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The Effect of Intercritical Annealing Time on the Microstructures and Mechanical Properties of an Ultrafine Grained Dual Phase Steel Containing Niobium

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Abstract

An ultrafine grained dual phase (UFG DP) steel containing niobium was produced by a new route utilizing simple cold-rolling and subsequent intercritical annealing of ferrite/martensite duplex starting structures. The effects of intercritical holding time on the microstructural evolutions and mechanical properties were studied. The results showed that increasing intercritical holding time enhnaced the volume fraction of martensite and decreased the grain size of ferrite. Tensile testing also revealed good strength–elongation balance for UFG DP steels (UTS×UE > 100 Jcm^{-3}), in comparison with the commercially used high strength steels. The variations of strength, elongation and fracture behavior with intercritical holding time were correlated with the microstructural features.

Keywords: Dual phase steel, Cold-rolling, Intercritical annealing, Mechanical characterization, Fracture behavior.

1. Introduction

Dual phase (DP) steels characterized by microstructures consisting of a soft ferrite matrix with hard martensite islands have received a great deal of attention due to their unique properties such as high strength, good formability, low yield ratio, high initial work hardening rate, continuous yielding and weldability [1-3]. Therefore, they are widely used for automotive applications to reduce weight and achieve higher crash resistance [4]. To obtain the dual phase microstructure, the steels are austenitized between the austenite formation start and finish temperatures (i.e., Ac_1 and Ac_3 temperatures, respectively) and then rapidly quenched [5].

In view of developing lighter and stronger DP steels with the optimized properties for occupants' safety and fuel efficiency, further strengthening of DP steels is required. While carbon is the most important element for strengthening steels, it has detrimental effects on weldability and formability [5]. Grain refinement is an effective tool to achieve the combination of strength and ductility [6, 7].

Recently, ultrafine grained (UFG) DP steels have been obtained with a thermomechanical treatment. Calcagnotto et al. [8] produced UFG DP steels by some large strain warm deformation by starting the ultrafine ferrite/cementite microstructure. They showed that the martensite fraction was increased with increasing holding time; however, the ferrite grain size was not significantly increased due to a longer intercritical time. Ahmad et al. [9] applied hot-rolling process in various intercritical temperatures. They indicated that changing volume fractions of martensite with hot-rolling in the intercritical region had a great influence on the tensile properties of DP steels. They showed that both yield and tensile strength were increased by grain refinement. Niakan and Najafizadeh [5] studied the effect of hot-rolling parameters and Nb content on the tensile properties of DP steels. They showed that increasing the reduction of hot-rolling percentage enhnaced the yield and tensile strength and decreased the total elongation. Hot-rolling and warm deformation were not very promising due to huge deformation induced at higher temperatures. Azizi-Alizamini et al. [10] developed UFG DP steels by rapid intercritical annealing of fine ferrite-carbide aggregates. The major drawback of the rapid intercritical annealing approach was found be very high heating and cooling rates (300 ºC/s and 1000 ºC/s, respectively).

According to the above researches, grain refinement with the simultaneous improvement of mechanical properties was attempted by changing starting microstructures and processing ways. However, these processing routes were found to suffer from some drawbacks. Nevertheless, further studies could be useful to overcome these limitations.

In the present study, UFG DP structures with simultaneously improved mechanical properties were fabricated by employing some uncommon cold-rolling

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and intercritical annealing of a ferrite-martensite duplex starting microstructure. A systematic study was conducted accordingly to investigate the effect of intercritical holding time on microstructural evolutions, tensile behavior and the fracture mechanism of UFG DP steels containing Nb microalloying element.

2. Materials and Experimental Procedures

The chemical composition of the steel used in this investigation is given in Table 1. The steel was casted by the copper boat vacuum induction melting (CBVIM) process.

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

Nb	C	Mn	Cr	Si	Fe
0.12	0.14	0.95	1.00	0.30	Bal.

The as-received steel and ferroniobium with a total weight of 25 gr were put inside an indentation (boat) on a copper tube. Then, the copper tube was centered inside a quartz tube to seal it from the atmosphere. Finally, the induction coil was placed around the quartz tube. Copper boat melting was carried out in a sealed pure Ar (99.9998 Vol.%) atmosphere using a radio frequency (RF) induction generator with the working frequency of 350 kHz and the nominal power of 45 kW. Before CBVIM, the steels were pickled by a solution of 60 percent hydrochloric acid (HCl) in water at the temperature of 25 °C.

The cast samples were first homogenized at 1000 °C for 2 h and this was followed by furnace cooling and hot-forging at 1100 °C to a thickness of 4 mm. In order to prevent severe decarburization, cast iron swarfs were used to protect the samples during homogenization. The samples dimensions were about $50\times20\times4$ mm^3. The thermomechanical procedure used to produce the ferrite-martensite DP microstructures is schematically presented in Fig. 1.

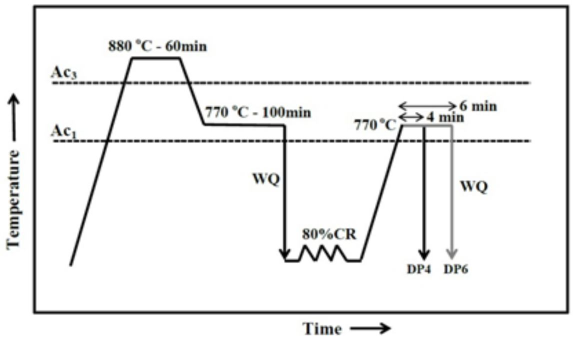

Fig.1. Thermomechanical cycle used to produce UFG-DP steels. Ac_1 and Ac_3: austenite formation start and finish temperatures during heating, respectively; WQ: water quench; CR: cold-rolling.

The ferrite-pearlite microstructures were first heated to the austenitization temperature of 880 °C for 60 min and this was followed by intercritical annealing at 770 °C for 100 min and water quenching. Then, the resulting duplex ferrite-martensite structures were subsequently cold-rolled up to 80% using a laboratory mill with a reduction of about 0.05 mm at each pass. Finally, the cold-rolled samples were heated to the intercritical temperature of 770 °C and held for 4 and 6 min and then water quenched. The DP steels were coded as the DP4 and DP6 depending on the intercritical holding time of 4 and 6 min, respectively.

Specimens for microstructural analysis were mounted, ground and polished till 4000 grit finished and polished with one micrometer alumina suspension. After polishing, the specimens were etched with 2% nital solution for 3s and this was followed by 10 % potassium metabisulfite ($K_2S_2O_5$) for about 5s. Microstructures were characterized using optical microscopy (OM) and scanning electron microscope (SEM, Philips XL30) techniques. The size and the volume fraction of phases were established by Image J software (V 1.48, US National Institute of Health). Three tensile specimens with a gage length of 12.6 mm were machined parallel to the rolling direction for each specimen by using electro-discharge machining (EDM) method. Room temperature tensile tests were carried out at a constant cross head speed of 1 mm/min with a Hounsfield H50KS machine. Fracture surfaces of the specimens were analyzed by SEM and Image J software.

3. Results and Discussion

3.1. Microstructural analysis

Fig. 2 shows the microstructure of homogenized steel at 1000 °C for 2 h after it was subjected to furnace cooling. It became clear that the microstructure consisted of 75 vol% ferrite with a grain size of about 13±2 µm and 25 vol% pearlite.

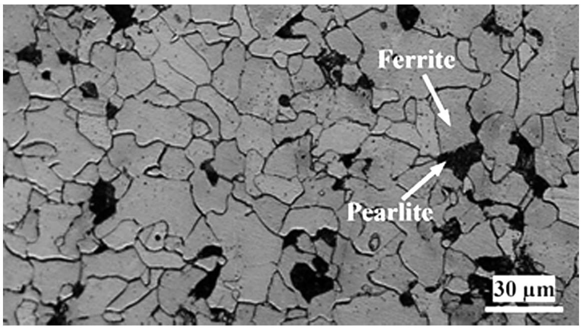

Fig. 2. OM micrograph of the steel homogenized at 1000 °C for 2 h and followed by furnace cooling.

Intercritical annealing at 770 °C for 100 min was followed by water quenching, resulting in a duplex microstructure composed of ferrite matrix and martensite networks (Fig. 3).

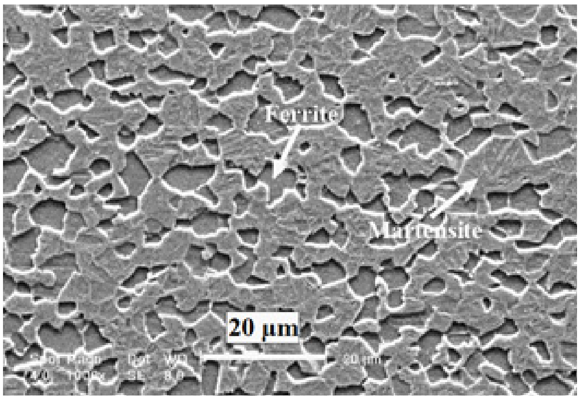

Fig. 3. SEM micrograph of duplex microstructure developed by intercritical annealing at 770 °C for 100 min and followed by water quenching.

It should be noted that when the initial ferritic-pearlitic steel was austenitized and then followed by slow cooling to the two phase (α+ γ) region, ferrite was nucleated at the austenite grain boundaries and grew into the austenite grain. After holding at the intercritical region, the retained austenite was transformed into martensite by quenching in water. The final microstructure consisted of martensite networks surrounded by ferrite. This microstructure was selected as the initial structure prior to cold-rolling. As reported by Okitsu et al. 11), cold reduction of this duplex microstructure was performed without cracking. The volume fraction of martensite (V_m) and average ferrite grain size (df) were about 54% and 4.3±1.2 µm, respectively.

The duplex microstructure was then 80% cold-rolled, heated to the intercritical region (770 °C held for 4 and 6 min) and subsequently, water quenched to form UFG DP microstructures (Fig. 4).

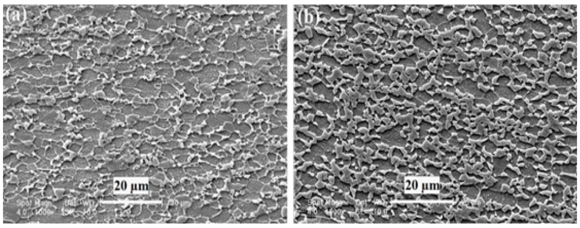

Fig. 4. SEM micrographs of UFG-DP structures developed by 80% cold-rolling and intercritical annealing of duplex microstructures at 770 °C followed by water quenching: (a) DP4 and (b) DP6.

Cold-rolling of the duplex microstructure resulted in increasing the stored energy due to the high dislocation density, providing the driving force for the ferrite recrystallization upon intercritical annealing. As shown in Fig. 4, with increasing the holding time, the V_m was increased. In fact, increasing holding time enhnaced the austenite volume fraction and then transform it to martensite upon quenching in water. Moreover, the d_f was decreased by increasing the holding time. It was found that higher amounts of the martensite phase caused finer ferrite grains 12). As shown in Fig. 4, the martensite islands size was decreased by increasing the holding time. The effect of holding time on martensite islands size and morphology could be attributed to the mechanism of the austenite formation during intercritical annealing. In fact, in the grain refinement by cold-rolling and intercritical annealing with starting the duplex ferrite-martensite microstructure, ferrite recrystallization kinetics, austenite formation kinetics and the interaction between both phenomena depend on three factors 13):

(i) The presence of deformed ferrite grains with internal strain (i.e. strain energy) may accelerate the ferrite recrystallization process.

(ii) Kinetics of austenite formation primarily depends on the available interface area, which is the nucleation site for austenite.

(iii) At the lower heating rates, the deformed ferrite grains are recrystallized fully before the austenite formation.

When the duplex microstructure was cold-rolled and intercritical annealed, the austenite nuclei at recrystallized ferrite/ferrite interfaces resulted in fine martensite particles. Due to the lower intercritical time in DP4 steel, austenite did not have enough time to be distributed uniformly throughout the microstructure, thereby resulting in the formation of some coarse martensite after quenching.

3.2. Tensile properties

The true stress-strain curves and the corresponding tensile data are presented in Fig. 5 and Table 2, respectively.

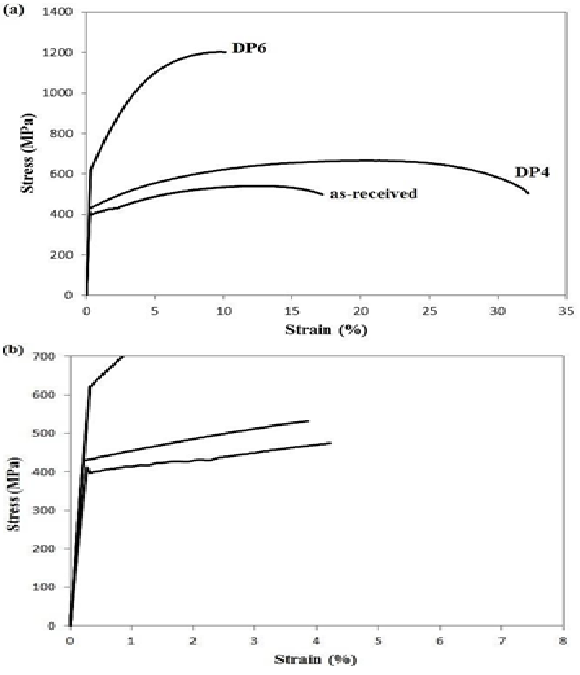

Fig. 5. (a) The engineering stress-strain curves and (b) magnified yield region of studied DP steels and the as-received sample.

Table 2. The microstructural characteristics and tensile properties of the present steel specimens

Steel	V_m (%)	d_f (µm)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)	Toughness (MPa)	UTS×UE ($J\ cm^{-3}$)
as-received	-	-	350±15	540±13	7.5±0.80	17.22±1.72	86	40.50
DP4	45	2.82±1.82	430±12	665±20	28.15±2.90	32.18±2.14	196	187.20
DP6	62	1.40±0.37	621±17	1203±10	9.61±0.3	12.90±1.10	126	115.60

V_m: martensite volume fraction; d_f: average ferrite grain size; YS: yield strength, UTS: ultimate tensile strength; UE: uniform elongation; TE: total elongation.

As shown in Fig. 5, the DP steels showed a continuous yielding behavior and a low yield ratio in contrast to the as-received sample. The austenite to martensite transformation from intercritical region resulted in generating residual stresses in the ferrite and producing geometrically necessary dislocations (GND) in the ferrite close to the ferrite/martensite interface. The residual stresses are thought to be responsible for lowering the elastic limit, while the unpinned GNDs are assumed to contribute to the continuous yielding behavior of DP steels [14]. In addition, it was found that the DP steels had higher yield strength (YS) and ultimate tensile strength (UTS) compared to those of the as-received specimen. The higher strength of the DP steels is known to be due to the presence of the matrensite as a hard phase. It can be seen that both YS and UTS were increased by increasing intercritical holding time. The maximum UTS was obtained in the DP6 steel. The effect of holding time on strength could be attributed to the higher V_m and lower d_f [15] .However, increasing intercritical holding time caused a decrease in both TE and UE. The toughness values were obtained by the measurement of the area under the engineering strain–stress curves, which was an indication of the required energy to break the material. It could be seen that the toughness of DP4 and DP6 steels was about 56 and 26 percent higher than that the as-received steel, respectively.

The energy absorption capability is an important factor in vehicle crashworthiness and can be established by strength–elongation (UTS×UE) balance. Fig. 6 shows UTS×UE versus UE plot used to compare the DP steels developed in this study with those of the previous works [9, 10, 12, 15-18].

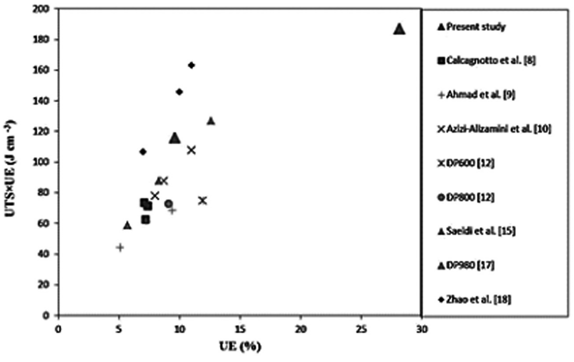

Fig. 6. Comparison of UTS×UE versus UE for various DP steels.

3.3. Fracture mechanism

Fig.7 shows the tensile specimens after failure. Clearly, most parts of the fracture surfaces exhibited dimples, but their size and distribution were different.

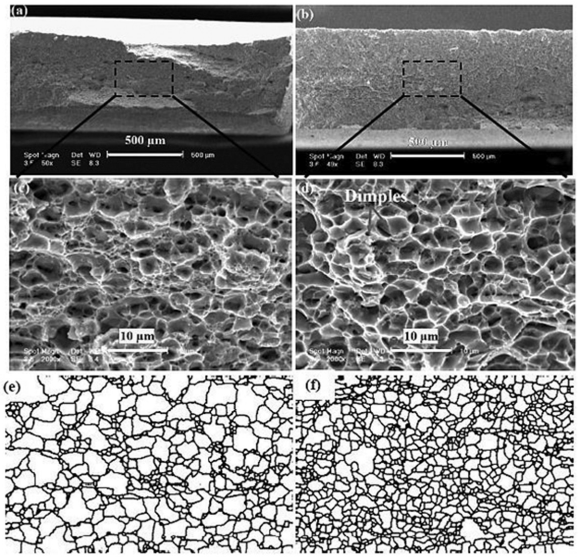

Fig.7. SEM micrographs of the fracture surfaces and corresponding processed images of (a,c,e) DP4, (b,d,f) DP6 tensile specimens.

This suggested that the prominent mode of fracture was ductile. One should note that the ductile fracture occurs in three stages in DP steels [19, 20]: (i) void nucleation, (ii) void growth and (iii) void coalescence. Void nucleation in the deformed samples exhibited three distinct processes: (a) martensite particles cracking, (b) decohesion at the ferrite/martensite interface and (c) necking and breaking of martensite particles. Void growth and coalescence have also been related to high local strains associated with necking [20]. It is known that the ferrite–martensite interfaces are the dominant places for voids nucleation that can promote the creation of the high density of initial voids [21]. Table 3 shows the average and maximum dimple size of the fracture surfaces of DP4 and DP6 steels. It is clear that with increasing intercritical holding time, the maximum and average dimples size was decreased. In this case, the number of dimples per unit area on the fracture surface depended on the number of nucleation sites and the plasticity of the material [15, 18].

In fact, with increasing nucleation sites, void growth could be limited by of intersecting and linking up the neighboring dimples. So, the final fracture surface appearance consisted of many small dimples[15]. According to Fig. 7 and Table 3, with increasing the intercritical holding time, the average ferrite grain size and martensite particle size were decreased. The higher fraction of ferrite/martensite interfaces in the DP6 steel could cause the lower dimple size.

Table 3. Average and maximum dimples size of the fractured surface of the investigated DP steels.

Steel	Average dimples size (μm)	Maximum dimple size (μm)
DP4	1.738	5.837
DP6	0.803	5.235

4. Conclusions

The effects of intercritical annealing time on the microstructural evolution and the mechanical properties of the UFG-DP steels were studied. The main conclusions that can be drawn from the study are as follows:

1- Increasing intercritical holding time increased the volume fraction of martensite and decreased the average grain size of ferrite.

2- The investigated DP steels showed superior mechanical properties in terms of toughness (196 and 126 MPa for DP4 and DP6 steels, respectively) and energy absorption capacity (116 and 187 J cm^{-3} for DP4 and DP6 steels, respectively) compared to commercially high strength steels and the as-received sample.

3- The prominent fracture mode of the investigated DP steels was ductile; however, the size and distribution of dimples were different.

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