Dual-scale correlation of mechanical behavior in duplex low-density steel

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The deformation behavior of duplex low-density steel was analyzed by correlation between macroscale uniaxial tension and nanoscale indentation. A dramatic difference in the tensile behavior was observed between two specimens obtained under specific heat-treatment conditions, despite these specimens having the same chemical composition and similar microstructures. In order to understand this difference, the intrinsic mechanical properties of each phase were analyzed based on nanoindentation results considering the Hall–Petch relationship. In addition, the mechanical stability of retained austenite was investigated by in situ electron backscattered diffraction.

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Reducing carbon dioxide emissions and improving fuel efficiency have become global issues for the automotive industry. Accordingly, much research has been devoted to developing many kinds of advanced high-strength steels (AHSSs), such as dual-phase and transformation-induced plasticity steels, in order to reduce the weight of automotive parts [1–4]. Interest in low-density steels with high strength and toughness is emerging in automotive applications, and many kinds of low-density steel have been developed [2,5–8]. Among the low-density AHSSs, duplex low-density steels containing light substitutional elements have attracted attention as these offer a balance between strength and ductility [2,5–7]. These duplex steels, which are composed of ferrite and retained austenite, show complex deformation behavior, which arises from the interactions among the different microstructural constituents. To ensure reliable alloy and processing designs of the duplex low-density steels, it is necessary to understand the influence of the intrinsic behavior of their microstructural constituents on the macroscale deformation behavior, including the plastic yielding and deformation-induced martensitic transformation (DIMT) of retained austenite [5,9].

In this study, a dramatic difference in the macroscale tensile behavior in terms of the yield strength, yield point and strain hardening was observed in two specimens of duplex low-density steel with the same nominal chemical composition, obtained under specific heat-treatment conditions. In order to understand the difference in the macroscale mechanical behavior despite the similar microstructures, the intrinsic mechanical properties of each phase were measured by nanoindentation and analyzed according to the Hall–Petch relationship. In addition, the mechanical stability of retained austenite was investigated by in situ electron backscattered diffraction (EBSD) and energy-dispersive X-ray spectroscopy (EDX) combined with transmission electron microscopy (TEM).

An alloy with a chemical composition of Fe–0.23C–8.1Mn–5.3Al–0.01Si (wt.%) was prepared by vacuum induction melting. The specific process conditions of the alloy have been presented in our previous work [6]. Two low-density steel specimens were prepared for experiments by cold-rolling and annealing at 800 °C (T800) and 900 °C (T900) for 2 min in an infrared heating furnace. The macrosopic tensile behaviors of both
steels were measured with sub-sized specimens according to ASTM-E8 M.

Nanoindentation tests were performed using a Hysitron Tribolab nanoindentation system in load control at a constant loading rate of 200 μN s⁻¹. A Berkovich-type indenter with a half angle of 65.3° was used. To investigate the intrinsic mechanical properties of each phase in T800 and T900 without the grain boundary effect, two different indentation methods were applied for each phase in two specimens. For δ-ferrite, which has a large grain size compared to the size of indenter tip, 100 indentations were performed in a 10 × 10 rectangular array with a spacing of 5 μm between each indentation. To exclude the grain boundary effect, only the indentation marks located inside the δ-ferrite grains with sufficient distance from the grain boundaries were selected for the analysis, based on the EBSD band contrast map of nanoindentation regions [10]. In the case of α-ferrite and austenite grains with smaller grain size, a scanning probe microscope (SPM) was used in the nanoindentation system, which made it possible to evaluate the grain morphologies and to precisely identify the areas that had been previously analyzed by EBSD [11]. Guided by the EBSD and SPM images, individual α-ferrite and austenite grains were selected for nanoindentation.

The stability of the retained austenite grains in both specimens during tensile deformation was analyzed using in situ EBSD. The microstructure change along with DIMT phenomena was measured during tensile deformation inside the chamber of a scanning electron microscope [12]. The chemical composition in the retained austenite region inside the chamber of a scanning electron microscope was determined from the nanoindentation of each phase in the specimens. The dashed lines on the curves are the theoretical Hertzian elastic solution, assuming that the indenter tip is spherical at shallow depths [13]:

\[ P = \frac{4}{3} E_s \sqrt{Rd^3}, \]

where \( P \) is the load applied by indenter, \( R \) is the radius of indenter tip and \( E_s \) is the effective modulus. \( R \) was determined to be 200 nm from a calibration using standard fused quartz. \( E_s \) is obtained from the unloading portion of the load–displacement curve fitted with a power law [14]. In the loading curves of δ- and α-ferrites of both specimens, sudden displacement excursions, called “pop-ins”, are observed for most indentations. Before the pop-ins, the experimental loading curves match the theoretical curve well, indicating an elastic response before pop-in. Clear pop-in is not detected in the loading curve of the austenite phase in both specimens. It is known that this pop-in phenomenon is closely related to the dislocation nucleation at the elastoplastic transition, and the difference in pop-in phenomenon between ferrite and austenite arises from the different mechanism of dislocation nucleation in body-centered cubic and face-centered cubic structures [15,16].

With regard to the initial yield strength of each phase, the maximum shear stress underneath the indenter, \( \tau_m^0 \), at the elastoplastic transition might become a parameter with which to compare the macroscale mechanical behavior with the nanoscale indentation. It can be calculated from Hertzian analysis [13]:

\[ \tau_m^0 = 0.31 \left( \frac{6P_{m}E_s^2}{\pi^3R^2} \right)^{1/3}. \]

Figure 1 shows EBSD phase maps of T800 and T900, respectively. Both steels contain δ-ferrite with layered structure along the rolling direction, small sized α-ferrite, and metastable austenite (γ). The bimodal size distribution in ferrite grains is caused by aluminum which stabilizes ferrite to remain stable even at high temperatures where hot-rolling was performed. The remaining elongated coarse phase is δ-ferrite, while the small-sized α-ferrite is formed by annealing after cold-rolling. The grain size of each phase and the volume fraction of austenite in specimens obtained from the EBSD measurements are listed in Table 1. The grain size of δ-ferrite is about 10 μm, and the grain sizes of α-ferrite and austenite are <1 μm in both specimens. T900 has larger grain sizes of α-ferrite and austenite, a smaller grain size of δ-ferrite, and a larger austenite fraction compared to T800.

Table 1. Grain size of each phase and austenite fraction of the alloys obtained from EBSD.

<table>
<thead>
<tr>
<th>Sample</th>
<th>δ-Ferrite (μm)</th>
<th>α-Ferrite (μm)</th>
<th>Austenite (%)</th>
<th>( f_t ) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T800</td>
<td>10.90</td>
<td>0.68</td>
<td>0.54</td>
<td>36.7</td>
</tr>
<tr>
<td>T900</td>
<td>8.62</td>
<td>0.79</td>
<td>0.70</td>
<td>41.4</td>
</tr>
</tbody>
</table>
where $P_m$ is the first load beyond which Hertzian elastic behavior is not observed. The $\tau_m^0$ values of all phases in the specimens were determined to be in the range 4.9–8.3 GPa, which corresponds to $G/10$ to $G/16$, where $G$, the shear modulus of steel at room temperature, is about 80 GPa [17]. These values of $\tau_m^0$ are within the range of values for the theoretical strength of a crystalline material [18], indicating that the elastoplastic transition in the specimens are probably not the result of movement of the existing dislocations, but of dislocation nucleation.

Since $\tau_m^0$ was determined from measurements inside each grain, the effect of grain size on yielding should be considered in order to evaluate the substantial initial yield stress of each phase from the viewpoint of the nanoindentation scale. The well-known Hall–Petch relationship was combined with the intrinsic $\tau_m^0$ at the elastoplastic transition measured by nanoindentation as follows:

$$\tau_m = \tau_m^0 + 0.5b\kappa_y/\sqrt{d},$$

where $\tau_m$, $d$ and $\kappa_y$ are the maximum shear stress reflecting the grain size effect, the mean grain size and the Hall–Petch constant, respectively. Since the Hall–Petch constant is normally obtained under a uniaxial stress state, a conversion factor of 0.5 was applied to the shear stress state based on the Tresca yield criterion. The constant, $b$, reflects the almost 10-fold difference between the maximum shear stress for a single grain (4.9–8.3 GPa) and the yield stress of polycrystalline material (561 and 718 MPa in Fig. 1). In fact, the value of $b$ should depend on the indenter radius due to size-dependent pop-in stresses [19]. Here, $b$ is assumed to be 10. It is generally known that austenite has slightly higher $\kappa_y$ values than ferrite in steel [20,21]. $\kappa_y$ values reported for duplex stainless steels of 0.21 MPa m$^{0.5}$ for ferrite and 0.32 MPa m$^{0.5}$ for austenite were used [20]. The above equation is valid under the assumption that the plastic yielding is mostly controlled by dislocation nucleation rather than movement of existing dislocations.

Since the specimens are well annealed, Eq. (3) is expected to work effectively.

Figure 2a,b shows the maximum shear stresses underneath the indenter upon initial yielding for each phase in the T800 and T900 specimens, respectively. The solid and dashed lines indicate the theoretical Hertzian elastic solution. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
ing. A sub-grain boundary, defined by a misorientation angle of $2^\circ-5^\circ$, is exclusively observed in a small number of δ-ferrite in T800. This comes from the slightly insufficient recovery of T800 during the annealing process in comparison with T900.

Figure 4e shows that DIMT is detected in T900 at only 3.9% strain, and increased as the macroscopic strain is increased, whereas DIMT could not be observed in T800 at up to 12.4%. The differences in micro-structural changes during the deformation of the two specimens can be explained by the mechanical stability of retained austenite in both specimens. The mechanical stability of retained austenite is usually ascribed to its size and shape [22], chemical composition [23], partitioning of strain [24] and crystallographic orientation [7]. Of these, the crystallographic orientation is likely to be a minor factor in this research, because the austenite phases in both specimens have similar texture distributions. The grain size of the retained austenite in T800 is smaller than that in T900, and hence T800 has higher mechanical stability of the austenite phase with respect to grain size. In addition, since the initial yielding in T900 is controlled by the retained austenite phase according to the analysis of $\tau_m$, the strain might concentrate in the retained austenite phase in T900 during tensile tests, resulting in the DIMT at an early stage of the deformation of T900. However, since the weakest phase in T800 is δ-ferrite, the strain of austenite in T800 is smaller than that of δ-ferrite. Therefore, T800 appears to be better at protecting austenite grains by strain partitioning than T900.

The difference in austenite stability between T800 and T900 can also be understood in terms of the difference in chemical composition. TEM/EDS and XRD analyses gave compositions of 0.551 wt.% C–9.18 wt.% Mn and 0.451 wt.% C–8.99 wt.% Mn for T800 and T900, respectively. It is confirmed that the austenite phase in T900 has a lower content of C and Mn compared to T800, and these elements are austenite stabilizers. This indicates that the intrinsic mechanical properties of austenite in T900, which is relatively dilute, are softer than those in T800 as shown in Figure 3. In addition, the lower C and Mn content of the austenite in T900 makes the austenite less stable than that in T800.

In this study, a dual-scale correlation between macro-scale tensile test and nanoscale indentation of a duplex low-density steel was carried out, in order to explain the dramatic differences in macro-scale tensile behavior in two specimens obtained under specific heat-treatment conditions. From nanoindentation, first, the maximum shear stress at the elastoplastic transition for each phase in both specimens was determined. The effect of grain size on yielding was considered in order to evaluate the substantial initial yield stress of each phase from the viewpoint of the nanoindentation scale using the Hall–Petch relationship.

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