Materials Science & Engineering A 739 (2019) 31–36

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

The effect of hydrogen charging on the evolution of lattice defects and phase composition during tension in 316L stainless steel

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ARTICLE INFO

Keywords:
316L stainless steel
Hydrogen charging
Tension
Dislocation density
Martensitic transformation

ABSTRACT

The effects of hydrogen charging on the evolution of strain-induced lattice defects and phase transformation were investigated in 316L stainless steel. The initial material was obtained by cold rolling to a thickness reduction of 20%. The surfaces of some of the initial samples were cathodically charged with hydrogen. Afterward, both the charged and uncharged samples were subjected to tension until failure. The dislocation density and twin-fault probability in the charged and uncharged specimens during tension were compared. We found that hydrogen charging reduced the degree of increase in the dislocation density and twin-fault probability during the application of tension to the same strain level. Significant martensitic phase transformation was observed in the uncharged samples tested to the strain of 10% or higher. In the hydrogen-charged samples, only a slight increase in the martensite phase fraction was detected. A correlation between the α′-martensite fraction and the dislocation density was found for the studied samples, suggesting that the lower degree of martensitic phase transformation in the charged 316L steel was caused by the smaller amount of stress developed due to the lower dislocation density. In accordance with the differences observed in the phase composition and defect densities, the hydrogen-charged material exhibited lower surface hardness.

1. Introduction

Hydrogen embrittlement in austenitic stainless steels continues to attract considerable attention because this phenomenon has a significant impact in many functional and structural applications, such as hydrogen storage and transportation [1–3]. For example, oxide-induced crack closure in steels was found to be reduced by hydrogen, leading to accelerated crack propagation [4]. One apparent cause is that the hydrogen diffusion rate in the α′-martensite phase is higher than that in the γ-austenite phase; therefore, in the former phase, the effect of hydrogen on crack propagation is higher [5–7]. Consequently, stainless steels with lower austenite fraction usually exhibit higher propensity to hydrogen embrittlement. Among the numerous types of austenite stainless steel available, 316L stainless steel is less susceptible to hydrogen-assisted fracture due to the higher stability of the austenite phase against martensitic transformation [3,5,6]. Accordingly, 316L steel is a frequently used structural material in hydrogen environments despite its still remaining potential issues with hydrogen embrittlement.

Indeed, numerous studies have investigated the influence of hydrogen-charging on the microstructure and mechanical behavior of austenitic stainless steels [2,8–10]. Previous studies showed that hydrogen embrittlement in steels strongly depends on many factors, such as hydrogen-charging conditions [11], hydrogen concentration [12,13], hydrogen diffusivity [9] and the chemical composition of the alloy [6,14,15]. Therefore, the study of hydrogen embrittlement is quite complicated since many variables must be experimentally controlled in order to achieve accurate results. Hydrogen embrittlement is likely to occur if the concentration of hydrogen in steel is above a certain limit [13]. In addition, hydrogen-assisted fracture is very sensitive to the stacking fault energy (SFE) and slip localization [16,17], making it dependent on the chemical composition of the steel [8,18]. The large nickel content of ~ 10 wt% in 316L stainless steels plays a significant role in resistance against hydrogen-assisted fracture; however, similar effects for other alloying elements have not been observed [2,8]. The effect with higher nickel content was attributed to the increase of SFE in steels, leading to better stability of the austenite phase [18], which increases the resistance against hydrogen-assisted fracture. The carbon content in 316L steel is very small (in the range of 0.014–0.043%) and has no significant effect on hydrogen embrittlement [8].

In many metallic materials, the dissolved hydrogen interacts with

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https://doi.org/10.1016/j.msea.2018.10.005
Received 18 July 2018; Received in revised form 1 October 2018; Accepted 6 October 2018

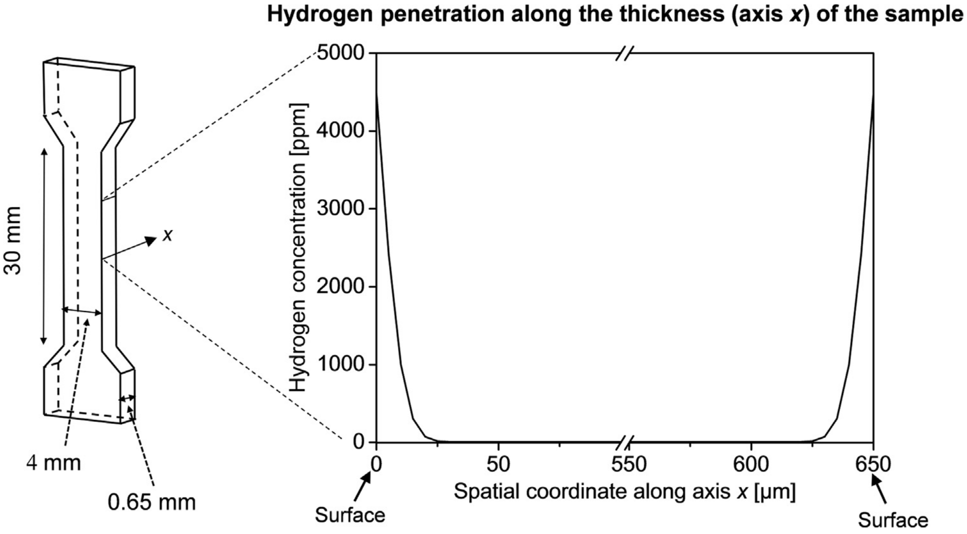

Fig. 1. The predicted hydrogen distribution along the thickness of the specimens.

lattice defects, thereby influencing the plastic behavior. A previous study reported that the presence of hydrogen in 316L steel caused a softening effect [9]. This softening effect and the early fracture were attributed to the enhanced local creation of vacancies during deformation in hydrogen-charged samples [19]. The higher strain-induced vacancy concentration may increase the ease of dislocation climb, leading to softening. In addition, the higher vacancy content may lead to earlier agglomeration into voids, thereby promoting the fracture of the samples charged with hydrogen, i.e., it contributes to hydrogen embrittlement. In addition, the interaction between hydrogen and dislocations may influence the mechanical properties of metals. On the other hand, some researchers suggested that hydrogen in metals and alloys reduces the lattice friction stress; therefore, the dislocation movement in the crystal lattice is easier, resulting in softening [16]. According to the concept introduced by Birnbaum and Sofronis known as hydrogen enhanced localized plasticity (HELP) [16], the hydrogen atoms segregated to dislocations can reduce the energy of interaction between the dislocations by reducing the elastic stress field. Consequently, dislocations can move at lower applied stress levels in hydrogen-charged samples.

It is well known that α′-martensite with a body-centered cubic (bcc) structure is more rigid than face-centered cubic (fcc) γ-austenite. However, the hydrogen embrittlement in stainless steels cannot be attributed to the less ductile nature of α′-martensite. Rather, hydrogen-charging usually leads to a smaller fraction of α′-martensite during straining due to the lower level of internal stress in the initial austenite phase as discussed above [15,20,21]. When the α′-phase forms, the hydrogen concentration in the neighboring γ-austenite increases due to the lower solubility of hydrogen in the martensite phase [22]. The larger hydrogen concentration in the remaining γ-austenite between the α′-phase regions leads to a softer γ-phase. This microstructure exhibits a higher propensity to strain localization in the γ-phase, which may contribute to hydrogen embrittlement in 304 and 316L steels [22].

Although the effect of hydrogen on mechanical performance in stainless steels has been studied in numerous papers, a systematic investigation of the influence of hydrogen charging on the lattice defect structure in 316L steel is still missing from the literature. In this paper, the effect of hydrogen on the dislocation density and twin-fault probability developed during tension in a 316L stainless steel is studied. In order to carry out the investigation with good statistics, defect density measurement was performed using X-ray line profile analysis. In addition, the α′-martensite fraction and hardness were compared in hydrogen-charged and uncharged samples. This study contributes to a better understanding of the effect of hydrogen on the defect structure and the concomitant mechanical properties of 316L steels.

2. Experimental materials and procedures

The material used in the present study was a 316L stainless steel with an alloying element composition of 17.20% Cr – 8.97% Ni –2.13% Mo – 1.03% Mn – 0.77% Si – 0.48% Cu – 0.35% Co (in wt%). First, the 316L stainless steel material was annealed at 1373 K for 1 h and then quenched in water to room temperature. This thermal treatment resulted in a single-phase γ-austenite microstructure. The annealed plates were then cold-rolled until they could achieve a 20% reduction in thickness. This material was considered the initial state in this study. Dog-bone-shaped tensile specimens were machined from the initial material with a gauge length, breadth, and thickness of 30.0, 4.0, and 0.65 mm, respectively. Some specimens were treated with a cathodic hydrogen-charging process. The hydrogen-charging was carried out at a temperature of 85 °C for ~ 60 min in a 0.5 M H_2SO_4 solution containing 250 mg L^{-1} As_2O_3 as a hydrogen recombination poison. A current density of 200 A m^{-2} was applied between the tensile samples and the platinum anode. Hydrogen concentration measurement was carried out using a LECO RH 404 analyzer and the average hydrogen content in the entire volume of a test sample was determined by averaging the values obtained from three different test samples. The measured average hydrogen content after charging was 94 ± 9 ppm for the entire volume of the specimen. Based on this measured average value, the spatial distribution of the hydrogen concentration was determined using a model of the diffusion-controlled charging process as described in Ref. [14]. This model is applicable because unlike the gas-phase charging method, a laboratory-scale cathodic charging process usually results in relatively short hydrogen diffusion distances in the sample [23]. Fig. 1 shows the predicted hydrogen distribution along the thickness of the charged specimens. It is apparent from the hydrogen distribution that significant hydrogen content can be found only in the outermost surface layers within the approximate thickness of 30 μm. The hydrogen-charged and uncharged samples were deformed by tensile testing to a selected engineering strain of ~ 10% and until failure (~ 18%), with a constant strain rate of 10^{-3} s^{-1} at room temperature.

The microstructure of the initial material was investigated using an FEI Quanta 3D scanning electron microscope (SEM). For the electron backscattered diffraction (EBSD) experiments, first the surface was mechanically polished with SiC abrasive papers. The last step in this process was carried out with a 4000 grit paper. Then, two-step polishing was performed using a colloidal silica suspension (OP-S) with particle sizes of 1 μm and 40 nm. Finally, the surface was