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Austenite and precipitation in secondary-hardening ultra-high-strength stainless steel

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ABSTRACT

Ferrium S53 is a new secondary-hardening ultra-high-strength stainless steel, with more Cr than other Co-Ni ultra-high-strength steels. Austenite has a great effect on the fracture toughness of this metal, and the carbides that are precipitated are the main strengthening phase of Ferrium S53 steel; in this study, its microstructural changes during heat treatment were observed and analyzed by X-ray diffraction (XRD), Optical microscope (OM), SEM, and transmission electron microscopy (TEM). About 40.85% retained austenite in the quenched sample is decreased to less than 5% after cryogenic treatment and tempering. In the final sample, block austenite is retained austenite, and film austenite around 5 nm in width is reverted austenite. Inside the martensite lath, parallel twin-like structures are actually segregated M_2C precipitates, and most of them are smaller than 20 nm. These precipitates can be aggregated into a several-micron-long parallel zonal microstructure.

1. Introduction

Secondary-hardening ultra-high-strength steel (UHSS) with high Co-Ni is widely used in aeronautics and astronautics, as these have both high strength and high toughness [1]. Due to the addition of the strong-carbide-forming elements Mo and Cr, fine-scale M_2C carbides are precipitated in this steel as it ages [2, 3]. M_2C carbide has an HCP group structure that can have high coherency with BCC iron and can precipitate on a sufficiently fine scale to provide high strength levels [2, 4]. For the toughness of secondary-hardening UHSS, the distribution and stability of austenite (including retained austenite and reversed austenite) is an important influencing factor [5–7]. Further, to improve toughness, prolonged tempering is usually employed to dissolve metastable cementite precipitates, which are quite coarse and therefore limit the fracture toughness of secondary-hardening UHSS [2, 7, 8].

Representative secondary-hardening UHSSs include HY180, AF1410, and AerMet 100 [9–11]. HY180 was developed by the United States Steel Corporation (U.S. Steel) in 1965. It is primarily used in deep-water submarines, because its strength does not meet the requirements for aircraft [12]. In 1978, a higher-strength steel, AF1410, was developed. AF1410 has a higher content of C and Co than HY180, which improves its toughness and performance. Created using improved melting techniques and some alloy modifications, Schmidt and Hemphill developed a novel high-performance steel in the 1990s, called AerMet 100 [6, 11]. AerMet 100 achieves an optimum combination of strength, plasticity, and toughness at a tempering temperature of 482 °C [5].

These secondary-hardening UHSS have good strength and toughness and increase the damage tolerance of Aircraft Structure Parts. However, the steels are susceptible to corrosion, and protective plating (Cadmium plating) is required for this steel to serve in corrosive environments [13]. The service environment may degrade the mechanical properties: this is called environment-assisted cracking (EAC) [14]. The phenomenon of stress corrosion cracking (SCC) is a typical example of this phenomenon in steel [13]. In 2003, QuesTek Innovations LLC developed a stainless UHSS (UHSSS) called Ferrium S53 using a design methodology. This steel has a similar strength to AerMet100 but better resistance to general corrosion and SCC [15]. To improve its corrosion resistance, Ferrium S53 steel contains 10% Cr, a figure that exceeds that of other UHSS.

As this steel was only recently developed, in 2003, and was first used in landing gear in 2010, few studies of its microstructure and precipitation have been reported. The present study conducted a detailed microstructural analysis of Ferrium S53 in the heat treatment process. Optical microscope (OM) and SEM were used to analyze the transformation of the microstructure. X-ray diffraction (XRD) and transmission electron microscopy (TEM) were used to characterize this steel's austenite. The morphology and structure of the precipitates in

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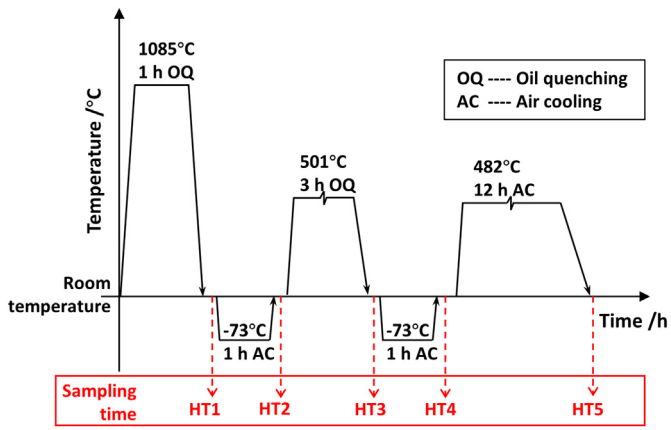

Fig. 1. Heat treatment process and the sampling time of specimens HT1–HT5.

the steel are also characterized by TEM.

2. Materials and Experimental Methods

Ferrium S53 for this study was produced by vacuum induction melting followed by vacuum arc remelting (VAR). The compositions (wt pct) were Fe–0.20C–14.2Co–5.7Ni–9.8Cr–2.0Mo–1.0 W–0.3 V. The VAR ingot was forged as a 45 mm square, and all the samples (15 mm × 15 mm × 15 mm) used in this experiment were cut from the steel bar. Then, the samples were heat treated using the processes indicated in Fig. 1. HT5 was heat treated using the standard and suggested heat treatment processes for Ferrium S53 [16]. The specimen was first austenitized at 1085 °C for 1 h and quenched in oil, and then it was immediately transferred to a mixture of dry ice and alcohol (−73 °C) for 1 h and heated in air to room temperature. After cryogenic treatment, the samples were reheated to 501 °C for 3 h and quenched in oil to room temperature. Then, they were cryogenically treated again (using the same process as the first time), and then aged at 482 °C for 12 h while being air cooled. HT1–HT4 were treated in ways corresponding to the following states: solution treated, primary cryogenic treated, primary tempered, and secondary cryogenic treated, respectively.

XRD measurements were carried out to determine the volume fraction of austenite. Data collection was conducted using Cu Kα radiation and the angular interval 35–105°. A direct comparison method is used to determine the austenite volume fraction as follows [17]:

$$V_\gamma = \frac{1}{1 + \frac{R_\gamma I_\alpha}{R_\alpha I_\gamma}}$$

V_γ is the volume fraction of austenite, and I_γ and I_α are the integrated intensities of the austenite and martensite peaks, respectively. The diffraction lines of $(220)_\alpha$, $(211)_\alpha$, $(200)_\gamma$, $(220)_\gamma$, $(311)_\gamma$ are selected to measure the integrated intensities.

To observe the microstructures, metallographic samples were etched in a solution of 1.5 g $CuCl_2$ in 33 mL HCl and 33 mL H_2O at room temperature. The microstructures of HT1–HT5 were observed using an OLYMPUS OM and further analyzed with an ULTRA-X55 field–emission scanning electron microscope with an energy-dispersive X-ray spectrometer (EDS). Samples for TEM analysis were subject to twin-jet electro-polishing at −25 °C to −30 °C, using a solution of 12.5% perchloric acid and 87.5% alcohol. The microstructure, sub-structure, and precipitates were observed and analyzed with a TEM (Tecnai G^2 20) with EDS at a 200-kV operating voltage.

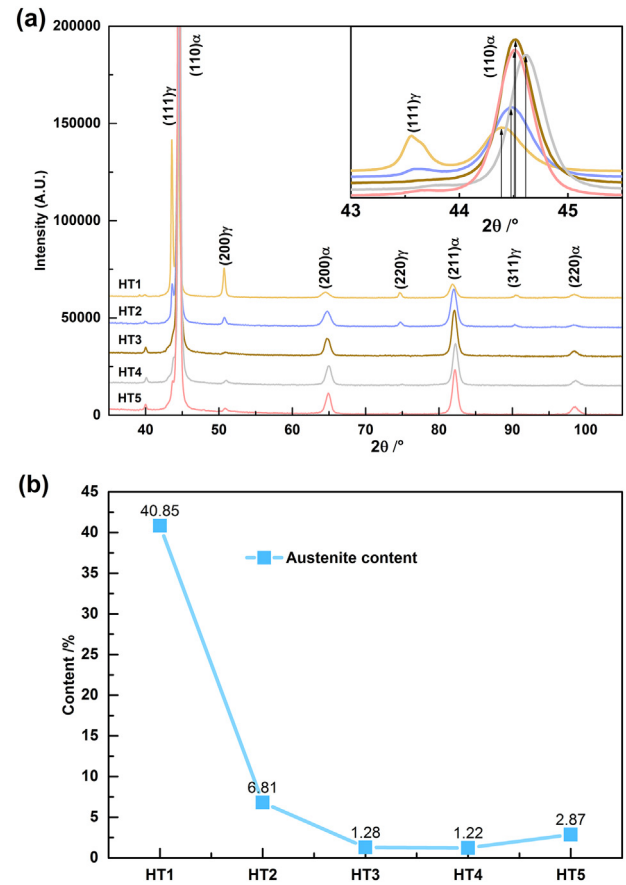

Fig. 2. XRD results. (a) X-ray diffraction patterns of martensite and austenite for all samples; (b) austenite content for all samples.

3. Results and Discussions

3.1. XRD Results

XRD examinations for HT1–HT5 were performed and the results are shown in Fig. 2. It is clear that martensite emerges as the main phase and austenite exists throughout, no matter whether it is in the quenched or tempered sample.

Ms. and Mf are the martensite start and finish temperature, and the M_s of these secondary-hardening steels are usually very low, the experimental Ms. temperature given by QuesTek Innovations LLC is 100 °C [18]. So the transformation of austenite to martensite is incomplete if the cooling rate during quenching is insufficient. This implies that a large amount of austenite will be retained [11, 19, 20]. The XRD result of this experiment, where oil quenching was used for Ferrium S53 steel, is shown in Fig. 2a, where it can be seen that 40.85% austenite remains in the HT1 sample, which indicates that the transformation was incomplete. This austenite that is retained is unstable and will change to martensite if more transformation energy is placed in the steel. The cryogenic and tempering treatments are usually used to promote the transformation to martensite [6, 8, 21]. Cryogenic treatment can provide greater undercooling for the transformation, so it usually immediately follows quenching. Differently from cryogenic treatment, however, tempering treatment promotes transformations by providing decomposition energy, and martensitic transformation occurs in the subsequent process of cooling [11, 21]. Therefore, further decomposition of residual austenite stems from the tempering temperature and cooling rate of this method. The higher the temperature and the faster the cooling rate, the more complete the transformation [21–23]. As Fig. 2 shows, austenite contents continuously decrease

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