Effects of temperature cycling and nitrogen on the stability of microstructures in austenitic stainless steels

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ABSTRACT

The influence of temperature cycling from room temperature to 77 K on nitrogen-alloyed austenitic stainless steels, Fe24Mn13Cr0.4N, Fe24Mn18Cr3Ni0.6N, Fe1Mn19Cr8Ni0.2N and Fe17Mn14Cr1Ni0.4N, has been studied by optical microscopy and scanning electronic microscopy (SEM). Results show that the phase transformation from austenite to ε martensite takes place due to the temperature cycling and stress concentration, whereas nitrogen can stabilize the austenitic microstructures greatly. Finally, the mechanism of the phase transformation and the structure of ε martensite are discussed in detail.

1. Introduction

Austenitic stainless steel is one of the most important categories of stainless steels, accounting for nearly 70% of the total amount of stainless steels that have been produced. It has many advantages, such as excellent cryogenic properties, anticorrosion and biocompatibility. Therefore, it has found extensive application in low temperature technology, chemistry, ocean technology, food industry, biomedicine, petrochemical processing, etc. Moreover, alloyed with nitrogen, austenitic stainless steel will exhibit a more stable austenite structure [1–4], better mechanical properties [5–8] and better corrosion resistance [9,10].

One of the most important applications of high-nitrogen austenitic stainless steels (HNASS) is as structural materials at low temperature [11–14]. There are certain basic requirements for this category of applications: (1) high yield strength; (2) superior toughness property at low temperature; (3) nonmagnetic; and (4) a stable austenitic microstructure. Because the martensite phase (α′ or ε) is ferromagnetic, the nonmagnetic property of the HNASS will deteriorate or even be lost if the phase transformation from austenite to martensite occurs during application at cryogenic temperature. Therefore, it is vital to investigate the microstructural stability of this kind of engineering material, which will greatly affect its reliability in such service. Moreover, in most circumstances, HNASS must endure the temperature cycling from room temperature to cryogenic temperature and, at the same time, withstand considerable stress. In this article, a special experimental set up was designed to simulate cryogenic service conditions for HNASS. The microstructural stability of four kinds of HNASS with different nitrogen content was investigated and the influence of temperature cycling and nitrogen content are discussed.

2. Experimental Details

The materials tested in this experiment are Fe24Mn13Cr0.4N (#1), Fe24Mn18Cr3Ni0.6N (#2), Fe1Mn19Cr8Ni0.2N (#3) and

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Table 1 – Compositions of the experimental materials

Materials	Composition, wt.%					
	Mn	Ni	Cr	N	C	Fe
1#	24.62	0.10	13.18	0.44	0.050	Balance
2#	24.22	3.23	18.03	0.62	0.051	Balance
3#	1.29	8.28	18.68	0.22	0.11	Balance
4#	14.90	1.41	17.36	0.39	0.17	Balance

Fe17Mn14Cr1Ni0.4N (#4). Their chemical compositions are shown in Table 1. They were melted in a vacuum furnace and then forged into 160 mm thick plates. A solution treatment (1100 °C for 1 h, water cooling) followed, which resulted in the austenitic structure. Next, the four alloys were processed into samples 16.0 mm in width and 40.0 mm in length. The thickness of the samples is 1.0 mm for alloys #1, #2 and #4 and 2.2 mm for #3, respectively. The experimental set up is shown in Fig. 1. The distance between the two armbraces is 30.0 mm. The tip of the screw is on the center of each sample; as it is screwed in the sample is deflected. In this experiment, three levels of deflections, 0.5 mm, 1.0 mm and 1.5 mm, were applied to all samples except for the samples of #3, which were thicker and only the 0.5 mm deflection was utilized. Samples of each of the HNASS steels were divided into two groups for different experiments. One group of samples was put into liquid nitrogen for 10 min and then removed. The temperatures of the samples returned to room temperature after about 20 min. For another group of samples this procedure was repeated three times. Each sample had been given a specific deflection for the cryogenic experiments. Finally, samples were taken from the set-up and cut cross-sectionally along the center to make metallographic specimens. These specimens were polished, then etched with nitric acid (20%), hydrochloric acid (40%) and glycerine (40%). The microstructures were characterized by optical microscopy EPIPHOT-300 and scanning electron microscopy (SEM) JXA-840.

3. Results and Discussion

In this experiment, some of the samples were plastically deformed, depending on the deflection level used. The room temperatures $\sigma_{0.2}$ and $\sigma_{\max}$ of all samples are listed in Table 2. These were calculated by using the parameters of the experimental set up and the equations of Dai et al. [14,15]. From Table 2, it is clear that samples of the #1, #2 and #4 steels are plastically deformed when the deflection is greater than 0.5 mm. The amount of deformation will obviously be greater for increased deflection. For samples of #3 steel, a deflection of only 0.5 mm will cause plastic deformation.

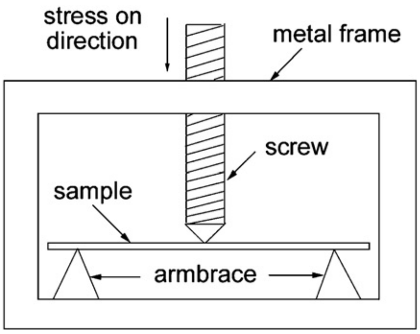

Fig. 1 – Schematic of experimental set up.

The threshold temperature of martensite phase transformation of these four materials can be predicted. Based on the equations for the transformation temperature of martensite ($M_{\varepsilon s}$ for ε martensite) by Dai et al. [16], the calculated $M_{\varepsilon s}$ of the #1, #2 and #4 steels are all lower than 4 K. Hence, from the thermodynamics point of view, no martensite should form at the liquid nitrogen environment (77 K). However, the calculated $M_{\varepsilon s}$ of steel #3 is higher than 77 K. So samples of #3 can transform from austenite to martensite when they are immersed into liquid nitrogen.

The experimental results, however, are contrary to the calculation prediction. The particular method used in this experiment, temperature cycling to 77 K while all samples were under stress, has made the phase transformation from austenite to martensite possible. The microstructures of the four materials after 0.5 mm deflection and one temperature cycle are shown in Fig. 2. Some amount of martensite is clearly seen in Fig. 2(c) and (d) for materials #3 and #4, respectively. In Fig. 2(a), there is less martensite for material #1, whereas no martensite was found in material #2, Fig. 2(b). Obviously, the amount of martensite that is formed is affected by the nitrogen content, as can be inferred from Table 1. Moreover, samples of #1, #4 are in the elastic region for deflections of 0.5 mm. Therefore, the transformation of ε martensite occurs in these lower-nitrogen materials under elastic loading only.

Microstructures of materials #1 and #3, deflected 0.5 mm and given three temperature cycles, are shown in Fig. 3. It is obvious that the amount of martensite increases with the number of temperature cycles. Moreover, with increased level of deflection, the plastic deformation becomes more intense, further favoring the phase transformation. The microstructures of #1, deflected 1.0 mm and 1.5 mm and subjected to three temperature cycles can be seen in Fig. 4. Along with Fig. 3 (a), it is seen that the extent of the martensite transformation is in direct relation with the deflection level. Moreover, there is some plastic deformation for material #1 at deflections greater than 0.5 mm. And, the greater the plastic deformation, the more martensite phase that forms. Lots of dislocations, stacking faults, slip lines and deformation twins result from the plastic deformation; this can greatly enhance the transformation of austenite to ε martensite under temperature

Table 2 – The $\sigma_{0.2}$ and $\sigma_{\max}$ of experimental materials at room temperature (300 K) with different deflections

Materials	$\sigma_{0.2}$ (300 K) MPa	$\sigma_{\max}$ (0.5 mm) MPa	$\sigma_{\max}$ (1.0 mm) MPa	$\sigma_{\max}$ (1.5 mm) MPa
1#	433	431	858	1281
2#	488	444	885	1320
3#	390	928	–	–
4#	454	435	867	1294