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Effect of the Heat-treatment Temperature on the Mechanical Properties and Microstructural Evolution of Cold-rolled Twinning-induced Plasticity Steel

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Abstract: TWIP steels with 70% cold-rolled reduction were heated at 500, 600, 700, 800, 900, 1000, and 1100 °C. Then, the properties before and after heating were examined through tensile and hardness experiments. The microstructures were also analyzed by optical microscopy and transmission electron microscopy. The relationship between the properties and microstructure was examined as well. Finally, the evolution process of cold-rolled microstructures during heating was discussed in detail. Moreover, some conclusions can be drawn, and the heating evolution characteristics are described.

Key words: TWIP steel; cold rolled; static recrystallization; dislocation walls

1 Introduction

As a result of mechanical twin formatting during deformation at ambient temperature, twinninginduced plasticity (TWIP) steel can possess the outstanding combination of strength and formability. Thus, TWIP steel can be used to produce automobile bodies with reduced weight, low energy consumption, and improved safety^[1-4]. Consequently, TWIP steel is attracting significant research interest in the field of steel manufacture. To date, the main production technologies of TWIP steel include hot rolling, cold rolling, and heating. The heating temperature is vital not only to the mechanical properties and microstructural evolution of cold-rolled (CR) TWIP steels, but also to their future utilization. Therefore, correlative investigation processes are important for the application of these steels.

When heated at 500 °C, the microstructure of CR TWIP steel evolves through a recovery mechanism accompanied by a reduction in dislocation density and unchanged morphology of deformation twins. Then, the yield strength (YS) of 1 200 MPa and total elongation (TE) of 8% can be obtained, and the strength would be higher but the plasticity would be lower^[5]. When heated from 575 °C to 625 °C, partially recrystallized submicron grains occur in the CR microstructure, and YS of 1 000 MPa and TE of 29% can be obtained. At 700 °C, the microstructure consists of fine and ultrafine completely recrystallized grains (RGs), and YS decreases to 430 MPa and TE increases to 60%^[6]. However, the grain size significantly influences the dislocation structure, nucleation, and growth of deformation twins during plastic deformation^[7]. Thus, if CR reduction and heated temperature were better controlled, a grain size of 1.8 µm would be realized at 700 °C, with YE of 550 MPa and TE of $50\%^{[8]}$.

Previous investigations have indicated that CR TWIP steels can achieve various microstructures at different heating temperatures^[9]. Thus, heated CR steels display diverse properties. Notably, the dislocation motion, formation and growth of RGs are the direct cause of the various properties of CR steels. However, to date, studies on the evolution of

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the CR TWIP microstructure and its relationship with properties during heating are limited. This paper aimed to investigate the influence of the heating temperature on the microstructural evolution and properties of CR TWIP steels.

2 Experimental

2.1 Materials

The experimental steel was first melted in a medium-frequency induction furnace and cast into an ingot with dimensions of 130 mm \times 160 mm \times 2100 mm. The chemical composition of the investigated steel was Fe-0.052C-24.26Mn-0.9Si-1.85Al-0.008P-0.001S (wt%). The ingot was then used as an electrode for electroslag remelting, and the final investigated ingot with dimensions of 150 mm \times 300 mm \times 1200 mm was solidified in a copper crucible. Afterwards, the ingot was heated at 1 100 °C for 0.5 h and hot rolled to a 30 mm-thick plate. The plate was machined to 600 mm \times 400 mm \times 30 mm, hot rolled to 3 mm from 1 200 $^{\circ}$ C to 800 $^{\circ}$ C, and then cooled to room temperature. The hot-rolled plate was subjected to cold rolling to 0.9 mm with a 70% reduction. Finally, the CR plates were heated to 500, 600, 700, 800, 900, 1 000, and 1 100 $^{\circ}$ C for 5 min and quenched with water.

2.2 Examination of properties and microstructures

Tensile tests were conducted at a strain rate of 10^{-4} s⁻¹ using test pieces with 70 mm gauge length, 20 mm gauge width, and 0.9 mm gauge thickness at room temperature. Hardness was measured with a Rockwell hardness meter (Wolpert Wilson 2000). An Axiovert 25 CA (Zeiss) optical microscope was used to examine the optical microstructural evolution from cold rolling to heating at various temperatures. The detailed microstructural morphology was examined by transmission electron microscopy (TEM) using a JEM-2100 TEM system, which was operated at an acceleration voltage of 200 kV. The samples for TEM

were cut out from plates before and after heating along the rolling direction. The samples were mechanically polished to a thickness of 30 μ m. Thin foils were prepared using a double-jet electrolytic polisher at a voltage of 30 V and a temperature of about -20 °C. The electrolyte contained 5vol% perchloric acid and 95vol% alcohol.

3 Results

3.1 Properties

The variations in hardness, strength, TE, and product of strength and elongation (PSE) before and after heating are listed in Table 1. With increasing heating temperature, the strength and hardness decrease whereas the TE and PSE increase. The tensile strength (TS) of CR TWIP decreases to 1050 MPa because of low CR reduction^[5]. The onset temperatures of recovery and recrystallization are higher, and the investigated steels possess the properties obtained at 800 °C for TWIP steel with 80% CR reduction until 900 °C. The TWIP steel gains improved strength and excellent plasticity at TE > 70%. The largest PSE reaches 528.99 MPa•%, which is higher than that of third-generation automotive steel^[9]. The highest variations in hardness, YS and TS are 27 HRB, 218 MPa and 111 MPa, respectively, from 600 °C to 700 °C, which are significantly higher than those at the other temperatures. Moreover, the change of YS is larger than the change in TS. Nevertheless, the variations in TE and PSE reach a maximum at 21% and 129 MPa•%, respectively, from 900 °C to 1 100 °C. As indicated in the Ref.[10], TWIP steels exhibit better impact toughness at the temperature ranging from -196 °C to 20 °C, the impact absorbing energies of the TWIP steels exceed 100 J from 20 to -50 °C. Even at -196 °C, the impact toughness of TWIP steel can reach 50 J.

3.2 Microstructural characterization

3.2.1 Optical microstructure

The metallographic structures of as-achieved steel

Heating temperatures/ $^{\circ}$ C Hardness/HRB Tensile strength/MPa Yield strength/MPa Total elongation/% PSE^{*}/(MPa \cdot %) 87 1050 842 73.5 0 500 110.88 81 1008 824 11 600 72 987 810 19 187.53 29 700 45 876 582 254.04 800 38 568 38 308.94 813 900 30 762 536 50 388.62 1 0 0 0 25 729 475 71 517.59 21 1 1 0 0 687 420 77 528.99

Table 1 Variations in the properties of CR TWIP steels heated at different temperatures

*PSE: product of strength and elongation

and CR steels heated at various temperatures are shown in Fig. 1.

Fig.1 Optical microstructure of (a) CR TWIP steel and after heating at various temperatures; (b)500 °C; (c)600 °C; (d)700 °C; (e)800 °C; (f)900 °C; (g)1 000 °C; (h)1 100 °C

Heating at increasing temperature enables the microstructures in CR TWIP steels to recover, recrystallize, and grow with new grains. Fig.1(a) presents the CR microstructure. The original austenitic grain boundary and deformation are not observed because of severe deformation. However, the dark deformed area and light area without deformation co-exist. When heated at 500 °C, the morphological changes of CR are minimal, and the deformation area lightens with decreased dislocation density that is induced by recovery, as shown in Fig.1(b). With increased temperature to 600 °C, cake-like compressed austenite grains are observed and a few new RGs are observed along the grain boundary, as shown in Fig.1(c). Fig.1(d) shows that more RGs form, with some of them growing inward and some taking shape inside the austenitic grains. At 800 °C, recrystallization is completed and fine new grains comprise the microstructure, which can be seen in Fig.1(e). With further increased temperature, the RGs grow and heating twins form, as shown in Figs.1(f)-1(h).

3.2.2 TEM observation

Fig.2 shows the TEM images of two parts in the CR TWIP steel. The straight bundles can be observed in Fig.2 (a), indicated by cycles. The bundles are single deformation twins, ascertained by the selected area electron diffraction (SAED) patterns in the top right corner of Fig.2(a). The deformation twins are cut off and not continuous for severe deformation, with some twins and dislocation taking the form of island structures. The length of twins is only hundreds of nanometers, which is significantly shorter than that of twins forming under tensile^[2, 11] and dynamic^[10, 12] deformation, as indicated by circles in Fig.2(a).

The orientation, length and width of twins in the two cycles in Fig.2(a) are different. The microstructure in the smaller cycle shows island structures, and the twins in it are thinner than those in the larger cycle. Two twin systems are observed in the large rectangle in Fig.2(b), and a black block structure also forms around the microstructure in this site. Austenite without deformation can be observed in Fig.2, ascertained by the SAED pattern in the top right corner of Fig.2 (b) and indicated by A and B in Fig.2(a) and (b). At the same time, austenite is divided by black bands induced



Fig.2 TEM images of two different sections in CR TWIP steel



by dislocation tangling or block structure produced by deformation twins interacting with the dislocation.

4 Discussion

4.1 Relationship between properties and micro-structure

4.1.1 CR TWIP steel

The yield ratio of CR TWIP steel is still 0.8. Thus, a certain plastic deformation occurs from yield to fracture depending on the microstructural morphology of CR TWIP steel. Original austenitic structures without any deformation feature are observed because of grain orientation transformation to <101>, which does not favor deformation twin formation^[13]; thus, the straight appearance of twins changes in the succeeding deformation. However the austenite grains with changed orientation can still form twins during tensile deformation, producing CR TWIP steel with specific plasticity. The interaction between partial dislocations and deformation twins in CR TWIP steel leads to higher critical resolved shear stress (CRSS) and higher YS.

4.1.2 Heated steels

The effects of the heating temperature on the YS, TS, hardness, and TE differ because their microstructural evolution varies with the temperature. During heating, the dislocation density decreases and dislocation walls form first. Thus, YS decreases because of the decrease in CRSS induced by a reduction in the restraint of partial dislocation. Then, new RGs generate and develop, but the restraint of the partial dislocation continues to decrease; in turn, YS gradually decreases. As shown in Figs.1(c), 1(d), and 3, the degree of recrystallization increases most rapidly, and the reduction in YS reaches the maximum. Similarly, Ueji^[8] found that the size of newly formed fine grains is about 2 µm when the static recrystallization of the investigated steel is completed. Simultaneously, the steel can achieve a YS of 500 MPa and TE of 50%. Identical with the cause of YS change, the hardness shows a corresponding variation with microstructural evolution. However, the TS of TWIP steel is related to work hardening, which strengthens the matrix as well as enhances the ability to resist the generation and propagation of crack (GPC). Thus, the TS of CR TWIP steel is the highest. With increasing heating temperature, the degree of softening of the CR microstructure increases, whereas TE decreases because of the inability to resist GPC. TE and the corresponding PSE are proportional to the deformation twin density; thus, they increase with increasing heating temperature and their variation reaches the maximum from 900 $^{\circ}$ C to 1 000 $^{\circ}$ C.

4.2 Microstructural characteristics 4.2.1 CR microstructure



Fig.3 TEM images of CR TWIP steel heated at various temperatures: (a) and (b)500 °C; (c) and (d)600 °C; (e) and (f) 700 °C

Due to the stacking fault energy of TWIP steel^[14,15], the planar dislocation structure forms through the movement of partial dislocations at the initial compression stage^[10,16,17]. Then, deformation twins nucleate at the point of stress concentration and grow thereafter^[18]. Consequently, deformation can continue. The orientation of austenitic grains significantly affects the entire process^[19]. Deformation twins completely develop and multisystem twins are created, as shown in Fig.2(b). During the subsequent deformation, twins interact with dislocations and become curved, accompanied by orientation changes. The twins are even cut apart and resemble an island together with dislocation. Finally, the microstructures of CR TWIP steel are composed of severe deformation sections and a non-deformation area.

4.2.2 Evolution of CR microstructure during heating

The morphology of deformation twins does not change when CR steel is heated at 500 $^{\circ}$ C, as shown in Figs.3(a) and 3(b). However, dislocation walls and subgrains form through the movement of partial dislocations, inducing reduced dislocation density, which has not been observed in previous studies^[5-8]. Therefore, twins and dislocations do not simultaneously disappear^[5]. Dislocation walls form through the movement of dislocations driven by thermal energy, thereby providing nucleation sites for the subsequent recrystallization.



Fig.4 Schematic of the evolution procedure of CR TWI steel under heating

With increasing temperature, RGs gradually form. However, unlike austenitic stainless steel, deformation twins do not form to provide preferential nucleation sites for new austenitic grains^[20]. The shape of the new RGs in Fig.3 shows that the nucleation sites are located in the severely deformed area, e.g., dislocation walls formed though recovery. Furthermore, the nucleation temperature in this area is low, which favors new grain growth. Therefore, different grain sizes can be observed in the microstructure generated at 700 $^{\circ}$ C, shown by Figs.3 (e) and 3(f). Considering the same atomic arrangement of the original and new austenitic grains, RGs initially grow inward for the original austenite, and then swallow up the deformation structure to achieve recrystallization.

No transformation of atomic arrangement occurs during recovery and recrystallization of the CR TWIP steels. The entire process only involves the change in austenitic morphology through atomic movement under thermal activation. This process can be regarded as the disappearance of dislocation and deformation twins. However, the thermal stability of deformation twins is higher than that of dislocation. Therefore, dislocations move first and form highly dense walls. The dislocation walls subdivide the original austenitic grains and surround deformation twins. Afterwards, new RGs nucleate at the dislocation site with high density, eg, dislocation tangles or walls, and then achieve recrystallization by swallowing up the original austenite and deformation twins. Fig.4 presents a schematic of the evolution process of CR TWIP steel under heating.

5 Conclusions

a) The microstructures of CR TWIP steels are composed of subdivided deformation twins, islands generated by interaction between twins and dislocation, as well as the original austenite of rotated orientation. Therefore, the steels possess high YS and TS, together with a certain plasticity.

b) Given the various determinants and heating microstructures, the maximum variations corresponding to the temperature intervals of hardness, strength, TE, and PSE of the CR heated steel also differ. The largest variation in TE is smaller than that in YE.

c) The heating evolution characteristic of CR microstructure can be described as follows: dislocations move first and form walls with high density or subgrains, which subdivide original austenitic grains and surround deformation twins. Then, new RGs nucleate at the dislocation site with high density, *eg*, dislocation tangles or walls. The recrystallization process is achieved by successively swallowing up the original austenite and deformation twins.

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