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Effect of hydrogen charging on the microstructure of duplex stainless steel

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Abstract

The influence of hydrogen on the microstructure of austenitic–ferritic stainless steel was investigated. Thin foils of this steel subjected to cathodic hydrogen charging were examined using transmission electron microscopy. The effect of hydrogen charging time on the microstructure evolution was studied. The characteristic evolution of the α and γ phases microstructures were observed. The microstructural transformations in the γ phase consist of an increase of the number of stacking faults and dislocation density. Another specific type of structural changes was revealed in the α phase by electron diffraction. The hypothesis of ferrite strong grain refinement was considered. It is suggested that the character of the interphase boundary affects the mode of cracking. Possible explanations of the effects observed are discussed.

Keywords: Metals; Transmission electron microscopy; Grain boundaries

1. Introduction

Although austenitic–ferritic duplex stainless steels (DSS) have an improved resistance to corrosion cracking compared with austenitic stainless steels, they suffer from hydrogen embrittlement (HE) in aqueous solutions. The influence of hydrogen on the microstructure of austenitic and ferritic stainless steels has been described in many articles (e.g. [1–3]). Its influence on the DSS microstructure has been reported to a lesser extent [2,4,5].

The main problem is that hydrogen behaves differently in the ferrite (α) and austenite (γ) phases. In the fcc γ phase, hydrogen has a higher solubility but a lower diffusivity than it has in the bcc α phase [6]. Some authors [7] suggest that interphase boundaries are effective trap sites and can be responsible for the DSS degradation under hydrogen attack.

The aim of this study is to describe the evolution of the DSS microstructure during a hydrogen charging process. Our concern is centred on the relationship between interfaces structure and microcracks modes.

2. Experimental

The material used in this study is 00H26N6 austenitic ferritic duplex stainless steel. The chemical composition of the steel is given in Table 1.

The material was subjected to a thermomechanical treatment applied in two variants A and B (see Table 2) in order to produce two series of specimens (A and B) with the same volume fraction of austenite and ferrite of about 50% but different types of orientation relationships (ORs) between the α and γ phases.

The A treatment led to the precipitation of the γ phase in the form of elongated grains embedded in the ferritic matrix. With the B treatment, a fully recrystallized, duplex microstructure with equiaxed α and γ grains was obtained.

An analysis of the interphase boundaries (IBs) structure revealed that, in state A, most of the γ grains have orientations close to the Kurdiumov–Sachs (K–S) or Nishiyama–Wassermann (N–W) ORs with respect to the ferritic matrix. In state B, the recrystallization that occurs during γ growth destroys the habitual N–W and K–S orientation relationships between the γ and α phases and leads to random misorientation between these phases. This means that the main difference between the two states results in special IBs structure that is characterized by low

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Table 1
Chemical composition of 00H26N6 steel

Element	C	Ni	Cr	Mn	P	S	Si	Mo	N
Weight (%)	0.03	6.0	26.5	0.24	0.005	0.014	0.21	0.04	295 ppm

Table 2
Schematic description of the thermomechanical treatments

Treatment	A	B
Step 1	Cold rolling up to 50%	Annealing 1260 °C–30 min in vacuum, oil quenching
Step 2	Annealing 1260 °C–30 min in vacuum, oil quenching	Cold rolling up to 50%
Step 3	Annealing 960 °C–1 h, oil quenching	

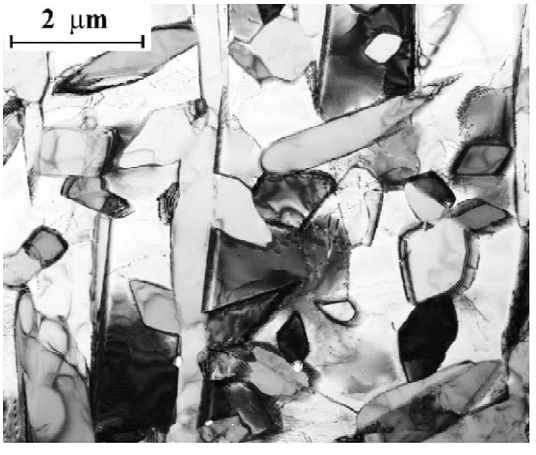

Fig. 1. A – state microstructure.

energy in the state A (where IBs fulfill special ORs) and is random in the state B (for which IBs deviate from special ORs) [8,9].

The microstructures of the A and B steel states are shown in Figs 1 and 2.

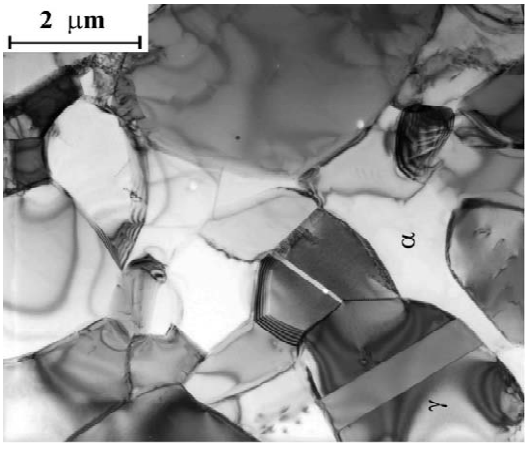

Fig. 2. B – state microstructure.

The samples for transmission electron microscopy (TEM), in the form of discs 3 mm in diameter and 0.2 mm thick were prepared by two-sided electrolytic thinning. The microstructure was examined in an electron microscope operated at 100 kV.

Hydrogen charging of previously observed thin foils was carried out electrolytically in 0.5 M H_2SO_4 at room temperature. A platinum counter electrode was used. The charging experiments were performed up to 3 h with a current density of 35 mA/cm^2.

3. Results and discussion

Post-charging TEM observations allow controlling the evolution of the DSS microstructure. In both A and B states the first microstructural changes occur in the α phase and consist of an increase of dislocation density (Fig. 3). Then, stacking faults appear in austenite and their density increases with the duration of the hydrogen charging process.

After 3 h of charging, strong changes can be observed in

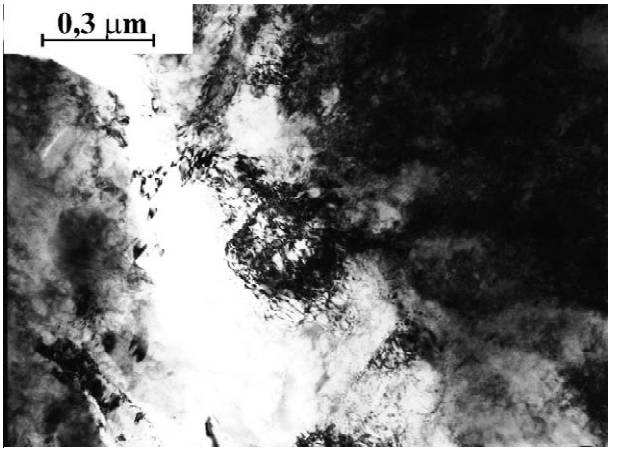

Fig. 3. Ferrite – accumulation of dislocations.

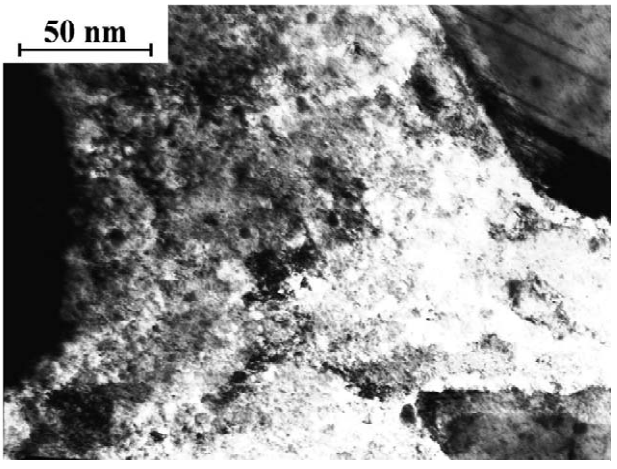

Fig. 4. Ferrite – bright field image.

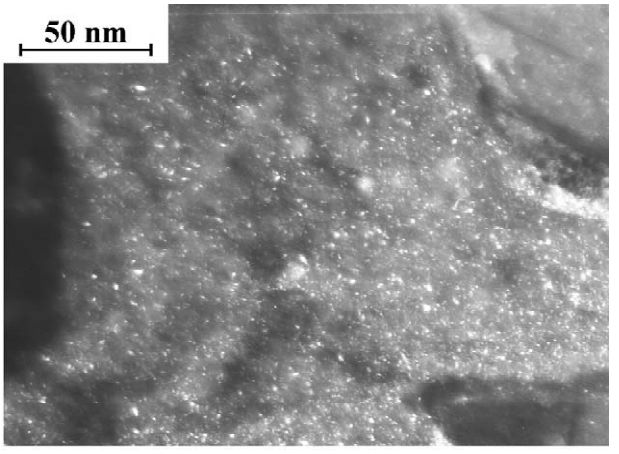

Fig. 6. Ferrite – dark field image.

the regions where the ferrite grains have a high dislocation density. On the diffraction patterns corresponding to these regions, distinct diffraction rings appear. The dark field image taken from the ring indicates that it corresponds to the very small grains of nanometric size (Figs. 4–6). It can be thus postulated that the strong grain refinement occurred in ferrite grains. This effect has been already observed in ferritic steel by Szummer et al. [2].

A question arises: what are the reasons for the observed changes? The appearance and the increase of the dislocation density in ferrite may result from the stresses induced by hydrogen absorption into the interstitial sites [10]. Moreover, the dislocations constitute effective traps for hydrogen atoms [11,12]. Therefore, the dislocations may enhance the hydrogen absorption leading to additional stresses and further dislocation accumulation. The observed subsequent strong changes of the α microstructure may be due to the accumulation of high dislocation density. Alternatively we may suppose that the dislocation accumulation and possible grain refinement in ferrite are due to hydride formation. However the strongly perturbed microstructure images did not permit us to verify which of these hypotheses is true. To settle this question further studies are needed.

The microstructure changes in the γ phase may also result from absorption of hydrogen, which reduces the stacking fault energy leading to the increase of stacking faults density [13].

Other effects that were revealed are the microcracks, which first appear in the α phase and then in the γ phase and α/γ IBs. The microcracks nucleate after 20 min of charging. In the A state they mostly have a transgranular character, and in the B state rather an intergranular character (Figs. 7–8). The microcracks observed were a few micrometers in length. In case of γ phase they are straight and seem to be parallel to the stacking fault {111} plane. This confirms the observations of Oltra et al. [14].

Wang [15] proposed a model that explains HE in iron and steels. This model assumes that hydrogen atoms segregate to grain boundaries and provoke decohesion. It fits well the case of high energy boundaries, but does not correspond to the low energy boundaries. The tendency of hydrogen atoms to segregate is generally higher in dis-

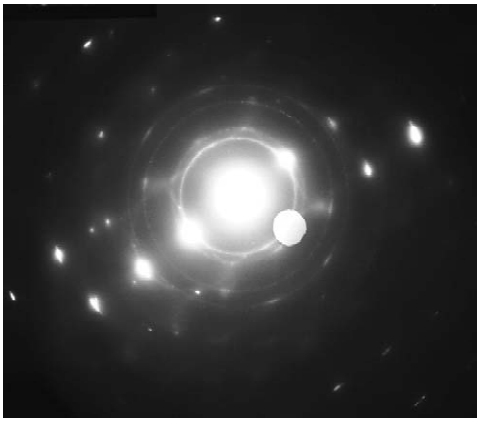

Fig. 5. Ferrite – diffraction. Circle marks the ring from which was taken dark field image.

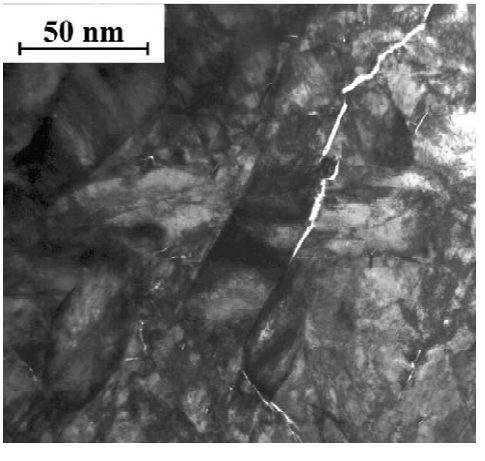

Fig. 7. Intergranular microcrack along the interphase boundary.

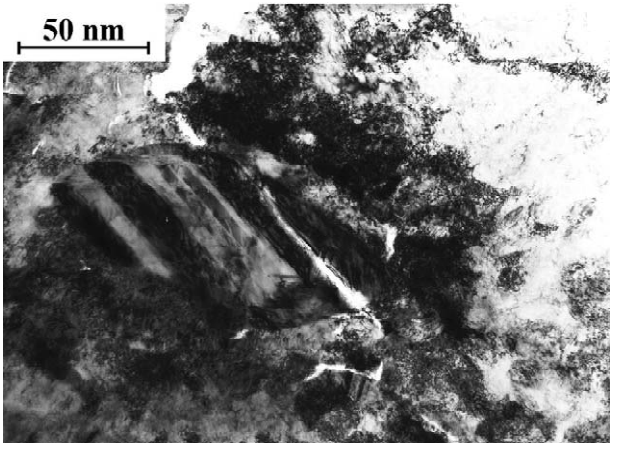

Fig. 8. Transgranular microcrack along the stacking fault in austenite.

ordered, high energy boundaries than in low energy, ordered interfaces. Therefore, the latter are more resistant to an intergranular fracture than former ones. In our case, the high energy interphase boundaries occur in B specimens where we observe intergranular cracks, whereas in A specimens with low energy boundaries, the cracks have rather a transgranular character.

4. Conclusions

Microscopic examinations of the samples charged with hydrogen allowed us to observe microstructure evolution.

Under the influence of hydrogen the α and γ phases behave differently. The α phase gradually decays and undergoes a strong change, probably connected with a grain refinement. In the γ phase the number of stacking faults significantly increases.

An increase of the charging time favours the formation of microcracks. Their character depends on the predominant kind of boundary. In the steel state with low energy boundaries a majority of the observed microcracks is transgranular. Intergranular microcracks mostly occur in the steel state with high energy boundaries.

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