and shear-

deformation behavior, with cylindrical and hat-shaped specimens. The hat-shaped specimens, originally designed by Meyer and Manwaring [19], were used to generate forced shear bands. Finally, the specimens were analyzed by the Rigaku D/max2500 XRD, FEI Helios G4 CX scanning electron microscopy (SEM), and Thermo QuasOr electron backscatter diffraction (EBSD). A focused ion beam (FIB) instrument was used to accurately prepare the FEI Themis Z Double Cs Corrector transmission electron microscope (HRTEM) samples inside the shear-band. A SuperX energy-dispersive spectrometer (EDS) and precession electron diffraction (PED) device were also utilized. The nano-hardness analyses were performed by an instrumented Hysitron TI980 nanoindenter.

The initial microstructure of the as-received LDRHEA alloy is composed of a single B2 intermetallic compound with a grain size ranging from 400 to 600 µm, as presented in Figs. 1(a) and S1. The density of the alloy measured by the Archimedes method is about ~ 5.4 \times 10³ kg.m⁻³, which is a little lower than the LDRHEAs reported by Stepanov et al.[9]. The quasi-static compressive true stress and true strain curves at room temperature are also plotted in Fig. 1 (a), which presents a considerable strain-hardening ability at ambient temperature during plastic deformation. With strain rates of 10⁻³, 10⁻², and 10⁻¹ s⁻¹, the yield stresses of 751 MPa, 912 MPa, and 940 MPa were achieved, respectively. The stress vs. strain curves at high strain rates are shown in Figs. 1(b) and (c). When subjected to a high strain rate of $\dot{\varepsilon} \sim 10^3$ s⁻¹, the



Fig. 1. The mechanical behavior of the Ti₃NbVAl alloy at room temperature: (a) true stress-strain curves of cylindrical specimens under quasi-static with SADP and XRD patterns of the master-material inserted; (b) true stress-strain curves of cylindrical specimens under dynamic compression with macroscopic images of the deformed cylindrical specimens; (c) shear stress-strain curves under the dynamic-impacting condition with deformed hat-shaped specimens; (d) strain-rate sensitivities at low and high strain rates.

yield stress of σ_y increases to ~ 1,400 MPa. This tendency is comparable with the dynamic strength of the Ti-6Al-4V alloy. Thus, the Ti₃NbVAl LDRHEA exhibits an outstanding work-hardening ability under both static and dynamic conditions. Furthermore, the nominal failure strain is larger than that of the Ti-6Al-4V alloy under both static and dynamic loading conditions, which indicates a superior toughness of the Ti₃NbVAl LDRHEA alloy, as compared to the benchmark low-density tit anium alloy of Ti-6Al-4V [23].

At higher strain rates, the hat-shaped specimens exhibit a yield shear stress at ~ 650 MPa at the strain rate of ~ 10^5 s^{-1} , as presented in Fig. 1 (c). A critical shear strain for shear localization with a high value of ~16 is found in the specimen which is much larger than that found in CrMnFeCoNi HEA [22]. It is suggested that this alloy also presents excellent shear resistance under a deviatoric strain rate of 10^5 s^{-1} . Furthermore, the fluctuations in the stress-strain curves at different strain rate of $2 \times 10^{5} \text{ s}^{-1}$, which are supposed to be the result from the dispersion effect of stress waves [24].

$$m = kT/\tau v^* \tag{1}$$

where *k* is the Boltzmann constant, *T* is the temperature, τ is the stress, and ν^* is the activation volume. The higher strain-rate sensitivity of this LDRHEA, compared with conventional BCC alloys, should be caused by a smaller activation volume for dislocation motion in the LDRHEAs. This behavior is also observed in the single FCC Al_{0.3}CoCrFeNi and CoCrFeMNi HEAs, compared with conventional FCC alloys [18,19].

With the increase of the shear strain and strain rate, shear localization becomes an important failure mechanism. The shear bands are found in the hat-shaped specimen at a critical shear strain of ~ 16 [Fig. 2 (a)]. Shear bands can be found along the largest shear-stress direction within the designed shear region. The width is about 20 - 30 μ m. Cracks can be seen at the tips of the shear band. Staker [25] has proposed an expression for the critical shear strain for shear-band formation based on the competition between the strain-hardening and thermal softening before shear localization by neglecting the strain-rate hardening effect:

$$\gamma_c = \frac{n\rho C_p}{-\frac{\partial r}{\partial T}} \tag{2}$$

where ρ is the density, C_p is the specific-heat capacity, n is the strainhardening power index for the constitutive equation ($\tau = \tau_0 + K\gamma^n$, where τ_0 is the yield stress, and K is the constant), and the term, $\partial \tau / \partial T$, is the thermal-softening parameter. From Eqn. (2), the value of $n\rho C_p$ can be regarded as the hardening component, while the $\partial \tau / \partial T$ is the competitive softening counterpart. The critical shear strain of ~ 16 in LDRHEA is much larger than that in the FCC CrMnFeCoNi alloy as well as the other alloys [26]. A smaller thermal-softening effect and a larger strain-hardening effect can be predicted in LDRHEA.

A TEM specimen inside the shear band was prepared by FIB, as indicated in Figs. 2(b) and (e). The significantly refined grains with grain size ranging from 100 to 300 nm are observed inside the shear band, which is similar to the equiaxed grains obtained in a dynamically-impacted CoCrFeMnNi HEA [19]. Different orientations are clearly seen in the nano-sized grains, and the diffraction pattern in Fig. 2(c) exhibits a polycrystalline structure. No second phase is formed at the grain boundary, as seen in Fig. S2. A selected area diffraction pattern (SADP) taken along the [-100] zone axis is presented in Fig. 2(c). The crystal structure is shown to be a BCC type, which is different from the initial undeformed alloy. It provides an evidence of the phase transition from the B2 to BCC structure. The LDRHEA alloy changes the crystal structure from an ordered B2 to a disordered BCC structure, which carries plastic deformation under dynamic loading.

In addition, the microstructure in the interrupted specimens are also presented in Figs 2 (e)-(f). Multiple slip bands are found in the designed



Fig. 2. The shear structure in the hat-shaped specimen: (a)-(c) microstructure characterization of the specimen with formation of ASB; (d)-(f) microstructure characterization of the interrupted specimen; (a) and (d) macroscopic structure of the shear zone; (b) and (e) the FIB position and EBSD map; (c) and (f) the bright-field TEM image with the selected area diffraction pattern (SADP) inside the bands showing the crystal structures.

shear zone. The grains are clearly elongated and nanograins are scare found inside the slip bands. The phase structure also remains B2 (ordered BCC) structure, as presented in Fig. 2(f). It suggests that the B2 \rightarrow BCC transition may only take place once the shear strain exceeds a critical value. Such structure transition may also cooperates the high shear resistance of LDRHEA alloy.

With further analysis by EBSD and PED, the deformation features inside shear bands are clarified. Many dislocation lines caused by their pileup are clearly distinguished adjacent to the shear band, suggesting that there is plastic deformation through a dislocation glide, as shown in Figs. 3(a) and (b). It can be deduced that the deformation process of the LDRMEA alloy is as follows: First, with the increase of the applied strain, the grains deform to the elongated grains and dislocations in a single grain pile up along crystallographic directions (Fig. 4c); Second, subgrain boundaries form along the reorganized dislocations, and the continued deformation increases the sub-grain misorientation (Fig. 4d); Third, once the shear strain exceeds a critical value, the B2 (ordered BCC) crystal structure transforms into disordered BCC structure. Meanwhile, nano-sized equiaxed grains form by the rotation of grain boundaries and the formation of sub-grain boundaries (Fig. 4e) [27]; Finally, the shear band, which is composed of fruitful recrystallized equiaxed nano-grains, forms along the maximum shear-stress direction (Fig. 4f). This feature is quite different from the other alloys, such as the coarse-grained beta titanium alloy, which exhibits a large deformation area covering several grains near the shear band [28].

Figs. 3(c) and (d) illustrate the microstructure within the shear band measured by the PED technique. It shows clearly that the initial coarsegrained structure has been replaced completely by an ultrafine-grained one with a grain size of \sim 300 nm. The geometric necessary dislocations (GND) can be computed by numerically solving the Nye tensor [29]. Fig. 3(d) shows that these GNDs are predominantly located along the grain boundaries. The crystallographic texture inside the shear band can be inferred from Figs. S5(d-f), which are inverse pole figures of the rolling (RD), transverse (TD), and normal directions (ND), respectively. The absence of a strong texture inside the shear band indicates a fully recrystallized microstructure, which is consistent with the electron-diffraction pattern shown in Fig. 2(c).

In order to understand the influence of the drastic microstructural changes on the mechanical properties, nano-hardness measurements were performed across the shear bands and presented in Fig. 3(f). The hardness inside the shear band reaches a maximum value of ~ 4.2 GPa near the center of the shear band. Such a value decreases gradually to an average value of 3.7 GPa outside the band. This 15 % increase of the hardness is due to the Hall-Petch effect, where the grain size decreases and therefore, a larger fraction of the grain boundaries reduces the mean free path of dislocations. The hardness enhancement in a highly deformed recrystallized region of the LDRHEA is consistent with the most related works reporting that the adiabatic shear bands composed of recrystallized equiaxed nano-grains exhibit hardness enhancement. Such phenomena are also observed in the shear bands of the pure Ti [30], Ti-6Al-4V [23], pure iron [31], CrMnFeCoNi [32], Al_{0.1}CoCrFeNi [33], as well as Zr-based metallic glasses [34].

A comparison of the dynamic yield stress at the strain rate of $\sim 10^3$ s⁻¹ as a function of the density with other major light metals/alloys and the classical commercial alloys is presented in Fig. 5 (a). It can be seen that the Ti₃NbVAl LDRHEA exhibits a remarkable high dynamic yield strength with a relatively low density. Thus, its specific strength at elevated strain rates exceeds that of many other low-density engineering materials [10,19,23,30,35–49], such as Ti-6Al-4V and Al alloys. Furthermore, the absorbed strain energy of the Ti₃NbVAl alloy is quite



Fig. 3. Microstructure and nanohardness characteristics near or inside the shear band: (a) the orientation map and (b) Kernel Avg Misorientation map of the microstructure near the shear band; (c) orientation map and (d) geometrically-necessary dislocations (units in m⁻²) of the microstructure inside the shear band; (e) misorientation degree within different grains adjacent to the shear band; (f) nanohardness variation inside and outside the shear band.



Fig. 4. Microstructure evolutions in the Ti₃NbVAl alloy during severe plastic deformation: (a) initial grains, (b) a single grain, (c) pile-up of dislocations, (d) rotation of segments of the single grain, (e) formation of subgrains with B2 to BCC transition, and (f) shear band with equiaxed grains.



Fig. 5. Dynamic yield stress vs density (a) and absorbed strain energy (b) for the Ti₃NbVAl alloy in relation to some classical-engineering materials.

superior compared to the Ti-6Al-4V and many classical engineering alloys [50–52], as exhibited in Fig. 5(b). The absorbed strain energy reaches 10 GPa at a critical shear strain (also named a failure shear strain) of \sim 16. It, therefore, may serve in extreme load-bearing conditions.

In summary, excellent mechanical properties at elevated strain rates were found in a newly designed, low-density refractory Ti_3NbVAI highentropy alloy, which is manifested by a drastic increase of the yield strength from 751 MPa at a quasi-static strain rate of 10^{-3} s⁻¹ to ~ 1,400 MPa at a strain rate of $\sim 10^3$ s⁻¹, indicating a superior strain-rate hardening ability. The Ti₃NbVAl alloy possesses an outstanding resistance to shear localization and its shear strength can be up to approximately 650 MPa at a shear strain rate of 10^5 s⁻¹. The critical shear strain for the onset of the shear band reaches ~ 16, which is remarkably high among many engineering alloys. The recrystallized grains inside the shear band are substantially refined to $100 \sim 300$ nm, and a unique phase transition from a B2 to BCC phase is found during severe plastic deformation inside a shear band. The outstanding shear resistance as well as its intrinsic low mass density put Ti₃NbVAl in a strategically advantageous position. With a global comparison of the dynamic mechanical properties, the superior specific strength as well as high toughness, compared with the most commercial lightweight structural materials, renders it a viable candidate for load bearing applications.

Data availability

Data will be made available on request.

CRediT authorship contribution statement

Na Yan: Writing – review & editing, Writing – original draft, Resources, Methodology, Investigation, Funding acquisition, Conceptualization. **Shiteng Zhao:** Writing – review & editing, Investigation, Funding acquisition. **Zezhou Li:** Writing – review & editing, Investigation. **Rui Feng:** Investigation. **Peter. K. Liaw:** Writing – review & editing, Supervision, Funding acquisition. **Marc André Meyers:** Writing – review & editing, Supervision, Resources, Methodology.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.scriptamat.2025.116610.

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