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Extraordinary tensile properties of titanium alloy with heterogeneous phase-distribution based on hetero-deformation induced hardening

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ABSTRACT

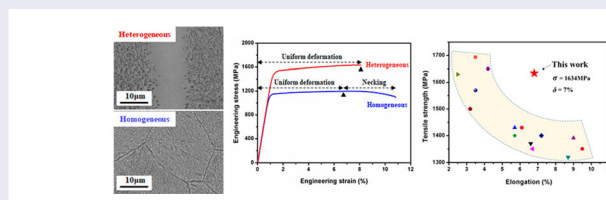
A heterogeneous ($\alpha + \beta$) titanium alloy with extraordinary tensile properties was prepared. The heterogeneous microstructures, consisting of soft β_{fiber} and hard ($\alpha + \beta$)_{matrix}, were achieved by controlling the distribution of β -stabilizing element. The heterogeneous alloy exhibits ultra-high strength of 1634 MPa with reasonable ductility, which is much higher compared to the homogeneous counterpart. The plastic incompatibility of the β_{fiber} and ($\alpha + \beta$)_{matrix} produces hetero-deformation induced (HDI) hardening, which is the main reason for the enhanced strength. Moreover, the α -twinning and martensitic transformation are responsible for the good ductility. This work provides a new strategy to achieve superior strength-ductility combination for multi-phase alloys.

ARTICLE HISTORY

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KEYWORDS

Titanium; heterogeneous structure; mechanical property; hetero-deformation induced (HDI) hardening



IMPACT STATEMENT

Heterogeneous phase-distribution was realized in an ($\alpha + \beta$) titanium alloy to achieve superior strength-ductility combination based on hetero-deformation induced hardening.

Achieving superior combination of strength and ductility for metals is an important object in high-technique industry [1]. Unfortunately, a gain in strength is usually accompanied by a sacrifice in ductility, resulting in a strength-ductility trade-off [2,3]. Extensive efforts have been paid on overcoming the strength-ductility trade-off dilemma. As for ($\alpha + \beta$) titanium alloy, the most typical high-strength titanium alloy, the mechanical properties are expected to be largely dependent on the grain size, fraction, morphology and distribution of α phase [4,5]. High strength is usually associated with fine and dense α phase. For example, by refining the α size, Mantri et al. [4] have tuned the ultimate tensile strength (UTS) of a Ti-15Mo-3Nb-2.7Al-0.2Si alloy from ~ 1 to 1.9 GPa. However, the precipitation hardening of α precipitates may reduce the ductility. Zhu et al. [2] have designed a strong and ductile Ti-5Al-4Zr-8Mo-7V alloy with fine and hierarchical α precipitates. While the refining of α

precipitates contributes to high strength, the hierarchical distribution of α precipitates helps relieve stress concentration and leads to a reasonable ductility. But, the improvement of ductility by arranging the distribution of α phase is limited. In particular, the uniform tensile elongation decreases to less than a few percent when the UTS is over 1300 MPa [6]. Therefore, imparting high strength without sacrificing too much ductility is one of the major challenges in high-strength titanium alloys.

Recently, the design of heterogeneous structures has achieved remarkable strength-ductility enhancement compared to their homogeneous counterparts [1,7,8]. In heterogeneous structures, soft and hard regions are mixed together, forming a high density of interfaces. During plastic deformation, the soft regions deform plastically more than hard regions, resulting in a gradient of plastic deformation [9]. The inhomogeneous plastic deformation will generate geometrically necessary

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dislocations (GNDs) to accommodate the strain gradient, and thereby leading to the hetero-deformation induced (HDI) hardening [10]. The HDI hardening can significantly enhance the strength-ductility combination. Nevertheless, the reported good combination of strength and ductility has been achieved mostly in single-phase alloys [7,8]. Dual-phase alloys usually exhibit higher strength, and have higher variability in microstructures and mechanical properties [4,11]. Therefore, it is important to introduce the hetero-structures in dual-phase alloys.

In the present work, we designed an ultra-high strength $(\alpha + \beta)$ titanium alloy with heterogeneous microstructures by controlling the distribution of β -stabilizing element, and the effect of HDI hardening on the mechanical behavior was investigated.

High purity ($> 99.5\%$) Ti, Al40V60 and Mo powders were used as raw materials to prepare the Ti-3Al-4.5V-5Mo alloys. The powders were mixed for 10 h under argon atmosphere. Then, the powder mixtures were compressed via cold isostatic pressing at a pressure of 200 MPa. The compacts were sintered at 1200°C for 2 h in vacuum, followed by furnace cooling. A heterogeneous microstructure with $(\alpha + \beta)_{\text{matrix}}$ and β_{particle} was obtained after sintering, as shown in Figure S1(a). To make a comparison, another group of as-sintered samples were annealed at 1200°C for 10 h to achieve a homogeneous microstructure, as shown in Figure S1(b). Cylindrical bars with a diameter of 40 mm and a length of 400 mm were cut from the as-sintered and as-annealed bulks, and then underwent hot swaging at 950°C . The as-sintered and as-annealed bars were swaged to a diameter of 8 mm followed by furnace cooling (hereinafter referred to as 'Heterogeneous' and 'Homogeneous', respectively).

Cylindrical tensile specimens with a gauge diameter of 3 mm and a length of 20 mm were cut from the as-swaged bars. Uniaxial tensile tests and load-unloading-reloading (LUR) tensile tests were conducted on a MTS Landmark test machine at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$. During LUR testing, the specimens were unloaded in a load-control mode to 350 N at an unloading rate of 3000 N min^{-1} . An extensometer was used to measure tensile strain. X-ray diffraction (XRD, Advance D8), scanning electron microscope (SEM, Helios Nanolab G3 UC), transmission electron microscope (TEM, Tecnai G2 F20) and electron probe microanalysis (EPMA, JXA-8530F) were used to study the microstructures. The micro-hardness was measured by using an IBIS Nano-indentation tester.

The microstructures of the hot swaged Ti-3Al-4.5V-5Mo alloys are shown in Figure 1. Obviously, the post-annealed alloy has a homogeneous microstructure (Figure 1(a-c)), while the alloy without annealing shows a heterogeneous microstructure (Figure 1(d-f)). Both the

two alloys consist of α and β phases. The phases distribute uniformly in the homogeneous alloy. But, the heterogeneous alloy has a non-uniform distribution of phases, with a fiber-structured single β phase in $(\alpha + \beta)$ binary phase matrix. Figure 1(f) shows a clear interface separating $(\alpha + \beta)_{\text{matrix}}$ and β_{fiber} . The corresponding elemental mapping of Figure 1(f) indicates that β_{fiber} is enriched with Mo, while Ti, Al and V distribute uniformly. It should be noted that although both Mo and V have high melting point, the diffusion coefficient of Mo ($\sim 2.5 \times 10^{-9} \text{ cm}^2 \text{ s}^{-1}$) is much lower than V ($\sim 2.0 \times 10^{-8} \text{ cm}^2 \text{ s}^{-1}$) in titanium [12,13]. Therefore, Mo-rich regions could form during sintering, while other elements diffused homogeneously. Since Mo is a strong β -stabilizing element [14], in the Mo-rich regions occur the formation of β particles, as shown in Figure S1(a). After swaging, β particles were stretched to β fibers, forming a heterogeneous microstructure. The elemental distributions in the homogeneous alloy were also measured, as shown in Figure S2, and the chemical compositions of individual phases in each alloy are shown in Table 1 (in supplemental material). Obviously, the elements distribute uniformly in the homogeneous alloy. The content of Mo in $(\alpha + \beta)_{\text{matrix}}$ of heterogeneous alloy is lower than that in the homogeneous alloy. The grain size, the phase fraction and the texture of the homogeneous alloy and the heterogeneous alloy ($(\alpha + \beta)_{\text{matrix}}$) are shown in Figures S3 and S4. The heterogeneous alloy shows the same texture character, but finer grain size and a slightly higher α phase fraction compared to the homogeneous alloy.

The details of the interface between $(\alpha + \beta)_{\text{matrix}}$ and β_{fiber} in the heterogeneous alloy are shown in Figure 2. The bright field (BF) images and the corresponding selected area electron diffraction (SAED) patterns further verify that the matrix is composed of β and α phases, while the fiber consists of single β phase. The interface separating $(\alpha + \beta)_{\text{matrix}}$ and β_{fiber} is clean, without obvious metastable phase. The micro-hardness of $(\alpha + \beta)_{\text{matrix}}$ is much higher than that of β_{fiber} , mainly due to the precipitation hardening caused by the fine α precipitates.

The tensile properties of heterogeneous and homogeneous Ti-3Al-4.5V-5Mo alloys are shown in Figure 3. The heterogeneous alloy shows an exceptionally high tensile strength of 1634 MPa, with a uniform elongation of 7%. The homogeneous alloy, as a contrast, has a tensile strength of 1205 MPa with a uniform elongation of 6% (the total elongation is 10.2%). The uniform elongation was determined based on the Considère criterion [15]. Obviously, the heterogeneous alloy achieves remarkable enhancement in strength without sacrificing ductility. Moreover, the heterogeneous alloy exhibits

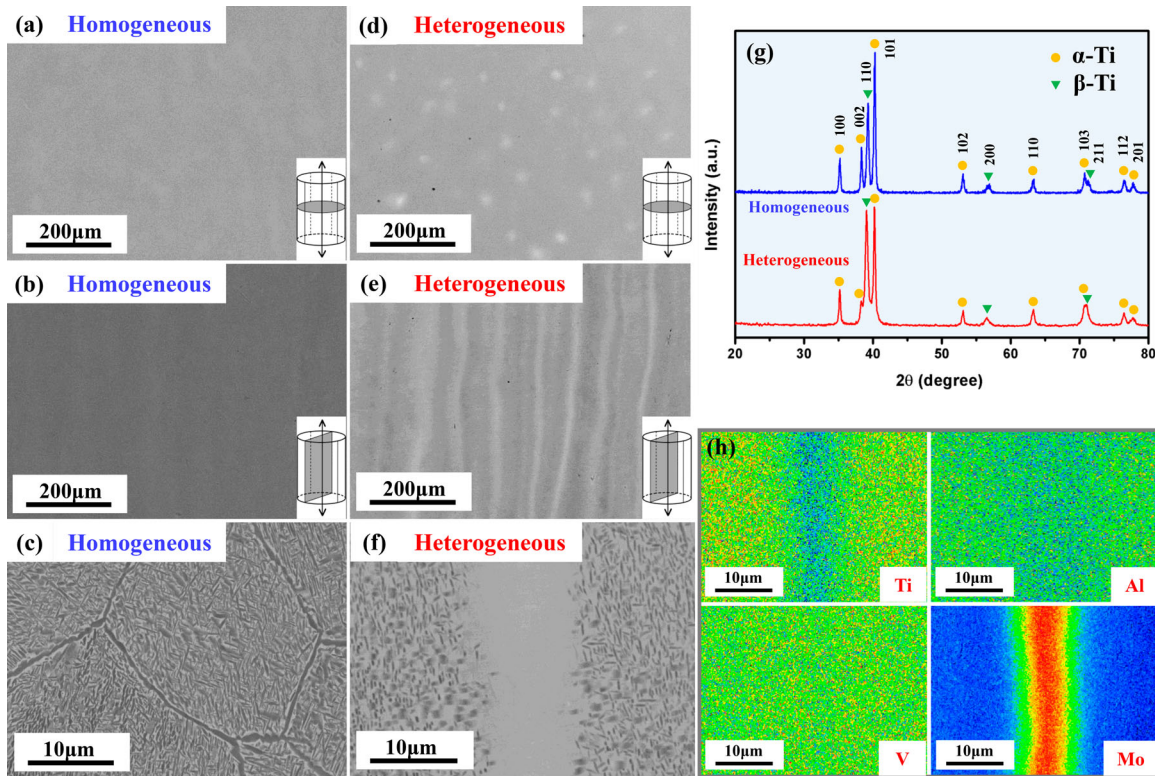


Figure 1. Microstructures of Ti-3Al-4.5V-5Mo alloy with homogeneous phase-distribution (a ~ c) and heterogeneous phase-distribution (d ~ f): (a) and (d) Low magnification micrographs of transverse section; (b) and (e) Low magnification micrographs of longitudinal section; (c) and (f) High magnification micrographs of longitudinal section; (g) XRD patterns of the alloys; (h) EPMA elemental mapping of corresponding region in (f).

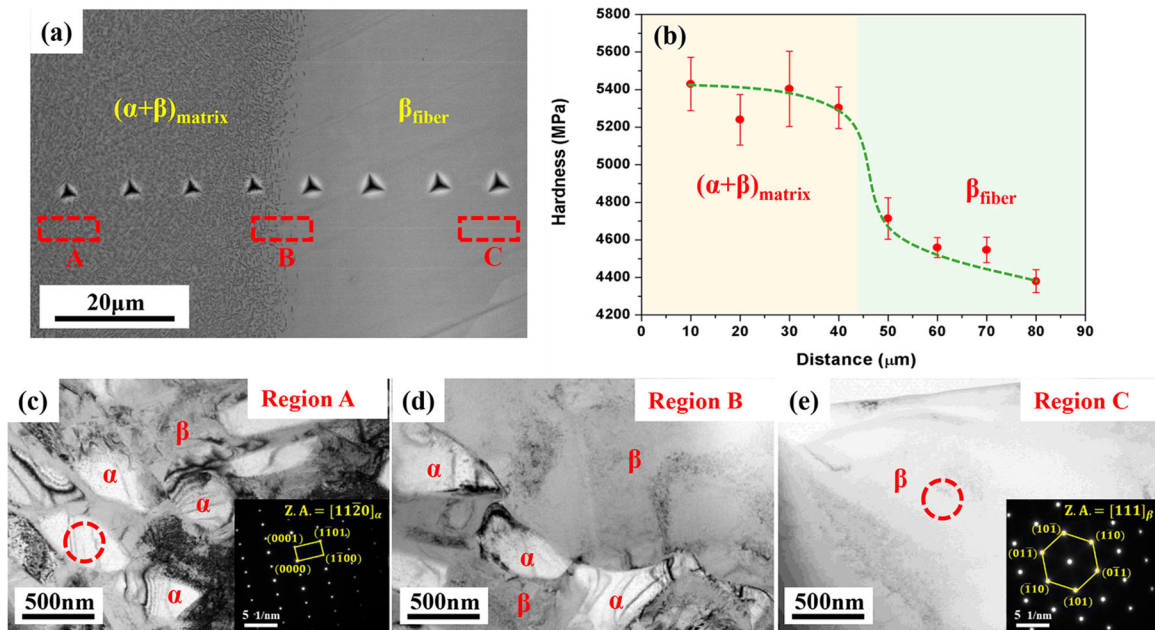


Figure 2. Details of the interface between $(\alpha + \beta)_{\text{matrix}}$ and β_{fiber} in heterogeneous Ti-3Al-4.5V-5Mo alloy: (a) Microstructure of the interface after nano-indentation testing; (b) Micro-hardness of $(\alpha + \beta)_{\text{matrix}}$ and β_{fiber} ; (c) TEM image in $(\alpha + \beta)_{\text{matrix}}$ region; (d) TEM image in $\beta_{\text{fiber}}/(\alpha + \beta)_{\text{matrix}}$ region; (e) TEM image in β_{fiber} region.

a higher work hardening rate of about 2 GPa than the homogeneous alloy. A comparison in tensile properties of the studied heterogeneous alloy with other high-strength

titanium alloys is presented in Figure 3(c) [2,4–6,16–20]. The high-strength titanium alloys (> 1300 MPa) are in the dilemma of the strength-ductility trade-off. However,

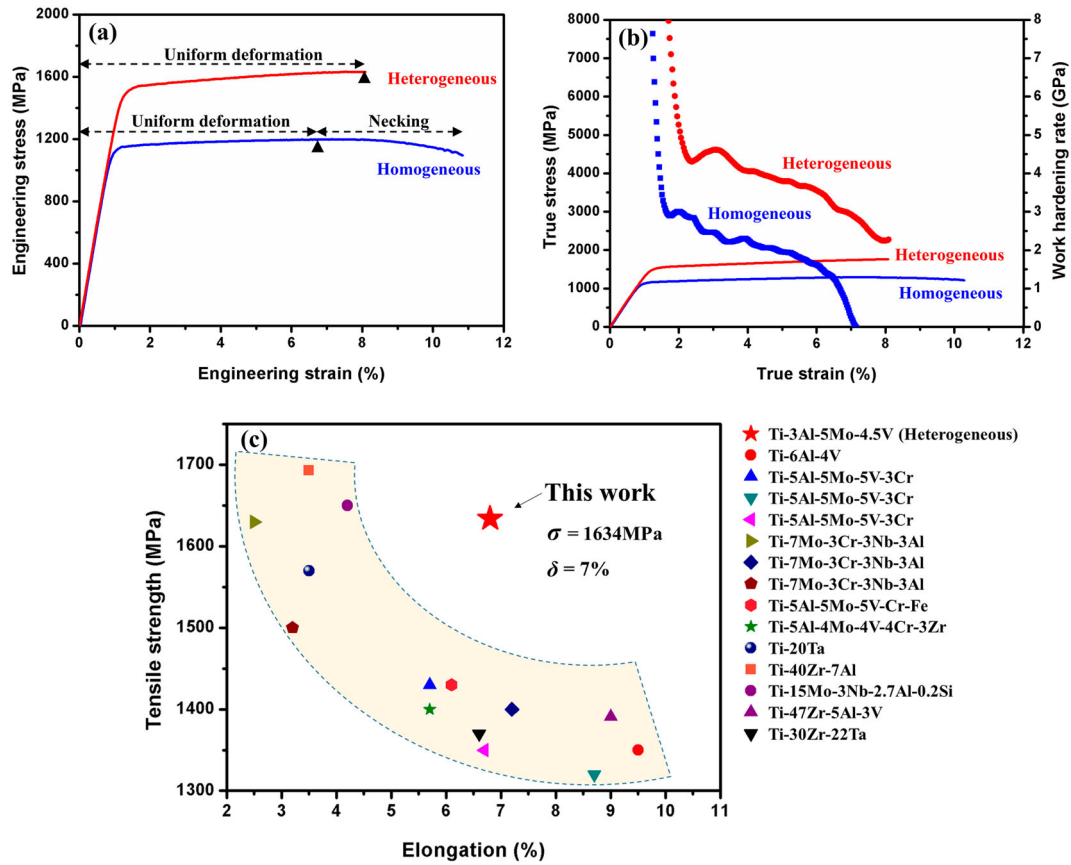


Figure 3. Tensile properties of the Ti-3Al-4.5V-5Mo alloys: (a) Engineering strain-stress curves; (b) True strain-stress curves and corresponding work hardening rates; (c) Comparison in tensile strength and uniform elongation of heterogeneous Ti-3Al-4.5V-5Mo alloy with other high-strength titanium alloys.

the heterogeneous Ti-3Al-4.5V-5Mo alloy shows a significant enhancement in strength-ductility combination.

The details around $\beta_{\text{fiber}}/(\alpha + \beta)_{\text{matrix}}$ domain interfaces after tensile testing were analyzed, as shown in Figure 4. Most dislocations were found around the interface (Figure 4(a)). The density of the dislocation in the interface is obviously higher than that in β_{fiber} and $(\alpha + \beta)_{\text{matrix}}$. The high magnification views of Region A and Region C clearly show the pile-up of dislocations near the interface. After tensile testing, some α -twins were observed around the interface, as shown in Figure 4(c ~ e). The BF image and the high resolution image (HRTEM) both indicate the occurrence of lamellar-like α -twins. Besides, the martensitic transformation (α'' phase) was also observed in β_{fiber} near the interface, as shown in Figure 5(g ~ i). The BF image and dark field (DF) image clearly show the nano-scaled α'' phase with needle morphology. The α'' phase transformation was only found in β_{fiber} near the interface rather than in the central of β_{fiber} or $(\alpha + \beta)_{\text{matrix}}$. Since there is no metastable phase or twin in the sample before tensile testing, the α -twining and the α'' phase transformation can be inferred to be induced by the tensile deformation.

Basically, strengthening mechanisms in polycrystalline materials are related to solid-solution hardening, grain-boundary hardening, dislocation hardening, and precipitation hardening [21]. However, the four mechanisms are not enough to explain the exceptionally high strength in heterogeneous materials. The mechanical mechanisms of heterogeneous alloys have been associated with the so-called HDI hardening [8,10]. Plastic deformation in the heterogeneous structure is inhomogeneous but continuous, producing strain gradient in interfaces or boundaries, which needs to be accommodated by geometrically necessary dislocations (GNDs). The multiplication and pile-up of GNDs produce long-range internal stress (back stress) in the soft domain, which could offset the applied shear stress, making the soft domain stronger. However, to balance the stress around the interface, there will be a stress opposite to the back stress in the hard domain, which was named as the forward stress. The interaction of the back stress and forward stress on the interface leads to increased density of GNDs, resulting in a strong hardening effect [10]. In this study, the GNDs pile-up is most likely around the $\beta_{\text{fiber}}/(\alpha + \beta)_{\text{matrix}}$ domain interface, as shown in Figure S5, so that the strain

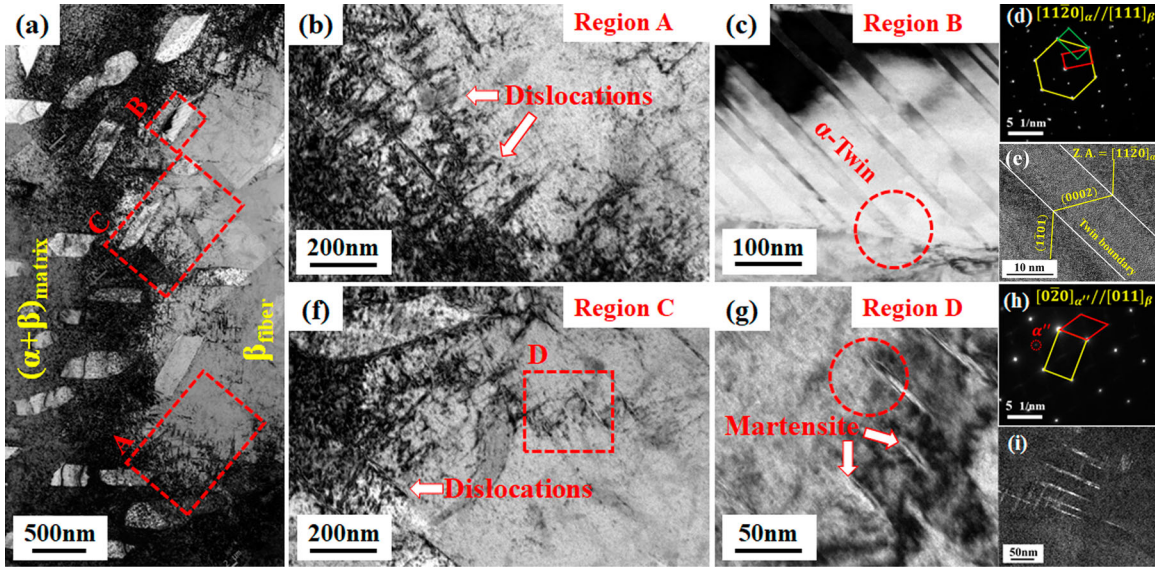


Figure 4. TEM images of heterogeneous Ti-3Al-4.5V-5Mo alloy after tensile testing: (a) Low magnification shows high density of dislocations around the interface between $(\alpha + \beta)_{\text{matrix}}$ and β_{fiber} ; (b) and (f) Dislocations pile-up in β phase near the interface; (c) Twining in α phase near the interface; (d) SAED patterns of α -twin and β phase; (e) HRTEM image of α -twin; (g) Martensitic transformation found in region D; (h) SAED patterns of α'' phase and β phase; (i) DF image obtained from the marked diffraction spot in (h) verifying α'' phase.

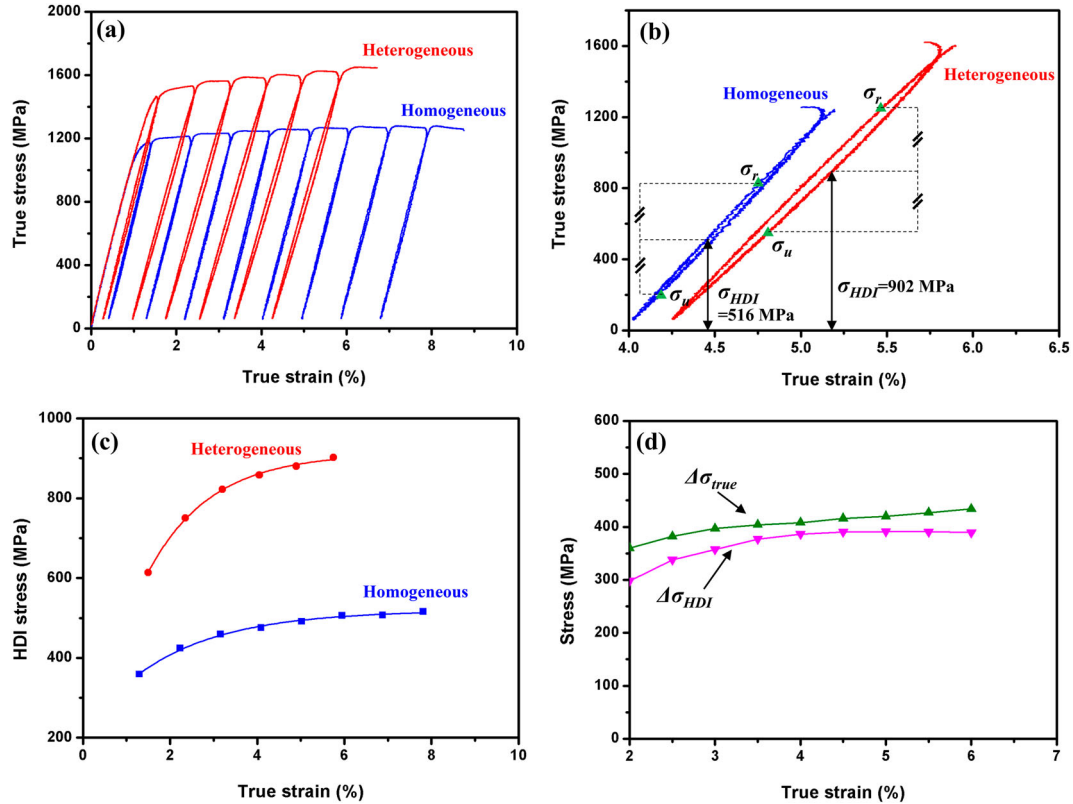


Figure 5. Analyses of HDI stress based on unloading-reloading testing: (a) Unloading-reloading behavior; (b) High magnification view of the hysteresis loops for determining the HDI stress; (c) HDI stress in homogeneous and heterogeneous alloys; (d) Analysis of true stress and HDI stress.

gradient around the $\beta_{\text{fiber}}/(\alpha + \beta)_{\text{matrix}}$ interface should be most significant. Hence, the HDI hardening could come into effect through three steps. Firstly, both soft β_{fiber} and hard $(\alpha + \beta)_{\text{matrix}}$ deform elastically. Secondly,

β_{fiber} begins to deform plastically while $(\alpha + \beta)_{\text{matrix}}$ stays in elastic deformation, resulting in a strain gradient in soft β_{fiber} side near the interface. To keep the deformation proceeds, GNDs are required to accommodate

the strain gradient, and are expected to generate in β_{fiber} near the domain interface. Accordingly, the accumulated GNDs can directly strengthen the soft parts through forest dislocation hardening and cross-slip mechanisms, leading to an improved yield strength [1,9]. Thirdly, with the strain increasing, both β_{fiber} and $(\alpha + \beta)_{\text{matrix}}$ undergo plastic deformation. However, the soft β_{fiber} still sustains more strain than the hard $(\alpha + \beta)_{\text{matrix}}$, causing a strain partitioning. In this stage, the strain gradient exists in both β_{fiber} and $(\alpha + \beta)_{\text{matrix}}$ near the domain interface. The GNDs are further stimulated to multiplication and pile-up around the interface, as shown in Figure 4, and thereby leading to high work hardening rate and increased UTS of the bulk alloy. Besides, the strain gradient is usually perpendicular to loading direction in fiber reinforced composites [22]. Since β_{fiber} structure is parallel to the loading direction, the direction of the strain gradient is expected to be perpendicular to the $\beta_{\text{fiber}}/(\alpha + \beta)_{\text{matrix}}$ interface. Therefore, the strain gradient should exist in a large area, due to the high density of $\beta_{\text{fiber}}/(\alpha + \beta)_{\text{matrix}}$ interface, causing significant HDI hardening. Moreover, the strain gradient will increase with the strain increasing, which means that the HDI hardening effect will also become stronger with the strain increasing [8]. The HDI hardening can be quantitatively analyzed through LUR test. The hysteresis loop reflects the Bauschinger effect, and represents the contribution of the HDI stress to the flow stress [7]. The analysis of the LUR behavior of the Ti-3Al-4.5V-5Mo alloys is shown in Figure 5. The hysteresis loops of the heterogeneous alloy are larger than the homogeneous alloy, indicating a stronger HDI hardening effect. The HDI stress, σ_{HDI} , can be estimated by $\sigma_{\text{HDI}} = (\sigma_r + \sigma_u)/2$, where σ_r and σ_u are the reloading and unloading yield stress, respectively [23]. Based on the calculation, it was found that the HDI stress of the heterogeneous alloy is much higher than the homogeneous alloy, and the HDI stress increases with the increasing tensile strain. The plots of HDI stress could be fit by an exponential function, as shown in Figure 5(c). The HDI stress of the heterogeneous alloy is about 260 MPa higher than the homogeneous alloy near the yield point. The yield strength of the heterogeneous alloy is about 300 MPa higher than the homogeneous alloy, as shown in Figure 3(a). It is apparent that to trigger yielding in the heterogeneous alloy, additional external stress needs to be supplied to move dislocations, so that the yield strength could be enhanced as much as the HDI stress contributes [8], particularly producing the back stress in soft β_{fiber} domains. Based on Figure 5(a and c), the differences in true stress ($\Delta\sigma_{\text{true}}$) and HDI stress ($\Delta\sigma_{\text{HDI}}$) of the two alloys were determined in Figure 5(d). Interestingly, the values of $\Delta\sigma_{\text{HDI}}$ and $\Delta\sigma_{\text{true}}$ are close, and the trends of $\Delta\sigma_{\text{HDI}}$

and $\Delta\sigma_{\text{true}}$ curves are also similar. Therefore, it can be inferred that the enhanced strength in the heterogeneous alloy is mainly caused by the HDI hardening. It should be noted that grain boundary strengthening and precipitation strengthening also contribute to the enhanced strength, since the heterogeneous alloy has a finer grain size and a higher fraction of α phase than the homogeneous alloy. But, these effects were evaluated to be not as obvious as HDI hardening, as presented in Figure S3.

Achieving high strength in titanium alloys is usually accompanied by limited ductility. For most $\alpha + \beta$ titanium alloys, precipitation strengthening is the most effective strengthening mechanism. But, an excessive precipitation strengthening could lead to a dramatic drop in ductility, because the high content of precipitate easily causes stress concentration. For the current heterogeneous Ti-3Al-4.5V-5Mo alloy, fortunately, the α -twining and the α'' phase transformation during plastic deformation can consume strain energy and relieve stress concentration [24]. Besides, the high HDI hardening rate (as shown in Figure 5(c)) could enhance the whole work hardening ability of the heterogeneous alloy [7], preventing it from early plastic instability and failure. As a result, while the heterogeneous deformation promotes the multiplication and pile-up of GNDs, leading to an increment in strength, both the deformation induced α -twining and α'' phase transformation relieve the stress concentration around the interface, helping maintain the ductility.

In summary, a heterogeneous $(\alpha + \beta)$ titanium alloy with soft β_{fiber} and hard $(\alpha + \beta)_{\text{matrix}}$ was successfully prepared by controlling the distribution of β -stabilizing element, Mo. The heterogeneous alloy has an ultra-high strength of 1643 MPa with a reasonable ductility. The superior combination of strength and ductility is apparently presented in comparison to other high-strength titanium alloys. The enhanced strength is mainly attributed to the HDI hardening induced by $\beta_{\text{fiber}}/(\alpha + \beta)_{\text{matrix}}$ interface, while the ductility is benefited from α -twining and the martensitic transformation.

Disclosure statement

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