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COMMENT

Identifying heterostructured materials

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ABSTRACT
Heterostructured materials comprise domains with markedly different properties whose interactive coupling produces synergistic effects beyond the rule of mixtures. As the field advances, a key question is how to distinguish heterostructured materials from conventional materials that also contain microstructural heterogeneities. Here, we identify defining characteristics based on the mechanisms underlying their superior mechanical performance. Forward stress plays a decisive role in governing the mechanical behavior of heterostructured materials but is negligible in conventional materials. Microstructurally, heterostructured materials contain plastically deformable hard domains. We also briefly discuss design strategies for representative heterostructures.

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Background

Since their inception in 2015 [1], heterostructured materials have rapidly attracted widespread attention in the materials research community and have evolved into a major research field. By design, heterostructured materials can exhibit mechanical and/or physical properties that are unattainable in their conventional homogeneous counterparts [2–4]. These superior properties arise from underlying mechanisms that are not captured in traditional materials textbooks. Importantly, heterostructured materials can be fabricated at large scale and low cost using existing industrial infrastructure, making them highly promising for practical applications [5].

Heterostructured materials were first formally defined by a group of thirteen researchers from America, Asia, and Europe [6]. This definition contains two essential elements: (1) heterostructured materials consist of heterogeneous domains with dramatically different mechanical and/or physical properties (typically differing by more than 100%); and (2) interactive coupling among these domains produces synergistic effects that exceed predictions based on the rule of mixtures. For conciseness, this definition can be restated as follows: *heterostructured materials consist of domains with markedly different properties, whose interactive coupling generates synergistic effects beyond the rule of mixtures*. It should be noted that the terms '*zone*' and '*domain*' are used interchangeably in the heterostructured materials literature and refer to the same concept [2, 3].

Although the above definition has been widely accepted by the materials research community, several concerns and misconceptions remain [7]. These largely stem from misunderstandings of the definition and the fundamental principles underpinning heterostructured materials. As increasing numbers of researchers enter this rapidly developing field, such misconceptions have led to growing confusion within the community.

Taken literally, the term 'heterostructured materials' may be interpreted as referring to any materials containing heterogeneities in their microstructure and/or crystal structure. Consequently, some researchers have extended the concept to encompass heterogeneities at all spatial scales, ranging from the atomic and nanometer scales to the micrometer and macroscopic scales. This overly broad interpretation is problematic and risks misleading new researchers entering the field.

At the atomic scale, virtually all materials—from single crystals of pure metals to polycrystalline high-entropy alloys—contain point defects such as vacancies and solute atoms. At the nanoscale, common engineering alloys (e.g. Al alloys, Mg alloys, and steels) frequently exhibit nanoprecipitates or short-range ordering. At the micrometer scale, grains generally show size distributions and crystallographic orientation variations, and many engineering

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alloys are intrinsically multiphase. Under such a misinterpretation, nearly all existing engineering metals and alloys would qualify as heterostructured at one or more length scales, rendering the concept of heterostructured materials both unnecessary and meaningless.

Principles of heterostructured materials

To avoid the aforementioned misconceptions and to delineate a clear boundary for this emerging field, it is essential to first understand the fundamental principles of heterostructured materials. In the following, we are only concerned with the structural heterostructured materials. Functional heterostructured materials have different physical mechanisms and won't be considered here.

For structural heterostructured materials, deformation is inherently heterogeneous, giving rise to an additional global stress, referred to as hetero-deformation-induced (HDI) stress [8]. This HDI stress leads to enhanced strength and strain hardening, thereby enabling a superior combination of strength and ductility. Experimentally, HDI stress can be measured using loading–unloading–reloading (LUR) tensile tests [9].

The origin and evolution of HDI stress are closely associated with the deformation behavior of heterostructured materials. A defining characteristic of these materials is that their hard domains are plastically deformable. During tensile loading, local plastic deformation initiates first in the soft domains, while the hard domains remain elastic. As shown in Figure 1(a), this mismatch in deformation generates strain gradient near the domain interface in the interior of the soft domain, which must be accommodated by geometrically necessary dislocations (GNDs). The accumulation of GNDs produces a long-range back stress in the soft domain [9]. The stress concentration at the head of the GND pile-up, in turn, induces forward stress in the adjacent hard domain. At this stage, however, the forward stress has only a limited influence on the macroscopic mechanical behavior, because the hard domain remains elastic.

At the domain interface, the back stress and forward stress cancel each other because they have equal magnitudes but opposite directions. The stress concentration at the head of a GND pile-up is given by $n\tau_a$ [11, 12], where n is the number of GNDs in the pile-up and τ_a is the applied resolved shear stress. Accordingly, the forward stress acting in the hard domain can reach values as high as $(n+1)\tau_a$. With increasing applied stress, the forward stress near the domain interface can become sufficiently high to initiate plastic deformation, leading to the formation of a localized plastic zone near the interface within the hard domain (see Figure 1(b)) [10].

The back stress strengthens the soft domains, producing a strengthening effect, whereas the forward stress softens the hard domains, resulting in a local softening effect. Because the back stress decays more slowly than the forward stress with distance from the interface [2, 8], as illustrated in Figure 1, the strengthening effect of the back stress cannot be globally offset by the weakening effect of the forward stress. This imbalance leads to a net strengthening contribution, manifested as hetero-deformation-induced (HDI) stress.

When the plastic zones in the hard domains percolate to link up, global yielding occurs. Thereafter, strain partitioning (hetero-deformation) continues to evolve between the hard and soft domains, with the soft domains accommodating a much larger fraction of the plastic strain. This ongoing strain partitioning promotes further accumulation of GNDs, resulting in increased back stress and forward stress, which together give rise to HDI strain hardening.

As discussed above, in heterostructured materials, forward stress plays a significant role in governing the mechanical behavior, particularly after yielding. Consequently, back stress alone is insufficient to physically explain the observed mechanical response, necessitating the introduction of the concepts of HDI strengthening and HDI strain hardening to account for the strengthening and strain-hardening behavior of heterostructured materials.

Identifying heterostructured materials

Based on the principles of heterostructured materials discussed in the previous section, two key characteristics can be used to identify them. The first is **microstructural**: heterostructured materials contain deformable hard domains. The second is **deformation physics-based**: forward stress plays a critical role in their mechanical behavior. These two criteria are interrelated—deformable hard domains provide a medium through which forward stress influences deformation.

To illustrate how to use the above characteristics to identify heterostructured materials, we consider ceramic-particle-reinforced metal matrix composites as an example. Conventional design strategies aim to uniformly distribute hard particles within the metal matrix, as schematically shown in Figure 2(a). In this case, the hard domains are ceramic particles, which are not plastically deformable. Consequently, forward stress cannot deform these hard particles and plays little role in the material's overall deformation behavior. Instead, geometrically necessary dislocations (GNDs) loop around the hard particles, generating back stress that strengthens the surrounding soft metal matrix. In other words, back

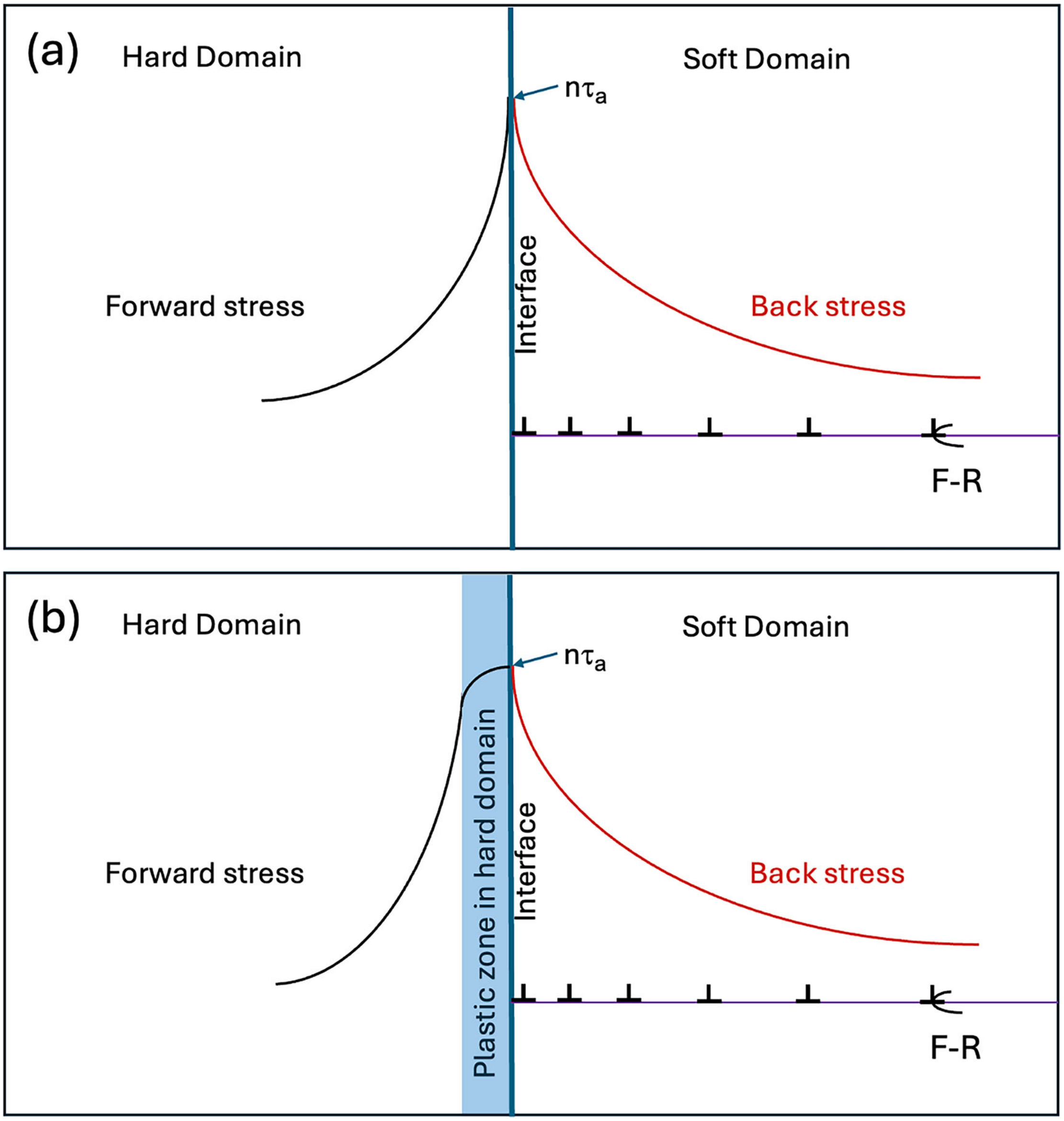

Figure 1. Schematic illustration of the generation of back stress, forward stress, and hetero-deformation-induced (HDI) stress. Geometrically necessary dislocations (GNDs) are emitted from a Frank–Read (F–R) dislocation source in the soft domain and pile up against the domain interface. The resulting stress concentration at the head of the dislocation pile-up induces forward stress in the adjacent hard domain. While the back stress and forward stress cancel each other at the domain interface, they decay at different rates away from the interface, collectively giving rise to the HDI stress. (a) Initial deformation stage, in which the hard domain remains fully elastic while dislocation slip has initiated in the soft domain. (b) A later stage, in which a plastic zone develops in the hard domain near the interface with the assistance of high forward stress. Global yielding occurs when the plastic zones in the hard domains percolate and link up [10].

stress alone governs the mechanical response. In fact, the concept of back stress was originally proposed to explain the mechanical behavior of this class of composites [13–17]. Therefore, such a composite is considered a conventional composite material.

In contrast, in a typical heterostructured composite, the hard particles are distributed non-uniformly, as illustrated in Figure 2(b). Here, hard domains correspond to regions with a high particle density, while soft domains are particle-free. The hard domains can undergo plastic deformation with the assistance of forward stress, although they accommodate less plastic strain than the soft domains due to strain partitioning. This combination produces **HDI strengthening**, enhancing yield strength, and **HDI strain hardening**, improving global ductility. Indeed, superior mechanical properties of heterostructured composites have been reported in the literature [18–24].

Using these two characteristics, typical heterostructures can be identified, including heterogeneous lamellar structures (HLS) [1, 25–29], gradient structure [9, 30–40], harmonic structure [41–48], layered structure

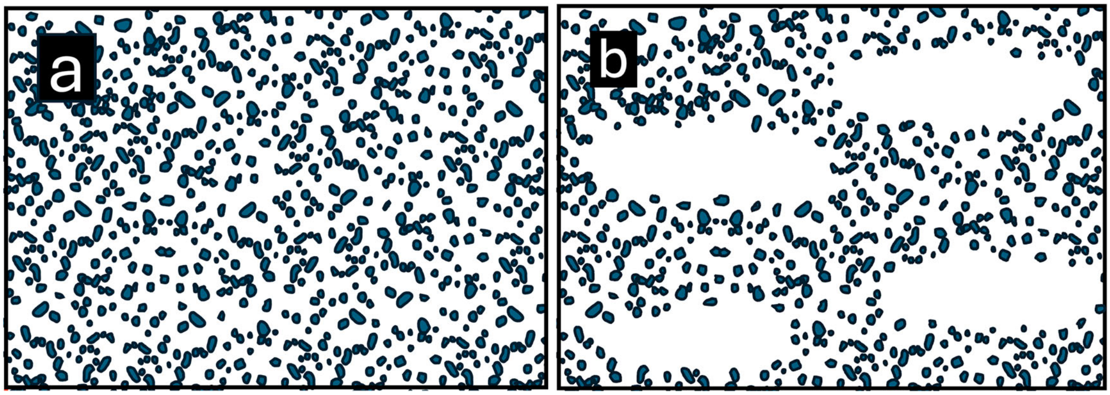

Figure 2. Schematics of (a) conventional ceramic-particle-reinforced metal matrix composites, and (b) heterostructured ceramic-particle-reinforced metal matrix composites.

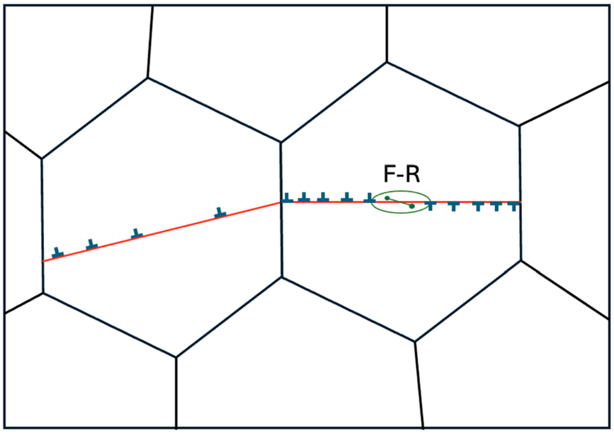

Figure 3. Schematic of transmission of geometrically necessary dislocations (GNDs) across a grain boundary. The GNDs are emitted from a Frank-Read (F-R) dislocation source.

[49–57], dual-phase structure [58–68], bi-modal/multi-modal structure [69–73], and heterostructured composite structure [18–24].

Back stress in conventional polycrystalline materials

Back stress typically influences the mechanical behavior of both heterostructured and conventional materials. Consider the case of conventional single-phase materials with relatively uniform grain sizes. In these materials, geometrically necessary dislocations (GNDs) generally pile up against grain boundaries, generating back stress (Figure 3). However, no distinct hard domains exist that could be plastically weakened, and thus the effect of forward stress is negligible.

Even if grain boundaries are considered as hard domains, the contribution of forward stress remains negligible because their thickness is only ∼0.5–1 nm [74–76]. In other words, for conventional materials with grain sizes larger than 1 µm, the grain-boundary volume fraction is effectively zero, and forward stress can be safely ignored.

On the other hand, stress concentration at the head of a GND pile-up can facilitate the transmission of GNDs into the neighboring grain, forming a new GND array, as illustrated in Figure 3. In this case, GNDs in both grains generate back stress, thereby strengthening the material. This mechanism has been observed experimentally through *in situ* transmission electron microscopy [77] and confirmed by dislocation simulations [48]. Accordingly, the HDI stress measured using loading–unloading–reloading (LUR) testing can be attributed to back stress.

These observations indicate that back stress exists in conventional materials and can significantly influence their mechanical behavior, whereas the contribution of forward stress remains negligible.

How to design heterostructured materials

It should be noted that not all heterostructured materials exhibit superior mechanical properties. To achieve such properties, heterostructures must be intelligently designed to generate substantial global HDI stress over a wide tensile strain range. The design principles have been delineated in a previous paper [10] and are summarized below.

First, the soft domains should be embedded within a hard-domain matrix. To realize this configuration, the volume fraction of soft domains should be less than 50%. According to the literature, the optimal volume fraction of soft domains typically lies in the range of 20–30% [3, 6].

Second, an appropriate spatial density of domain interfaces should be engineered. In heterostructured materials, high HDI stress arises from GND pile-ups at domain interfaces; therefore, a high interface density is desirable. This requirement makes certain soft-domain geometries particularly effective in enhancing the heterostructure effect, such as discontinuous lamellar structures [1] and fibrous structures [78–80]. For most metals and alloys reported to date, the optimal lamella thickness is on the order of a few micrometers [3], as determined by the balance between increasing interface density and providing sufficient space for GND accumulation.

Third, gradient domain interfaces are more advantageous than sharp interfaces, as they can generate additional strain hardening over a broader strain range.

Summary

In conclusion, heterostructured materials can be distinguished by the presence of plastically deformable hard domains alongside soft domains, unlike conventional materials, which lack such hard domains. Mechanistically, forward stress plays a critical role in deforming the hard domains, directly influencing the overall mechanical behavior of heterostructured materials. By contrast, back stress contributes to the deformation of both heterostructured and conventional materials. These fundamental microstructural features and deformation mechanisms provide a clear basis for differentiating heterostructured materials from conventional ones and highlight the central role of HDI stress in designing materials with superior mechanical performance.

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