High-cycle fatigue behavior of TWIP steel with graded grains: breaking the rule of mixture

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ABSTRACT
We report a method, named as T & A treatment, to improve the high-cycle fatigue (HCF) property of twinning-induced plasticity (TWIP) steel without sacrificing its ductility and work-hardening ability. A long-range gradient in grain size was obtained in the TWIP steel, whose HCF resistance is better than both coarse-grain and fine-grain steels, breaking the rule of mixture. The surprising HCF property of the graded grain structure is believed to originate from the large generation of geometrically necessary dislocations and the formation of hard core and soft shell construction during cyclic loading.

IMPACT STATEMENT
A new strategy, refers to long-range gradient in grain size, is proposed to improve the HCF property of TWIP steel. The graded structure exhibits better fatigue resistance than single unit.

Introduction
Nowadays, carmakers are increasingly building vehicles with body-in-white (BIW) designs, in order to improve both passenger safety and fuel economy [1,2]. The safety issue requires the BIW material an excellent energy absorption (passive safety for the possible cases of car crashes) and a good fatigue endurance (reliability regarding the vehicles normal use). The economy issue includes direct fuel costs and indirect emission costs, which is mainly environmental concerning. As an outstanding representative of advanced high-strength steels (AHSS), twinning-induced plasticity (TWIP) steel possesses high strength (1.4–1.6 GPa [3,4]) and excellent plastic elongation (90% [5]), and, therefore, becomes a potential candidate for BIW material. Unfortunately, the fatigue property of TWIP steel leaves much to be desired, which largely limits its more wide application. In fact, other industrially available automotive steels, like DP (Dual-Phase) and Q&P (Quenching and Partitioning) steels, show much better high-cycle fatigue (HCF) resistance than TWIP steel [6].

To date, some efforts have been spent on the improvement of HCF resistance of TWIP steels, and most of them regard pre-strain (pre-tension [7,8], pre-drawing [9], pre-rolling [10], etc.). The enhanced fatigue property of TWIP steel mainly comes from the significant improvement of strength after pre-deformation due to its high work-hardening ability, which has its origin from the universal formation of twin boundaries and dislocations in grains. However, the plasticity and energy absorption capacity are a trade-off with strength, which makes TWIP steel in a dilemma when applied in the automobile industry [11–13].

Here, we report a new method for the improvement of the HCF property of TWIP steel without sacrifice its ductility. By the torsion and annealing (T & A) treatment, a linear gradient in grain size was introduced into the TWIP steel. It was found that its HCF resistance
was higher than homogeneous structures, and the mechanisms for this are macroscopic strain gradient and heterogeneous hardening under uniaxial applied stress. Noting that this kind of gradient structure also shows better mechanical and low-cycle fatigue (LCF) properties than individual homogeneous structures, according to our previous works [14,15].

Materials and methods
A typical TWIP steel with chemical compositions of Fe-30Mn-0.6C (wt. %) was used in our study. The detailed processing in making such material was described elsewhere [16]. The starting material was a full austenitic processing in making such material was described elsewhere [16]. The starting material was a full austenitic material with a mean grain size around 40 μm). Round bar-shaped specimens were extracted from the starting material with gauge diameter of 8 mm and length of 12 mm, then we applied pre-torsion of 180° to the TWIP steel bar, followed by annealing for 10 min at 800°C in Ar atmosphere, obtaining the graded grain (GG) sample. For comparison, homogeneous material with fine grains (FG material with a mean grain size around 4 μm) was prepared by cold rolling and subsequent annealing: a part of the CG plate with dimensions of 200 × 40 × 40 mm³ was cold rolled from an initial thickness of 40mm to 15mm (62.5% reduction), followed by a recrystallization annealing at 700°C for 12 min and air cooling to room temperature. Stress-controlled axial pull–push fatigue tests with a stress ratio of R = −1 were performed on an Instron 8850 instrument. A sinusoidal loading waveform signal in the frequency of 30 Hz was applied. The hardness was tested by an AMH43 full automatic microhardness tester with a load of 500g and a holding time of 15 s. Two or three-dimensional surface damage morphologies of the fatigued specimens were investigated by an Olympus OLS400 confocal laser scanning microscope (CLSM). Internal microstructures were studied by an LEO Supra 35 field emission scanning electron microscope (SEM) equipped electron backscatter diffraction (EBSD) component, and an FEI Tecnai F20 transmission electron microscope (TEM).

Results and discussion
Figure 1(a–c) presents microstructures in the GG sample at different radial positions, r/R = 0, 0.5, 1, respectively. The EBSD images reveal a gradient grain size along the radial direction: coarse grains with an average size of 40 μm distribute in the center of cylindrical bar (Figure 1(a)); the average grain size reduces a little at r/R = 0.5, accompanied by several deformation twins in some grains (Figure 1(b), red line means twin boundary); the grain size decreases rapidly to an average value of 4 μm in outermost layer near surface (Figure 1(c)). For comparison, microstructures in CG and FG samples are also displayed in Figure 1(d,e). The lattice distortion in GG sample is higher than which in CG and FG samples, considering the larger misorientation in grains of GG sample. The lattice distortion is believed to result from the residual strain after torsion deformation and partial recrystallization [14]. Grain size information of the three samples is summarized in Figure 1(f). Clearly, the grain size of GG sample commonly falls in the range from FG to CG.

The HCF results for the three samples are shown in Figure 2(a). Surprisingly, the GG sample exhibits a higher HCF resistance than both CG and FG samples, which evades the rule of mixture (ROM). For CG sample, the fatigue limit (σ_y) reaches above 312 MPa, which is close to the value of yield strength (σ_y) [16]. The extremely high fatigue ratio (σ_y/σ_y ≈ 1) makes the HCF damage of TWIP steel quite different from other steels. Most engineering steels fail at stress level less than σ_y during stress-controlled HCF, and their plastic deformation/damage mainly restrains in micro-scale zone [17]. However, the HCF damage of the present material concerns much more on macro-scale plastic deformation. Coupled with its high work-hardening ability, strength and hardness enhanced largely after fatigue, especially for the CG sample. As we can learn from Figure 2(b), the hardness of CG sample increases as high as 42% after fatigued at Δσ = 375 MPa, which is even higher than that of FG sample, though CG is lower than FG before cyclic loading due to the Hall-Petch effect. For the GG sample, the hardness increases along the radial direction before fatigue, while an opposite trend of hardness vs. position is found after fatigue. In other words, the hardness increases much higher in the central CGs than in the outer FGs for the GG sample during fatigue.

To decipher the effect of grain size and distribution on the fatigue behaviors of TWIP steel, it is necessary to investigate their microstructures and the corresponding deformation mechanisms with respect to these properties. Figure 3(a–c) give typical surface damage morphologies of the three samples observed after cyclic loading at Δσ = 375 MPa. According to Figure 3(a), notable persistent slip bands (PSBs), sometimes with multiple directions, and numerous cracks (pointed out in the inset) were easily found on the surface of fatigued CG specimen. While for the FG and GG specimens, whose surfaces are both covered of FGs, mild slip traces commonly with only one direction and much less (if there are some) cracks were observed (Figure 3(b,c)).

In order to effectively compare the surface deformation behaviors of the three samples, the PSB spacing and
Figure 1. Microstructures in annealed GG, CG, and FG samples of Fe–30Mn–0.6C TWIP steel. EBSD images (a–e) of microstructures in (a) GG, \( r = 0 \)R; (b) GG, \( r = 0.5 \)R; (c) GG, \( r = 1 \)R; (d) CG; (e) FG. (f) Evolution of grain size as a function of the radial position in the GS sample. Note that average grain sizes of CG and FG samples are also included in (f) for comparison.

Figure 2. (a) The stress amplitude–fatigue life relation of the GG, CG, and FG samples. (b) Variations of hardness before and after fatigue.

height were measured and summarized, and Figure 3(d) illustrates the measuring process by CLSM. According to the statistical results from Figure 3(e), both height and spacing of PSBs reduce significantly with decreasing the grain size for the homogeneous samples, which means much more slip planes activating in fine grains to participate the plastic strain, and thus a higher deformation homogeneity. The average spacing and height of PSBs on
Figure 3. Characterization of surface damage morphology after fatigued at $\Delta\sigma/2 = 375$ MPa. CLSM images of surface intrusions and extrusions of (a) CG, (b) FG and (c) GG. (d) Sketch map of height and spacing measurements of slip bands. (e) The relationship between height/spacing and grain-size distribution. Note that all the CLSM images were taken directly from the cambered bar surfaces (in gauge section) of fatigued samples.

the GG sample surface are numerically close to that on the FG surface, which is supposed to result from the similar surface states, loading condition, and thus the surface damage mode.

Figure 4 provides typical dislocation structures in CG, FG and GG samples after fatigue. As one can see, well-developed veins, which results from the accumulation of mutually trapped primary edge dislocation dipoles [18], dominates the dislocation configuration in the CG sample (Figure 4(a)). As a comparison, the dislocation density in the FG sample is much lower, and the vein is seldom found except for some dislocation network caused by dislocation tangling (Figure 4(b)). The above finding corresponds to the hardness results as displayed in Figure 2(b), where CG sample exhibits bigger increment of hardness than FG sample after fatigue, considering the strengthening caused by dislocation multiplication. For the GG sample, the remaining dislocation structure is different at various positions, i.e. vein is the primary damage microstructure at central core coarse grains (Figure 4(c)), while dislocation network is commonly found in fine grains at surface layer (Figure 4(d)), and that is why hardening is more significant at core compared to surface layer.

The above observation on surface morphology and internal microstructure may indicate that there are two factors contributing to the remarkable increment of hardness (or strengthening) in the present TWIP steel samples after HCF. One is the large-scale plastic deformation occurring during cyclic loading, and actually, nearly all the grains activate abundant slip planes (see Figure 2(a–c)). The other factor stems from the dislocation slip mode. Compared to planer slip, which usually prevails in other materials with short-range ordering (SRO) structure or low-stacking fault energy (SFE), cross slip happening in the present TWIP steel shows a higher hardening ability due to the interaction of dislocations from different slip systems. Though the hardening difference is quite slight between the two dislocation slip modes in a single slipping, it will accumulate and enlarge cycle by cycle during fatigue, finally leading to wide variations in hardness [19].

The difference in dislocation configurations in the CG, FG, and GG samples after fatigue are believed to relate to the Hall-Petch effect of TWIP steel [14]. For the homogeneous samples, CG exhibits a larger plastic strain response than FG, due to a lower yield strength, and thus more slip systems activate under the same loading stress. The activation of secondary slips, on the one hand, enhances the dislocation density; on the other hand inhibits the dislocation gliding on the primary slip plane, which both contribute to the remarkable hardening. That is why the hardness of CG is even higher than that of FG after fatigue. It should also be noted that the prevailing of secondary slipping is awfully detrimental to fatigue damage of TWIP steel, though the improvement
Figure 4. TEM images of dislocation structures after cyclic loading at $\Delta \sigma / 2 = 375$ MPa: (a) CG; (b) FG; (c) GG, $r = 0R$; and (d) GG, $r = 1R$.

of hardness (or strength) helps reduce the plastic strain during cyclic loading. In the case of grain with multiple-slips operating, vein structure is much easier to form [20,21], leaving numerous point defects (mainly vacancies [22]) at the boundaries between veins and channels [23]. As a comparison, the dislocation annihilation rate is relatively lower in the grains with only one slip system operating, for the weaker interaction of dislocations from other cross-slip planes and the absence of high/low-density regions of dislocations. The vacancies will concentrate and precipitate to form microscale cracks which may later grow into the fatigue cracks [24], due to their tendency to cluster [25]. In addition, the operation of the secondary slip system will inhibit the dislocation gliding on the primary slip plane inevitably, and thus some potential slip planes in the primary slip are locked. Only slip planes with sufficient dislocations pileup at the junction of primary and secondary slip systems can overcome the resistance and continue further slip. As a result, slips (plastic deformation) in CG are commonly restrained in certain thick PSBs, while PSBs in FG are much more homogeneously distributed (Figure 2(e)). The gathered slips in CG facilitate the vacancies concentration, and thus the initiation of micro-cracks.

According to the above observation and analysis, it is believed that dislocation slip and damage mechanisms are different at central core and on the surface layer for the GG sample (Figure 5(a)). Similarly, the plastic strain in the GG sample is also graded distributed in the initial cycle, considering the different mechanical properties (or hardness) at various positions. To be specific, the center exhibits the larger strain than the surface layer, due to its low strength relating to coarse grain in the Hall-Petch relationship. The higher strain in the center leads to higher cyclic hardening and faster fatigue damage. However, it should be noted that, the internal fatigue damage does not decide to the total fatigue life of the GG sample, because all the cracks causing fatigue failure to start from the surface, and actually, the large improvement of strength at the center helps inhibit the damage not only for the reduction of plastic strain but also for the newly formed strain gradient after several cycles of loading.

The strain gradient in GG sample formed during fatigue benefits the HCF property at last in two ways. Firstly, no matter the positive strain gradient (strain increase with the radial direction) in the later cycles or the negative strain gradient (strain decrease with the radial...
Figure 5. (a) Schematic representation of damage mechanisms at different positions of the GG sample; (b) sketch showing the formation of geometrically necessary dislocation.

direction) in the initial cycles, both of them will introduce the geometrically necessary dislocations (GND) [26–28], which further improve the strength of fatigue sample. As illustrated in Figure 5(b), a gradient of strain, $\partial \gamma / \partial y$, requires a density of GNDs,

$$\rho_{GND} = \frac{1}{b} \left( \frac{\partial \gamma}{\partial y} \right),$$  \hspace{1cm} (1)

where $b$ is the Burgers vector [26]. The formation of long-range GNDs results to an additional hardening in the whole GG sample, and thus reduces the plastic strain under the stress-controlled loading. Secondly, the hard core and soft shell structure (referring to the positive strain gradient) formed in the later cycles helps restrain the strain of surface layer, and leads to prolong the lifetime for fatigue crack initiation. Specifically, the soft shell deforms plastically earlier than the hard core, due to the lower yield strength. After yielding of soft shell, the outer soft layer deforms easier and more than the central hard core under the same loading condition, i.e. the outer layer strain along the work-hardening curve and the central core strain further along the elastic part of stress–strain curve (maybe contain a part of plastic strain later) to the same loading stress. Thus, the positive gradients in displacement and strain occur across the cross-section. Meanwhile, internal stresses arise between the hard core and soft shell, due to the strain continuity. For example, the outer layer is subjected to a compressive stress from the core and the core receives a tensile stress from the outer layer under the tension part of cyclic loading. As a result, the actual stress in the outer layer equals to the applied stress plus the inner reversal stress from the core, causing stress drop on the sample surface. Similarly, the stress drop phenomenon in the surface layer also exists in the compressive case of cyclic loading. The stress drop in the surface layer of the GG sample is believed to greatly improve the fatigue life, considering cracks leading to fatigue failure all initiate on surfaces under the present HCF.
Conclusions
A linear gradient in grain size was introduced into an Fe-30Mn-0.6C TWIP steel via pre-torsion and annealing, and the GG structure exhibits a higher HCF resistance than homogeneous structures with similar grain size. The unique cyclic hardening and damage mechanism of GG sample caused by strain gradient help inhibit fatigue crack initiation on the surface, and may provide new and important implications for the optimized microstructural design of the high fatigue performance material without sacrificing other mechanical properties.

Disclosure statement
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