Journal of Alloys and Compounds 588 (2014) 509–513

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jalcom

Temperature dependence of sensitization on tensile pre-strained AISI 304 stainless steels

Lv Jinlong, Luo Hongyun *

School of Materials Science and Engineering, Beijing University of Aeronautics and Astronautics, Xueyuan Road 37, Beijing 100191, China

ARTICLE INFO

Article history:
Received 9 April 2013
Received in revised form 28 September 2013
Accepted 10 November 2013
Available online 20 November 2013

Keywords:
Metal
Heat treatment
Tension test
Corrosion
Dislocation

ABSTRACT

With the increase of strain, the degree of sensitization of 304 stainless steels increased and desensitization did not occur in 240 h at 380 °C. Although the stress relaxation appeared, the reversed transformation of strain-induced martensite was scarcely observed. Therefore, strain-induced α′-martensite could lead directly to increase of degree of sensitization. Unlike the sensitization at lower temperature, tensile pre-strain promoted desensitization in higher temperature at 575 °C and 675 °C. The higher the sensitization temperature was, the lower the degree of sensitization was. The higher temperature promoted reversed transformation of strain-induced α′-martensite and facilitated recovery and recrystallization of reversed transformed austenite, which resulted in decreased degree of sensitization.

1. Introduction

Austenitic stainless steels are the most widely used corrosion–resistant alloys. However, chromium-rich carbide precipitates ($M_{23}C_6$) form along the grain boundaries, when these steels having sufficient carbon contents are heated at 500–850 °C. The areas adjacent to the grain boundaries suffer depletion of free Cr and this phenomenon is known as sensitization [1]. The sensitized grain boundaries are known to provide easy propagation path for stress corrosion cracking of austenitic stainless steels in chloride solutions [2]. Moreover, the sensitization could induce martensite formation near grain boundary and between austenite/martensite interface [3]. The previous study showed that deformation-induced martensite caused rapid sensitization at below 600 °C and could also produce rapid healing (desensitization) [4]. Types 316L and 316LN did not develop martensite upon cold rolling, therefore, they were not sensitized at 500 °C [5]. Povich and Rao [6] showed that intergranular stress corrosion cracking occurred due to the low temperature sensitization (LTS) for 304 stainless steels components in boiling water reactors for about 10 years at 300 °C. Transmission electron microscopy (TEM) studies have shown that no new carbides nucleated in the boundaries during LTS, however, the existing carbides increased in size by chromium diffusion [6]. Moreover, special grain boundary [7], effective grain boundary energy (EGBE) [8], grain size [9], the grain boundary area [9], recrystallized structure [10] and texture [10] also affected the degree of sensitization (DOS). An attempt was made in the present paper to study the effect of low temperature and high temperature sensitization treatment on the DOS of the tensile strained austenitic stainless steels.

2. Experimental details

The material used is AISI 304 stainless steel plate with chemical compositions (wt.%) as follows: 0.05 C, 1.75 Mn, 0.8 Si, 17.15 Cr, 9.08 Ni, 0.024 P, 0.024 S and balanced Fe. The as-received samples were annealed for 30 min at 1050 °C. The solid solution strips were uniaxially tensile strain to 10%, 20%, 30% and 40% engineering strain at strain rate of $1 \times 10^{-4}\ s^{-1}$ in laboratory air. The solid solution and strained specimens were heat-treated at 380 °C for 240 h, 575 °C for 5 h and 675 °C for 5 h following water quenching, respectively. The volume fraction of α′-martensite in specimen was evaluated according to Ref. [11].

$$V_{\alpha'} = \frac{(1/n)\Sigma_{j=1}^{n}(I_{\alpha'}^{j}/R_{\alpha'}^{j})}{(1/n)\Sigma_{j=1}^{n}(I_{\gamma}^{j}/R_{\gamma}^{j}) + (1/n)\Sigma_{j=1}^{n}(I_{\alpha'}^{j}/R_{\alpha'}^{j})} \quad (1)$$

where n, I and R_i is the number of peaks of the phase used in calculation, the integrated intensity of the reflecting plane and the material scattering factor (R_i), respectively. Cu Kα (0.154056 nm) radiation at 40 kV and 40 mA at 4°/min was used for X-ray diffraction (Rigaku ultima IV) analysis.

The electrochemical cell employed in this study was made of glass beaker with the three electrodes. Very high density graphite and a saturated calomel electrode (SCE) were used as the counter and the reference electrodes, respectively. The electrochemical measurements were performed using CHI660B controlled by a PC. The double loop electrochemical potentiokinetic reactivation (DLEPR) technique experiments were performed according to Ref. [12]. The DLEPR was conducted using 0.5 M H_2SO_4 + 0.01 M KSCN solution. The DLEPR experiments were started after nearly steady state open circuit potential (E_{ocp}) had been reached (about 30 min), and the potential swept in the anodic direction at 1 mV s^{-1} until the potential of

* Corresponding author. Tel.: +86 10 82339905; fax: +86 10 82317128.
E-mail addresses: ljlhit@126.com (J. Lv), luo7128@163.com (H. Luo).

0925-8388/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.jallcom.2013.11.048

0.3 V_{SCE} was reached, then the scan was reversed until the E_{ocp}. The working electrode had been cathodically cleaned by polarization at -1.0 V_{SCE} for 5 min before passive film was formed at open circuit potential (OCP).

In the present study, the DOS can be determined from the current peak values by using the following equations:

$$DOS_1 = (I_r/I_a) \times 100\% \quad \text{for pre-strain} \leqslant 20\% \tag{2}$$

$$DOS_2 = \{(I_{r1}/I_a) + (I_{r2}/I_a)\} \times 100\% \quad \text{for pre-strain} \geqslant 30\% \tag{3}$$

where I_a and I_r are the maximal values of activation and reactivation current.

For the EBSD procedure, the surface of specimens was subsequently electro-polished in a solution of $HClO_4$:CH_3COOH = 2:8 (volume fraction) under 30 V for 20 s. The indexed rates of Kikuchi patterns were better than 85% and some reached 90%. HKL channel 5 software was used for collecting and indexing Kikuchi patterns. The Brandon criterion can be expressed as follows:

$$\Delta\theta_{max} = 15^{\circ}\Sigma^{-1/2} \tag{4}$$

3. Results and discussion

Fig. 1a–d compare the X-ray diffraction patterns of the strain specimens without and with sensitization. The solid solution stainless steel is single phase austenite. The X-ray diffraction patterns indicate no existence of any other phase. With the increasing of engineering strain, the intensities of austenite peaks gradually decrease, while α′-martensite and ε-martensite peaks appear in Fig. 1a [13]. The initial strain-induced α′-martensite almost keeps unchanged up to 240 h at 380 °C in Fig. 1b. However, XRD patterns show a decrease in the half width of the diffraction peaks of α′-strain-induced martensite. It could be attributed to a stress relaxation [14]. A great quantity of strain-induced α′-martensites reversed to austenite with the increasing of temperature in Fig. 1c and d. The content of residual strain-induced α′-martensite after sensitization is shown in Fig. 1e. At 380 °C the strain-induced α′-martensite is scarcely changed, while the higher temperature promotes reversed transformation of strain-induced α′-martensite. The thermal stability of the strain-induced α′-martensite can affect degree of sensitization, which will be discussed subsequently.

The DLEPR results are shown in Fig. 2a–d. The significant activation peaks are observed in Fig. 2a, however, the reactivation peaks are not observed. The significant activation and reactivation peaks can be observed in Fig. 2b, except for the sample without strain. Two reactivation current peaks are observed for the sensitized specimens with 30% and 40% pre-strain in Fig. 2c and d, which is very little reported in the literature [12]. Fig. 3 displays that the magnitude of the DOS increases with the increasing pre-strain at 380 °C and strain does not induce desensitization. However, the DOS decreases with the increasing pre-strain at 575 °C, except for the specimen with 10% pre-strain. The DOS decreases first and then increases with the increasing pre-strain at 675 °C. At 575 °C and 675 °C, the DOS of strained specimen is lower than that of specimen without pre-strain. Therefore, strain induces

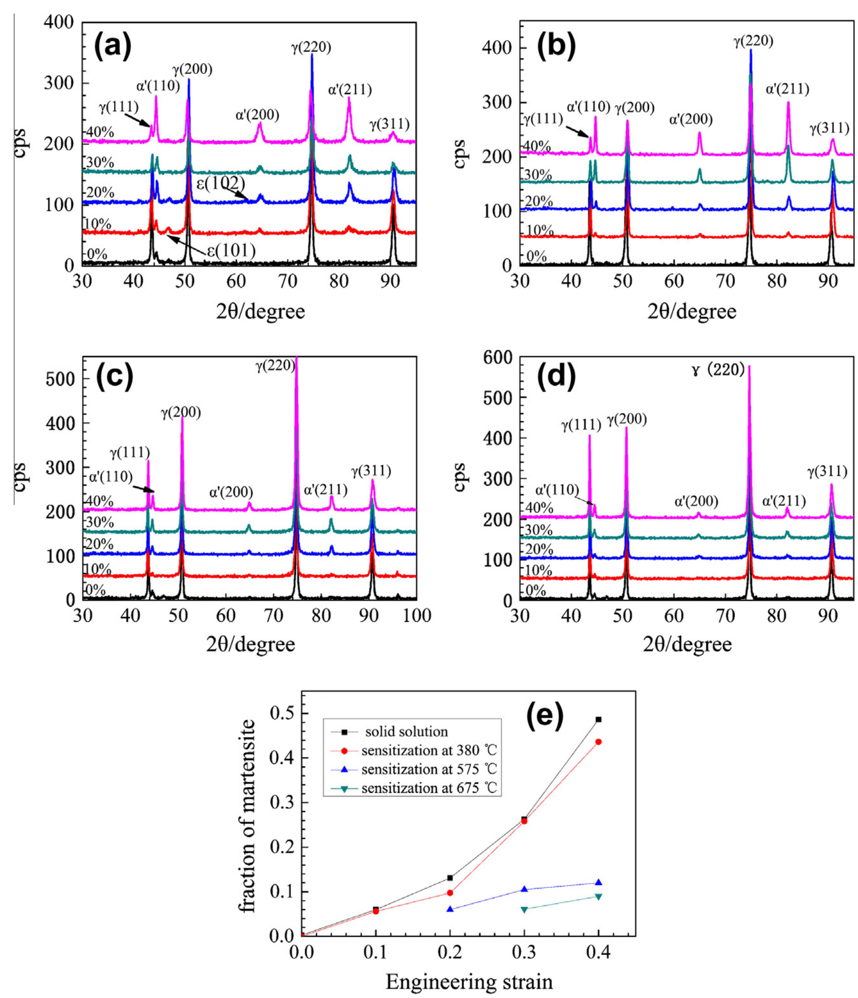

Fig. 1. The X-ray diffraction patterns of AISI 304 austenitic stainless steels introduced at (a) different strain amount for the as-received specimens, (b) sensitized at 380 °C for 240 h for the pre-strained specimens, (c) sensitized at 575 °C for 5 h for the pre-strained specimens and (d) sensitized at 675 °C for 5 h for the pre-strained specimens. (e) The relationship between the engineering strain and martensite phase content.