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Deformable reinforcing particles facilitate the development of novel metal matrix composites

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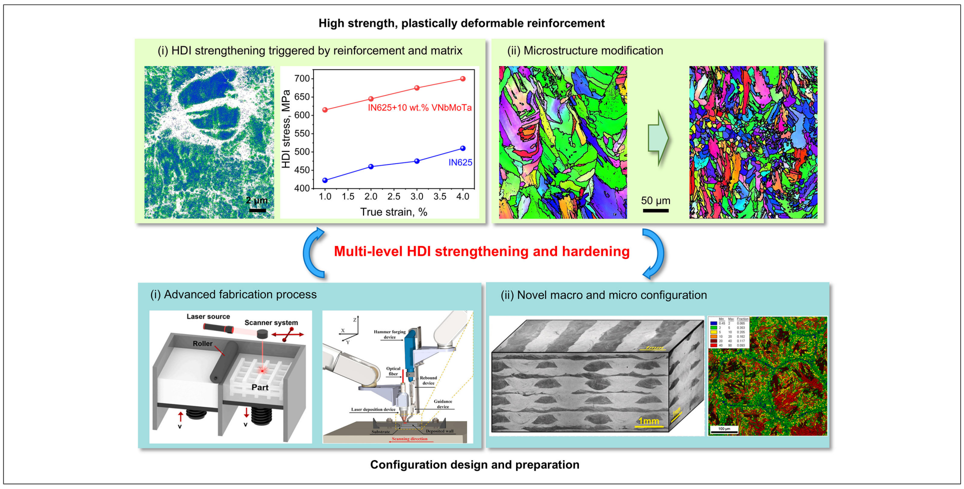

Abstract

Conventional rigid reinforcing particles in metal matrix composites (MMCs) typically induce limited or single-level hetero-deformation induced (HDI) strengthening and hardening effect, thereby restricting the further enhancement of mechanical properties. We propose to incorporate deformable reinforcing particles (e.g., medium-/high-entropy materials) into metallic matrices to overcome the above limitation, and verify the feasibility in the laser powder bed fusion of VNbMoTa-reinforced Inconel 625 composites. The VNbMoTa particles demonstrated superior storage capacity for geometrically necessary dislocations, triggering significant HDI effects and resulting in excellent room- and elevated-temperature tensile properties. It is encouraging that the HDI effect arises between the deformable particles and the matrix, distinct from the current method of generating HDI effects among different parts of the matrix. This innovative strategy enlightens that deformable reinforcing particles are conducive to activating multi-level HDI effects in MMCs through coordinated interactions among reinforcing particles and matrix for better mechanical properties.

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HDI STRENGTHENING AND HARDENING AND ITS ACHIEVEMENT IN METAL MATRIX COMPOSITES

Heterostructured materials represent an emerging class of advanced materials characterized by architectured heterogeneous zones with substantial mechanical and/or functional property contrasts[1,2]. These materials typically demonstrate superior performance compared to their homogeneous counterparts, primarily attributed to the well-documented hetero-deformation induced (HDI) strengthening and hardening mechanisms[3]. While metal matrix composites (MMCs) inherently belong to this material category, they exhibit remarkably limited HDI contributions, with yield strength predominantly governed by the rule of mixtures. This limitation primarily originates from the inadequate geometrically necessary dislocation (GND) accumulation capacity within constrained hard zones (e.g., reinforcing particles in MMCs). Crucially, conventional non-deformable reinforcing particles, such as TiC[4,5], TiB_2[6,7], Y_2O_3[8,9], demonstrate negligible GND storage capability, fundamentally restricting the HDI strengthening and hardening potential.

To achieve substantial HDI strengthening and hardening in MMCs, prevailing strategies focus on leveraging reinforcing particles to modify matrix microstructures, thereby creating architectured heterostructures with alternating soft/hard domains. For instance, it is well known that TiBw particles can refine the grain structure of titanium alloys. Liu *et al.* engineered a bimodal grain structure in Ti-6Al-4V composites by strategically controlling the spatial distribution of nano-TiBw particle clusters[10]. This approach yielded distinct coarse-grained (5.8 μm average) and fine-grained (42.3 μm average) regions, resulting in a dual-phase composite exhibiting synergistic strength-ductility enhancement, which surpassed both monolithic matrices and homogeneous composites through activated HDI mechanisms. Furthermore, parametric optimization revealed that reducing cluster diameters (< 15% size reduction) and inter-cluster spacing (> 30% density increase) further improved room-temperature tensile properties. However, this microstructure-driven paradigm predominantly exploits matrix-mediated HDI effects while underutilizing the intrinsic deformation compatibility between reinforcing particles and the matrix - a critical yet overlooked avenue for multi-level HDI activation.

HIGH-STRENGTH, PLASTICALLY DEFORMABLE PARTICLE-REINFORCED METAL MATRIX COMPOSITES

Recently, we proposed a strategy to induce a remarkable HDI strengthening effect through deformation incoordination between reinforcing particles and the matrix. The key to the strategy was to choose high-strength reinforcing particles with excellent GND storage capacity. We confirmed the validity of the aforementioned strategy in the selective laser melting of nickel-based composite material[11]. Given that nickel-based superalloys are typically utilized under high-temperature conditions, the selected reinforcing particles need to possess outstanding high-temperature mechanical properties while maintaining a good GNDs storage capacity. Simultaneously, to guarantee a favorable interface bonding, we hold the view that the elements constituting the reinforcing particles should preferably be the strengthening elements of nickel-based superalloys. Based on the above deliberations, we ultimately selected the VNbMoTa high-entropy alloy (HEA) as the reinforcing particle. The matrix was chosen as Inconel 625 superalloy. The VNbMoTa-Ni interface demonstrated a two-layer feature, with one layer being BCC-typed HEA and the other layer being composed of alternatively distributed Laves particles and nano-scale grains, as shown in Figure 1A. Surprisingly, the introduction of the deformable VNbMoTa largely suppressed the softening behavior of the Inconel 625 superalloy at 650 °C, making the elevated-temperature tensile properties comparable to the room-temperature tensile properties as shown in Figure 1B. The two-layer interface was quite robust with no cracking after both room- and high-temperature tensile tests, as shown in Figure 1C. Remarkable HDI strengthening contribution was induced by the strong and ductile VNbMoTa particle and the soft matrix, resulting in excellent room-temperature tensile properties, as shown in Figure 1B and D. The outstanding inhibition effect on the high-temperature softening behavior and the significant HDI strengthening contribution caused by the reinforcing particle and matrix are difficult to observe in the MMCs reinforced with non-deformable particles.

It should be noted that utilizing HEAs as reinforcements is not unprecedented. Currently, significant research focuses on HEA-reinforced MMCs[12-14], with matrices encompassing aluminum alloys[15-23], copper alloys[24,25], magnesium alloys[26-28], steels[29-32], and titanium alloys[33-36], as summarized in Table 1. However, these studies exhibit several limitations that contrast with our previous work. First, reinforcement selection is predominantly limited to CoCrNiFe-series HEAs, which possess melting points comparable to steels and lower than those of titanium alloys. Consequently, in melting-solidification-based forming processes, it is often challenging to retain a significant volume fraction of HEA reinforcements. For example, CoCrNiFe-series HEA particles are rarely observed in laser powder bed fusion (LPBF)-fabricated steel matrix composites[30-32]. Second, additive manufacturing (AM) is less commonly employed for fabricating such MMCs. This limitation is significantly influenced by the first point, as AM is inherently a melting-based process. Third, the matrix materials are typically restricted to low-melting-point alloys (e.g., aluminum alloys), with insufficient consideration given to high-melting-point alloys, particularly nickel-based superalloys.

Our previous investigation addressed the current research gap: the additive manufacturing of HEA-reinforced nickel matrix composites. Conventional CoCrNiFe-series HEA particles are incompatible with this system due to their melting points being comparable to the nickel matrix, hindering their effective retention during additive manufacturing. Consequently, screening suitable HEA reinforcements represents a primary distinction of our research. The VNbMoTa HEA was selected based on three critical criteria: high melting point (~2,500 °C), plastic deformability, and superior mechanical properties, particularly at elevated temperatures. A second key distinction is that our study, for the first time, demonstrates the triggering of the HDI strengthening between the plastically deformable VNbMoTa reinforcements and the matrix. Although the HDI strengthening effect in our previous study remains single-level, employing plastically deformable reinforcements enables the potential achievement of multi-level HDI strengthening in metal matrix composites (MMCs).

SINGLE- AND MULTI-LEVEL HDI STRENGTHENING AND HARDENING

We believe that the strategy is very promising in the field of developing novel MMCs with multi-level HDI strengthening and hardening effects and will attract widespread attention within the MMC research community.

Conventional HDI strengthening and hardening effects predominantly operate at a single level

Since the concept of heterostructured materials was proposed, researchers have developed various types, including lamellar structures[37], voxelized structures[38,39], gradient structures[40], core-shell structures[41], and multi-phase structures[42]. Although these heterogeneous structures enhance material performance, the HDI strengthening and hardening effects they induce remain confined to a single level. For example, Tan *et al.* utilized laser-aided additive manufacturing to fabricate voxelized hetero-structured steel[39], and the resulting HDI strengthening and hardening originated from the interaction between the alternatively deposited 420 stainless steel and C300 martensitic steel, as illustrated in Figure 2A. Similarly, Wu *et al.* produced a heterogeneous lamella structure in eutectic high entropy alloy through cold rolling followed by annealing[37]. The significant HDI strengthening and hardening effect arose from the interaction between the fine-grain regions and the coarse-grain regions, as shown in Figure 2B. Likewise, Wang *et al.* fabricated a harmonic (core-shell) structure in Ti-6Al-4V alloy using spark plasma sintering[41]. They achieved HDI strengthening via the interaction between fine-grain regions (average size: 5 μm) and coarse-grain regions (average size: 30 μm), as demonstrated in Figure 2C.

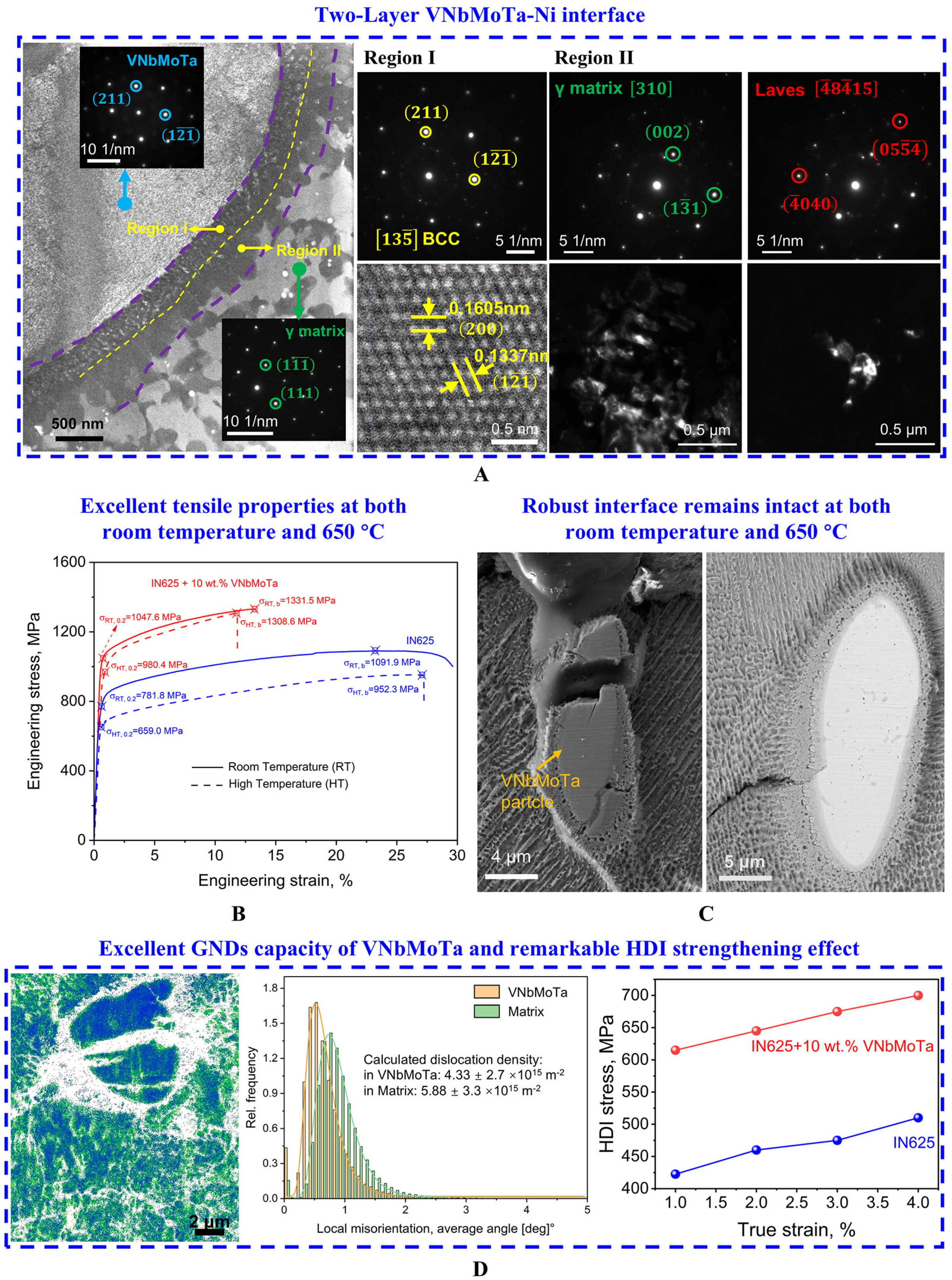

Figure 1. Additive manufacturing of VNbMoTa reinforced Inconel 625 superalloy. (A) Two-layer VNbMoTa-Ni interface; (B) Excellent room- and high-temperature tensile properties; (C) Robust interface with no cracking after both room- and high-temperature tensile tests; (D) Excellent GNDs capacity of deformable VNbMoTa particle and significant HDI stress induced by hard VNbMoTa and soft matrix. This figure is quoted with permission from Sui *et al*.[11].

Table 1. HEA reinforced metal matrix composites

Matrix	HEA reinforcement	Forming process	Ref.
Aluminum alloys	AlCoCrFeNi	Friction stir processing	[15]
	$CoFeNiCrTi_{0.4}Al_{0.6}$	Ball milling, hot pressing, hot extrusion	[16]
	CoCrFeMnNi	Stir-squeeze casting	[17]
	$Al_{0.5}CoCrFeNi$	Stir casting	[18]
	$Al_{0.8}CoCrFeNi$	Friction stir processing	[19]
	CrNiCuAlFe	Ball milling, spark plasma sintering	[20]
	$FeCoNi_{1.5}CrCu$	Static pressing	[21]
	$AlCoCrFeNi_{2.1}$	Friction stir processing	[22]
	CoCrFeNi	Friction deposition	[23]
Copper alloys	FeCoNiCrAl	Friction stir processing	[24]
	TiZrNbTa	Ball milling, spark plasma sintering	[25]
Magnesium alloys	$Al_{0.5}CoCrFeNi_2$	Ball milling, spark plasma sintering	[26]
	CoCrFeNiMoTi	Multi-pass friction stir processing	[27]
	$Al_4CrFeMnTi_{0.25}$	Stir casting	[28]
Steels	AlCoCrFeNi	Sintering	[29]
	FeCoNiCr	Laser powder bed fusion	[30]
	FeCoNiAlTi	Selective laser melting	[31]
	FeCoNiAlTi	Laser powder bed fusion	[32]
Titanium alloys	CoCrFeCuTi	Microwave sintering	[33]
	$CoCrFeNiMo_{0.2}$	Hot pressing sintering	[34]
	FeCoNiCr	Spark plasma sintering	[35]
	$Al_{0.5}CoCrFeNi$	Discharge plasma sintering	[36]

Multi-scale heterogeneous microstructures do not inherently produce multi-level HDI strengthening and hardening effects

The multi-scale heterostructures reported to date typically combine grain heterogeneity with second-phase particle heterogeneity. For instance, Dasari *et al.* developed a heterogeneous microstructure in an $Al_{0.5}Co_{1.5}CrFeNi_{1.5}$ HEA featuring grain size heterogeneity and B2 precipitate size heterogeneity[43]. Similarly, Miao *et al.* reported a heterogeneous microstructure in a $(CoCrFeNi)_{94}Ti_2Al_4$ HEA exhibiting grain size heterogeneity and $L1_2$-phase distribution heterogeneity[44]. Recently, Ma *et al.* fabricated a dual heterogeneous microstructure in a metastable β titanium alloy characterized by grain size heterogeneity and ω precipitate content heterogeneity[45]. In these studies, grain heterogeneity consistently induces HDI strengthening. However, heterogeneity in second-phase particle size, content, or distribution often fails to induce HDI strengthening. The primary reason is that second-phase particles are frequently difficult-to-deform materials. Acting as hard zones, they lack the requisite plastic deformation capability to generate the HDI strengthening effect.

High-performance, plastically deformable reinforcements are essential for achieving multi-level HDI strengthening and hardening

As established in prior analysis, the fundamental limitation impeding multi-level HDI effects resides in the difficult-to-deform nature of conventional second-phase/reinforcement particles, which prevents their heterogeneity from generating HDI effects. Our previous study directly addresses this limitation[11]. We demonstrate that when high-strength, deformable reinforcements (e.g., VNbMoTa HEA) are employed, HDI strengthening is triggered between the particles and the matrix. This particle-matrix interaction establishes

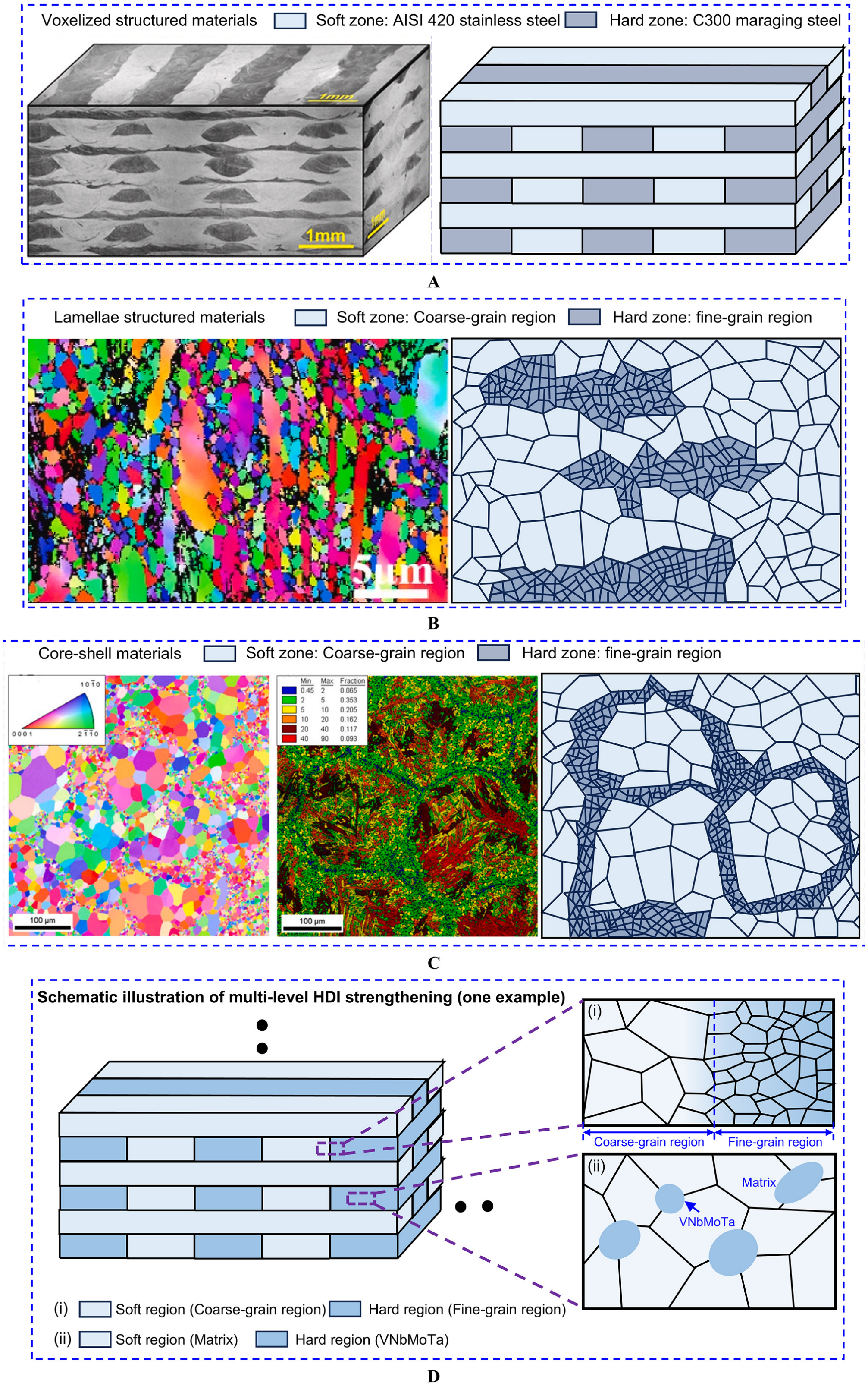

Figure 2. HDI strengthening effect in different heterostructured materials. (A) Voxelized structured material[39] (This figure is quoted with permission from Elsevier); (B) Lamellae structured material[37] (This figure is quoted with permission from Elsevier); (C) Core-shell material[41] (This figure is quoted with permission from Elsevier); (D) One example of multi-level HDI strengthening effect.

the foundation for realizing multi-level HDI effects. Figure 2D exemplifies one such composite structure. In this design, the multi-level HDI strengthening can be induced through both the interaction between fine-grain and coarse-grain regions and the interaction between the VNbMoTa particles and the matrix.

Multi-level HDI strengthening and hardening are applicable to other composite systems

Although the multi-level HDI strengthening mechanism was proposed based on a VNbMoTa HEA-reinforced nickel matrix composite, it is suggested that this mechanism remains applicable to other composite systems. The critical factor lies in the selection of suitable reinforcement. The following three criteria are proposed for developing similar metal matrix composites: (1) The plastically deformable reinforcing particles must exhibit superior target mechanical properties compared to the matrix, as this is fundamental for generating HDI strengthening and hardening; (2) The reinforcing particles must remain stable during the forming process. For methods like powder sintering, the melting point of the reinforcement is less critical. However, for melt-solidification processes, a significant melting point difference (>1,000 °C) between reinforcement and matrix is preferable to ensure a wide processing window and substantial microstructural adjustability; (3) The reinforcement-matrix interface requires meticulous design, as not all high-strength, plastically deformable reinforcements can effectively trigger the HDI strengthening mechanism. For instance, in Ti particle-reinforced Al or Mg matrix composites, brittle phases typically form at the Ti-Al or Ti-Mg interface[46-49], hindering effective load transfer and limiting the HDI strengthening. This limitation motivated the selection of HEA as the reinforcement in our previous study[11]. HEAs generally possess broad compositional ranges, increasing the likelihood of forming a compatible new HEA material at the HEA-metal interface, thereby promoting strong bonding and effective load transfer. It should be emphasized that HEAs represent one promising, but not the exclusive, class of reinforcing materials capable of inducing multi-level HDI strengthening. We believe that these criteria are considered essential for designing composites where reinforcement-matrix interactions are leveraged to achieve significant multi-level HDI contributions.

CONCLUSION

In a short summary, deformable particles, especially strong and ductile medium/high-entropy alloys, are promising reinforcing materials. They are able to induce multi-level HDI strengthening and hardening effects, which contribute to developing novel metal matrix composites with better performances to meet different application requirements.

DECLARATIONS

Authors' contributions

Conceptualization and study design: Sui, S.
Data curation, formal analysis, and investigation: Qi, Y.; Qi, J.
Administrative and technical support: Ma, D.
Manuscript writing, review, and editing: Sui, S.; Qi, Y.; Qi, J.; Xu, C.
Project administration and supervision: Sui, S.; Xu, C.

Availability of data and materials

Not applicable.

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Conflicts of interest

All authors declared that there are no conflicts of interest.

Ethical approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

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