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Graphene-decorated bimodal pure metal with high strength and ductility

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ABSTRACT

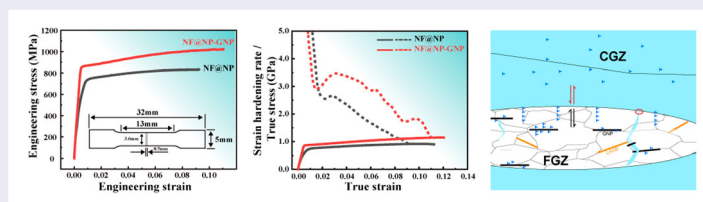
Heterostructured design and microcrack management mitigate the strength-ductility dilemma in metallic materials. Here, we demonstrate a simultaneous enhancement of strength and ductility in graphene-decorated bimodal pure nickel, achieving a strength increase to 1 GPa and an 18% improvement in elongation by designing heterostructure with graphene, metal powders, and metal foams. The results show that grain refinement, hetero-deformation-induced hardening, and activated stacking faults mainly cause the strength-ductility synergy. Additionally, while dispersed microcracks nucleate within the fine grain zones, their propagation is constrained by the coarse grain zones. This study provides new insights into improving the strength-ductility combination in metallic materials.

ARTICLE HISTORY

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KEYWORDS

bimodal structure; graphene; microstructure design; mechanical properties; microcrack behavior



1. Introduction

Benefiting from the exceptional mechanical properties of nanocarbon materials such as graphene, graphene derivatives, and carbon nanotubes, nanocarbon-reinforced metal matrix composites exhibit enhanced strength and stiffness [1]. This makes them promising candidates for the next generation of metallic structural materials. However, the incorporation of nanocarbon may lead to reduced ductility and crack tolerance, limiting the application of nanocarbon/metal composites [2,3].

This challenge can be alleviated by designing a heterogeneous structure containing zones with distinct strength and ductility properties, such as soft and hard zones. The non-homogeneous plastic deformation and steep strain gradients between the soft and hard zones induce a large number of geometrically necessary dislocations (GNDs)

in the soft zones during plastic deformation, thereby inducing hetero-deformation-induced (HDI) hardening due to the mutual constraint between the heterogeneous zones [4–8] and enhancing strain hardening [9,10]. However, due to significant deformation incompatibility between the soft and hard zones, as well as the limited plastic deformation capability of the hard zone, cracks often nucleate at the interfaces between these two zones or within the hard zone before the deformation capacity of the soft zone is fully exhausted, leading to rapid catastrophic failure of the material. For example, in bimodal pure nickel (Ni), shear bands form in the hard zones and rapidly propagate into both the soft and hard zones during tensile deformation [11]. Similarly, in the deformation process of layered Ti–Al metal composites, interface strain incompatibility and strain localization in the Ti

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layers also lead to the nucleation of cracks at a low macro strain [12].

Optimization methods typically involve prolonging the annealing time to soften the hard zone, thereby reducing the initiation of cracks at the interface or in the hard zones [13]. However, this often results in decreased strength. Additionally, Ma et al. suggested that optimizing the interface spacing between the soft and hard zones can produce the highest hardening capacity and ductility without sacrificing strength [14,15]. Furthermore, in low-melting-point metals, techniques such as hot extrusion can be used to elongate and arrange coarse and fine grain zone into a lamellar structure. For example, Al and Mg alloys produced using the ball milling-sintering-hot extrusion method demonstrate simultaneous enhancements in both strength and ductility/toughness [16–18]. Although cracks may still nucleate in the hard zone, they do not merge into catastrophic macrocracks but are arrested or blunted by the ductile zones with high strain hardening capability [18]. However, while this method is feasible for low-melting-point metals, it is challenging to apply to high-melting-point metals, like Cu, Ti, and Ni.

In this study, we used powder metallurgy followed by thermomechanical processing to fabricate graphene-decorated bimodal pure Ni. This strategy innovatively assembles graphene nanoplatelets (GNP)/Ni composite powders within nickel foams to form the fine grain and coarse grain zones of the bimodal composites. Mechanical tests show a simultaneous enhancement of strength and ductility. The investigation reveals that the strength-ductility synergy originates from the novel design of the bimodal grain structure and the incorporation of graphene, which activates multiple strengthening and ductilizing mechanisms, including grain refinement, higher HDI hardening, activated stacking faults, and unique crack behavior. Our strategy provides a new approach for preparing high-performance metallic structural materials.

2. Materials and methods

Figure 1(a) schematically illustrates the preparation method of the as-sintered graphene-reinforced composite coin. The Ni powders (> 99.9%, Shanghai Pantian Powder Materials Co., Ltd.) and GNP (Suzhou Tanfeng Graphene Technology Co., Ltd.) were uniformly mixed using planetary ball milling. Then, the composite powders were ultrasonically dispersed and uniformly filled into the pores of Ni foams (> 99.0%, Yiyang Foam-metal New Material Co., Ltd.). The Ni foams filled with composite powders were stacked and then sintered into bulk coins using the spark plasma sintering method at 1100°C for 5 min under an applied load of 50 MPa in an

argon atmosphere. The sintered coins were cold-rolled to 1 mm thickness and vacuum-annealed at 400°C for 1 h. The as-annealed composite added with GNP was named NF@NP-GNP, while the counterpart without GNP was named NF@NP. The detailed raw materials, experimental process, and characterization tests can be seen in Supplementary Note 1.

3. Results and discussion

The Ni powders are spherical with an average diameter of ~ 500 nm (Figure 1(b)), constituting the FGZ in the NF@NP-GNP and NF@NP composites. Meanwhile, the NF comprises a three-dimensional interconnected skeleton, 50 μm in width, forming the CGZ in both composites (Figure 1(c)). The received GNP tend to agglomerate, but after 10 h of ball milling, they effectively disperse within the NPs with minimal damage, as evidenced by the slight increase in the intensity ratio of I_D/I_G from 0.91 to 0.93 (Figure 1(d–g)) [19].

The as-sintered NF@NP-GNP composite coin exhibits a distinctive three-dimensional bimodal structure composed of FGZ and CGZ (Figure 2(a and b)). The average grain size of the FGZ and CGZ is approximately 4.56 and 48.60 μm , respectively (Figure 2(c)). After cold rolling and subsequent annealing, the CGZ undergoes significant elongation. It can be observed from distinct rolling orientations that the deformed composite still maintains a three-dimensional bimodal structure, with the FGZ embedded within the elongated CGZ (Figure 2(d–h) and Figure S1). In the CGZ, numerous shear bands induced by cold rolling appear inside the grains, as depicted in Figure 2(d and g). The grain orientation spread (GOS) images (Figure 2(e and h)) indicate that the grains in the CGZ have not undergone recrystallization, and high-density dislocations persist within the grains both in the NF@NP and NF@NP-GNP composites [20].

However, a significant difference in recrystallization behavior is observed in the FGZ of these two composites. In the NF@NP composite, the deformed grains are nearly completely recrystallized, with the proportion of high-angle grain boundaries (HAGB) reaching 79.9%, an average grain size of 7.50 μm , and hardness of 4.31 GPa, which is lower than that of the CGZ (4.98 GPa), as shown in Figure 2(e, f, and k). Conversely, in the NF@NP-GNP composite, only a few sporadic recrystallized grains are observed, with most remaining deformed. Among these, 66.5% of the grain boundaries are low-angle grain boundaries (LAGB), the average grain size (2.50 μm) is only one-third of that in the NF@NP. The FGZ hardness reaches 4.99 GPa, indicating a high density of dislocations and delayed recrystallization within the grains (Figure 2(h and i)). This suggests that the graphene/Ni

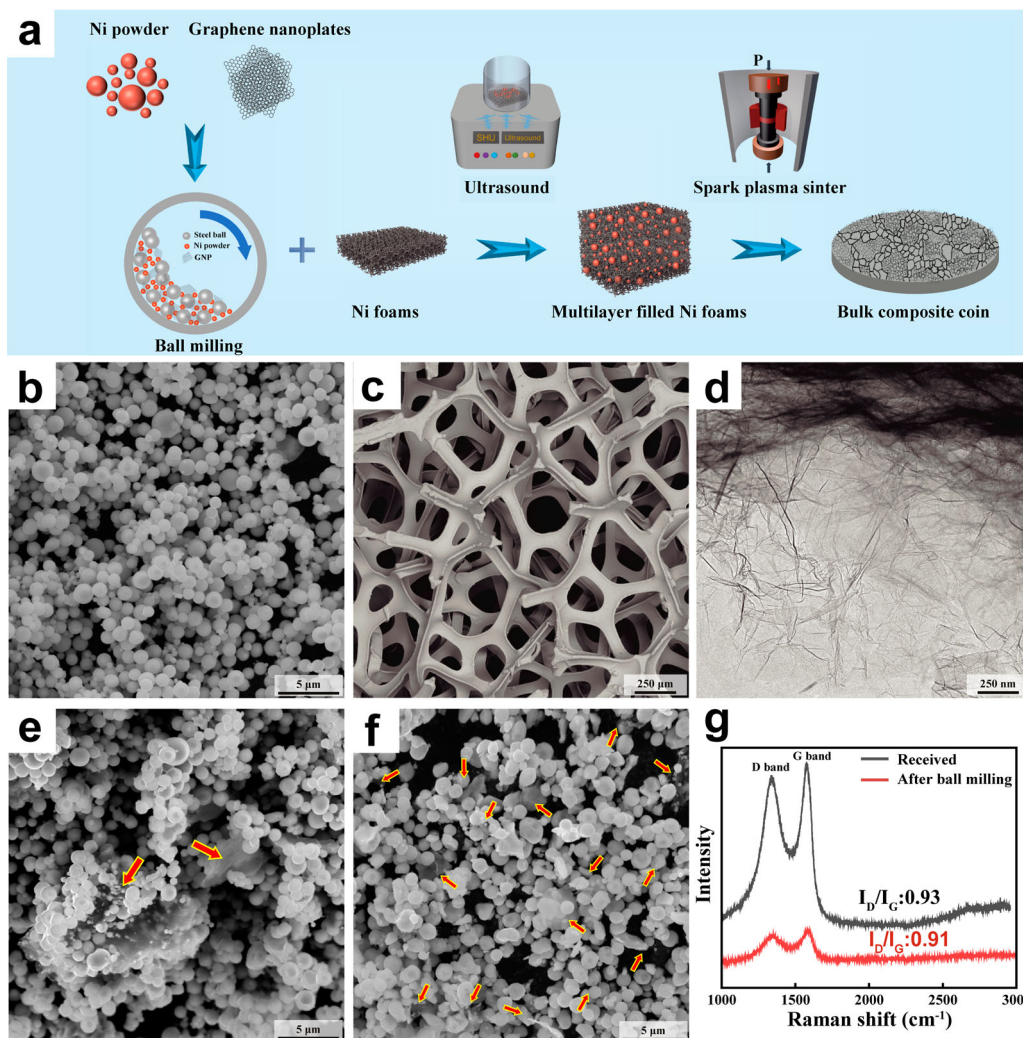


Figure 1. Sample preparation and raw materials. (a) Schematic illustration of the preparation process for the bulk composite coin. (b) Scanning electron microscope (SEM) images of (b) Ni powders and (c) Ni foams. (d) Transmission electron microscope (TEM) image of the received GNP. (e) and (f) SEM images of the mixed composite powders after 2 and 10 h of ball milling, with arrows indicating that the GNP were well dispersed in Ni powders. (g) Raman spectroscopy of the GNP before and after ball milling.

interface pinned by GNP hinders grain boundary migration and inhibits grain coarsening during annealing [21]. Low-energy Ar⁺ ion beams were employed to grind the surface of the NF@NP and NF@NP-GNP composites (Figure 2(j) and Figure S2). The presence of GNP was detected in the FGZ of the NF@NP-GNP composite.

The NF@NP-GNP composite exhibits improved mechanical properties compared to NF@NP, with a yield strength of ~860 MPa, a tensile strength of ~1022 MPa, and a uniform elongation of ~11.0% (Figure 3(a) and Table 1). It is widely acknowledged that introducing hard reinforcements into metallic matrix often enhances the strength of composites but sacrifices ductility, especially when the strength reaches 1 GPa. Fortunately, our strategy increases tensile strength to gigapascal level and enhances uniform elongation by 18% (Figure 3(e)). This strength-ductility combination surpasses previously

reported Ni matrix composites, as shown in Figure 3(d and e).

To further explore the origin of the improved strength-ductility synergy, the strain hardening rates ($\theta = \partial\sigma/\partial\epsilon$) of the NF@NP and NF@NP-GNP composites were evaluated based on their true stress-strain curves (Figure 3(b)). The strain hardening curve of the NF@NP composite begins at an initial value of ~2.65 GPa and exhibits an almost linear decrease with increasing tensile strain. Conversely, the strain hardening curve of the NF@NP-GNP composite commences from a higher initial value of ~3.5 GPa. It gradually decreases stepwise, indicating a higher initial strain hardening capability and additional hardening mechanisms, effectively delaying the onset of necking [22,23].

Cyclic load-unload-reload (LUR) tensile tests were conducted to evaluate the evolution of HDI stress and

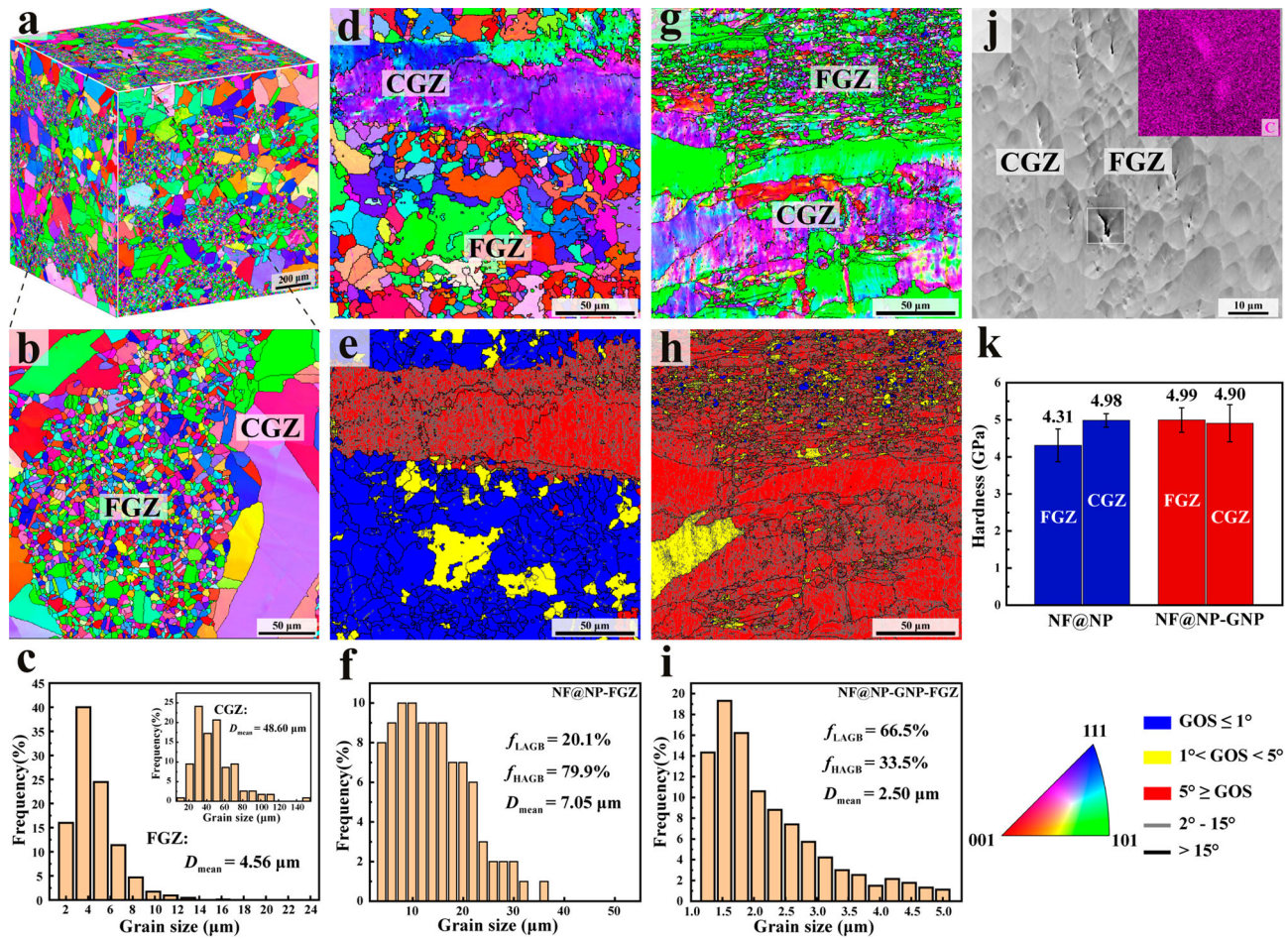


Figure 2. Microstructural characterization of the NF@NP and NF@NP-GNP composites. (a) Three-dimensional inverse pole figure (IPF) image depicting the as-sintered NF@NP-GNP composite coin. (b) Enlarge view of the FGZ and CGZ of the as-sintered NF@NP-GNP composite coin. (c) Grain size statistics in the FGZ and CGZ in (b). (d), (e), (g), and (h) IPF and GOS images of the NF@NP and NF@NP-GNP composites. (f) and (i) grain size statistics in the FGZ of the NF@NP and NF@NP-GNP composites. (j) SEM characterization of the GNP in the FGZ of the NF@NP-GNP composite. The inset energy dispersive spectrometer (EDS) image confirms the enrichment of carbon elements. (k) Nanoindentation test results of the NF@NP and NF@NP-GNP composites.

effective stress with strain in both the NF@NP and NF@NP-GNP composites (Figure 3(c) and Figure S3) [5,38]. As strain increases, the HDI stress and effective stress of the NF@NP-GNP composite consistently surpass those of the NF@NP composite. Furthermore, the NF@NP-GNP composite demonstrates a more pronounced increase in HDI stress and effective stress than the NF@NP composite. This indicates that incorporating graphene effectively enhances the HDI hardening and dislocation hardening, achieving improved mechanical properties.

The EBSD results of the NF@NP and NF@NP-GNP composites before and after a strain of 0.04 are shown in Figure 4(a–f) and Table 2. In the NF@NP composite without tensile deformation, due to recrystallization, the FGZ exhibits a Kernel Average Misorientation (KAM) value of 0.93°, 50% lower than the KAM value (1.88°)

in the CGZ, consistent with the microhardness testing results. After a strain of 0.04, the Δ KAM value (indicating the difference in the KAM values before and after tensile strain) in the CGZ increases by 0.32°, significantly higher than that in the FGZ (0.09°), indicating that more strain occurs within the CGZ. If the CGZ with higher hardness undergoes sufficient deformation, the specimen will exhibit higher flow stress, resulting in increased strength. However, Figure 4(c) shows the strain localization near the shear band (indicated by black arrows), resulting in significant flow localization and shear band softening [39]. Conversely, with both the CGZ and FGZ of NF@NP-GNP having Δ KAM values of 0.11°, shear band flow localization in the CGZ is alleviated, while strain concentration is dispersed across both the FGZ and CGZ, as well as their interfaces, as shown in Figure 4(f) and Table 2.

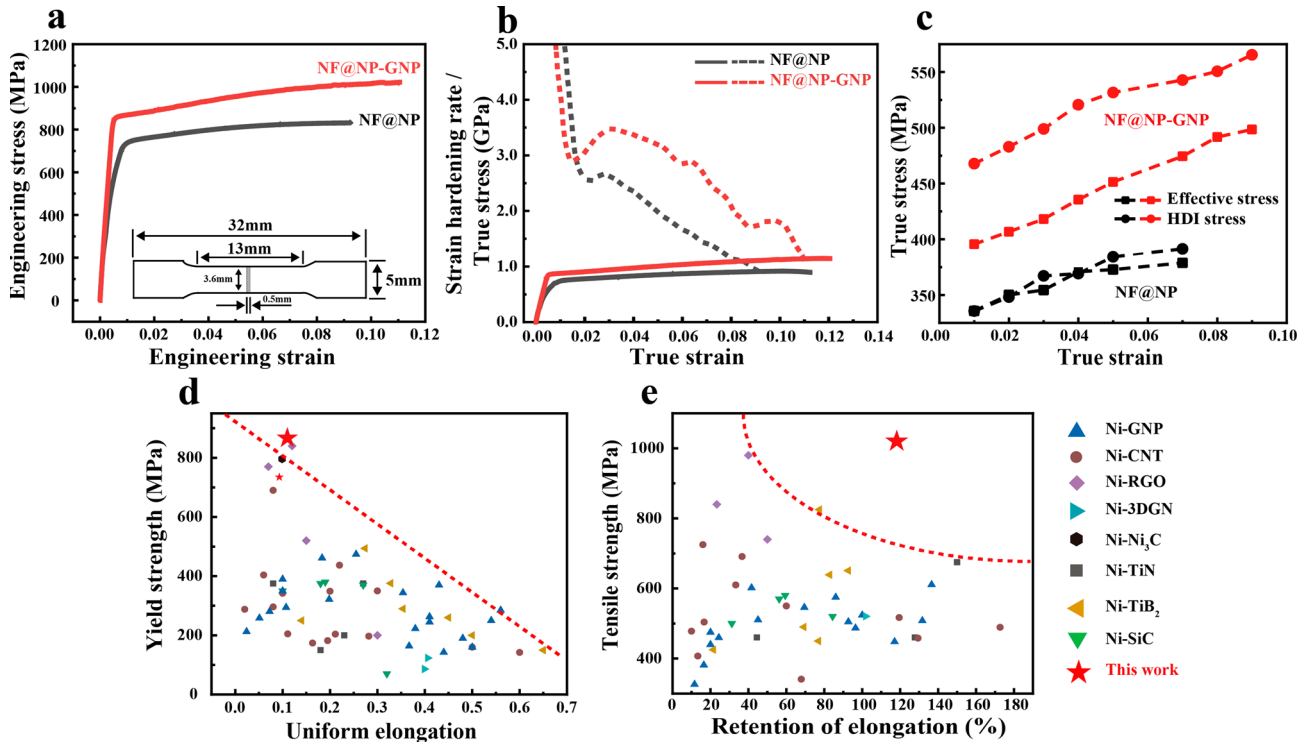


Figure 3. Mechanical properties of the NF@NP and NF@NP-GNP composites. (a) Quasi-static uniaxial tensile engineering stress–strain curves of these two composites. (b) True stress and the associated strain hardening rate versus true strain curves. (c) Evolution of HDI stress and effective stress versus true strain for both composites. (d) Comparison of yield strength versus uniform elongation with previously reported Ni matrix composites [24–37]. (e) Comparison of tensile strength versus retention of elongation ($\varepsilon/\varepsilon_M$ where ε represents the uniform elongation of the composite, and ε_M represents the elongation of the matrix) with previously reported Ni matrix composites [24–37].

To explore the origin of higher HDI stress and effective stress in the NF@NP-GNP composite, the interface between CGZ and FGZ after tensile deformation was carefully characterized (Figure 4(g–i)). The FGZ consists of elongated and equiaxed grains featuring dense dislocation cells, dislocation walls, and microbands within the grains [40,41]. Meanwhile, the CGZ exhibits larger grains, as observed in the dark-field image shown in Figure S4. High-resolution Transmission Kikuchi Diffraction (TKD) with a 7 nm step size offers more detailed information about the interface of coarse and fine grains. A notable dislocation density gradient emerges near the interface, with the dislocation density inside the grains reaching $5 \times 10^{15} \text{ m}^{-2}$ and at the interface reaching $3 \times 10^{16} \text{ m}^{-2}$ (the inset in Figure 4(h)). This accumulation of geometrically necessary dislocations (GNDs) generates long-range back stress near the interface, activating the HDI hardening effect during tensile deformation [42].

Moreover, as exceptional reinforcements with ultra-high mechanical properties, the GNP introduced within the FGZ also participate in the deformation process (Figure 4(j)). Figure 4(k) shows the corresponding

Table 1. Mechanical properties of the NF@NP and NF@NP-GNP composites.

Composite	Yield strength (MPa)	Tensile strength (MPa)	Uniform elongation (%)
NF@NP	735 ± 32	832 ± 54	9.3 ± 0.6
NF@NP-GNP	860 ± 24	1023 ± 65	11.0 ± 0.3

inverse Fast Fourier Transform (IFFT) image, which displays a high density of dislocations accumulation (indicated by ‘T’-shaped symbols) in the Ni matrix near the GNP/Ni interface. This indicates that GNP effectively block dislocation propagation during deformation, consistent with previously reported results [43,44]. The dislocations generate long-range back stress, which prevents further dislocation emission and motion, causing higher flow stress required for further deformation [45]. Additionally, they are even capable of activating stacking faults (SFs) (Figure 4(l)), which are challenging to observe under room-temperature standard tensile tests of pure Ni or Ni matrix composites due to their high stacking fault energy [46]. The formation of stacking faults (SFs) in this work is due to the special structural design and

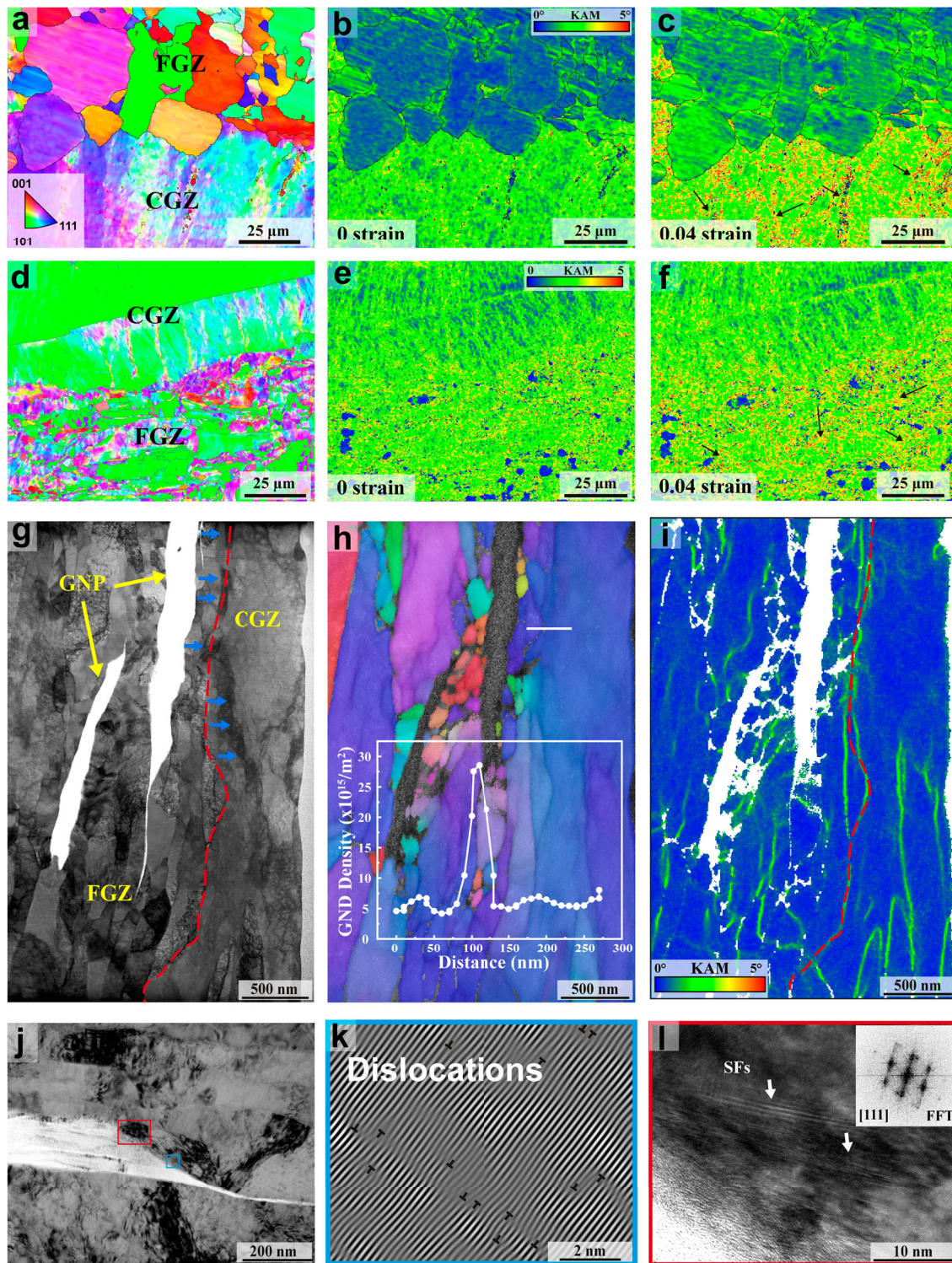


Figure 4. Deformation behavior and hardening mechanisms in the NF@NP-GNP composite. (a) and (d) EBSD IPF image illustrating the interface between CGZ and FGZ in the NF@NP and NF@NP-GNP composites. (b) and (c) Corresponding KAM images of the area shown in (a) before and after a strain of 0.04. (e) and (f) KAM images of the area shown in (d) before and after a strain of 0.04. (g)-(i) STEM, TKD, and corresponding KAM images of the interface between CGZ and FGZ in the NF@NP-GNP composite after a strain of 0.04. The inset in (h) shows the geometrically necessary dislocation density at the interface between CGZ and FGZ (marked by a white line). (j) Enlarged view of the interface between GNP and FGZ after a strain of 0.04. (k) Inverse fast Fourier transform (IFFT) image of the blue box in (j), indicating the presence of high-density dislocations at the interface. (l) High-resolution TEM and inset fast Fourier transform (FFT) images of the red box in (j), indicating the presence of SFs in the FGZ near GNP.

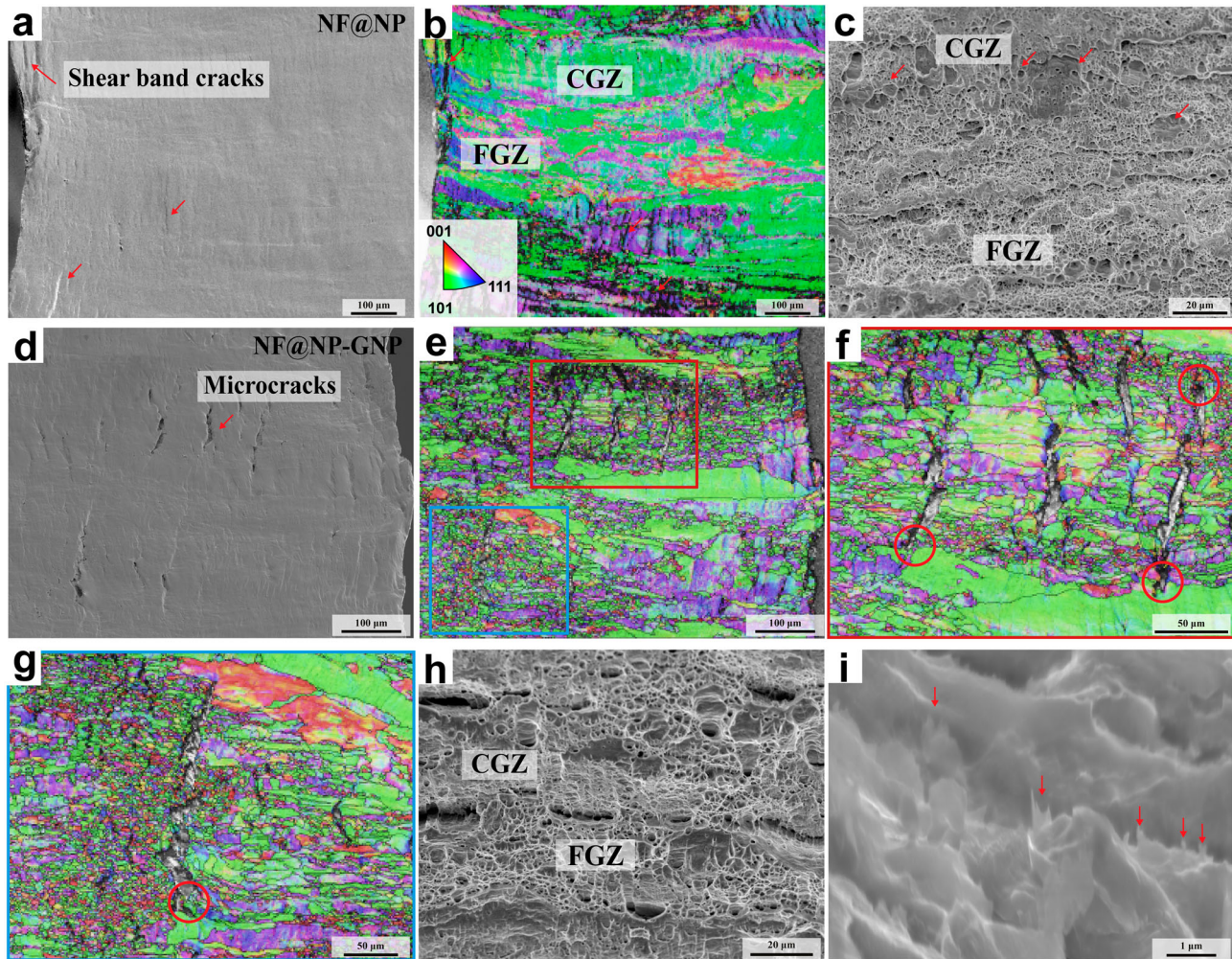


Figure 5. Fracture mechanism of the NF@NP and NF@NP-GNP composites. (a)–(c) SEM and EBSD IPF images of the fractured end of the NF@NP composite. (d) and (e) SEM and EBSD IPF images of the fractured end of the NF@NP-GNP composite. (f) and (g) Enlarged view of the red and blue boxes in (e). (h) The fracture surface of the NF@NP-GNP composite. (i) An enlarged view of the fracture surface shows pulled-out graphene.

the introduction of GNP. In turn, these SFs enable further strain hardening and toughening mechanisms that enhance the strain hardening of the composite [47,48].

The damage-evolution mechanisms of the NF@NP and NF@NP-GNP composites were carefully characterized to understand the initiation and propagation of microcracks. In the NF@NP composite, strain localization occurs in the shear band of the CGZ, resulting in all cracks observed across the fracture end cross-section originating from the shear bands (Figure 5(a and b)). These shear band cracks result in early catastrophic failure, occurring before the deformation capacity of FGZ is fully exhausted [39,49]. The fracture surfaces within the CGZ exhibit shear dimples (indicated by red arrows in Figure 5(c)), indicating microvoids coalesced along shear band planes during the deformation [50].

Conversely, in the NF@NP-GNP composite, strain localization primarily occurs in the FGZ, leading to

Table 2. Comparison of KAM differences before and after plastic deformation.

Sample	Domain	Strain	KAM(°)	Δ KAM(°)
NF@NP	FGZ	0	0.93 ± 0.43	0.09
		0.04	1.02 ± 0.42	
		0	1.88 ± 0.67	0.32
		0.04	2.2 ± 0.72	
NF@NP-GNP	FGZ	0	2.26 ± 0.94	0.11
		0.04	2.37 ± 0.97	
	CGZ	0	1.77 ± 0.72	0.11
		0.04	1.88 ± 0.74	

the initiation of high-density microcracks within this zone (Figure 5(d–g)), which is completely different from the NF@NP composite. Initially, these microcracks are blunted due to the extrinsic ductilizing effect of graphene, which acts as a bridge and deflector, dissipating fracture energy [27,51,52]. This is evidenced by the observation

of graphene pull-out and torn graphene tips at the fracture surface (Figure 5(i)). As the microcracks propagate further, they traverse the entire FGZ and are blocked at the interface between the CGZ and FGZ (Figure 5(e–g)). These numerous microcracks, completely constrained within the FGZ, efficiently buffer stress concentration and facilitate plastic deformation of the FGZ, as evidenced by the deeper and larger dimples compared with those in the FGZ of NF@NP. Additionally, the fracture surface of the CGZ in the NF@NP-GNP composite displays mixed ductile and shear dimples, suggesting that the addition of GNP also changed the stress triaxiality of the CGZ. This change mitigates the flow localization in the shear band and prolongs its stable plastic deformation [50].

4. Conclusions

In summary, graphene-decorated bimodal pure nickel was fabricated using powder metallurgy followed by thermomechanical processing. The composite exhibits a good combination of mechanical properties, with tensile strength exceeding 1 GPa while maintaining 11% uniform elongation, which surpasses previously reported Ni matrix composites. The novel design of the heterogeneous grain structure and the incorporation of graphene activates/enhances multiple strengthening and ductilizing mechanisms such as grain refinement, HDI hardening, activated stacking faults, and unique microcrack behavior. This study provides new insights into the design of metallic structural materials.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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Data availability statement

The data that support the findings of this study are available from the corresponding author, [Z. Shen], upon reasonable request.

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