Available online at www.sciencedirect.com

SciVerse ScienceDirect

Scripta Materialia 66 (2012) 956–959

Scripta MATERIALIA

www.elsevier.com/locate/scriptamat

Hydrogen effects on cathodically charged twinning-induced plasticity steel

J.A. Ronevich,[a,*] S.K. Kim,[b] J.G. Speer[a] and D.K. Matlock[a]

[a] *Advanced Steel Processing and Products Research Center, Department of Metallurgical and Materials Engineering, Colorado School of Mines, 1500 Illinois Street, Golden, CO 80401, USA*

[b] *POSCO Technical Research Laboratories, Gwangyang 545-711, Pohang, Republic of Korea*

Available online 20 December 2011

Cathodically charged twinning-induced plasticity (TWIP) steel was evaluated to determine the effects of brief hydrogen charging on the tensile properties. Ductile fracture mechanisms and bulk tensile properties were unaffected by hydrogen contents of up to 9.5 ppm. However, surface microcracks were observed to depths comparable to calculated hydrogen penetration depths, up to 6 μm for samples charged for 6 h. These results highlighted the limited applicability of using pre-charged tensile samples to assess the potential for delayed cracking of austenitic TWIP steels.

Keywords: Tension test; Austenitic steels; Hydrogen embrittlement; Fracture; Twinning induced plasticity steels (TWIP)

Delayed fracture of high-strength steels has become a growing concern with the development of advanced high-strength steels (AHSS), e.g. dual-phase (DP), transformation-induced plasticity (TRIP) and twinning-induced plasticity (TWIP) steels. TWIP steels are austenitic and represent one specific AHSS class which typically exhibits higher strengths and ductilities than observed in ferritic-based AHSS steels (i.e. DP and TRIP). However, delayed cracking has been observed in deep-drawn cups produced from various TWIP steel grades [1–3]. Recent studies [1–3] showed that TWIP with aluminum additions has reduced sensitivity to delayed fracture, due to residual internal hydrogen from the processing. The enhanced resistance to delayed fracture of Al-alloyed TWIP steels was suggested to result from an increase in stacking fault energy by aluminum which promoted twinning and suppressed martensite formation [1–3]. Recently [4] tensile testing of selected pre-charged AHSS grades was used to assess the importance of microstructure on ductility loss as a function of hydrogen content, and the results revealed that the TWIP steels studied (both with and without an Al addition) exhibited limited sensitivity to hydrogen charging. The purpose of this study is to examine further the response of hydrogen-charged TWIP steels in order to provide a better interpretation of the observed lack of hydrogen sensitivity in pre-charged tensile samples.

The material examined in this study consisted of 1.2 mm thick TWIP steel with the following composition (in wt.%): 0.6C, 1.5Al, 18Mn, 0.016P, 0.046S, 0.31Si, 0.38Cr, 0.04V, 0.03Nb [4]. Following established procedures from related AHSS hydrogen embrittlement studies [4], ASTM E-8 tensile samples were cathodically charged at 10 mA cm^{-2} in 1 N H_2SO_4 electrolyte with an addition of 10 mg l^{-1} As_2O_3 at room temperature. The times were varied between 1 and 6 h to obtain systematically varied charged hydrogen contents. Hydrogen contents were determined on coupons using the RH-404 Hydrogen LECO® hot extraction method and thus values reported are bulk measurements [4]. To remove oxides prior to charging, all samples were lightly ground (through 600 grit) in the rolling/tensile direction. Tensile samples were removed from the charging bath, dried, and placed into an electromechanical tensile frame within 1.5–2 min following the charge. Tests were performed at 3.3×10^{-4} s^{-1} and a 50.8 mm extensometer was used to measure the displacement. Selected TWIP steel samples were examined by field emission scanning electron microscopy (FESEM) to study the fracture morphology and surface cracks.

Engineering stress–strain curves for hydrogen-charged TWIP steel are shown in Figure 1 with hydrogen

* Corresponding author. Tel.: +1 303 273 3090; fax: +1 303 273 3016; e-mail: jronevic@mines.edu

1359-6462/$ - see front matter © 2011 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.
doi:10.1016/j.scriptamat.2011.12.012

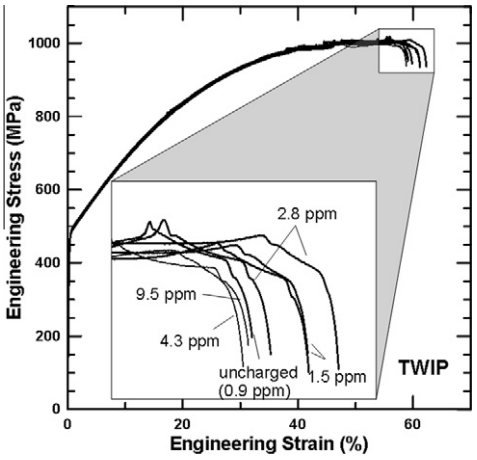

Figure 1. Engineering stress–strain curves for uncharged and hydrogen-charged TWIP-Al tensile samples. The reference hydrogen content measured on the uncharged TWIP sample was 0.9 ppm.

contents measured for each sample. An uncharged TWIP steel specimen is also included. In all TWIP steel samples, stress–strain curves exhibit serrations from approximately 30% engineering strain to failure, similar to previous work [5]. Strains to failure of uncharged and hydrogen-charged tensile samples ranged from 56% to 61% and hydrogen contents ranged from 0.9 (measured in the as-received sample) to 9.5 ppm. Post-uniform elongation consisted of both diffuse and localized necking. Consistent with previous results [4], negligible changes in work-hardening behavior or strain to failure were observed between charged and uncharged TWIP steel, suggesting that the macroscopic mechanical properties were not affected by the charged hydrogen.

Even though the macroscopic tensile properties exhibited no apparent signs of hydrogen damage, the potential effects of hydrogen on microscopic fracture phenomena were evaluated by comparing fracture morphologies and deformed sample surfaces in uncharged and charged tensile samples. FESEM analysis showed that for all conditions, charged and uncharged, fracture occurred by ductile rupture, i.e. void coalescence, indicating that hydrogen did not affect bulk fracture morphology.

Figure 2 shows FESEM images of metallographic mounts from near-surface regions of three different conditions of TWIP steel, with images oriented as shown in the sample schematic included in Figure 3a. Each micrograph consists of the mounting material, i.e. bakelite, at the top of the image and the TWIP steel edge and bulk material in the as-polished condition at the bottom. Figure 2a, an FESEM image of uncharged TWIP steel deformed to failure, reveals small amounts of surface roughness due to preparation and deformation. Figure 2b, an FESEM image of undeformed TWIP steel charged for 1 h to a hydrogen concentration of 4.3 ppm, shows no evidence of hydrogen damage due to charging. Figure 2c and d show FESEM images of TWIP steel charged to 4.3 ppm and tensile tested to failure. Surface cracks were observed to penetrate a few microns into the bulk material. Similar results were observed on all samples charged and tensile tested. Some regions of the cracks contain bakelite which had seeped into the cracks during mounting and which electrically charged in the electron microscope, resulting in intense contrast between steel and bakelite. All surfaces of the samples were exposed to hydrogen pick-up during charging; however, the broad sheet surfaces were examined due to their large area relative to the corners and through-thickness surfaces.

Figure 3a and b show sheet surfaces of tensile samples charged to 4.3 ppm hydrogen in the as-charged (Fig. 3a) and deformed to failure (Fig. 3b) conditions and were taken from the same tensile sample used for Figure 2b and c, respectively. Scratches introduced from light grinding, to remove the oxide, in the longitudinal direction are evident in Figure 3a, but, consistent with Figure 2b, there is no observable damage due to hydrogen charging. However, when the TWIP steel was charged with 4.3 ppm H and deformed to failure, cracks perpendicular to the grinding direction and tensile axis were observed as shown in Figure 3b. Grain size varied from 3 and 15 μm, which when compared to Figure 3b may account for the initiation of cracks at grain boundaries. Further propagation was dominated by stresses perpendicular to the tensile axis.

The results of this study have revealed the presence of near-surface hydrogen damage in a TWIP steel with 1.5 wt.% Al which exhibited macroscopic tensile properties otherwise unaffected by hydrogen. This TWIP steel has been shown in previous studies [1–3,6] on delayed fracture in heavily cold-drawn cups to exhibit reduced susceptibility to hydrogen-induced fracture. To understand the source of the near-surface damage, hydrogen concentration profiles were calculated for charging times of 1 and 6 h to estimate the depth of hydrogen penetration into austenite at room temperature. Several assumptions were made and the corresponding boundary conditions are listed in Eq. (1) with the resulting solution shown in Eq. (2) [7] for an infinite, i.e. non-depleting, source. The initial concentration, C, of hydrogen in the steel was assumed to be zero, i.e. residual hydrogen levels were neglected. The surface concentration, C_S, was assumed constant as the charging process provided a continual supply of hydrogen at the imposed constant current and constant solution pH. The hydrogen concentration at infinity (Eq. (1)) was assumed to be zero, i.e. it was assumed that due to the low diffusivity of hydrogen at room temperature, the 1.2 mm thick sheet steel could be treated as a semi-infinite plate:

$$C(x,0) = 0; C(0,t) = C_s; C(\infty,t) = 0 \quad (1)$$

$$C(x,t) = C_s erfc\left[\frac{x}{2\sqrt{Dt}}\right] \quad (2)$$

Figure 4 shows predictions of Eq. (2) for times of 1 and 6 h, the upper and lower limits of charging used in this study. A value of 10^{-12} cm^2 s^{-1} [8,9] was used for hydrogen diffusivity in austenite at room temperature (i.e. 23 °C). The concentration is observed to decrease considerably immediately below the surface. The normalized results are applicable to any surface potential (C_s). For the 1 h charging time, hydrogen remains negligible at a depth of 3 μm. The longest charging time used in this study was 6 h, which yielded a measured