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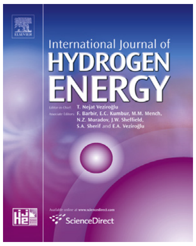

Hydrogen-induced cracking mechanism of precipitation strengthened austenitic stainless steel weldment

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ABSTRACT

Precipitation strengthened austenitic stainless steels are widely used in hydrogen related applications. However, their applications may face hydrogen damage resulting in hydrogen-induced delayed failure. Results show that the weld is more sensitive to fracture and hydrogen-induced failure than the matrix. High density curved dislocations, abundant of large size precipitates and considerable γ' precipitates coarsening are found in the weld. Large size precipitates are found to be major hydrogen traps and preferential microcrack nucleation sites. The γ' precipitates coarsening make the weld more ductile than the matrix. With the decrease of the applied stress, hydrogen-induced cracking mechanism in the weld changes from brittle transgranular fracture to brittle intergranular fracture.

Introduction

Hydrogen embrittlement, in the forms of hydrogen-induced ductility loss, hydrogen-induced cracking, hydrogen-induced delayed fracture, happens via hydrogen adhesion, permeation, diffusion and localized concentration in materials [1–5]. It is the primary factor of materials failure in petrochemical engineering, nature gas, nuclear power, hydrogen energy, chemical processing, aircraft and spacecraft industries [6–8]. With the development of these fields, better hydrogen resistant alloys are needed for harsh hydrogen environment applications. In addition to have good resistance to hydrogen embrittlement, the alloys also need to have excellent mechanical properties in the hydrogen rich environment.

Due to the relatively smaller hydrogen diffusion coefficient, better cryogenic properties and better resistance to hydrogen embrittlement than other types of alloys, austenitic alloys are widely used in hydrogen related applications [9–17]. But the yield strength of common single-phase austenitic alloys is in the range of 200–500 MPa [9,13,16]. That is too low to carry high intensity of work pressure. Fe–Ni based alloys, including A-286 series and Incoloy series, embed coherent γ' [$Ni_3(Ti,Al)$] precipitates in austenite substrate to get strengthening, owning the high yield strength in the range of 650–850 MPa [18–21]. These Fe–Ni based alloys have improved mechanical properties, preserving small hydrogen

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diffusion coefficient and less hydrogen embrittlement susceptibility [12,13,19–21]. Investigations showed that these traditional A-286 series alloys would lose about half of the ductility in the tensile tests after hydrogen charging, and accompanied by a change of fracture mode from ductile fracture to brittle-appearing intergranular fracture [22]. This embrittlement was attributed to the unfavorable precipitates, including η (Ni_3Ti), G ($Ni_{16}Ti_6Si_7$) and carbides (e.g. TiC) [22–25]. The coherent γ′ phase does not contribute to hydrogen embrittlement before it loses coherency with austenite substrate [22], which is related to particle size and strain [22,24,26,27]. Alloying and heat treatment (especially aging condition) were studied to minimize these unfavorable precipitates. Minimizing P (Mn) can increase γ′/γ misfit hence decrease the reduction area loss [28]. It has shown that by adding trace boron, η precipitate can be impeded [29,30]. Reducing carbon can reduce the carbide content. In addition, the aging treatment effect on γ′, η, G and carbides precipitates were also studied [31,32].

For large scale applications, it is always necessary to join various parts together. Welding is the most common way to achieve this purpose. However, the welding process generally causes differences between the weld and matrix microstructures, such as grain coarsening of the heat affected zone, casting structure of the fusion zone, residual stress, supersaturated vacancies, some large inclusions and different types of precipitate phases [33–36]. And, these differences are always difficult to be eliminated by heat treatment or other ways. Inevitably, the presence of these differences has implications for hydrogen transport, deformation mechanism, crack initiation and growth mechanisms within the weld [37–39]. Some investigations about other types of stainless steel also showed that the welds were more sensitive than the matrix to hydrogen embrittlement due to the microstructure of the weld region [40–43].

As a high energy density fusion welding technique, electron beam welding is widely used in joining precipitation strengthened austenitic stainless steels on account of its narrow heat affected zone, less contamination and high penetration depth [44,45]. Normally, electron beam welding is applied pre-aging so as to precipitate γ′ phase. Recent research has shown that electron beam weld has very different features than the matrix [44,45]. The fusion zone is rich in dendrites. Chemical composition and the γ′ precipitates distribution are remarkably inhomogeneous. Ti segregation at interdendritic regions results in nonuniform distribution of γ′ precipitates. This microstructural inhomogeneity can induce the inhomogeneity of mechanical properties and step-like fracture topography [45,46].

However, detailed precipitates properties in the electron beam weldment with the precipitation strengthened austenitic stainless steel especially that of γ′, η, G phases, carbides and their effects on hydrogen embrittlement are still missing. In this work, constant load tensile tests with dynamic charged hydrogen were carried out to simulate the service condition. Scanning electronic microscope (SEM) was employed to study the weldment microstructure and the fracture surface. Transmission electron microscope (TEM) was used to study the weldment precipitates. *In-situ* tensile tests were also conducted with TEM to reveal the propagation of cracks in real time. Finally, microstructure, precipitates properties and the relationship with hydrogen-induced cracking of the studied precipitation strengthened austenitic stainless steel electron beam weldment are clarified.

Experiment details

Table 1 shows the chemical composition of precipitation strengthened austenitic stainless steel used in this research. The plate samples were annealed at 1253 K for 1 h, and then quenched into water. The treated plate was full-penetration butt-welded in a vacuum electron-beam welding apparatus, then aged at 1013 K for 8 h to eliminate internal stress and obtain a near peak-aged microstructure.

A small specimen was then polished and etched in 50% aqua regia glycerol etchant for 2–3 min. The width of the weld was about 2 mm. The specimen was examined with an SEM. Single cycle electron beam welding could result in a fusion zone composed of a columnar grain zone and an equiaxed grain zone. The microstructure of the matrix and the weld is shown in Fig. 1. The matrix shows typical austenite with the average grain size of 40–50 μm and some annealing twins, as shown in Fig. 1(a). There is no heat-affected zone with large grains near the matrix/weld interface can be observed, as shown in Fig. 1(b). The weld shows cast microstructure with clear fusion lines, columnar grains and small equiaxed grains. The columnar grain zone results from the fast heat dissipation of the weld-matrix metal interface [45] and directional solidification, as shown in Fig. 1(c). The equiaxed grain zone results from the low heat dissipation rate in the weld center. Due to the very narrow weld width, the equiaxed grain zone is very small compare to the columnar grain zone, as shown in Fig. 1(d).

Fig. 2 is an illustration of the plate tensile specimen with the thickness of 0.6 mm. All macroscopic tensile specimens were prepared with the same dimensions. The weld was positioned at the center of the tensile specimens. The specimens with or without the weld were then polished down to 1200 mesh SiC abrasive paper. Tensile tests were carried out in the air at room temperature with a strain rate of 5×10^{-4}/s. All of the specimens with the weld fractured in the weld zone. Table 2 shows the mechanical properties of the matrix and the weld. The yield strength and ultimate tensile strength of the weld are lower than the matrix.

Polished specimens without any applied stress were charged in 0.5 mol/L H_2SO_4 + 0.25 g/l As_2O_3 solution with a current density of 400 mA/cm^2. Hydrogen damage on the surface of the charged hydrogen specimen was examined by SEM.

Table 1 – Chemical composition (wt.%) of the precipitation strengthened austenitic stainless steel that was used in this study.

Element	C	Mn	Si	Ni	Cr
Content	≤0.02	Minimize	0.10/0.30	29/32	13.5/16.5
Element	Mo	Al	Ti	V	Fe
Content	1.0/1.60	0.10/0.40	1.8/2.4	0.15/0.35	Balance

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