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Microstructure and Mechanical Properties of 06Cr13Ni4Mo Steel Treated by Quenching–Tempering–Partitioning Process

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A heat treatment process, quenching–tempering–partitioning (Q–T–P), has been applied to a low carbon martensitic stainless steel 06Cr13Ni4Mo. By using this process, ultrafine reversed austenite can be obtained at room temperature. The microstructures of the reversed austenite and the martensite matrix were characterized by transmission electron microscopy (TEM) and energy dispersive spectroscopy (EDS) in detail. The results show that the ultrafine reversed austenite is enriched in Ni resulting in the austenite stability at room temperature. Two new types of nano-scale carbide precipitates are found in the martensite matrix. Detailed analysis suggests that the two nano-scale precipitates can be identified as ω phase and λ phase carbides, respectively. The orientation relationship between the ω phase and matrix is $[011]\alpha//[2\bar{1}\bar{1}0]\omega$ and $(2\bar{1}1)\alpha//(01\bar{1}0)\omega$, while that between the λ phase precipitate and matrix is $[011]\alpha//[0001]\lambda$ and $(\bar{2}00)\alpha//(1\bar{2}10)\lambda$. For the present steel, the ultrafine reversed austenite and carbide precipitates obtained by Q–T–P treatment provide a good combination of high strength and toughness.

1. Introduction

The amount and stability of retained or reversed austenite at room temperature is of critical importance for improving the ductility and toughness of steels by the transformation-induced plasticity (TRIP) effect[1,2]. The chemical composition of strong austenite stabilizers such as C or Ni has a great influence on its stability. Especially for the martensitic stainless steels such as Fe–Cr–Ni–C system, the phenomenon occurring during austenite reversion is complex due to the high content of alloying elements[3]. For low carbon martensitic stainless steel 06Cr13Ni4Mo, conventional quenching and tempering heat treatments have long been applied to produce the reversed austenite[4–7]. It has been confirmed that the reversed austenite can only be obtained in the intercritical tempering temperature range of 580–650 °C (slightly above the austenite start temperature A_s), where the local Ni enrichment is sufficient to stabilize the reversed austenite during cooling to room temperature[6]. When the tempering temperature is higher, the reversed austenite is unstable at room temperature due to the dilution of Ni. This will lead to the decrease in strength of materials and even cannot meet the requirement for ductility although it is improved during tempering resulting from dislocation recovery and so on[8].

In the present study, we propose a novel treatment termed as the quenching-tempering-partitioning (Q–T–P) in order to provide another way to produce reversed austenite-containing microstructures. In this new treatment, the as-quenched alloy was tempered at higher temperature to provide a martensitic–austenitic mixed microstructure. Then the partitioning temperature was chosen to be at a lower temperature to ensure more Ni to segregate to the local reversed austenite[4]. The morphology and chemical composition of the reversed austenite and martensite obtained by the novel process were characterized by using transmission electron microscopy (TEM) and energy dispersive spectroscopy (EDS). As will be shown below, this process could generate microstructures of both ultrafine reversed austenite and the martensite with high density of dislocation. And there exists two types of nano-scale precipitates at room temperature, which are identified to be the ω phase and λ phase, respectively, as reported in Ref. 9,10. Usually, the ω phase is widely studied in numerous group IV and V transition metals such as Ti and their alloys[11,12], which exists in the metastable bcc-type β phase through phase transformation during cooling or under static/dynamic pressure[13,14]. The crystal lattice of the ω phase is coherent with the bcc lattice and has hexagonal lattice parameters of $a_\omega = \sqrt{2} \times a_{\mathrm{bcc}}$, $c_\omega = \sqrt{3}a_{\mathrm{bcc}}/2$[10]. Ping and Geng first designated the ω phase in a commercial spring steel[9]. The λ phase was identified to be a hexagonal close packed crystal structure with lattice parameters of $a = b = 0.573 \pm 0.005$ nm and $c = 1.206 \pm 0.005$ nm, which was first reported by Tirumalasetty et al.[10]. The ultrafine

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reversed austenite and the carbide precipitates are considered to provide a good combination of high strength and toughness for this steel.

2. Experimental

The experimental material used in this study is 06Cr13Ni4Mo steel and the chemical composition is Fe–0.05C–11.36Cr–3.84Ni–0.46Mo–0.5Mn–0.008P–0.002S (in wt%). The transformation temperatures A_s and A_f (austenite start and finish temperatures) of this steel are approximately 580 °C and 807 °C, respectively[6]. The alloy ingot was prepared by vacuum melting and casting. The cast ingot was forged to a slab of 35 mm in thickness and then hot rolled to 12 mm thick plate using several passes with an average reduction ratio of 10% per pass. The samples of 10 mm × 12 mm × 12 mm in size cut from the hot-rolled plate were homogenized and austenitized at 1050 °C for 1 h and water quenched. A sample of 0.1 mm × 10 mm × 10 mm in size was prepared for the in-situ high temperature X-ray diffraction (HT-XRD) studies performed on the Bruker D8 Discovery diffractometer (Cu-$K\alpha$ radiation, $\lambda = 0.154056$ nm). The as-quenched sample was heated to 680 °C at 1 °C/s and held for 30 min, and then subsequently cooled to 580 °C at 1 °C/s and held for 45 min. After that, the specimen was cooled to room temperature at 2 °C/s. The heat treatment route was schematically shown in Fig. 1. The HT-XRD data were collected at each step and the volume fraction of reversed austenite was calculated according to the following equations.

$$V_\gamma + V_\alpha = 1 \tag{1}$$

$$V_\gamma = 1.4 I_\gamma / (I_\alpha + 1.4 I_\gamma) \tag{2}$$

where V_γ and V_α are the volume fractions of austenite and martensite, respectively, and I_γ and I_α are the integrated intensities of $(111)_\gamma$ and $(110)_\alpha$ peaks, respectively[15]. Thin foil was prepared for transmission electron microscopy (TEM) analysis by means of ion milling. A JEOL 2100F microscope equipped with energy dispersive spectroscopy (EDS) operated at 200 kV was used to observe the microstructure.

The samples after the same austenitization mentioned above were subjected to tempering and partitioning treatment, i.e. the as-quenched samples were tempering at 680 °C for 2 h, and then directly cooled to 580 °C for 2 h for partitioning. For comparison, the conventional treatment of quenching + tempering + tempering (Q–T–T) was also carried out, i.e. the as-quenched samples were tempered at 680 °C for 2 h and air-cooled to room temperature, and then heated to 580 °C for 2 h, followed by air cooled to room temperature. The room temperature tensile and standard Charpy V-notch impact specimens were prepared from the as-treated samples, and their dimensions are 4 mm in width × 2 mm in thickness × 15 mm in gauge length and 10 mm × 10 mm × 55 mm, respectively.

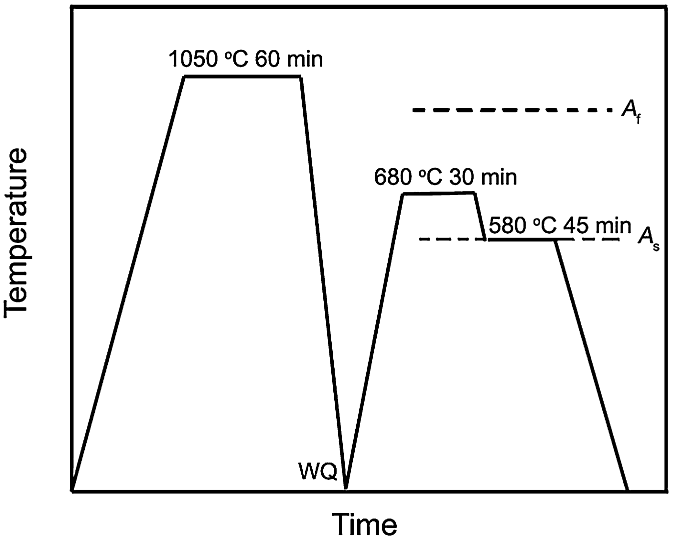

Fig. 1. Schematic diagram of the heat treatment route.

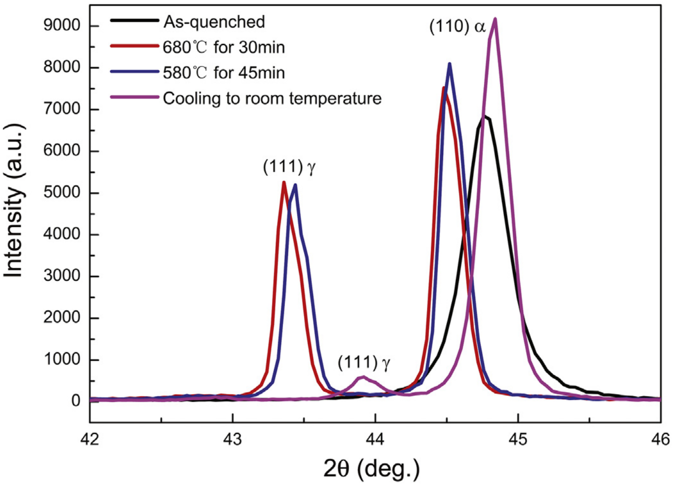

Fig. 2. In situ HT-XRD patterns of the sample at the as-quenched state, 680 °C for 30 min, 580 °C for 45 min and room temperature, respectively.

3. Results and Discussion

3.1. Volume fraction of the reversed austenite after Q–T–P treatment

Fig. 2 shows the in-situ HT-XRD spectrum of the samples under different heat treatment processes. It can be seen that no peak of the retained austenite was found from the as-quenched sample, which is in agreement with our previous results[5]. When the sample was heated to 680 °C, the peak of the reversed austenite was obvious and about 48% reversed austenite could be obtained, which was not decreased when the sample was cooled to 580 °C. The shift of the peaks to higher 2θ position during cooling is attributed to the thermal contraction. When the sample was cooled to room temperature, it is noticeable that the peak of the reversed austenite can still be observed. This indicates that some reversed austenite is stable and remains untransformed to martensite during cooling. The volume fraction of the stable reversed austenite at room temperature is about 8%. Nearly half martensite is fresh, which is directly transformed from the austenite during cooling. The newly formed martensite is considered to have a high density of dislocation.

3.2. Characterization of the reversed austenite after Q–T–P treatment

Fig. 3 shows the typical TEM images of the reversed austenite in the sample after Q–P–T treatment. The ultrafine reversed austenite laths in a [011] orientation shown in Fig. 3(a) are identified by electron diffraction in Fig. 3(b) and the reversed austenite size is of several hundreds of nanometers. Furthermore, some ultrafine reversed austenite coexisted with the $M_{23}C_6$ carbides along the martensite lath boundaries were also observed in the sample, which are in agreement with that in Ref. 4. Table 1 summarized the weight concentrations of Fe, Cr and Ni in the reversed austenite and