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Influence of nanoparticle reinforcements on the strengthening mechanisms of an ultrafine-grained dual phase steel containing titanium

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ABSTRACT

To reduce cost, optimise mechanical properties and improve process tolerance, a series of 1000 MPa grade ultrafine-grained dual phase (DP) steels with nanosized precipitates have been developed based on the C–Si–Mn–Ti alloy system. The grain size of ferrite in ultra-high strength DP steels ranges from 1.1 to 1.7 μm. The amount of precipitations in the annealed sheet is much more than that in the hot-rolled plates with the highest distribution frequency being between 5 and 10 nm. The grain refinement and precipitation strengthening interact significantly and have a considerable effect on yield strength. Therefore, the strengthening effects cannot be expressed as a simple linear relationship. A modified root-mean-square (RMS) relationship has been proposed to express the yield strength for dual phase steel with obvious grain refinement and precipitation strengthening.

1. Introduction

In automotive industries, reducing weight, increasing performance and rationalising production methods are motives for the development of advanced high-strength (AHSS) and ultra-high-strength steels (UHSSs) [1–3]. As a type of AHSS, dual phase (DP) steel contains soft ferrite with hard dispersed martensite islands. The enhancements in formability with these strength and ductility combinations make DP steels strong candidates for structural applications [4–7].

There are many strengthening mechanisms for DP steels, such as solid–solution strengthening, phase strengthening, refinement of grain size strengthening and precipitation strengthening. Huang and Gwo [8] studied Fe–2%Si–1.5%Mn steels with three levels of carbon content (0.10, 0.14 and 0.19 wt%), indicating that the ultimate tensile strength of DP steels increased as the volume fraction and the tensile strength (carbon content) of martensite increased. Although this is the simplest way to strengthen DP steel, higher carbon content plays a negative effect on its weldability and surface quality. The 1000 MPa grade DP steels have been produced by a continuous annealing line cooled by a water-quenching process [9,10]. The quenching rate is 1000–2000 °C/s, and as a result the carbon content could be less than 0.13%. However, the tremendous cooling rate limits the thickness of plates, and the water-quenching equipment is not commonly available in factories around the world.

Delince et al. [11] developed fine-grained DP steels produced by swaging to investigate the physics-based model for strain hardening. The grain size is between 1.5 and 2.4 μm. Calcagnotto et al. [12] obtained the fine-grained (2.4 μm) and ultrafine-grained (1.2 μm) ferrite/martensite DP steels by large strain warm deformation at different temperatures and subsequent intercritical annealing. The ultimate tensile strengths are 964 and 1037 MPa for the 2.4 and 1.2 μm sized DP steels, respectively. However, swaging, large strain warm deformation combined with subsequent intercritical annealing and equal channel angular pressing [13] are not efficient for industrial production.

Microalloyed steels generally contain Nb, V, or Nb, V, Ti elements, which could precipitate to retard austenite recrystallization and grain growth, and play a significant role in precipitation strengthening [14–17]. Titanium is beneficial because it combines with nitrogen at relatively high temperature, preventing grain growth. Niobium can effectively retard recovery and recrystallization during the hot rolling leading to ferrite grain refinement. Vanadium is less effective in grain refinement but behaves in a manner similar to niobium [18]. For example, Ti–Mo-bearing high strength low alloy (Nanohiten) sheet steels exhibit high tensile strength up to 780 MPa with excellent formability by producing a ferritic matrix structure mixed with nanosized carbides [19]. The precipitation strengthening is two or three times higher than

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that of conventional HSLA hot-rolled sheet steels. Chen et al. [20] reported that titanium molybdenum complex carbide ((Ti, Mo)C, ~5 nm) can strongly maintain nanoscaled sizes and has the largest contribution to hardness as compared to titanium carbide (TiC, ~15 nm) and titanium niobium complex carbide ((Ti, Nb)C, ~10 nm) fabricated by hot rolling. However, due to the high price of molybdenum, niobium and vanadium, the development of titanium microalloyed steels is still much more economically viable.

In this present study, therefore, to reduce cost, optimise mechanical properties and improve process tolerance, we attempted to investigate the microstructure, mechanical properties and its dependence on processing parameters in C–Si–Mn–Ti ultra-high strength ultrafine-grained ferrite/martensite dual phase steels with nano precipitates. A modified model for strengthening mechanism was also proposed to express the yield strength of experimental steels as function of lattice friction stress, solid solution, grain refinement, precipitation and dislocation.

2. Experimental procedure

The chemical composition of the experimental steel in weight percent is C (0.16%), Si (0.62%), Mn (1.94%), Ti (0.082%), S (0.0062%), P (0.011%), N (0.0032%), and Als (0.06%). Experimental steels were smelted in a 50 Kg vacuum induction furnace. Forging billets were heated to 1200–1250 °C for 30 min, and then hot-rolled from 60 to 5 mm, with the coiling temperature being about 560 °C. The as-rolled steels were pickled and cold rolled to 1.2–2.3 mm.

A Gleeble-3800 thermo-mechanical simulator was used to simulate the annealing process. Cold-rolled plates were heated to 780–820 °C and held for 100 s, then slowly cooled to 680 °C. Next, the samples were fast cooled to overaging temperature (170–280 °C) by gas-jet cooling. The cooling rate was approximately 35 °C/s. There were six different annealing processes. The corresponding relation between soaking temperature and overaging temperature during continuous annealing is shown in Table 1. Other annealing parameters were identical to study the effect of soaking and overaging temperature on the microstructure and properties of DP steels.

The specimens for microstructure characterization were prepared by grinding down to 2000 grit sand paper, and then mechanically polished by polishing cloth with diamond paste of 3 μm and 1 μm. Specimens were etched with 4% Nital for 15 s to provide enough contrast of martensite and ferrite under optical microscopy (OM) and scanning electron microscopy (SEM, Supra 55 VP). The volume fraction and grain size of martensite were estimated by Image Tool Software. The volume fraction of retained austenite was investigated by electron backscattered diffractometry (EBSD, HKL channel 5). Carbon extraction replicas were analysed by transmission electron microscopy (TEM, JEM-2010) and energy dispersive X-ray fluorescence spectrometer (EDX). Precipitates in Ti microalloyed steels were obtained by electrolysation and extraction, and then X-ray diffraction was used to determine the phase constitution. The mass fraction of precipitates was quantitatively measured by using the chemical method [21,22]. Besides, the nano precipitates size distribution was analysed through 3014 X-ray diffracto-spectrometer/Kratky small angle scattering goniometer [23].

Table 1
Schedules of continuous annealing process.

Annealing process	Soaking temperature (°C)	Overaging temperature (°C)
L1	780	280
L2		240
L3		170
L4	820	280
L5		240
L6		170

3. Results

3.1. Mechanical properties

A series of ultra-high strength dual phase steels were developed based on C–Si–Mn–Ti alloy system, including high strength low ductility steel and low strength high ductility steel fabricated by various annealing processes. Fig. 1 shows the change in tensile properties with annealing parameters in C–Si–Mn–Ti DP steels. The annealing processes L2 and L5 correspond to high strength (HS) and high ductility (HD) steels (Fig. 1), respectively. The annealed samples at a higher soaking temperature (820 °C) show a lower tensile strength (TS), lower yield strength (YS, also called $\sigma_{p0.2}$), higher elongation (El_{50}) and higher yield ratio (YR) than those annealed at a lower soaking temperature (780 °C). Meanwhile, a higher overaging temperature increases the ductility and reduces the TS, YS, and YR. The microstructure and mechanical properties of HS and HD steels are summarized and compared to the hot-rolled acicular ferrite and pearlite steel with a high yield ratio of 0.90 obtained in this study. However, after being cold-rolled and annealed, both HS and HD steels show a notably low yield ratio of less than 0.55. As shown in Fig. 1, the YR is as little as 0.49 for the sample processed at L3, which means that the gas-jet cooling annealing process tends to produce the DP steel with a low yield ratio of 0.49–0.55, which is much lower than that processed by water quenching (0.63–0.93 [9]).

3.2. Microstructure

Fig. 2 shows the final microstructure of high strength and high ductility DP steels fabricated by different annealing processes. The optical micrographs (Fig. 2a and b) show a uniform ferrite phase (bright contrast) with an embedded martensite phase (dark contrast), which was also confirmed by SEM (Fig. 2c and d) and TEM (Fig. 4a and b) images. The change in volume fractions of martensite and austenite with soaking–overaging temperature (Fig. 3) shows that the volume fraction of martensite in low soaking temperature steels at 780 °C ranged from 66.3% to 68.9%, which is higher than that in high soaking temperature steels at 820 °C (55.8–58.9%). The relatively higher volume fraction of martensite in HD steel (~68.2%) than that in HS steel (~58.9%), as shown in Table 2, is mainly because the high soaking temperature increases the volume fraction of austenite and reduces the alloy element unit volume, which promotes the formation of epitaxial ferrite during cooling in annealing process. Meanwhile, the overaging temperature has a slight influence on the volume fraction austenite for all experimental steels. The volume fraction of austenite has been determined to be less than 1% by EBSD analysis.

On the other hand, the mean grain sizes of ferrite are measured to be 1.1 μm and 1.7 μm for HS and HD steels, respectively. This formation of ultrafine-grained ferrite in both steels is known to be primarily attributed to grain refinement induced by Ti addition, the high dislocation density and low-angle grain boundaries as a result of cold-rolled acicular ferrite. It was worth noting that the martensite islands become dominant in HS steel, whereas such islands are mainly replaced by the decomposed martensite in HD steel. This was associated with an inhomogeneous carbon distribution in different austenite regions formed during the intercritical annealing treatment of DP steels [24]. The tempered martensite was generated by low carbon content austenite. As the austenite