JOURNAL OF IRON AND STEEL RESEARCH, INTERNATIONAL. 2015, 22(5): 451-456

Effect of Deep Cryogenic Treatment on Formation of Reversed Austenite in Super Martensitic Stainless Steel

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Abstract: The effect of deep cryogenic treatment on the formation of reversed austenite (RA) in super martensitic stainless steel was investigated. RA was found to form in steels without (A) and with (B) deep cryogenic treatment. The volume fraction of RA initially increased and then decreased with increasing tempering temperature over 550－750 ℃ for the two steels, which were quenched at 1050 ℃. In addition, for both with and without deep cryogenic treatment, the RA content reached a maximum value at 650 ℃ although the RA content in steel B was greater than that in steel A over the entire range of tempering temperatures. Furthermore, the hardness (HRC) of steel B was greater than that of steel A at tempering temperatures of 550－750 ℃. From these results, the basic mechanism for the formation of RA in steels A and B was determined to be Ni diffusion. However, there were more Ni-enriched points, a lower degree of enrichment, and a shorter diffusion path in steel B. It needed to be noted that the shapes of the RA consisted of blocks and stripes in both steels. These shapes resulted because the RA redissolved and transformed to martensite along the martensitic lath boundaries when the tempering temperature was 650－750 ℃, and a portion of RA in the martensitic lath divided the originally wider martensitic laths into a number of thinner ones. Interestingly, the RA redissolved more rapidly in steel B and consequently resulted in a stronger refining effect.

Key words: reversed austenite; super martensitic stainless steel; deep cryogenic treatment; diffusion transformation

As an extension of traditional heat treatment, deep cryogenic treatment often refers to the process of cooling a material in liquid nitrogen at 196 ℃[1]. Deep cryogenic treatment can promote austenitic transformation, martensite decomposition, and changes in ultrafine carbides in steel[2]. It is effective for not only increasing the mechanical properties and service life of these materials significantly but also stabilizing their size, improving their uniformity, and reducing deformation. In addition, deep cryogenic treatment is also advantageous because it is a low-cost, simple, environmentally friendly operation that can be performed without destroying the workpiece[3-5].

Super martensitic stainless steels have been developed to solve the problem of brittle chromium carbide precipitation at grain boundaries during the welding of traditional martensitic stainless steels[6-8]. In super martensitic stainless steels, the carbon content is reduced to less than 0.07%, while the contents of nickel (3.5%－6.0%) and molybdenum (1.5%－2.5%) increased. Thus, these steels have high strength, good plasticity, and weldability as well as good corrosion resistance and are lower cost materials. Therefore, super martensitic stainless steels are particularly attractive to the oil industry[9-11].

Reversed austenite (RA), which was first reported by a Swede who published a patent for Cr-Ni-Mo martensitic stainless steel undergoing a martensitic shear reversion during the tempering process, is stable at room temperature and below. It is distinguished from retained austenite by the conditions of its formation[12]. Studies have revealed that with different heat treatments, it is possible to control the RA content and improve the mechanical properties of steels[13,14]. RA is typically obtained by quenching and tempering super martensitic stainless steel, and the mechanism of transformation is diffusional rever-

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sion[15,16]. In addition, Ni enrichment prevents the austenite from transforming into martensite during cooling. However, the number of Ni-enriched areas is small and the concentration of Ni is high.

Therefore, to confirm the role of Ni diffusion in RA formation and the ability to control the formation of RA in super martensitic stainless steels, the quantity and microstructure of the RA phases in two steels subjected to two different heat treatments (quenching and tempering; quenching with deep cryogenic treatment and tempering) were investigated.

1 Experimental Procedure

Ultra high-purity super martensitic stainless steel was designed and melted in a vacuum induction melting furnace. The ingots were hot forged into round bars with a diameter of 15 mm. The chemical composition of the experimental steel is shown in Table 1. To obtain complete lath martensite, the samples were quenched in oil at 1050 ℃ for 0.5 h using a vertical type furnace. The specific heat treatments for steels A and B are shown in Table 2. The volume fraction of austenite in the each steel sample subjected to tempering at different temperature and holding time was determined using X-ray diffraction (XRD) analysis with Co radiation from 40° to 120°. The morphology and distribution of the austenite in the martensitic matrix of each sample was investigated with transmission electron microscopy (TEM), and the elemental concentration distributions of the microstructures were determined using energy dispersive spectrometry (EDS).

Table 1 Chemical composition of tested steels mass%

C	Mn	Si	Cr	Ni	Mo	Cu
0.021	0.40	0.27	14.78	6.5	2.04	1.44

Table 2 Heat treatments for steels A and B

Steel	Heat treatment
A	Quenched at 1050 ℃ for 0.5 h+tempered at 550−750 ℃ for 2 h
B	Quenched at 1050 ℃ for 0.5 h+liquid nitrogen (−196 ℃) +tempered at 550−750 ℃ for 2 h

2 Results and Discussion

2.1 Effect of deep cryogenic treatment on the RA content

After quenching at 1050 ℃, the microstructure of the super martensitic stainless steel consisted of quenched martensite and retained austenite, and the volume fraction of retained austenite was 1.95%. Variation in the volume fraction of RA with tempering temperature is shown in Table 3. After tempering, the volume fraction of RA in both steels clearly increased, and the formation of new austenite, or RA, was confirmed. When the tempering temperature ranged from 550 to 650 ℃, the volume fraction of RA increased as the tempering temperature increased and reached a maximum in both steels at 650 ℃ (A: 28.04%; B: 45.24%). In addition, over this range of tempering temperatures, the RA content in steel B was greater than that in steel A. Interestingly, when the tempering temperature was greater than 650 ℃, the volume fraction of RA decreased gradually as the tempering temperature increased, indicating that the RA transformed back to martensite above 650 ℃. Notably, in steel B, which was subjected to deep cryogenic treatment and thus had a higher phase change driving force and greater internal energy, the rate of conversion of RA was greater than that in steel A.

Table 3 Volume fraction of austenite following tempering at different temperatures

Tempering temperature/℃	Volume fraction of austenite/%	
	Steel A	Steel B
550	10.21	13.79
600	21.37	41.22
650	28.04	45.24
700	24.47	21.03
750	21.56	20.03

Plots of the volume fraction of RA and hardness as a function of the tempering temperature are shown in Fig. 1. It can be seen in Fig. 1(b) that in both steels, the hardness initially decreased with increasing tempering temperature, reaching a minimum at 650 ℃. Notably, this trend for the hardness is opposite to that of the RA content (Fig. 1(a)). These results suggest that the RA is a soft phase, and the greater the RA content, the lower the hardness. Therefore, tempering promoted improvement of the toughness of the materials. It can also be seen in Fig. 1(a) that the RA content in steel B was greater, but hardness was smaller than that in steel A over the entire range of tempering temperatures. These results are mainly due to the presence of martensite in steel B: the martensite acted as a carbon-supersaturated solid solution in which the lattice parameters for Fe and the vacancy concentration decreased at