Mechanism of Balanced Strength and Ductility in High-Strength Low-Alloy Steel

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Abstract: A high-strength low-alloy steel with balanced strength and ductility was reported. A product of the strength and elongation (PSE) at a break of ~19 GPa% was obtained. The microstructure of the material was investigated by scanning electron microscopy, electron backscattered diffraction, and transmission electron microscopy methods. Phase transformation follows the K–S orientation relationships. Interconnecting structures generate due to the variant interactions within one prior austenite grain. The multi-phase microstructure containing both soft and hard phases contributes to good plasticity. The homogeneously distributed NbC nanoparticles make up the loss of strength ascribed to the soft retained austenite and keep the strength at an extremely high level.

Keywords: high-strength low-alloy steel; phase transformation; precipitation; TEM; EBSD

1. Introduction

High-strength low-alloy (HSLA) steels with low carbon content and a small addition of alloying elements (such as Cu, Nb, Ti, etc.) are attracting more and more attention due to the demand for improving ductility without sacrificing strength in advanced metallic materials [1–3]. Due to such comprehensive properties, HSLA steels can be used in long-distance gas pipelines, automotive parts, offshore platform structures, etc. [4–6]. With the addition of micro-alloying elements, HSLA steels can be strengthened through a combination of precipitate hardening, solid solution strengthening, and grain refinement strengthening [7–9].

The microstructures of steels could consist of single-, dual-, or multi-phase. Complex face-centered to body-centered cubic phase transformation can take place in steels during thermomechanical processes through a coordinated movement of atoms [10,11]. The introduction of alternative phases can certainly change the behaviors of the steels. Single-phase steel, even with ultra-fine grain size, can exhibit extremely high yield strength, but also may show early failure due to the limited strain-hardening rate [12,13]. Therefore, the microstructure of the steel should also be considered in order to regulate the mechanical properties. Multi-phase steel consisting of both soft and hard phases is preferred to balance the strength and ductility through the interactions among different phases [14–16]. The mechanical response of the steel depends on the volume fraction, morphology, and distribution of different phases. For example, film- and blocky-like retained austenite exhibit high and low thermal and mechanical stability, respectively [17,18].

Apart from the contributions of different phases, precipitate strengthening has been proved as an effective strengthening mechanism in HSLA steels. Precipitates with coherent or incoherent microstructures exhibit a dislocation shearing mechanism or Orowan dislocation bypassing mechanism [19,20]. Higher coherency of the precipitate/matrix interface means better stability of the precipitate [21]. With the growth or coarsening of precipitates, the interface with the matrix shows a loss of coherency. The particle growth is mainly controlled by the interfacial energy, the solubility limit, and the diffusivity of...
the precipitates [21]. For example, Kapoor et al. [22] found that Cu precipitates show a slower coarsening rate compared with the coexisting NiAl-type precipitates in ferrite steel, as the segregation of Ni and Al at the Cu/Fe interface can lower the interfacial energy. Multicomponent carbides, i.e., (V,M)C, can show finer morphologies compared with the single element carbides, due to the lower solubility of the former in the matrix [23]. It was also reported that an incoherency interface can lead to a decreased surface energy, resulting in a decrease in strength [24,25]. Thus, a coarsening of precipitation should be avoided.

Recently, a new series of HSLA steels with balanced strength and ductility has been developed. The introduction of the in-situ nanoparticles during the casting process can strengthen the steel without sacrificing the plasticity of the materials. There is a lack of research on the strengthening mechanism of the combination of multi-phase and precipitates. In this study, the microstructure of the heat-treated steel was carefully investigated via scanning electron microscopy (SEM), electron backscattered diffraction (EBSD), and transmission electron microscopy (TEM). The contributions of different phases and the precipitations on the mechanical responses were discussed in detail.

2. Experimental Methods

The HSLA steel used in this study was received as as-rolled plates with a thickness of 24 mm. The chemical compositions of the HSLA steel determined by inductively coupled plasma atomic emission spectroscopy (ICP-AES) are listed in Table 1. The reheating temperature, the rolling temperature, and the rolling reduction rate are 1250 °C, 970–1150 °C, and 20–32%, respectively. The detailed manufacturing process can be found in Ref [26]. The heat treatment process was as follows. The as-rolled plate was firstly austenitized at 860 °C for 1 h followed by water quenching to obtain a martensitic structure. Then the plate was reheated at 720 °C for 1 h and water-quenched to get a microstructure which is a mixture of martensite and ferrite. Finally, the plate was tempered at 560 °C for 1 h.

Table 1. Chemical compositions of the HSLA steel (wt%).

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>V</th>
<th>Ti</th>
<th>Nb</th>
<th>Al</th>
<th>P/ppm</th>
<th>S/ppm</th>
</tr>
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<tr>
<td>0.06</td>
<td>0.08</td>
<td>1.27</td>
<td>0.53</td>
<td>4.71</td>
<td>0.53</td>
<td>0.03</td>
<td>0.007</td>
<td>0.06</td>
<td>0.09</td>
<td>74</td>
<td>14</td>
</tr>
</tbody>
</table>

Specimens were then cut from the heat-treated section for mechanical tests and microstructure observations by wire cutting. Specimens for mechanical tests were ground by SiC papers to 2000 grit progressively. To investigate the homogeneity of the mechanical properties, specimens at six different positions throughout the whole plate were chosen. The exact positions of each specimen were identified in Figure 1. Specifically, for microstructure investigation, specimens were obtained at P4. The dimension of dog-bone specimens for tensile tests was 1.5 mm in thickness, 3 mm in width, and 20 mm in gauge length. Cubic specimens with the dimension of 10 × 10 × 10 mm were prepared for SEM and EBSD observation. Thin foils with a thickness of ~0.4 mm were cut for TEM observation. Uniaxial tensile tests were conducted with a constant strain rate of 10^{-3} s^{-1} using a CMT 4105 machine (Jinan Liangong Testing Technology Co., Ltd., Shandong, China) at ambient temperature along the rolling direction (RD) and transverse direction (TD).

![Figure 1](image-url)
Samples for SEM observation were mechanically ground and polished. Samples for EBSD observation were mechanically ground and polished, followed by electrochemical polishing with 4 wt% nitric acid-ethanol. SEM/EBSD observation was carried out by a LEO 1450 scanning electron microscope (Leo, Oberkochen, Germany). The observation was focused on the TD-RD plane of the sample. The voltage was 20 kV and the step size was 0.1 µm. Obtained data were processed using HKL Channel 5.0 software. The reconstruction of prior austenite grains was performed based on EBSD data, using MTEX toolbox in MATLAB (version R2021b, MathWorks, Inc., Natick, MA, USA) [27]. For TEM observation, thin foils were mechanically ground to a thickness of ~80 µm. Discs with 3 mm diameter were then punched from thin foils, followed by ion-milling. TEM observations were performed in a JEM-2100 scanning transmission electron microscope (JEOL Ltd., Tokyo, Japan) at the acceleration voltage of 200 kV.

3. Results and Discussion
3.1. Mechanical Response

Figure 2 shows the mechanical properties of the heat-treated plates at different positions identified in Figure 1. Three dashed lines from the top to the bottom in Figure 2a–c represent the maximum, the average, and the minimum value, respectively. The mean value with the standard deviation of the yield strength, the elongation, and the yield ratio are presented. At first glance, the mechanical properties of the plate show a good consistency throughout the plate, as the yield strength, the elongation, and the yield ratio of the specimens are ranging from 827–852 MPa, 22–24%, and 0.9–0.93, respectively.

Since the specimen shows a very good homogeneous mechanical property, one specimen was chosen to study the stress–strain curve and the microstructure in detail. Figure 2d shows the stress–strain curves at uniaxial tension with a strain rate of $10^{-3}$ s$^{-1}$ for the HSLA steel specimens at P4 along the TD. The results indicate that the yield strength (YS), the ultimate tensile strength (UTS), and the total elongation (TE) at the break of the specimen are $830 \pm 1.77$ MPa, $928 \pm 9.6$ MPa, and $20.8 \pm 0.4\%$, respectively. The product of the strength and elongation (PSE) at break is ~19 GPa%, which is relatively high (see the red star in Figure 2e). On one hand, PSE is a property to represent the combination of strength and ductility. On the other hand, the HSLA steel shows high strength without
sacrificing ductility, whose position is located very close to the top right corner of the UTS-elongation map. Both of them indicate that the HSLA steel has a very good balance of strength and ductility.

3.2. Microstructure Observations

Figure 3 shows the SEM microstructure of the heat-treated sample. The structure mainly consists of tempered martensite, where carbides distribute randomly on the ferrite matrix. Apart from it, several pieces of blocky-form ferrite (highlighted by white dashed lines in Figure 3b) exist, which are considered formed during the heat treatment between Ac1 and Ac3 and the subsequent water quenching process. Figure 4 shows the EBSD maps of the heat-treated sample. The inverse pole figure (IPF) map shows that the grains exhibit random orientations based on the colors designated in the stereo projection triangle inserted in the top right corner in Figure 4a. Low-angle grain boundaries (LAGBs, defined as boundaries with a misorientation angle between 5–15°) and high-angle grain boundaries (HAGBs, defined as boundaries with a misorientation angle larger than 15°) are represented by white lines and black lines, respectively, in Figure 4a and b. To analyze the phase map in Figure 4b, it can be found both face-centered cubic (FCC) and body-centered cubic (BCC) structural phases appear in the HSLA steel. A fraction of the 6.53% FCC phase was calculated. This FCC phase is considered retained austenite, with a grain size ranging from 0.47 to 0.82 µm.

![Figure 3](image1.png)

**Figure 3.** (a) SEM image of the HSLA steel; (b) enlarged SEM image of the selected area enclosed by the dashed line rectangle in (a).

![Figure 4](image2.png)

**Figure 4.** EBSD maps of the HSLA steel: (a) inverse pole figure (IPF) map; (b) phase map of ferrite (blue) and retained austenite (red); (c) correlated misorientation angle distribution between ferrite variants; and (d) correlated misorientation angle distribution between retained austenite.
To further discriminate the BCC phase information, a band contrast (BC) map and grain average BC map were also drawn in Figure 5. Discriminations of different phases are generally dependent on the map quality. For example, martensite contains more defects, which show poorer EBSD map quality, and thus can be distinguished from ferrite, even though both of them are BCC structural phases. A detailed description of the method can be found in Ref. [28]. Figure 5a is the BC map, where bright bands and dark bands represent high-quality and low-quality patterns. Overall, the patterns show a relatively average level of quality, despite the poor image qualities at grain/phase boundaries. Figure 5b also verifies that no distinct threshold of BC is observed in the original BC profile. To avoid the influence of the poor image qualities at grain boundaries, the grain average BC map and profile were calculated in Figure 5c, d. No threshold is detected in the grain average BC profile, which suggests that the BCC phase in this study is a relatively homogeneous structure. Together with the heat retreatment process and the SEM image, we can conclude BCC phase should be tempered martensite.

Figure 4. EBSD maps of the HSLA steel: (a) inverse pole figure (IPF) map; (b) phase map of ferrite (blue) and retained austenite (red); (c) correlated misorientation angle distribution between ferrite variants; and (d) correlated misorientation angle distribution between retained austenite.

Figure 5. EBSD analysis for phase determination: (a) original BC map; (b) grain average BC map; (c) original BC profile; and (d) grain average BC profile.

Figure 4c, d shows the misorientation angle distributions in the ferrite (tempered martensite) and the RA regions, respectively. As can be seen in Figure 4c, the peaks are ranging around 0–10° and 50–60°, whereas there is a lack of misorientation angles ranging from 20–50°, which is a typical Kurdjumov–Sachs (K–S) orientation relationship that ferrite grains nucleate from the prior austenite grains. The austenite–austenite interface distribution in Figure 4d shows a sharp peak around 60°, where the angle is associated with the misorientation axis at <111> orientation. Thus, the peak indicates Σ3 twin boundaries in retained austenite regions.

Although most of the prior austenite grains were transformed into tempered martensite, a small amount of them were retained, staying at prior austenite grain boundaries/triple junctions (marked by black arrows) or within the tempered martensite (marked by white arrows) in Figure 4b. By using the MATLAB toolbox MTEX [27], the parent austenite can be reconstructed as shown in Figure 6a. To be noticed, the colors in Figure 6a do not represent the grain orientations but help to identify the prior austenite grains. Figure 6b and c show the normalized histograms of the deviation angles of the paired parallel planes and directions between austenite and ferrite based on orientation relationship models. By far, several orientation relationships (i.e., K–S, N–W, Bain, and Pitch) have been reported in austenite/ferrite transformation in steels, and the orientation relationships of these models are listed in Table 2 [10,29–31]. Therefore, if the deviation angle is close to zero, it means that the transformation can be well explained by the model. By comparing
the misfit angle distributions of all four models, the deviation angles for both the plane and the direction in the K–S model mainly range from 0–5°, which confirms that the K–S model can be adopted for the analysis of the phase transformation in these kinds of HSLA steels in this study.

![Figure 6](image)

**Figure 6.** Ferrite variant analysis: (a) reconstruction of prior austenite grains; (b–e) distribution of deviation angles from K–S, N–W, Bain, and Pitch OR models; (d) a prior austenite grain containing ferrite with complex interconnecting structure; (e) corresponding point-to-point misorientation angle distribution along the red arrow in (d); (f) K–S ORs simulated from experimental [001]bcc pole figure of the prior austenite grain in (d); (g) [001]bcc pole figure of the prior austenite grain in (d); (h) a prior austenite grain containing variant pairs; (i) corresponding point-to-point misorientation angle distribution along the red arrow in (h); (j) K–S ORs simulated from experimental [001]bcc pole figure of the prior austenite grain in (h).

<table>
<thead>
<tr>
<th>Name</th>
<th>Orientation Relationship</th>
<th>Planes</th>
<th>Directions</th>
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<tbody>
<tr>
<td>Bain</td>
<td>[100]γ // [100]α</td>
<td>&lt;100&gt;γ //&lt;110&gt;α</td>
<td></td>
</tr>
<tr>
<td>Pitch</td>
<td>[100]γ // [110]α</td>
<td>&lt;110&gt;γ //&lt;111&gt;α</td>
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One prior austenite grain was selected to study the variant selection in detail in Figure 6d. Figure 6f shows the K–S orientation relationships predicted from experimental data. Figure 6g shows the experimental [001] pole figure of the tempered martensite corresponding to Figure 6d. The positions of [001] planes of retained austenite are indicated by the symbol “A”. Variant pairs following K–S ORs (see the distributions in Figure 6f,g), which are commonly activated in low carbon steels [32] with small misorientations, can also be activated in certain prior austenite grains as shown in Figure 6d. The misorientation...
angle distribution across the prior austenite grain shown in Figure 6e confirms the existence of LAGBs. Even though some of them may be annihilated during the heat treatment process, an intense proportion remains (in Figure 4c,d). Previous studies have confirmed good strain compatibility of LAGBs in bi-crystal Cu alloys and that easy slip transmission across LAGBs occurs during cyclic loadings [33]. Based on this scenario, alloys containing large amounts of LAGBs are designed or fabricated to achieve both high strength and high ductility [34,35]. Therefore, we assume that LAGBs in the HSLA steel can also contribute to good plasticity. Unlike the variant pairs in Figure 6d, a complex structure containing a large number of HAGBs is observed in Figure 6h. It can be seen from Figure 6h that the retained austenite lies at the center of three “Bain circles”, confirming that the K–S orientation relationship fits well between the γ and α phase. Moreover, compared to the theoretical “Bain circle”, missing parts can be observed in the actual “Bain circles”, indicating that all K–S variants were not activated simultaneously. In other words, a variant selection occurs with favorable nucleation of some orientations. As can be seen in Figure 6h, different variants with distinct orientations impinge on each other rather than merge, i.e., ferrite grain G2 (Euler angles (105.0, 40.1 27.6)) grows until it reaches the boundary of ferrite grain G1 (Euler angles (246.8, 22.4, 59.7)). For further analysis, G1 and G2 belong to two different crystallographic packets (marked by black arrows in [001]bcc pole figure in Figure 6k) and the misorientation angle between them is 54.9° (Figure 6i). This type of interaction can lead to a complex interconnecting structure and further introduce a significant grain refinement, which in turn results in grain refinement strengthening. Furthermore, HAGBs among variants act to strain accommodation, which can further benefit crack tolerance [36]. In sum, the very complex matrix containing large amounts of HAGBs and LAGBs can result in the increment of strength and ductility.

To further analyze the good mechanical properties in the HSLA steel, TEM and HRTEM were performed to study the precipitation behaviors. Figure 7a is the bright field (BF) TEM image of the matrix. The selected area electron diffraction (SAED) patterns of the matrix area and the retained austenite are inserted at the top right and bottom right corner of the figure, respectively. The index confirms the ferrite matrix, which was taken along the [011] zone axis. Round-shaped retained austenite (marked by a dashed red circle) entangled with dislocations and round-shaped nano-sized precipitates (marked by red arrows) can be observed lying in the matrix. To be noticed, this kind of retained austenite was also detected by EBSD in Figure 4a,b, with a fraction of ~6.53%. Compared with film-like retained austenite, blocky retained austenite shows a higher phase transformation tendency during deformation [37,38]. Therefore, it is assumed that the round-shaped retained austenite shows poor stability and might transform easily to martensite during deformation, leading to an increase in elongation. Furthermore, the entangled dislocations around retained austenite indicate that retained austenite acts as a dislocation sink during the thermomechanical process. A very similar effect of retained austenite on absorbing dislocations during the deformation process has been confirmed in Q-P-T martensite/bainite steel [39,40]. Thus, both effects would be beneficial to the good ductility in the HSLA steel.

Apart from the retained austenite, round-shaped precipitates with a diameter of several nanometers are randomly distributed in the matrix (see the BF TEM image with a higher magnitude in Figure 7b). The strain incompatibility between the soft-retained austenite phase and the hard-tempered martensite phase can cause void initiation to increase the risk of fracture, whereas the introduction of randomly distributed precipitates in the matrix can synergistically improve the strength and plasticity [41,42]. High-resolution TEM (HRTEM) and EDS were conducted to determine the type of the precipitates in Figure 7c,d. By measuring the atomic spacing of the crystallographic planes, it is determined that the precipitate has a d-spacing of ~2.2 Å. Together with the EDS result shown in Figure 7d, the lattice plane is corresponding to the (200) plane of NbC. When the α-Fe is transferred from the γ-Fe, there is a Baker–Nutting (B–N) orientation relationship between MC-type carbide and α-Fe, where (100)MC is parallel to (100)α-Fe [43]. Therefore, a lattice misfit of ~23.1%
between (100)\(\text{NbC}\) and (100)\(\alpha\)-Fe can be calculated, which is similar to the semi-coherent relationship between NbC precipitate and \(\alpha\)-Fe matrix, with a theoretical lattice misfit of 29.2\% reported in previous studies [44,45]. It is believed that the semi-coherent precipitation interface with the matrix is beneficial to obstructing incoming dislocations, which in turn improves the strength [46]. However, the interactions between the nano-sized precipitations and the dislocations are dynamic processes, thus it is rather difficult to understand the interactions via ex-situ characterization methods. Both the shearing mechanism and Orowan bypass mechanism can contribute to the strengthening, depending on the size of the precipitations. Seidman et al. [47] reported a transition from the shearing mechanism to the Orowan bypass mechanism as the precipitate size is over 2.1 nm. As can be seen in Figure 7b,c, the size of most of the NbC precipitates is ~10 nm, therefore, the Orowan bypass mechanism might dominate. We assume that the homogeneously distributed NbC nanosized precipitates in \(\alpha\)-Fe contribute to the high strength of the HSLA steel.

**Figure 7.** Precipitates in the HSLA steel: (a) BF TEM image; (b) distribution of the precipitates; (c) HRTEM of the precipitate; and (d) EDS analysis of the area A in Figure 7b.

### 4. Conclusions

In this study, the microstructure of HSLA steel with a balanced strength–ductility mechanical property was investigated by SEM, EBSD, and TEM. The ultimate tensile strength and the elongation at break are 928 ± 9.6 MPa and 20.8 ± 0.4\%, respectively. The microstructure consists of both hard and soft phases, containing a large fraction of LAGBs. Phase transformation during the thermomechanical process obeys K–S orientation relationships. Variants with distinct orientations interact with others belonging to different crystallographic packets, forming a complex interconnecting structure. The homogeneously distributed NbC precipitates, showing an incoherent relationship with the matrix, contribute to the high strength. It is proposed that the multi-phase microstructures containing homogeneously distributed NbC precipitations might be responsible for the balanced mechanical properties, yet further studies are needed to investigate the evolution of multi-phase microstructures during deformation and the interactions between NbC and dislocations.
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Conflicts of Interest: The authors declare that they have no known competing financial interests or personal relationship that could have appeared to influence the work reported in this paper.

References


43. Pushkareva, I.; Allain, S.; Scott, C.; Redjaimia, A.; Moulin, A. Relationship between Microstructure, Mechanical Properties and Damage Mechanisms in High Martensite Fraction Dual Phase Steels. *ISIJ Int.* 2015, 55, 2237–2246. [CrossRef]


