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Optimizing strength-ductility synergy in lightweight steel via heterogeneous design: discontinuous fibrous ferrite

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ABSTRACT

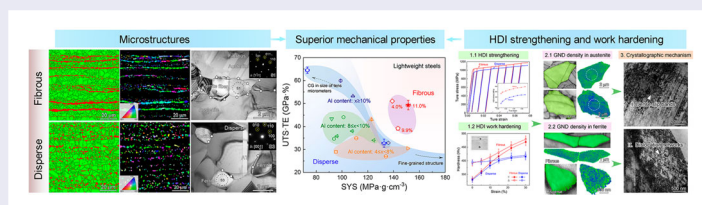
Disperse ferrite is generally embedded in lightweight steel, yet exhibits decent performance, thereby prompting a question: can heterogeneous design optimize it? Here, two representative structures, featured with discontinuous fibrous and dispersed ferrite separately embedded in the austenite matrix, were constructed as examples to address the issue. A heterostructure with fibrous ferrite shows a remarkable product (49 GPa·%) of ultimate strength and total elongation, standing out among other lightweight steels. Superior strength-ductility synergy is primarily attributed to hetero-deformation-induced strengthening and hardening. Abundant hetero-interactions imparted by phase boundaries, dense slip bands in austenite and dislocation networks inside ferrite serve as underlying physical mechanisms.

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KEYWORDS

Lightweight steel; heterogeneous structure; fibrous ferrite; strength and ductility; HDI strengthening and work hardening



1. Introduction

Lightweight steel has garnered substantial attention in the field of advanced automotive and aerospace applications due to its low density and decent mechanical performance [1]. The low density is enabled by the incorporation of a notable amount of light element (Al) [2], leading to the generation of ferrite or B2 phase. As reported in many lightweight steel [3–6], ferrites with a volume fraction of 4–30% are dispersedly integrated into the austenite matrix, giving rise to the typical duplex structure. Dispersed ferrite grains are relatively harder than the austenite matrix, which are hard zones in the duplex structure. Unique deformation mechanism of microband-induced plasticity [7] and dynamic slip band refinement [8] dominated by planar slip, combined with synergistic deformation [9] near the dispersed hetero-phase zones are responsible for the decent mechanical performance. However, due to the simplified hetero-phase zone design,

the dispersed structure is unable to fully engender hetero-deformation-induced (HDI) strengthening and strain hardening [10], supported by geometrically necessary dislocation (GND) pile-ups for coordinating interface strain gradient [11].

Recently, a notable strategy for optimizing mechanical performance has emerged as constructing heterogeneous structures [12–14], such as lamellar structures [15,16], heterogeneous laminates [17,18] and discontinuous fiber-like structures [19,20]. Among them, the discontinuous fiber-like structure has numerous heterogeneous interfaces, which sufficiently promote synergistic deformation and fracture toughness [20,21]. Assigning discontinuous fibrous ferrite in lightweight steel may provide a potential opportunity for further amplifying the HDI strengthening and strain hardening [22]. Therefore, two pivotal inquiries arise: (1) which structure can engender the optimal strength-ductility synergy, the

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discontinuous fibrous one or dispersed one? (2) what is the underlying physical mechanism behind it? These critical questions hold paramount implications for the structural design of high-performance lightweight steel and other heterostructured materials.

Here, taking the austenite/ferrite lightweight steel as an example, two distinct structures, featured with discontinuous fibrous and dispersed ferrite individually embedded in the austenite matrix, were regulated cautiously by thermal-mechanical processing. The heterostructure with discontinuous fibrous ferrite exhibits remarkable ductility at a high strength level, which produces a high product (49 GPa·%) of ultimate strength and total elongation (UTS·TE). The physical mechanisms, responsible for the exceptional strength-ductility synergy, were fastidiously unveiled through mechanical evaluation and microstructural investigation.

2. Materials and methods

The nominal composition of the lightweight steel investigated in the present work is Fe-25Mn-11Al-0.5W-0.3C (wt.%). The as-cast steel has a density of 6.66 g/cm³ as measured by Archimedes principle [1], which is 15% lighter than pure Fe (7.87 g/cm³). The as-cast steel was homogenized at 1250°C for 12 h and forged into bars with a diameter of 15 mm at a temperature range of 950–1100°C. Then, solution-treating at 1050°C for 2 h was done and the bars were water-quenched. By this, the coarse-grained (CG) samples were obtained. Subsequently, thin plates with dimensions of 50 × 12 × 10 mm³ were cut from the bars and cold rolled from 10 to 1 mm in thickness. To obtain the two distinct dual-phase structures, the cold-rolled strips were then annealed at 920°C for 5 min and 880°C for 30 min (Figure S1), which were labeled as S_{Fibrous} and S_{Disperse} samples, respectively.

Dog-bone-shaped samples with a gauge dimension of 10 × 2 × 1 mm³ were prepared from the as-annealed strips. Quasi-static uniaxial tensile and cyclic load-unload-reload (LUR) tensile tests were conducted with a Shimadzu AG-100KN testing machine with a nominal strain rate of 5 × 10^{−4} s^{−1}. In the LUR tests, upon stretching to a designated strain, the specimen was unloaded to 20 MPa, followed by reloading to the next fixed strain. Vickers microhardness of austenite and ferrite was measured with a Mitutoyo MVK-H3 tester, using a load of 10 g for 15 s. The X-ray diffraction (XRD) scanning was performed using a Cu K α radiation at the rate of 0.02° s^{−1}. A FEI quanta 650F field emission electron microscope (FE-SEM) equipped with an electron backscattering diffraction (EBSD) system was used to characterize the microstructure. EBSD measurements

were performed with an accelerating voltage of 15 kV and a current of 10 nA. The step size for the before-tension specimen is 200 nm and that for the strained specimen is 20 nm. The strain-induced GNDs density for both samples is calculated by eliminating that of corresponding strain-free samples. Transmission electron microscopy (TEM) characterizations were conducted on an FEI Tecnai F20. TEM foils were mechanically ground to a thickness of ~80 μ m, followed by twin-jet electropolishing at 20 V and 253 K, in a solution of 90 vol% ethanol and 10 vol% perchloric acid, respectively.

3. Results and discussion

Fiber bands of ferrite extend over 40 μ m length along the rolling direction (RD) in the austenite matrix were successfully fabricated (Figure 1(a)). The fibers retain a fibrous structure in the normal plane although they tend to be flattened. The XRD pattern of the S_{Fibrous} sample (Figure S2) reveals an 18% volume fraction of ferrite. Figure 1(c1–c2) are phase maps and inverse pole figures (IPF) of the S_{Fibrous} sample. The typical fiber is composed of multiple grains, displaying random orientations. The austenite matrix exhibits an equiaxed-grained structure, containing high density of annealing twins. Furthermore, as shown in Figure 1(e1), the small-sized ferrite grains are sandwiched by adjacent coarse austenite grains, both of which have sharp grain boundaries and very low initial dislocation densities. This kind of grain structure was formed from full recrystallization though the annealing duration is short at 920°C. The average grain sizes of austenite ($\bar{d}_{\text{austenite}}$) and ferrite ($\bar{d}_{\text{ferrite}}$) are 5.0 and 1.9 μ m, respectively (Figure 1(c3)).

A conventional duplex structure composed of dispersed ferrite grains, uniformly distributed in three dimensions (Figure 1(b)), was also prepared by long-duration annealing. It exhibits a comparable content of ferrite with the S_{Fibrous} sample (Figure S2). The element contents between the two samples are comparable, with aluminum exhibiting a higher concentration in ferrite and manganese being more concentrated in austenite (Figure S3, Table S1). As displayed in Figure 1(d1–d2), the austenite matrix is dispersedly infused with equiaxed ferrite grains. The ferrite grains typically nucleate at the trigeminal grain boundaries of austenite, making the ferrite encompassed by surrounding austenite. As further indicated by Figure 1(e2), all the ferrite and austenite grains exhibit a fully recrystallized structure, similar to the S_{Fibrous} sample. The average grain sizes of austenite and ferrite are 5.2 and 2.0 μ m (Figure 1(d3)), respectively, in proximity to that of the S_{Fibrous} sample. Eventually, the phase contents, element contents, grain sizes and states and initial dislocation density of the

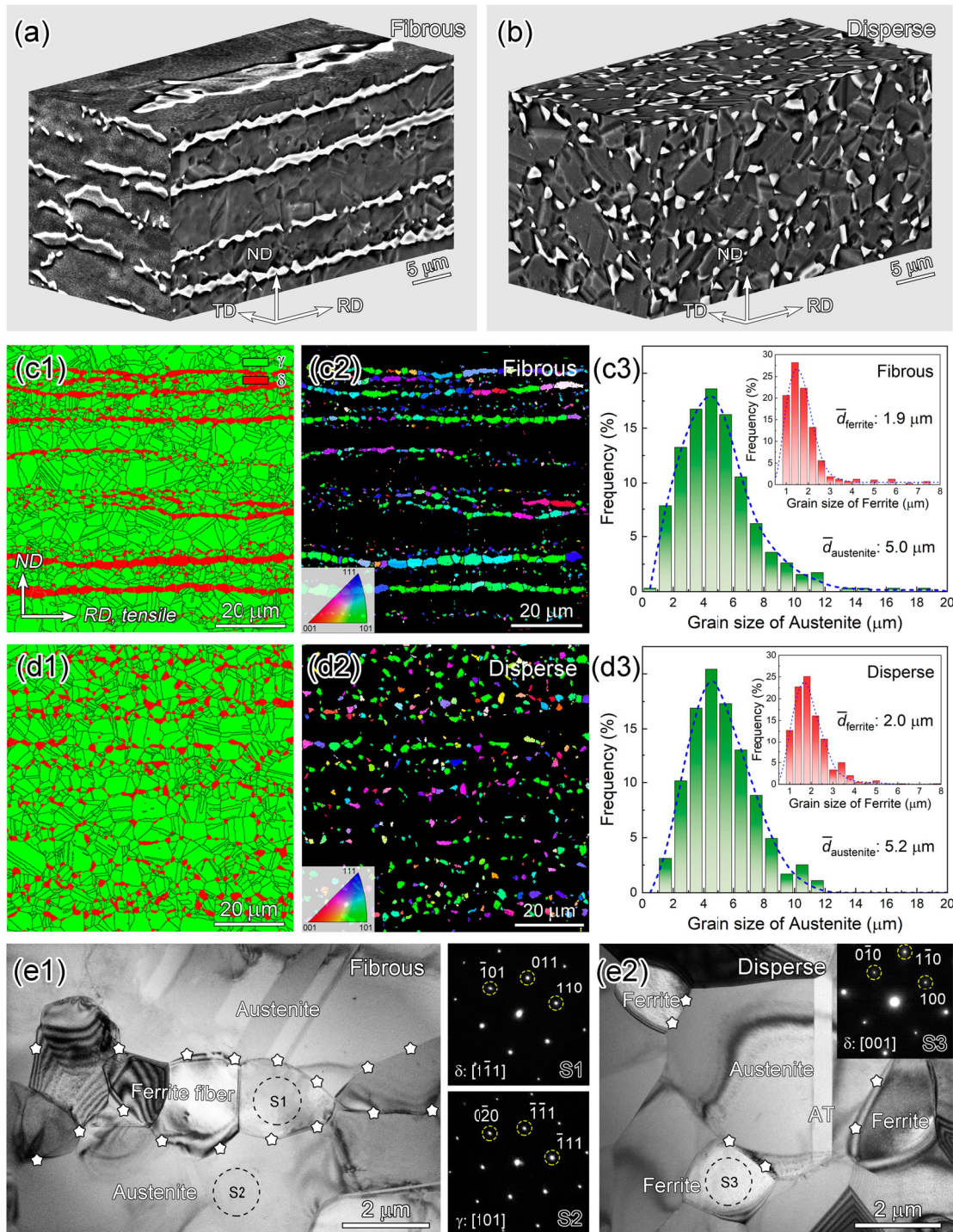


Figure 1. Microstructural characterizations of the S_{Fibrous} and S_{Disperse} samples. (a–b) SEM micrographs showing the fibrous and dispersed ferrite. In the coordinate, RD, TD and ND are the rolling, transverse and normal direction, respectively. (c1–d3) Phase maps (1), IPF maps of ferrite (2) and statistical distributions (3) of grain size: (c) S_{Fibrous} ; (d) S_{Disperse} . The tensile direction is aligned with the RD. (e) TEM morphologies of (1) fibrous ferrite; (2) disperse ferrite. S1, S2 and S3 represent the selected-area electron diffractions (SAED), corresponding to the locations in (e). Star-shaped markers represent phase boundaries. AT indicates an annealing twin.

S_{Fibrous} and S_{Disperse} samples resemble, in addition to the spatial configuration of ferrite, i.e. one is fibrous and another is dispersed. This structural differential arises from meticulous control of annealing parameters. Initially, the ferrite fibers are formed after cold-rolling [23]

(Figure S4). During annealing at 920°C for 5 min, the ferrite recovers and recrystallizes along fibers, retaining the fibrous structure. However, under longer annealing time (30 min), the ferrite tends to distribute dispersedly by grain-boundary diffusion [24,25]. The lower

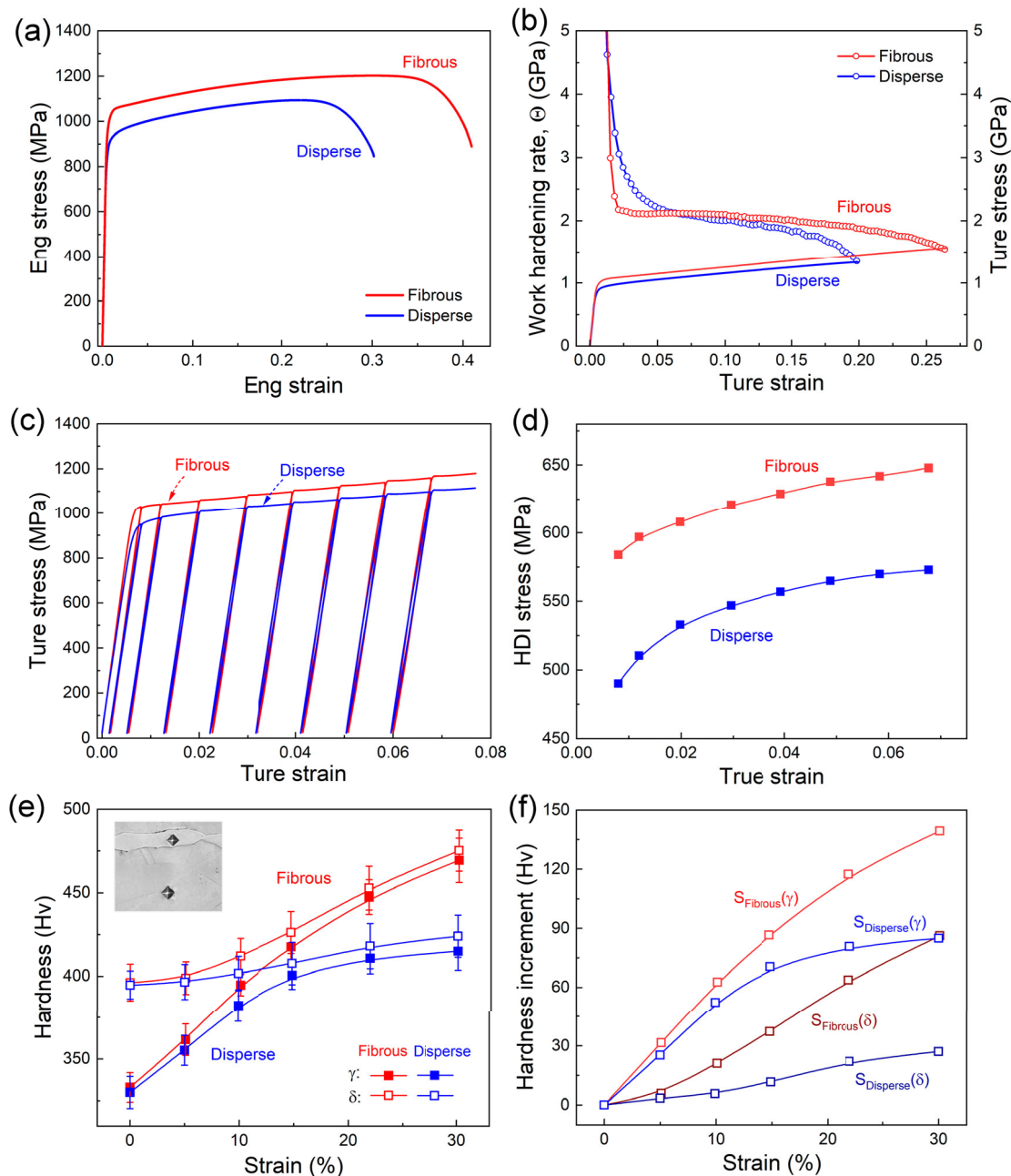


Figure 2. Tensile properties, HDI stress and hardness response of the S_{Fibrous} and S_{Disperse} samples. (a) Engineering stress–strain curves. (b) True stress and corresponding work hardening rate versus true strain curves. (c) LUR stress–strain curves. (d) HDI stress. (e) Hardness evolution of austenite and ferrite in the S_{Fibrous} and S_{Disperse} samples during straining. The inset in (e) represents typical indentation pits. (f) Evolution of hardness increment of each phase.

annealing temperature (880°C) for the S_{Disperse} sample is strategically chosen to maintain comparable grain sizes and phase contents between the two samples. These microstructural characteristics provide crucial prerequisites for the following structural-dependent mechanical superiority comparison.

As the engineering stress–strain curves shown in Figure 2(a), surprisingly, the S_{Fibrous} sample achieves an exceptional tensile elongation of 41% at a high tensile strength of 1202 ± 14 MPa. Specifically, the yield

strength (1008 ± 11 MPa) of the S_{Fibrous} sample is higher than that (883 ± 12 MPa) of the S_{Disperse} sample, and the plastic deformation capability is also almost 1.5 times greater. It is evident in enhancing the uniform elongation from $\sim 22\%$ to $\sim 32\%$, along with an extended total ductility from $\sim 30\%$ to $\sim 41\%$. On the other hand, the CG sample, also featured with disperse ferrite (Figure S5), demonstrates high ductility, but the yield strength (487 MPa) falls significantly below that of the S_{Fibrous} sample (Figure S6). These remarkable mechanical

discrepancies firmly clarify the mechanical superiority of discontinuous fibrous structures. Figure 2(b) illustrates strain hardening rate curves of the S_{Fibrous} and S_{Disperse} samples. After dropping to ~ 3 GPa, the downward trend of Θ gradually decelerates for both structures, whereas the S_{Disperse} sample decrease slower, which is associated with the intensified stress fields induced by the prolonged elastic-plastic transition behavior (Figure S7). With further increasing tensile strain ($\epsilon_{\text{ture}} > 6\%$), the attenuation of Θ in the S_{Fibrous} sample is weaker than that of the S_{Disperse} sample, signifying an enhanced strain hardening response.

Hetero-zones with mechanical incompatibility can provoke non-homogeneous deformation under applied strains, thus engendering long-range internal stress significantly to strengthen soft regions [10], which is known as HDI strengthening and hardening [26]. The LUR tensile tests were conducted to investigate the effect of HDI stress on the mechanical response, as presented in Figure 2(c). While using Yang et al.'s method [27], the HDI stress in both structures is significantly high,

reaching magnitudes of $500 \sim 650$ MPa (Figure 2(d)). It exhibits a relatively rapid rise at the low strain stage, followed by a gradual deceleration in the growth rate. The HDI stress of the S_{Fibrous} sample is higher than that of the S_{Disperse} sample, indicating stronger hetero-interactions, thereby resulting in elevated yield strength.

The hardening response of austenite and ferrite is evaluated by measuring their hardness at various strains. As shown in Figure 2(e), the austenite and ferrite in the S_{Fibrous} sample (red points) exhibit an accelerated hardening rate compared to that of the S_{Disperse} sample (blue points), especially at high strain stage, indicating an elevated strain hardening. Furthermore, the increased hardness of austenite surpasses that of ferrite in both structures (Figure 2(f)), which represents the softer austenite undertaking more applied strain than the harder ferrite. These differential strain hardening behaviors lead to the gradually decreased inter-phase heterogeneity between austenite and ferrite.

The pronounced HDI strengthening and strain hardening are inevitably accompanied by GNDs [28]. As

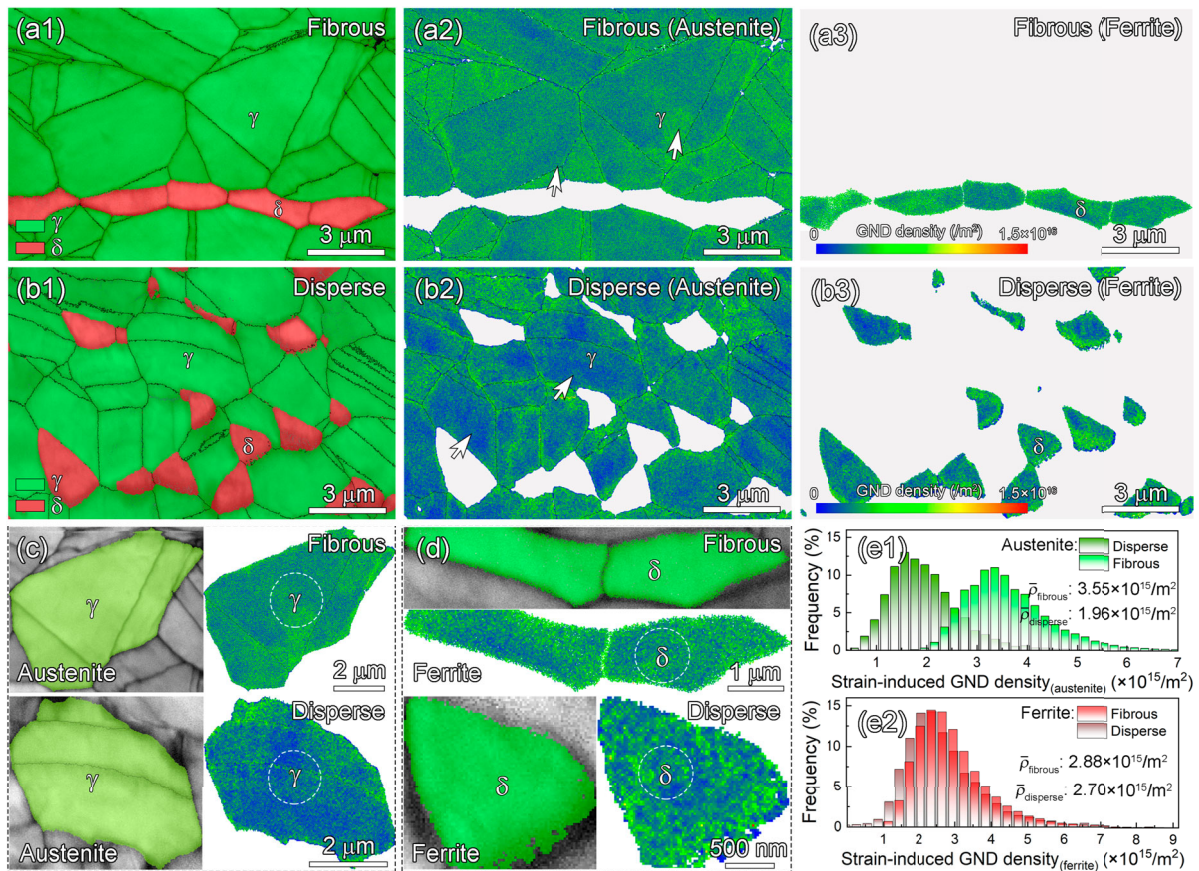


Figure 3. Distribution of GND density at the tensile strain of $\sim 5\%$. (a1–a3) Phase map and GND density maps of the austenite and ferrite in the S_{Fibrous} sample, respectively. (b1–b3) Phase maps and GND density maps of austenite and ferrite in the S_{Disperse} sample, respectively. (c) GND density distribution of typical individual austenite grain in the S_{Fibrous} and S_{Disperse} samples. (d) GND density distribution of typical individual ferrite grain in the S_{Fibrous} and S_{Disperse} samples. (e1–e2) Statistical distribution of strain-induced GND density in austenite and ferrite.

shown in Figure 3(a–b), the GND density in the S_{Fibrous} sample is higher than that in the S_{Disperse} sample, indicating a stronger heterogeneous deformation. Furthermore, by analyzing the individual austenite and ferrite grains with similar Schmid factors (0.45–0.46), GNDs concentrate not only near the phase/grain boundaries but also within the austenite interior (Figure 3(c)) in the S_{Fibrous} sample, which is scarcely discernible in the austenite interior of the S_{Disperse} sample. Meanwhile, the distribution inside fibrous ferrite is more uniform (Figure 3(d)), contrasting to the clustered distribution in the dispersed ferrite. As further clarified by statistical and calculated results in Figure 3(e1), the average strain-induced GNDs density of austenite in the S_{Fibrous} sample is $3.55 \times 10^{15}/\text{m}^2$, greater than that ($1.96 \times 10^{15}/\text{m}^2$) of the S_{Disperse} sample. The strain-induced GND density of ferrite in the S_{Fibrous} sample is also slightly ($2.88 \times 10^{15}/\text{m}^2$) higher than that of the S_{Disperse} sample (Figure 3(e2)).

For further revealing the crystallographic mechanism driving the superior strength-ductility synergy enabled by HDI strengthening and hardening, microstructural evolutions of the S_{Fibrous} and S_{Disperse} samples at strains of 1.5%, 12% and 22% were successively investigated (Figure 4). At the initial strain ($\sim 1.5\%$), several dislocation pile-ups prevail inside austenite in the S_{Fibrous} sample (Figure 4(a1, b1)). Coordinated with the preferential deformation of austenite, intersecting dislocations are activated in the adjacent fibrous ferrite (Figure 4(b1)). However, in the S_{Disperse} sample, individual dislocations scatter in the austenite (Figure 4(c1)). Accompanied by it, several disjointed dislocations are embedded in adjacent dispersed ferrites (Figure 4(d1)). These observations reflect that multiple slip systems are activated in the S_{Fibrous} sample despite the strain being small, contributing to stronger hetero-interactions.

At a tensile strain of $\sim 12\%$, the deformation of austenite in the S_{Fibrous} sample is dominated by

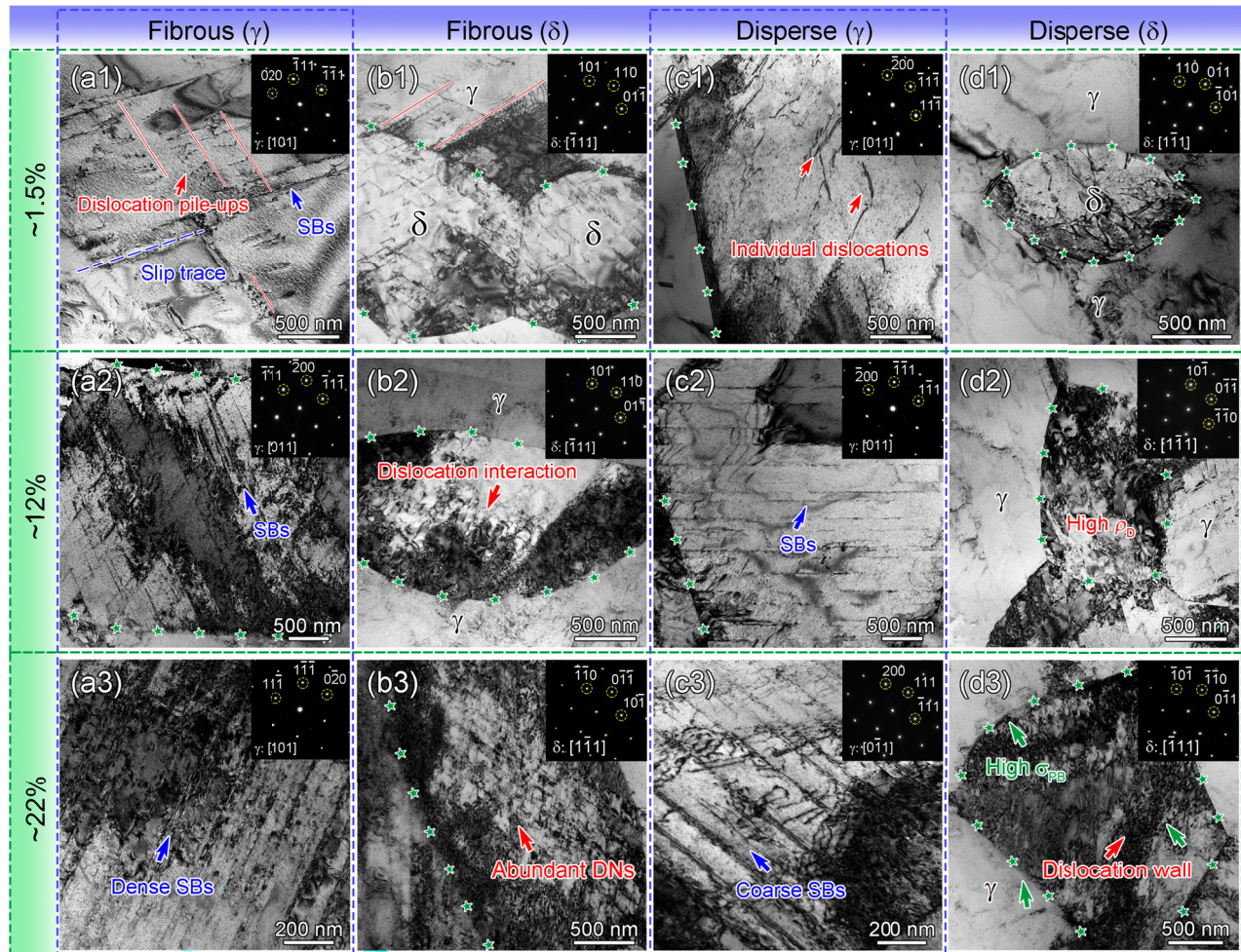


Figure 4. Microstructural evolutions of (a) austenite in S_{Fibrous} , (b) ferrite in S_{Fibrous} , (c) austenite in S_{Disperse} and (d) ferrite in S_{Disperse} at various tensile strains: (1) $\sim 1.5\%$; (2) $\sim 12\%$; (3) $\sim 22\%$. Inserts are corresponding SAED patterns. Red lines represent dislocation pile-ups. Star-shaped markers indicate phase boundaries. SBs in (a, c) represent slip bands. DNs and ρ_D in (b, d) represent dislocation networks and dislocation density, respectively.

slip bands, which penetrate through the entire grain (Figure 4(a2)). The adjacent fibrous ferrite is undergoing dislocation interactions from intersecting planes (Figure 4(b2)). In the S_{Disperse} sample, sparse slip bands are produced in austenite (Figure 4(c2)). The neighboring dispersed ferrite experienced extensive plastic deformation, resulting in a complex internal diffraction contrast (Figure 4(d2)). With further increasing tensile strain ($\sim 22\%$), it is evident that slip bands (Figure 4(a3)), entangled with plentiful dislocations, are further refined in the entire austenite with a spacing of ~ 51 nm. The denser slip bands arise from the high-stress levels, which are ensured by more sufficient GND pile-ups, complex stress state (Figure S8) and stronger interactions between fine slip bands and the austenite matrix. Alongside it, abundant dislocation networks emerge in the adjacent fibrous ferrite (Figure 4(b3)), contributing to the strain hardening by acting as strong dislocation impellers. However, in the S_{Disperse} sample, coarse slip bands with wider spacing indicate a relatively weak deformation. Dislocation walls and dislocation entanglements generate in the adjacent disperse ferrite (Figure 4(d3)), indicating an extremely high fracture tendency due to significant stress concentrations at the phase boundary and dislocation wall. Through the entire deformation process, the above crystallographic discrepancies between the two samples indicate that dense slip bands and high density of dislocation networks are exclusively triggered in the S_{Fibrous} sample under synergistic straining.

In the present lightweight steel, numerous phase boundaries provide a critical structural prerequisite for HDI strengthening and strain hardening [28–30]. The pronounced HDI effect is primarily dominated

by three aspects: (i) magnitude of heterogeneity; (ii) density of hetero-interfaces and (iii) distribution of hetero-interfaces. In the first aspect, the heterogeneity, i.e. elastic/plastic incompatibility between austenite and ferrite, can engender heterogeneous deformation under applied strains and induce strain gradients near phase boundaries, which is concurrently accompanied by dislocation pile-up (Figure 3(a–b), Figure 4(a1)). The GND pile-up generates back stress in the austenite and forward stress in the ferrite, eventually giving rise to the HDI strengthening and strain hardening [31]. In the second aspect, low-density hetero-phase boundaries are difficult to strengthen validly the entire sample owing to the weak HDI effect. Conversely, as for nano-dual-phase structures with excessive hetero-phase boundaries, the HDI hardening is hard to support synergistic deformation, primarily due to alterations in the dislocation interaction mechanism [32]. Appropriate high-density hetero-interfaces, like phase boundaries in the fibrous structure, can effectively accumulate numerous GNDs and promote the HDI effect. In the last aspect, the distribution of hetero-interfaces also plays a crucial role in promoting HDI strengthening and hardening. Various distributions of hetero-interfaces can alter the ways of hetero-interactions and the magnitude of GND pile-ups. Reasonable arrangement of the hetero-interfaces can further optimize the mechanical synergy.

Furthermore, it is found that the unique deformation substructures, i.e. dense slip bands in austenite, dislocation networks in ferrite and dislocation pile-ups near the consecutive ferrite-austenite interfaces are the underlying crystallographic mechanisms for the elevated HDI

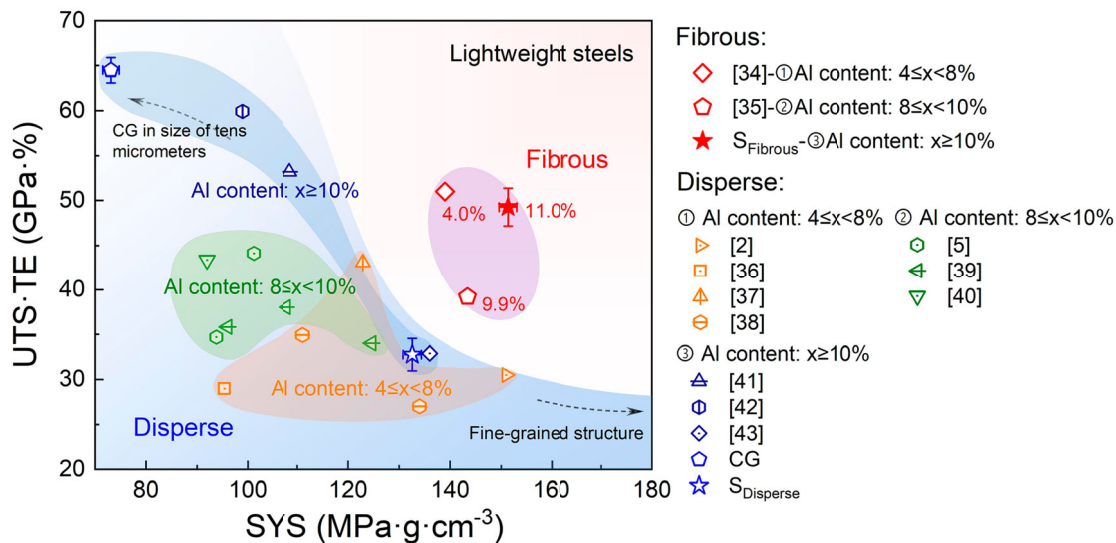


Figure 5. Comparison of mechanical properties (product of UTS·TE vs. SYS) of two structural (fibrous and disperse) lightweight steels with varying aluminum contents [2,5,34–43]. Detailed information for the mechanical property comparison is listed in Table S2.

strengthening and strain hardening in the S_{Fibrous} sample, which is not observed in the S_{Disperse} sample. On the one hand, the dense slip bands (Figure 4(a2–a3)) can generate significant incompatibility coupled with the austenite matrix, providing more hetero-zones for hetero-interactions [33]. On the other hand, the high forward stress drives the ferrite to deform plastically by the high density of dislocation networks (Figure 4(b3)), which tends to impede more dislocation slips. Finally, the relatively consecutive ferrite-austenite interfaces facilitate more sufficient dislocation pile-ups near these interfaces (Figure 4(a1, b1), Figure S8(a)).

Mechanical performances of lightweight steels with discontinuous fibrous and dispersed ferrite are compared across varying aluminum contents (Figure 5). Under three ranges of aluminum content: (i) $4 \leq x < 8\%$; (ii) $8 \leq x < 10\%$; (iii) $x \geq 10\%$ (corresponding density ranging from 7.42 to 6.47 g/cm³), the fibrous structure consistently manifests a superb product of UTS·TE, together with an outstanding specific yield strength (SYS). For instance, the S_{Fibrous} sample surpasses the S_{Disperse} sample in SYS by ~ 20 MPa/g/cm³ and in UTS·TE by a factor of 1.5. This comparison of mechanical properties across multiple compositions (densities) highlights that the fibrous structure is a universal strategy for optimizing mechanical performance in lightweight steels.

4. Conclusion

The optimized ferrite's distribution for achieving superb strength-ductility synergy in lightweight steel was comparatively unveiled by two typical structures, featured with discontinuous fibrous and dispersed ferrite individually embedded in the austenite matrix. Discontinuous fibrous structure exhibits an outstanding combination of high tensile strength (1202 MPa) and large total ductility (41%), which is far beyond the mechanical limit of dispersed structure. The excellent mechanical performance primarily originates from notable HDI strengthening and strain hardening. Underlying crystallographic mechanisms are attributed to the pronounced hetero-interactions near numerous consecutive ferrite-austenite interfaces, the high density of slip bands inside austenite and dislocation networks within ferrite. These findings highlight discontinuous fibrous structure as a promising strategy to achieve large uniform elongation at elevated strength levels in lightweight steels.

Disclosure statement

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

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Data availability statement

Data will be made available on request.

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