Development of a New Ultrafine/Nano Ferrite-Carbide Microstructure by Thermomechanical Processing

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Abstract The steel with the new microstructure, bimodal submicrometer equiaxed ferrite grains with uniformly distributed nanosized cementite particles, was manufactured by a new approach utilizing simple cold-rolling and subsequent annealing of a dual phase ferrite-martensite starting structure. The mean ferrite grain size and carbide size range of the specimen after 80% cold-rolling and subsequent annealing at 600 °C for 20 min were 0.35 μm and 70–140 nm, respectively. A combination of bimodal ultrafine ferrite and nanoscale carbides used as a more effective method for achieving an excellent balance in strength-ductility. The strength of the steel with the new microstructure increased to about 880 MPa (nearly 60% higher than that of the as-received state, e.g., 540 MPa), without significant loss of ductility.

KEY WORDS: Low-carbon steel; Dilatometry; Thermomechanical processing; Grain refinement; Mechanical properties

1 Introduction

Low-carbon steels containing carbon less than 0.2 wt% are the most widely used ferrous alloys in structural application. These steels consist of ferrite of large volume fraction with pearlite as the remainder and exhibit strength of ~ 400 MPa. To date, considerable effort has been devoted to enhancing the strength of these steels. Among the strengthening mechanisms, grain refinement strengthening is one of the effective methods of increasing strength without loss of ductility and toughness [1]. Several processing techniques have been developed to induce grain refinement in these steels. For example, Shin et al. [2] have used equal channel angular pressing (ECAP), Lei et al. [3]

and Ohmori et al. [4] have used warm deformation. But the potential of these processing routes to be employed industrially is very slim due to the steps involved in these individual processes. ECAP has limitation on sample size and shape; huge deformation at high temperatures is a major drawback of warm-rolling.

Several studies have attempted to achieve grain refinement by rolling and annealing of carbon steels with various carbon contents and starting microstructures. In the cases of ferrite-pearlite [5], martensite [6, 7] and tempered martensite [4] as starting microstructures before rolling, submicrometer ferrite microstructures have been obtained; but the ferrite grains contained residual dislocations inside. The dislocation density was decreased by annealing at elevated temperatures, but ferrite grain coarsening over 1 μm occurred simultaneously.

According to the above researchers, grain refinement was attempted by changing starting microstructures and processing ways. However, existing methods of improving strength are limited by the counter effect of ductility and toughness loss. Some research shows that the bimodal grain size distribution (BGSD) [8–12] and nanoscale

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second-phase precipitates [13] have been successfully used for improving the ductility of nano/ultrafine grain (UFG) metals.

The present investigation was aimed at creating a new microstructure for low-carbon steel, which was BGSD–UFG ferrite and nanosized carbide particles, exhibiting high strength without loss of ductility. The process was characterized by the starting microstructure before cold-rolling, which was a ferrite-martensite dual phase (DP) structure. In particular, the effect of rolling reduction and annealing time on microstructure evolution and mechanical properties determined to evaluate the processing window for the proposed thermomechanical stages.

2 Experimental

2.1 Materials

The steel used in this investigation was AISI 5115 with the chemical composition presented in Table 1. The material was received as a hot forged plate with the thickness of 6 mm. Hot forged sheets were cut to several specimens with 50 mm × 30 mm × 6 mm dimensions.

2.2 Thermomechanical Processing

Thermomechanical processing route performed on low-carbon steel to develop fine grained ferrite-carbide aggregates is shown in Fig. 1a. At first, the ferrite-pearlite structure was austenitized at 880 °C for 30 min in an electrical furnace followed by water quenching (WQ). In order to prevent severe decarburization, cast iron swarfs

were used to protect samples during heating. This stage was aimed at developing a fine martensite structure. Since martensite is a hard phase, cold-rolling (CR) operation without cracking is not possible. Therefore, the same austenitization treatment and intercritical annealing step (770 °C for 100 min) were introduced prior to cold-rolling to obtain a DP structure. In this study, the A_1 and A_3 temperatures (The lower and upper temperatures of two-phase ferrite and austenite region) were determined by a dilatometry experiment, as shown in Fig. 1b. The resulting DP structure was subsequently 50, 65, and 80% cold-rolled in a laboratory mill with rolling speed of 350 r/min and reduction of about 0.05 mm at each pass. Cold-rolled sheets were then annealed at 600 °C for 20 min and water quenched. This processing procedure resulted in ultrafine/nano ferrite-carbide microstructure. In addition, to fabricate a coarse-grained ferritic microstructure for comparison, the 50% cold-rolled sheet was annealed at 600 °C for 40 min.

2.3 Characterization

Microstructural analysis was carried out along the transverse direction (TD), i.e., the plane perpendicular to both the rolling direction (RD) and normal direction (ND). These sectioned specimens were mounted, ground, and polished till 4,000 grit finish followed by polishing with one micrometer alumina suspension and etched in 2% nital solution. Microstructures were characterized using Philips XL30 scanning electron microscopy (SEM). Grain and carbide particle size measurements were conducted using Image J software.

Hardness measurement was performed using a Vickers microhardness tester under a load of 10 N and dwell time of 5 s. Tensile specimens (15 mm gage length, 5 mm width, 1.5 mm thickness) were machined in RD, using electro discharge machining method. Tensile tests were carried out at a constant cross head speed of 1 mm/min with Hounsfield H50KS machine.

Table 1 Chemical composition of the investigated steel (wt%)

Element	C	Si	Mn	Cr	S	P
Composition	0.17	0.4	1.15	0.95	0.035	0.025

Fig. 1 **a** Thermomechanical processing developed to produce ultrafine/nano ferrite-carbide microstructures **b** dilatometry test experiment upon continuous heating by the rate of 1 °C/s

3 Results and Discussion

3.1 Microstructural Characterization

Figure 2a represents the as-quenched SEM microstructure of steel. This martensite structure is predominantly lath type because of its carbon content which is lower than 0.6 wt% [14]. In this SEM microstructure, a fine and complicated morphology of packets and blocks of the martensite is observed. The Vickers hardness of this initial martensite structure was about 368 HV. This made it difficult to perform cold reduction without cracking. Therefore, the process followed by intercritical annealing to develop the softer starting microstructure before rolling. Intercritical annealing followed by water quenching resulted in a DP microstructure composed of ferrite matrix and martensite islands (Fig. 2b). The ferrite and martensite volume fractions were 44 and 56%, respectively. For this starting DP structure the hardness dropped to about 249 HV, i.e., a significant drop of over 100 HV.

The 80% cold-rolled DP structure is shown in Fig. 2c. The ferrite matrix (dark gray region) exhibited a wavy microstructure elongated roughly in the RD and bent along the martensite islands (light gray region). This indicates that a complex plastic flow has occurred and higher strain has been introduced in the softer ferrite matrix due to the existence of the hard martensite phase. Such a strain

distribution between the soft phase and the hard phase is the key factor for the formation of ultrafine/nano ferrite-carbide microstructure in the present thermomechanical processing. As shown, the martensite islands were also deformed to some extent in cold-rolling. The strain introduced into martensite should be smaller than that into ferrite matrix, because martensite is much harder than ferrite.

After annealing at 600 °C for 20 min ultrafine/nano ferrite-carbide microstructures were obtained, as shown in Fig. 2d, e. The cold-rolled microstructure changed to equiaxed UFG ferrite including fine cementite particles during annealing. The cementite particles were nearly spheroidized and homogeneously dispersed within the UFG ferrite matrix. The average ferrite grain size (d_F) of the specimen was 0.35 μm . Some coarse grains nearly 1 μm seen in the lower part of Fig. 2d, probably formed mainly by recovery of ferrite regions such as the elongated and bent ferrite seen in the lower part of Fig. 2c. Those ferrite regions in the cold-rolled microstructure seem to be deformed to smaller plastic strain because of the relatively low density of surrounding martensite islands. Furthermore, the fact that the coarse ferrite grains in the annealed microstructure contained few cementite particles suggests that they were originally ferrite.

The cementite particles were much finer than the ferrite grains. There is a range of carbide sizes. Larger particles

Fig. 2 SEM microstructures of: **a** as-quenched, **b** intercritical annealed, **c** 80% cold-rolled, **d, e** annealed at 600 °C for 20 min steels produced by the processing routes illustrated in Fig. 1 (*F* ferrite, *M* martensite, *C* carbide, *RD* rolling direction, *ND* normal direction)

Table 2 Average grain sizes and mechanical characterizations of the steels developed under different conditions

Rolling reduction (%)	Annealing time (min)	d_F (μm)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
50	20	0.98	615 ± 5	643 ± 7	9.4 ± 0.6	19.9 ± 0.6
	40	1.48	544 ± 6	633 ± 7	8.7 ± 0.3	19.5 ± 0.5
65	20	0.64	706 ± 4	723 ± 7	5.8 ± 0.2	18.2 ± 0.8
80	20	0.35	880 ± 5	880 ± 5	7.6 ± 0.4	12.4 ± 0.6

d_F average ferrite grain size, YS: yield strength, UTS: ultimate tensile strength, UE: uniform elongation, TE: total elongation

Fig. 3 **a** Engineering stress–strain curves of the steel specimens obtained by various thermomechanical conditions and **b** relationship between ferrite grain size and yield strength

with an approximate average size of ~ 140 nm were mainly located at ferrite grain boundaries (Fig. 2d). Finer carbide particles with an average size of ~ 70 nm were generally distributed inside the grains (Fig. 2e). These results show a bimodal size distribution of ferrite grains and carbide particles. It is hard to find the present microstructure in the literature for low-carbon steels. The cases comparable to the present microstructure are the structures obtained through severe plastic deformation (SPD) and subsequent annealing [1, 15]. However, the ferrite grain size in these steels was much larger than that of the present new microstructure.

On the other hand, UFG ferritic microstructures were obtained by relatively low plastic strain through conventional cold-rolling in the present process, but an extremely high equivalent strain of over 4 or 5 is required to produce the UFG microstructure through SPD [2, 16].

The average ferrite grain size (d_F) of the produced steels under different conditions of rolling reduction and annealing time are listed in Table 2. After 50% rolling reduction, the d_F of the sample decreased by increasing annealing time from 20 to 40 min. It seems that increasing the d_F , can be related to the ferrite grains growth with increasing processing time [17]. In case of the 65% and 80% cold-rolled samples, d_F was smaller than that of 50%

rolling reduction. The structure became finer with increasing rolling reduction, because of the heavier prior deformation. Therefore, according to microstructural characteristics the finest structure can be achieved by annealing of the 80% cold-rolled DP microstructure at 600°C for 20 min.

3.2 Mechanical Properties

The engineering stress–strain curves and the corresponding mechanical characterizations are presented in Fig. 3a and Table 2, respectively. Yield strength (YS) and ultimate tensile strength (UTS) of the 80% cold-rolled sample after annealing at 600°C for 20 min reached about 800 MPa equivalent to over 60% and 150% enhancement compared to that of the as-received state (YS: 350 and UTS: 540 MPa), respectively. It is quite noticeable that, in spite of much higher strength, the steel with the new microstructure showed not only similar total elongation (TE) but also larger uniform elongation (UE) compared to the as-received steel (UE = 5.5% and TE = 12.5%). This means that significant strengthening by grain refinement was achieved in the present UFG steel. In agreement to other researches [8–13], a combination of BGSD-UFG ferrite and dispersive nanoscale carbides has been successfully

used for achieving an excellent balance in strength-ductility of low-carbon steel. Ohmori et al. [4] and Song et al. [18] also reported that finely dispersed globular cementite is beneficial in improving strength and ductility by enhancing the work-hardening rate of UFG steels.

Azizi-Alizamini et al. [19] fabricated a ferritic microstructure with bimodal ferrite grain size distribution by 50% cold-rolling and substantial annealing of a ferrite-martensite DP microstructure. The microstructure they obtained was a mixture of 40 vol% fine grains with size below 2 μm and 60 vol% coarse grains with sizes of 3–15 μm , but the strength of the material was unfortunately not so high (tensile strength of 550 MPa). This is probably because the rolling reduction they took (50%) was smaller than the present case (80%), and not enough to obtain uniform UFG structures like those shown in Fig. 2d.

Additionally, Okitsu et al. [17] fabricated fully annealed UFG ferrite grains and homogeneously dispersed cementite particles in low-carbon steel by 91% cold-rolling of a martensite-ferrite duplex microstructure containing 42 vol% martensite and short-time annealing. They obtained the high strength of 966 MPa, but the uniform elongation was very low of 0.6%. This is probably because they obtained UFG ferrite grains without BGSD.

As can be seen in Fig. 3a and Table 2, YS and UTS gradually increase with increase in rolling reduction and decrease in annealing time. The strength of steels generally increases with decreasing d_F , obeying the Hall–Petch equation [20]:

$$\sigma = \sigma_0 + k d_F^{-1/2}, \quad (1)$$

where σ refers to the YS or flow stress, and σ_0 and k are constants. Figure 3b represents the YS as a function of the inverse square root of average ferrite grain size ($d_F^{-1/2}$) and the fitted Hall–Petch relation. Accordingly, the higher strength of steels at higher rolling reduction and lower annealing times is mainly attributed to smaller d_F . Other researchers have observed that both the yield and tensile strength increased when the grain size of ferrite decreased [4, 21].

It is also noteworthy that the produced UFG steels with BGSD exhibits high UE and TE, which is considerably larger than that of the submicrometer ferrite single phase structures fabricated by other researchers [1, 22]. The 50% cold-rolled materials have the medium strength and sufficient elongation. This result suggests that 50% cold-rolled martensite-ferrite DP microstructure is favorable as a starting structure for the production of UFG steel when the required strength is not too high. This can be also an advantage in terms of industrial production. On the other hand, in the 80% cold-rolled materials, it is found that refinement of the structure leads to a great improvement in strength level without significant reduction in elongation.

4 Conclusions

In order to create a new microstructure for low-carbon steel showing high strength without significant loss of ductility, a new process was proposed. Low-carbon steel sheets were intercritically annealed and quenched to obtain martensite-ferrite DP structure. Steel sheet with DP microstructure was cold-rolled from 50 to 80% reduction in thickness, and then annealed at 600 °C for different times. The new microstructure revealed bimodal ultrafine ferritic grains and nanoscale carbide particles, and showed an excellent combination of high strength (880 MPa yield and tensile strength) and adequate ductility ($\sim 8\%$ uniform and $\sim 13\%$ total elongation) compared to conventional low-carbon steels. The formation of the UFG ferrite was considered to be attributed to the strain concentration in the soft ferrite matrix and also the mild deformation of the martensite regions in the cold-rolling.

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