



Hydrogen effects on microstructural evolution and passive film characteristics of a duplex stainless steel



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ABSTRACT

We revealed the effects of hydrogen on the microstructural evolution and passive film properties of a 2205 duplex stainless steel by the joint use of electron backscatter diffraction (EBSD), electron channeling contrast imaging (ECCI), X-ray photoelectron spectroscopy (XPS) and electrochemical measurements. The microstructural analysis results show that effects of hydrogen on the two phases are different: (i) in austenite, stacking faults are induced by hydrogen, and (ii) in ferrite, hydrogen causes an increase of the dislocation density. The XPS analysis revealed that hydrogen reduced the occurrence of Cr_2O_3 and nitrogen in the passive film, leading to the reduction of their overall thickness. Furthermore, for the first time we demonstrated that the hydrogen release time plays an important role in the electrochemical behavior of the hydrogen charged steel.

1. Introduction

The excellent corrosion resistance of stainless steel is due to the formation of a protective oxide film on the surface exposed to environment. The pitting corrosion, stress corrosion cracking, and corrosion fatigue are all correlated to the passive film. Therefore, understanding of the passive film is essential to the development of advanced materials with superior corrosion resistance. Moreover, the presence of hydrogen in materials can significantly influence the corrosion behavior as well as microstructure and mechanical properties [1–3]. Hydrogen does not just affect the bulk material but also interacts with the passive film of stainless steels. The transport of hydrogen through the passive film can significantly affect the corresponding corrosion processes [4,5]. Furthermore, hydrogen can accelerate the anodic dissolution and decrease Young's modulus of the passive film [6,7].

Although the essential role of hydrogen effects on stainless steel has been noted over the past years [8], the exact role of hydrogen in relation to the microstructural evolution and the resulting properties of the passive film are still not well understood. It should also be noted that most of the electrochemical tests conducted subsequent to hydrogen charging shown in previous literature [9,10] have ignored the hydrogen release time, that is the time elapsed between the end of hydrogen charging and the start of the ensuing testing. The corresponding effect of the release time on the electrochemical behavior of hydrogen charged materials has never been considered.

The aim of this work is to understand the hydrogen effects on a Cr-

Ni-Mo-N duplex stainless steel and the corresponding influence of the hydrogen release time. The steel contains α and γ phases with different hydrogen diffusion coefficients. We combined electron backscatter diffraction (EBSD) and electron channeling contrast imaging (ECCI) techniques to reveal the microstructural evolution induced by hydrogen. Also, X-ray photoelectron spectroscopy (XPS) was employed to disclose the hydrogen effects on the characteristics of the passive films.

2. Experimental procedures

The material used is a 2205 DSS (duplex stainless steel) from Avesta Inc., and the chemical composition in weight percent is 22.39% Cr, 5.68% Ni, 3.13% Mo, 0.17% N, 1.38% Mn, 0.39% Si, 0.023% P, 0.001% S, 0.014% C and Fe balanced. The sample was annealed at 1323 K for 60 min, followed by water quenching. The specimens used for hydrogen charging have a diameter of 10 mm and a thickness of 3 mm. All the specimens were ground with SiC paper and polished with diamond powder. Final polishing was performed with colloidal silicon suspension. Specimens were hydrogen charged at a current density of 15 mA/cm² at room temperature in 0.5 mol/L H_2SO_4 + 2.0 g/L $\text{CH}_4\text{N}_2\text{S}$ solution for 30 min, 60 min and 360 min, respectively. The diffusible hydrogen content in the sample was determined by measuring the cumulative desorbed hydrogen from 25 °C to 400 °C through thermal desorption analysis (TDA) at a heating rate of 26 °C/min. Hydrogen is typically not uniformly distributed in the samples after charging and corresponding models describing its distribution have been introduced in [11,12].

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Electrochemical measurements were conducted in 3.5 wt% NaCl solution with the three-electrode cell systems provided by Solartron system-potentiostat (model 1287A). A platinum sheet and saturated calomel electrode (SCE) were used as counter and reference electrodes, respectively. The specimens were placed as working electrodes. The polarization curves were obtained at sweep rates of 0.5 mV/s, starting from a potential of $-500 \text{ mV}_{\text{SCE}}$ below open-circuit potential to the transpassive direction.

EBS and ECCI analyses were carried out using a Zeiss-Crossbeam XB 1540 FIB SEM and a Zeiss-Merlin instrument. The ECCI is a SEM technique. It reveals highly resolved details of the microstructure on bulk samples [13] and is hence highly suited for observing the microstructure evolution induced by hydrogen [14]. The respective compositions of passive films exposed to different hydrogen charging times were measured by XPS using a Quantum 2000 ESCA microprobe system.

3. Results and discussion

3.1. Microstructural evolution

Fig. 1a and b show the EBSD phase and inverse pole figure (IPF) maps, respectively. As marked in Fig. 1a, four grains in the same sample were selected for ECCI analysis. Fig. 1c reveals a few dislocations both in the γ phase grains (grains '1' and '3') and in the α phase grains

(grains '2' and '4') prior to hydrogen charging. Following the charging of 30 min, the dislocations observed in the γ phase developed into stacking faults (Fig. 1d). The density of stacking faults observed after the initial 30 min charging was increased further by prolonged charging of 60 min in total (Fig. 1e). In some regions, the stacking faults tend to tie together into bundles. With the increase in the charging time to 360 min in total, the density of stacking faults significantly increased (Fig. 1f). In α phase grains, no stacking faults were observed after hydrogen charging, not even after the longest charging times of 360 min. However, dislocation movement induced by hydrogen was observed and the density of dislocations in the α phase was substantially increased after further prolonged charging (e.g. 360 min).

The enhanced dislocation movement and multiplication inside the α phase grains can be attributed to the absorbed hydrogen. The phenomenon of an increased density of hydrogen-induced stacking faults in the γ phase shown above suggests that hydrogen can cause a decrease of the stacking fault energy (SFE). Since the γ phase of 2205 DSS has already a relatively low intrinsic SFE (ca. 10 mJ/m^2) [15], the formation of stacking faults are readily promoted by the presence of hydrogen. This is consistent with earlier work reporting that stacking faults can serve as nuclei for the formation of ϵ or α' martensite [16].

3.2. Passive film analysis

Fig. 2 shows the measured XPS of the passive films that were formed

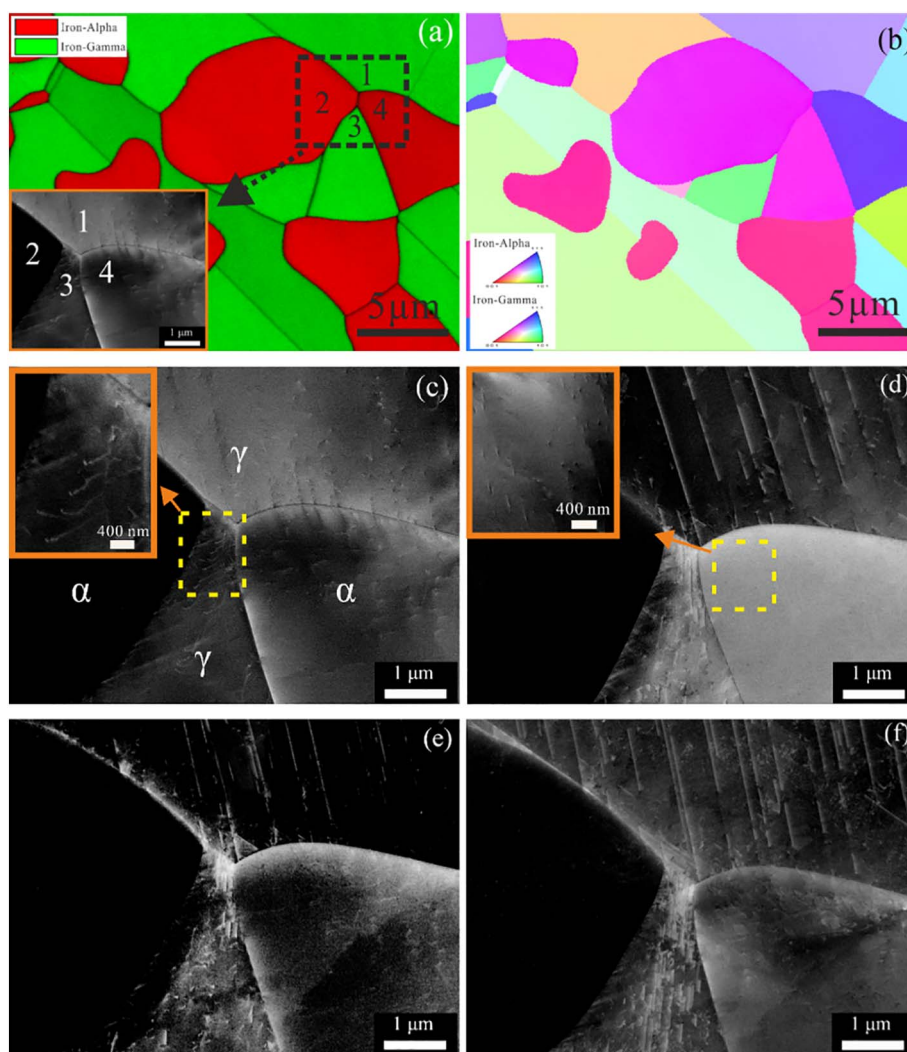


Fig. 1. Combined EBSD and ECCI analyses of samples exposed to hydrogen charging. (a) EBSD phase and (b) IPF maps; ECCI results corresponding to (c) no hydrogen and hydrogen charging time of (d) 30 min, (e) 60 min, and (f) 360 min. EBSD: Electron backscatter diffraction; ECCI: Electron channeling contrast imaging; IPF: Inverse pole figure.

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