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ORIGINAL REPORT



Eliminate the contradiction between temperature and toughness by grain-boundary delamination in heterogeneous ultrafine-grained lamellar steels

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ABSTRACT

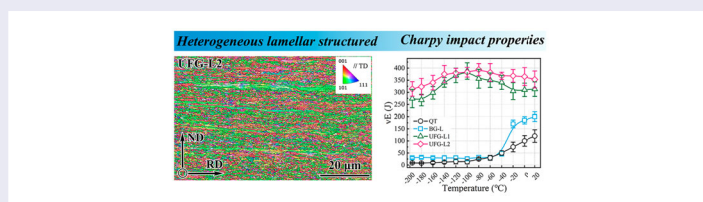
Heterostructured ferritic steels with bimodal-grained lamellar (BG-L) and ultrafine-grained lamellar (UFG-L) microstructure were prepared through a warm deformation process. The BG-L steel exhibits enhanced mechanical properties compared to conventional quenched and tempered (QT) steel. While the UFG-L steel demonstrates an outstanding combination of strength, ductility, and toughness. Furthermore, the UFG-L steels exhibit no ductile-to-brittle transition (DBT) from room temperature (RT) to liquid nitrogen temperature (LNT) and the Charpy impact energy remains as high as 314 J at LNT. The enhanced toughness at LNT can be attributed to the crack-arrester mechanism caused by grain-boundary delamination.

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KEYWORDS

Heterostructured steel; toughness; ductile-to-brittle transition temperature; mechanical properties; grain-boundary delamination



1. Introduction

Achieving a balance between the strength and toughness of steel materials has long been a pursuit in engineering applications [1–3]. Among the most widely used metallic materials, body-centered cubic (bcc) steels often experience an enhancement in strength at the expense of toughness [4,5]. Unfortunately, bcc steels are also prone to a severe ductile-to-brittle transition (DBT) phenomenon [6–9]. When the temperature is reduced to a certain degree, the material usually undergoes a sudden change from ductile fracture to brittle fracture. The essence of the DBT phenomenon in bcc steels lies in the unique three-dimensional core structure of screw dislocations, which exhibits significant thermal activation characteristics [9,10]. At cryogenic temperatures, these screw dislocations move much slower compared to the

edge dislocations. As a result, slip and plastic deformation are hindered, leading to a significant increase in yield strength and surpassing the strength required for cleavage fracture. Consequently, bcc steels often experience brittle cleavage fracture at cryogenic temperatures.

The introduction of heterogeneous lamellar structure is one of the most effective ways to resolve the DBT phenomenon [11–14]. Kum et al. [15] successfully prepared an ultra-high carbon steel/medium carbon steel laminated composite steel through a roll-bonding process. They achieved an extremely low ductile-to-brittle transition temperature (DBTT) of -140°C and ultra-high shelf energy ($> 325\text{J}$) as a result of the crack-arrester effect caused by the delamination between laminates. It has been reported that the weak bonding of the interface plays a crucial role in the delamination

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process [15]. Furthermore, Sun et al. [16] employed hot rolling and intercritical annealing processes to fabricate ferrite/martensite (F/M) layered steel in bainitic steel. The F/M layered microstructure effectively suppresses the strain localization and shear band propagation during impact processes. It achieved a remarkable impact energies of 401 J at room temperature (RT) and 245 J at liquid nitrogen temperature (LNT) [16]. However, it should be noted that not all heterogeneous lamellar structures can achieve excellent mechanical properties at cryogenic temperatures. Previous studies indicate that the heterogeneous lamellar steels still lose toughness at LNT, typically have an impact energy ranging from 10 to 30 J [17–19]. Achieving high impact energy at LNT and eliminating the DBT phenomenon from RT to LNT in low alloy steels remains a challenging task.

In this work, two categories of heterogeneous lamellar structure, namely, bimodal-grained lamellar (BG-L) and ultrafine-grained lamellar (UFG-L), were fabricated by warm deformation in a commercial low-alloy medium-carbon steel. Our systematic investigation reveals that the BG-L steel exhibits an excellent combination of strength and ductility, while the UFG-L steels present significantly higher toughness at cryogenic temperatures in the crack-arrester direction due to the prevalence of grain-boundary delamination mechanism. Furthermore, the impact toughness of BG-L and UFG-L steels in the crack-divider direction is also sufficiently high compared with the quenched and tempered (QT) steel specimens.

2. Materials and methods

An Fe-0.42C-0.95Cr-0.19Mo-0.25Si-0.88Mn (mass%) steel was used in this study for demonstration. A 180 kg ingot was produced in an induction melting furnace and homogenized at 1200°C for 8 h. Subsequently, the ingot was forged into billets with a sectional area of $260 \times 260 \text{ mm}^2$ at 1050°C, followed by water cooling to room temperature (RT). The process of preparing heterogeneous lamellar UFG steel can be divided into two steps (Figure 1(a–c)). In the first step, the lamellar microstructure was obtained by reheating the billet to 680°C for 6 h followed by a warm forging. Namely, the billet was forged into a sectional area of $85 \times 510 \text{ mm}^2$ (60% forging reduction). In the second step, the warm forged steel was cut into $85 \times 255 \text{ mm}^2$ plates, reheated to 650°C for 2 h, and subsequently warm rolled to thicknesses of 17 and 10 mm (93% and 96% rolling reduction, respectively). For ease of description, the warm forged and warm rolled steels were named BG-L, UFG-L1, and UFG-L2, respectively. In addition, quenched and tempered (QT) steel specimens with identical chemical compositions were

prepared for comparison (length of 160 mm, width of 80 mm and thickness of 75 mm), the QT steel was austenitized at 850°C ($A_{C3} + 50^\circ\text{C}$) for 1 h and subsequently water-quenched. The quenched steel was then tempered at 650°C for 2 h.

The microstructures were observed using a scanning electron microscope (SEM, Carl Zeiss Gemini 300) with an electron backscattering diffraction detector (EBSD, Oxford Symmetry S2) and a transmission electron microscope (TEM, JEOL JEM-2100F) equipped with focused ion beam (FIB) and transmission Kikuchi diffraction (TKD) tools. The grain size was measured by the line-intersect method along LD/RD and ND. The tensile tests were conducted on an AGS-X-50KN universal testing machine with a constant strain rate of $1.7 \times 10^{-3} \text{ s}^{-1}$, using dog-bone shaped tensile specimens with a gauge length of 50 mm, a cross-section of $15 \text{ mm} \times 2 \text{ mm}$ cut along LD/RD, in accordance with ASTM standard E8-E8M, the tensile specimens along ND and specimens' size are shown in supplementary material Fig. S7. The Charpy impact tests were conducted using 2 mm V-notch (ISO 148-1:2009) specimens in a temperature range from Liquid nitrogen temperature (LNT) to room temperature (RT), the crack-arrester type (the impact direction along ND, the notch along TD) and crack-divider type (the impact direction along TD, the notch along ND) specimens were using a PTM2302-C metal pendulum impact tester, as show in Figure 1(d).

3. Results and discussion

Figures 2 (a–c) schematically show the microstructure of BG-L, UFG-L1, and UFG-L2 steels. As the amount of warm deformation increases, the bimodal-grained lamellar microstructure gradually transforms into the ultrafine-grained lamellar microstructure. Figures 2 (d–i) display the orientation distribution maps and the corresponding grain-boundary (GB) maps for the longitudinal ND-LD or ND-RD sections. The corresponding GB distribution confirms that the proportion of HAGB continuously increases with the extent of warm deformation, reaching 37.7%, 61.3%, and 65.1% in the BG-L, UFG-L1, and UFG-L2 steels, respectively. The orientation distribution maps for the other two directions are shown in Fig. S1 in the supplementary material. It shows that λ -fiber is the dominant texture component in all three steels (ODF section at $\varphi_2 = 45^\circ$ in Fig. S2 in the Supplementary material). The strength of texture increases with the amount of warm deformation, and the texture type becomes a single plate texture type in the UFG-L2 steel [12,20–22].

Figures 3 (a–c) show the TEM morphologies of the BG-L, UFG-L1, and UFG-L2 steels, respectively. Zhu and Wu [23] classified ferrite grain size into

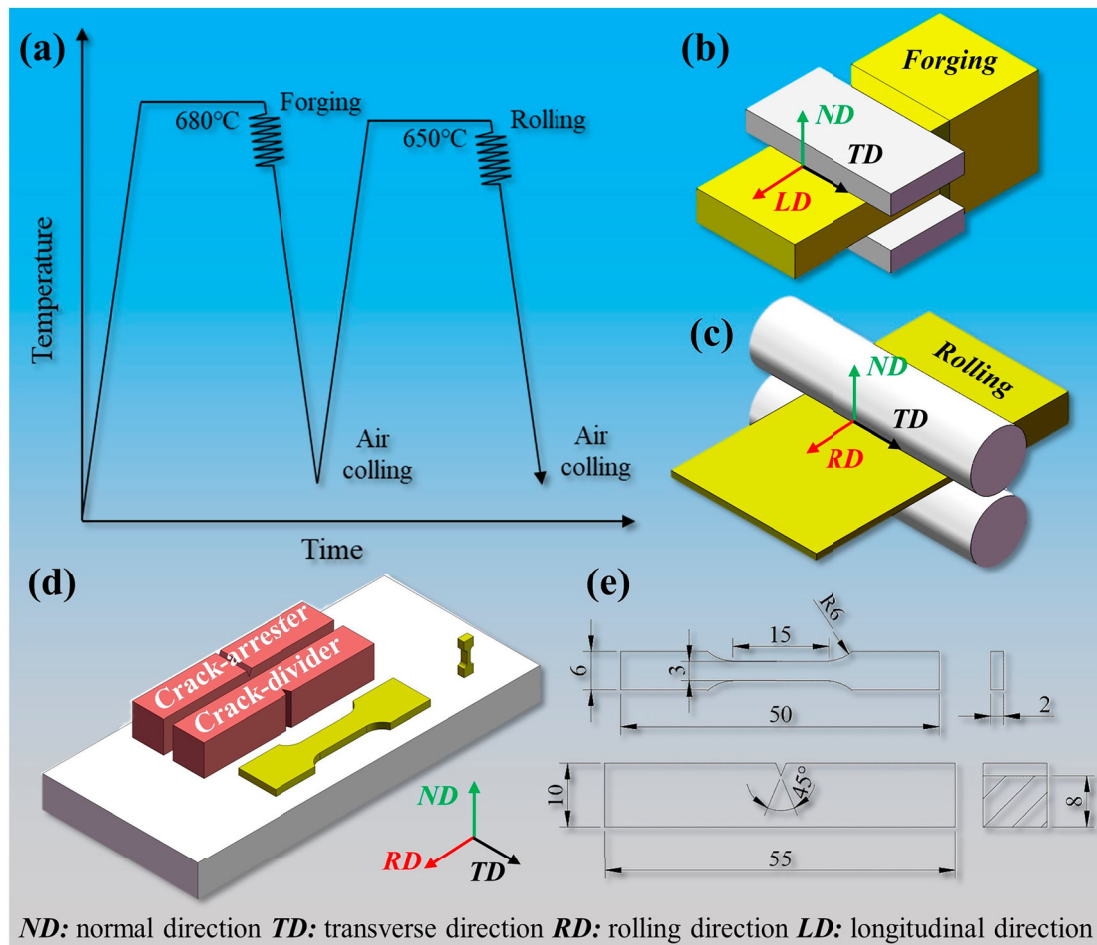


Figure 1. (a) Schematic diagram showing the two-stage warm deformation processes, (b) warm forging, (c) warm rolling, (d) sampling location for mechanical testing, (e) dimensions of tensile and impact specimens, where LD is the longitudinal direction, RD is the rolling direction, TD is the transverse direction, and ND is the normal direction.

three levels: ultrafine-grained (UFG, $d \leq 1 \mu\text{m}$), fine-grained (FG, $1 \mu\text{m} < d \leq 5 \mu\text{m}$), and coarse-grained (CG, $d > 5 \mu\text{m}$). The average grain size was measured by the line-intercept method (Supplementary Fig. S3). The grain size distributions of BG-L steel comprise two peaks in both the LD and ND. Those measured along the ND (the transverse grain size) correspond to CG ($5.6 \pm 0.5 \mu\text{m}$) and FG ($1.4 \pm 0.3 \mu\text{m}$), resulting in a bimodal-grained microstructure. Meanwhile, the transverse grain sizes of UFG-L1 and UFG-L2 steels are $0.45 \pm 0.2 \mu\text{m}$ and $0.30 \pm 0.2 \mu\text{m}$, respectively, as shown in Figures 3 (a1–c1). Furthermore, the average size of carbide particles is determined as $120 \pm 10 \text{ nm}$, $105 \pm 5 \text{ nm}$, and $100 \pm 5 \text{ nm}$ in the BG-L, UFG-L1, and UFG-L2 specimens, respectively (Supplementary Fig. S4). The carbide particles are confirmed to be Fe_3C based on the selected area diffraction (SAD) pattern and the enrichment of such elements as C, Cr, Mn suggested by the line and mapping scans (Supplementary Fig. S5).

Figure 4 (a) summarizes the Charpy impact properties of QT, BG-L (crack-arrester type), UFG-L1 (crack-arrester type), UFG-L2 (crack-arrester type) steels at different temperatures. For the BG-L steel, the impact energy and toughness decrease substantially with a decrease in test temperature. Consequently, the BG-L steel exhibits a typical DBTT of around -40°C , resulting in a rapid decrease in impact energy from 200 J at RT to less than 50 J at LNT. In contrast, the UFG-L1 and UFG-L2 steels do not show a DBT phenomenon. For comparison, the QT steel exhibits a DBTT from 20°C to -20°C . The high DBTT of QT steel may be caused by the chain-like distribution of carbide particles along the HAGB in the QT steel (Supplementary Fig. S6). These intergranular carbides make the cracks easy to nucleate and propagate during the impact tests [24–26].

The Charpy impact load-displacement curves are presented in Figure 4(b). The shape of the impact load-displacement curves is different among the present three

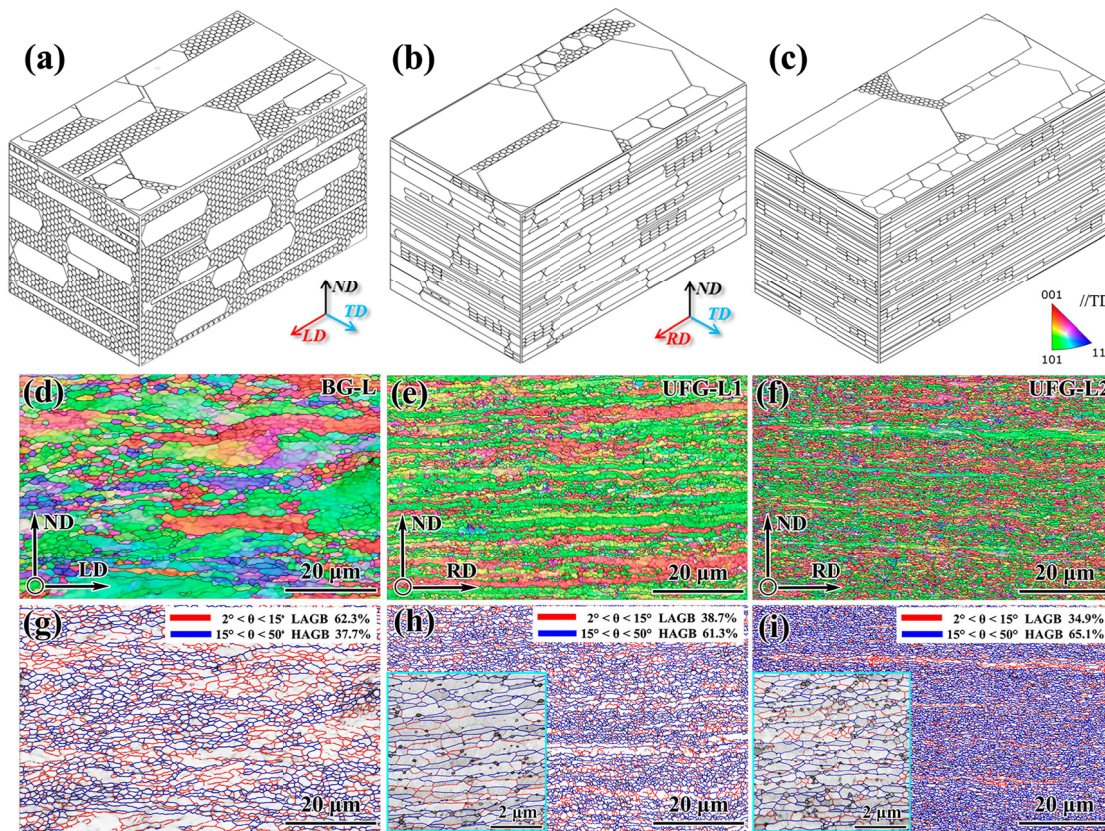


Figure 2. The heterogeneous lamellar microstructure of the steel after warm deformation: (a-c) schemes of three steels with heterogeneous lamellar microstructures, (d-f) orientation distribution maps and (g-i) corresponding GB maps. LAGB: low-angle grain-boundary, HAGB: high-angle grain-boundary.

steels. With increasing displacement, besides the commonly observed initial elastic and plastic deformations, the Charpy steel specimens in this study demonstrate crack arrest behavior. This phenomenon occurs when delamination stops unstable crack propagation, leading to a multi-peaked load-displacement curve similar to those reported in previous studies [12,25]. It is worth noting that a decrease in temperature causes a significant increment in the impact load of UFG-L1 and UFG-L2 steels. Similar behavior has been observed in the temp-forming steels, the delamination along lamellae led to an increment of impact load with decreasing temperature [27].

Figure 4 (c) shows the stress-strain curves at RT and LNT for the experiment steels with Heterogeneous lamellar microstructures. The tensile curves of QT steel are also shown in Figure 4 (c) for reference. The tensile properties are listed in Table 1. It can be seen that all the steels with lamellar microstructures exhibit significantly higher strength and elongation compared to the QT steel. At RT, the total elongation (TEL), yield strength (YS), and ultimate strength (UTS) of BG-L steel are 21.9%, 819.9 and 822.3 MPa, respectively; while those are as high as 18.8%, 910.3 and 935.9 MPa, respectively, for the UFG-L2 steel.

Table 1. Tensile properties of QT, BG-L, UFG-L1, UFG-L2 steels.

Steel	YS (MPa)	UTS (MPa)	TEL (%)
QT-RT	652.6	751.8	16.1
BG-L-RT	819.9	822.3	21.9
UFG-L1-RT	828.4	878.2	21.6
UFG-L2-RT	910.3	935.9	18.8
QT-LNT	1064.1	1069.7	9.1
BG-L-LNT	1072.5	1094.8	20.7
UFG-L1-LNT	1176.5	1129.9	18.3
UFG-L2-LNT	1263.6	1184.5	18.1

At LNT, the strength of all steels is much higher than at RT. It should be noted that the increase in strength of QT steel with decreasing temperature is accompanied by a significant decrease in TEL. However, the TEL remains nearly unchanged regardless of test temperature in the steels with heterogeneous lamellar microstructures, the tensile properties in the ND are shown in Fig. S7 in supplementary material.

A comprehensive investigation was conducted to compare the room temperature YS and the cryogenic Charpy impact energy (vE) of the heterogeneous lamellar UFG steels with other structural materials, including Maraging steels, Austenite steels, TWIP/TRIP steels, cryogenic steels, pipeline steels, high-entropy alloys (HEA), etc. in

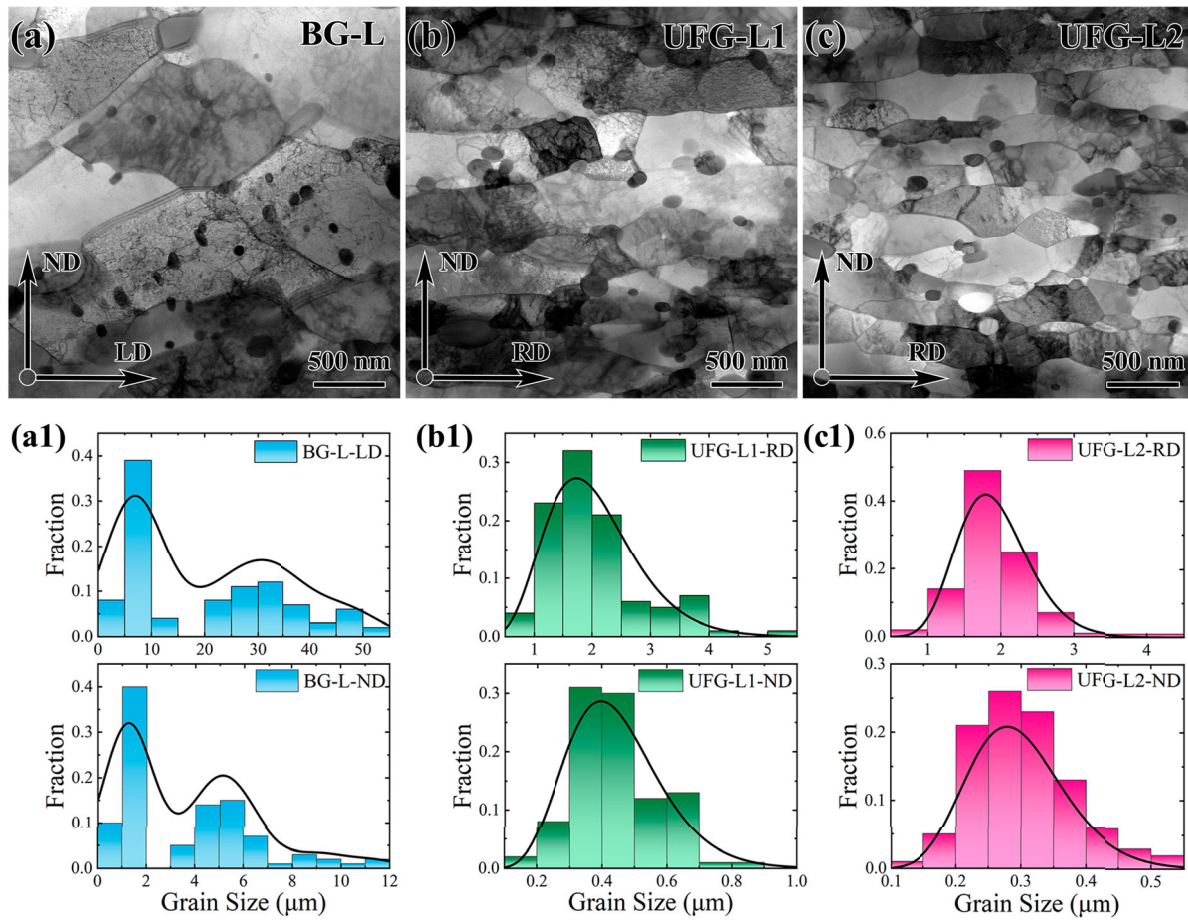


Figure 3. TEM images and grain sizes distribution of steels with heterogeneous lamellar microstructures; (a-a1) BG-L, (b-b1) UFG-L1, (c-c1) UFG-L2.

addition to laminated steels (Figure 4(d)). The UFG-L1 and UFG-L2 steels exhibit an excellent combination of strength and toughness. Impressively, at equivalent strength levels, UFG-L2 steel stands out with the highest recorded toughness. On the other hand, despite the similar cryogenic impact toughness between UFG-L2 steel and the HEA, the YS of UFG-L2 steel is three times higher than that of HEA.

The YS of experimental steels can be estimated by a summation of solid solution strengthening (σ_s), dislocation strengthening (σ_D), carbide particle strengthening (σ_p), grain-boundary strengthening (σ_g). Therefore, the YS can be expressed as:

$$\sigma_{YS} = \sigma_0 + \sigma_s + \sigma_D + \sigma_p + \sigma_g \quad (1)$$

where σ_0 represents Peierls stress of about 45 MPa as usually adopted for ferritic matrix steel [28], σ_s depends on the chemical composition, the solid solution strengthening can be estimated by the synthesis of the contributions of each alloying element, and the empirical formula is as

follows [29]:

$$\begin{aligned} \sigma_s &= \sum [X_i] \sigma_i \\ &= 5544[C + N] + 32[Mn] + 83[Si] + 678[P] \\ &\quad + 11[Mo] + 31[Cu] - 30[Cr] \end{aligned} \quad (2)$$

where $[X_i]$ is the weight percentage of the element X, σ_i is the solid solution strengthening contribution of this element. The σ_s term is 44.65 MPa for the experiment steel. Here, most of the carbon element exists at the grain-boundary and inside the grain in the form of Fe_3C , so the contribution of carbon to the σ_s is neglectable [30].

The contribution of dislocations to the YS can be evaluated by adopting the Taylor equation [31]:

$$\sigma_D = \alpha M G b \rho^{0.5} \quad (3)$$

here α is a constant equal to 0.15, M is the Taylor factor. The value of M can be calculated from the EBSD results. The M values of BG-L, UFG-L1, and UFG-L2 are 2.64, 2.45, and 2.36, respectively, as shown in Fig. S8 in the supplementary material. G is the shear modulus, $G = 76$

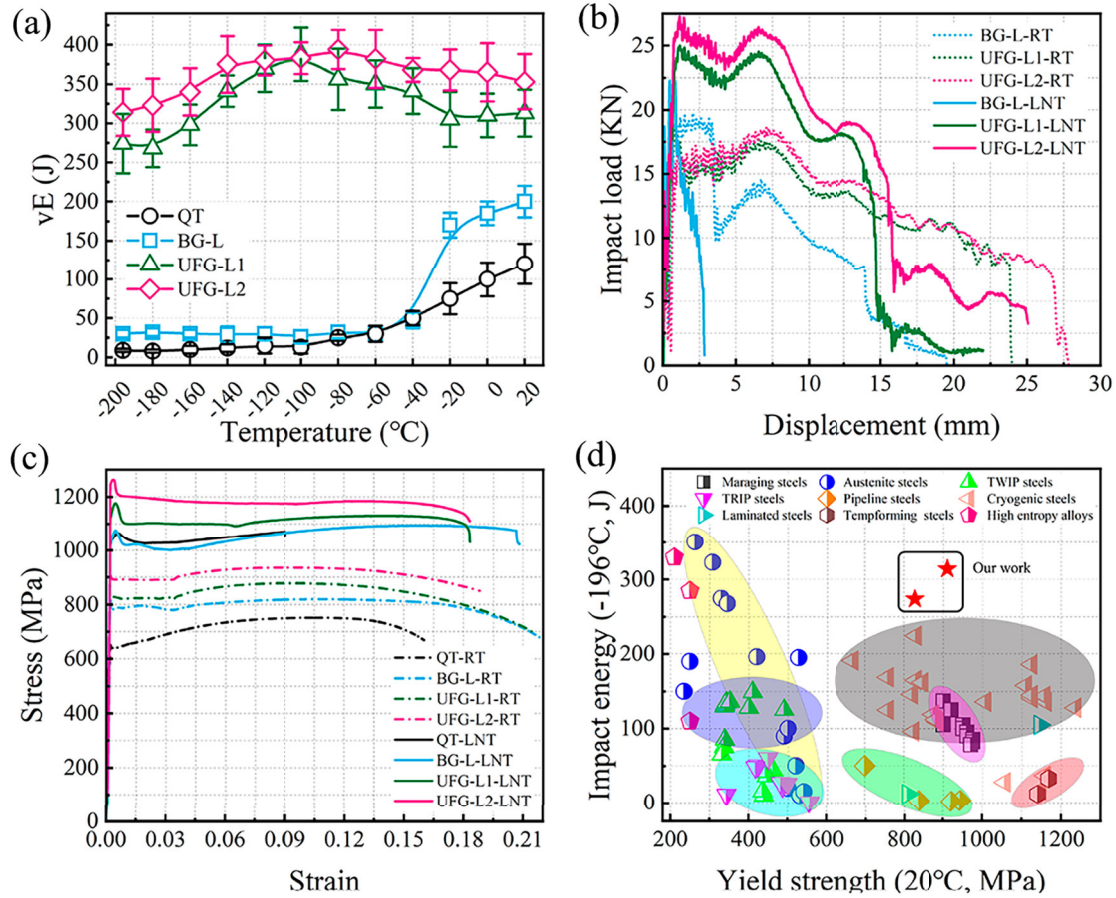


Figure 4. Mechanical properties of heterogeneous lamellar steels and QT steel; (a) temperature-dependent Charpy impact toughness (crack-arrester type); (b) load-displacement curves during impact test; (c) engineering stress-strain curves at RT and LNT; (d) Charpy impact energy vs yield strength of our work and other materials, the relevant comparative data are shown in supplementary material Table S1.

GPa, and b is the Burgess vector, $b = 0.248 \text{ nm}$ [32], ρ is the dislocation density. The dislocation density can be calculated by the Williamson-Hall's method based on the X-ray diffraction method (Supplementary Fig. S9).

The contribution of carbide particles to YS is shown in the following equation [33].

$$\sigma_p = 8995 \times \left(\frac{\sqrt{\varphi_p}}{d} \right) \times \ln(2.417d) \quad (4)$$

where φ_p is the volume fraction of carbides, d is the carbide particle size.

Meanwhile, σ_g can be used to represent grain-boundary strengthening [34]:

$$\sigma_g = \frac{K}{\sqrt{D_g}} \quad (5)$$

where D_g is the transverse grain size (along the ND), K is the grain-boundary strengthening factor, for ferritic matrix steel, $K = 0.24 \text{ MPa m}^{0.5}$ [22]. The contribution of various strengthening mechanisms to YS is listed in Table 2. By summing these contributions, the

Table 2. Strengthening contributions of YS for BG-L, UFG-L1, UFG-L2 steels.

Specimen	BG-L	UFG-L1	UFG-L2
d_p , nm	125	105	100
φ_p , %	14.08	12.86	12.57
D_g , μm	3.58	0.45	0.30
σ_p , MPa	153.73	170.08	175.01
σ_g , MPa	126.98	358.20	444.45
σ_D , MPa	469.11	286.16	257.28
Calculated values, MPa	794.47	859.09	921.39
Experimental values, MPa	819.9	828.4	910.3

calculated YS values closely match experimental results. It shows that the dislocation strengthening term dominates the strength of BG-L steel, while the grain boundary strengthening term dominates in UFG-L1 and UFG-L2 steels.

The fracture mechanisms have been investigated. Figure 5 (a–c) shows the TKD analysis of the impact fracture surface of UFG-L2 steel at LNT. The macroscopic morphology of impact fracture of other steels is shown in Fig. S10 in the supplementary material. The

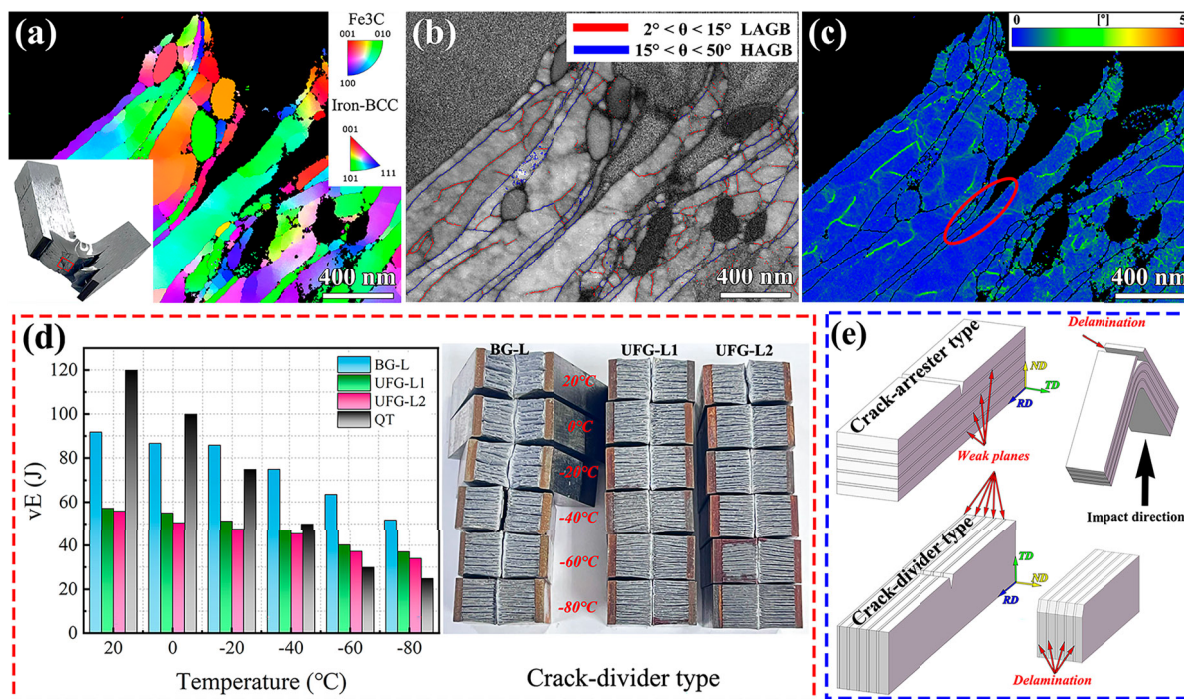


Figure 5. Impact fracture analysis of UFG-L2 steel by TKD: (a) the inverse pole figure map, (b) the grain boundary map, and (c) the kernel average misorientation map. (d) Charpy impact properties along the crack-divider direction and the impact fracture morphology. (e) Schematic diagram of delamination toughening mechanisms of steels with heterogeneous lamellar microstructure.

red rectangular box represents the FIB lift-out location. Notably, it can be observed that the two sides of the crack belong to different grains, indicating that intergranular fracture takes place along the grain boundaries [35]. Additionally, Kernel Average Misorientation (KAM) maps demonstrate that there is minimal plastic deformation at the crack tips, as illustrated by the red elliptical boxes in Figure 5(c). Consequently, grain-boundary delamination cracks propagate within the UFG-L1 and UFG-L2 steels readily without strong impediment. Thus, the incident crack is deflected to the vertical direction, the crack arrester mechanism prevails. After arresting, for the pure mode I loading condition, the stress intensity factor (K_I) ahead of the original crack tip reduces to a very low value and it undergoes a simple bending condition, hence the crack velocity along the pre-cracked direction approaches zero. Crack reinitiation is necessary to fracture the material within the state of near uniaxial tension [15,36,37]. Therefore, the impact toughness has been effectively improved.

Furthermore, the Charpy impact tests have also been conducted along the crack-divider condition (Figure 5e). Figure 5(d) shows the Charpy impact properties along the crack-divider direction of BG-L, UFG-L1, UFG-L2 steels at different temperatures. Although the Charpy impact properties of the BG-L and UFG-L steels at RT are slightly lower than that of the QT specimen, their Charpy impact properties at low temperatures is better than that

of the QT specimen as shown in Figure 5(e). It can be seen that the fracture mode is completely different from that of the crack-arrester type impact fracture. For BG-L steel, in the temperature range from RT to -20°C , the specimens did not break into two pieces, whereas UFG-L1 and UFG-L2 specimens split completely into two pieces from the middle. It shows that the number of lamellae of UFG-L2 is significantly higher than that in the BG-L specimen.

After warm forging and warm rolling, the weak interface is distributed parallel along the LD or RD plane. For the crack-divider type, the delamination of the weak interfaces can transform the main crack into a series of cracks along LD or RD planes, changing the tri-axial stress state at the crack tip into a biaxial planar stress state [38–40]. This interrupts the propagation of the main crack, and allows significant localized plastic deformation and necking on the ligaments in front of the main cracks. Recently, the crack-divider mechanism caused by multi-delamination at manganese-enriched prior-austenite grain boundaries has been adopted to toughen the 2.1 GPa D&P steel [41].

4. Conclusion

The present investigation developed heterostructured BG-L and UFG-L microstructure in a low-alloy ferritic steel by conventional warm deformation method.

It demonstrates that BG-L and UFG-L steels exhibit an enhanced combination of strength and toughness compared with conventional QT steel. Forest dislocation strengthening dominates the strength of BG-L steel, while grain boundary strengthening dominates the strength of UFG-L steel. The BG-L steel has improved impact toughness. The UFG-L steels exhibit exceptionally high toughness at LNT and the DBT effect has been eliminated due to the occurrence of grain-boundary delamination mechanism. Furthermore, the present BG-L and UFG-L steels also demonstrate sufficiently high impact toughness along the crack-divider direction.

Data availability statement

Data available on request from the authors.

Disclosure statement

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

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