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Effect of grain size on the hydrogen embrittlement sensitivity of a precipitation strengthened Fe–Ni based alloy

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ABSTRACT

The hydrogen embrittlement (HE) sensitivity of a precipitation strengthened Fe–Ni based alloy with various grain sizes was investigated by hot hydrogen charging experiment and tensile test. The results showed that HE sensitivity was reduced with decreasing grain size, even though the hydrogen concentration in the small-grained specimen was higher than the large-grained specimen. Fractographic features showed that intergranular fracture was less pronounced with decreasing grain size, which was related to the transport of hydrogen to the grain boundary by moving dislocations. During deformation, the number of dislocations expected in the slip bands was proportional to the grain size, leading to that a lower hydrogen concentration being accumulated at the grain boundary in the small-grained specimen.

1. Introduction

The precipitation strengthened Fe–Ni based alloys, such as A286 and JBK75, have been widely used as structural materials in the hydrogen environment because of their high strength, acceptable hydrogen performance and excellent corrosion resistance [1–3]. These alloys are strengthened by the precipitation of ordered coherent $\gamma'[Ni_3(Al,Ti)]$, which is formed during high temperature aging. However, investigations indicated that these alloys would lose about half of their ductility after hydrogen charging, accompanied by a change in fracture mode from ductile fracture to brittle-appearing intergranular fracture [1–7].

The susceptibility to hydrogen-induced fracture of precipitation strengthened Fe–Ni based alloys is closely related to their microstructures. It had been shown that intergranular fracture was correlated with the grain-boundary η phase because the incoherent interface between η phase and matrix was considered as a strong hydrogen trap [7,8]. Thereafter, Zhao et al. [9] found that addition of minor boron could retard the precipitation of η phase so as to improve the HE sensitivity of Fe–Ni based alloys. But intergranular fracture still occurred after hydrogen charging in the Fe–Ni based alloys. In addition, previous work suggested that the amount of γ' phase could influence the hydrogen-induced fracture behavior of precipitation strengthened Fe–Ni based alloys due to the enhanced slip planarity with the increasing amount of γ' phase [10].

Another important microstructural parameter is the grain size which influences the strength and ductility of alloys. However, the effect of grain size on the HE sensitivity is a controversial point. Martinez-Madrid et al. [11] found that a fine-grained iron had the lower HE sensitivity because of a lower hydrogen concentration at the grain boundary of fine grains. But in 4340 steel, the HE sensitivity could be improved with increasing grain size, reflected in a higher threshold stress intensity and lower crack growth rate [12]. Contrary to the above results, no appreciable differences in the HE sensitivity were observed in the micro-alloy steels with various grain sizes [13]. In this paper, various grain sizes were obtained through different heat treatments, in order to clarify the effect of grain size on the HE sensitivity of a precipitation strengthened Fe–Ni based alloy.

2. Experimental

A precipitation strengthened Fe–Ni based alloy was produced by vacuum induction melting technique with the chemical composition as follows: 30Ni–15Cr–1.3Mo–1.88Ti–0.36Al–0.24V–0.2Si–0.0008B–Fe bal. The ingot was homogenized at 1433 K for 20 h and then forged and rolled into 15 mm diameter bars. In order to produce the alloys with various grain sizes, specimens were solution treated at 1173 K, 1213 K, 1253 K and 1303 K for 1 h

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and water quenched. The solution-treated specimens were then aged at 1013 K for 8 h followed by air cooling and they were designated as 1173-SA, 1213-SA, 1253-SA and 1303-SA according to the solution temperature.

Specimens for optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and tensile test were spark machined from the bars. The Specimens for OM and SEM observations were mechanically polished, followed by electro-etching in a solution of 10% chromic acid. Thin foils for TEM analysis were prepared by double-jet polishing at 253 K in 10% perchloric acid ethanol solution and TEM examinations were conducted on FEI Tecnai G220 TEM. The tensile specimens were machined with the gauge sections of 5.0 mm in diameter and 25 mm in length. Tensile tests were carried out immediately at room temperature at a strain rate of $1.3\times10^{-3}\,s^{-1}$ after hot hydrogen charging experiment, which was conducted at 573 K for 240 h under a hydrogen pressure of 10 MPa. To quantify the HE sensitivity, an embrittlement index (EI) was defined according to the following equation:

$$\mathrm{EI}=\frac{RA_0-RA_H}{RA_0} \tag{1}$$

where RA_0 and RA_H are the reduction in area of hydrogen-charged and uncharged specimens, respectively. The hydrogen concentration of specimens after hydrogen charging was measured using a RH-404 hydrogen determinator (LECO) with an accuracy of ± 0.05 ppm.

3. Results

3.1. Microstructural observation

Fig. 1 shows the typical microstructure of specimens in the as-rolled and solution-treated conditions. For the as-rolled specimen, a γ polycrystalline structure is formed and no precipitates are observed in the matrix (Fig. 1a). After solution-treated at 1173 K, some lamellar precipitates (indicated by black arrows in Fig. 1b and inset) identified as η phase in the previous paper [14], are formed in the matrix. It has been demonstrated that the presence of η phase is detrimental to the HE sensitivity of Fe-Ni based alloys [7,8]. As shown in Fig. 1(c) and (d), η phase does not exist in the matrix when the solution temperature is raised above 1173 K, which is approximately the solvus temperature of η phase. So the solution temperature is selected above 1173 K in order to avoid the introduction of η phase.

The solution-treated specimens were aged at 1013 K for 8 h for the precipitation of γ' phase. As shown in Fig. 2, there are no obvious differences in the microstructure other than the grain sizes of the specimens by OM and SEM observations. The average grain size measured by the linear intercept method, increases from 32 μm to 95 μm by an increase in the solution temperature from 1213 K to 1303 K, as shown in Table 1.

3.2. Mechanical properties

The tensile properties of specimens with various grain sizes after hydrogen charging are summarized in Table 2. The increase in the grain size results in a decrease in the yield strength and ultimate strength, which is in accordance with the Hall–Petch theory [15,16]. In general, hydrogen has little effect on the strength of Fe–Ni based alloy, but a strong effect on the ductility [4,9]. As shown in Table 2, the EI of the heat-treated specimens are all over 30%, showing that an obvious decrease in the RA is induced by hydrogen-charging. For the 1303-SA with a grain size of about 95 μm, the EI is 44.6%. The reduced HE sensitivity is observed with decreasing grain size, indicating that the EI decreases from 44.6% to 34.8% as the grain size is reduced from 95 μm to 32 μm.

3.3. Fractography

Fig. 3 shows the the fracture surface of the tensile specimens with various grain sizes before and after hydrogen charging. As shown in Fig. 3(a–c), all specimens exhibit a typical dimpled feature of microvoid coalescence (MVC) before hydrogen charging. However, intergranular fractures (indicated by black arrows) are

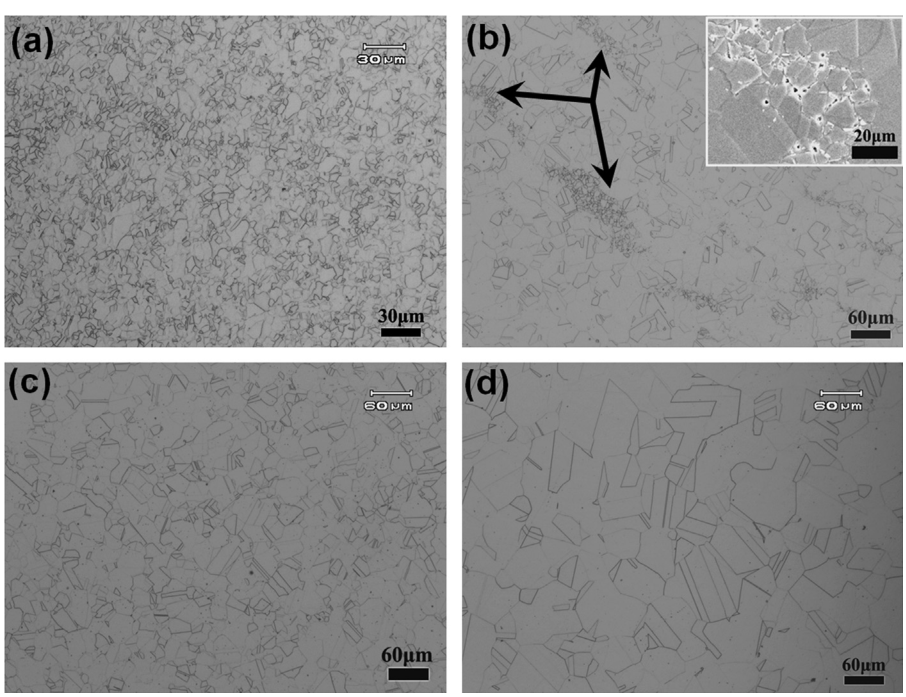

Fig. 1. Optical microstructure of the alloys in (a) as-rolled condition and after solution-treated at (b) 1173 K,(c) 1213 K, and (d) 1253 K. The inset in (b) is the higher magnification image of η phase.