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Effects of microstructures on hydrogen induced cracking of electrochemically hydrogenated double notched tensile sample of 4340 steel

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ABSTRACT

Quantitative fractographic characteristics of 4340 steel is demonstrated for a grain size range of 10 – 100 μm and hardness range of 41–52 HRC. Double-notched tensile samples were electrochemically charged in-situ with hydrogen in 0.5 m H_2SO_4 + 5 mg/l As_2O_3 solution for 0–40 min charging time. Hydrogen induced fracture initiations were analyzed by novel metallographic investigation of the “unbroken” notch while the overall fractographic behaviors were examined by the scanning electron microscopic imaging of the fracture surfaces of the actually broken notch. Effect of hydrogen was predominantly manifested as intergranular fracture for the harder samples and quasi-cleavage fracture for the softer counterparts. 10–40 μm samples showed the maximum intensity of the hydrogen induced fracture features (intergranular and/or quasi-cleavage) close to the notch which gradually reduced with increasing distance from the notch. The largest grained samples (100 μm) however showed brittle behavior even in absence of hydrogen with similar intensity of percent fracture features at all distance from the notch, while presence of hydrogen intensified the overall percent brittle fractures with their intensities being highest close to the notch. Finally, the brittle fracture characteristics of the hydrogen embrittled samples were shown to be distinguishably different from that of the liquid nitrogen treated samples of same grain sizes and hardnesses.

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1. Introduction

Embrittlement effect of hydrogen in steel is well known and well documented. Atomic hydrogen can be generated at the surface of metals and alloys by thermal dissociation of gaseous hydrogen [1] or by electrochemical decomposition of hydrogen bearing aqueous solutions [2] and vapor condensates [3]. Regardless of the source, the general principles of hydrogen adsorption at materials surface, its subsequent diffusion and metallurgical interaction with various microstructural features causing premature failure of the structure remain the same.

Failure mechanisms induced by hydrogen have always been controversial. Some mechanisms of hydrogen induced fracture are couched merely on thermodynamics principles and macroscopic observation, e.g., pressure theory [4], Hydride formation theory [5], mechanism of reduction of surface energy [6]. These mechanisms barely consider the role of hydrogen at the microstructural and/or sub-microstructural level. On the other hand, mechanisms such as hydrogen enhanced decohesion (HEDE) [7], adsorption induced dislocation emission (AIDE) [8], hydrogen enhanced localized plasticity (HELP) [9] demonstrated the role of hydrogen at microstructural and/or sub-microstructural levels.

AIDE theory argued that hydrogen is adsorbed at the trap sites and helps in the nucleation of dislocation and it subsequent emission from the crack tip. On the other hand HEDE theory propounded that the weakening and consequent decohesion of the grain boundary is due to inference of electrons contributed by hydrogen with the d-electron cloud of the transition metal atoms. HELP mechanism however is perhaps the most modern of all theories. HELP theory explains how hydrogen induced locally enhanced plasticity at the sub-microscopic level eventually leads to macroscopic brittle failure. According to this theory, hydrogen segregates at the dislocation and reduces the strain energy per unit length of the dislocation causing weaker interaction with any other strain field and reduces their mutual repulsive interactions. As a result, dislocation mobility is amplified locally, resulting a local plastic instability and damage nucleation. However, none of these individual models can explain all the observations associated to hydrogen induced failure and often times synergetic [10,11] effect of more than one mechanism (hybrid mechanism) provides

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Table 1
Composition of the double notch tensile specimens of AISI 4340 (provided by vendor).

Element	C	Cr	Mn	Ni	P	Si	S	Cu	Al	Mo	N
Composition (wt%)	0.4	0.79	0.74	1.69	0.007	0.29	0.014	0.18	0.036	0.24	0.009

Table 2
Post heat treatment PAGS and martensitic hardnesses and times of hydrogenation employed for different microstructures.

Austenitizing (°C)	870 (1 h)		1100 (1 h)		1175 (1 h)	
Tempering (°C)	350 (2 h)	500 (2 h)	350 (2 h)	500 (2 h)	350 (2 h)	500 (2 h)
PAGS (ASTM)	10.5 ± 0.5	10.5 ± 0.5	6.5 ± 0.5	6.5 ± 0.5	3.5 ± 0.3	3.5 ± 0.3
PAGS (μm)	10 ± 3	10 ± 3	40 ± 10	40 ± 10	100 ± 20	100 ± 20
Hardness (HRC)	52 ± 0.5	45.5 ± 0.5	50 ± 0.5	43 ± 0.5	48.5 ± 0.5	40.5 ± 0.5
H Charging time (min)	0,5,10,20	0,5,10,20,40	0,5,10,20	0,5,10,20,40	0,5,10	0,5,10

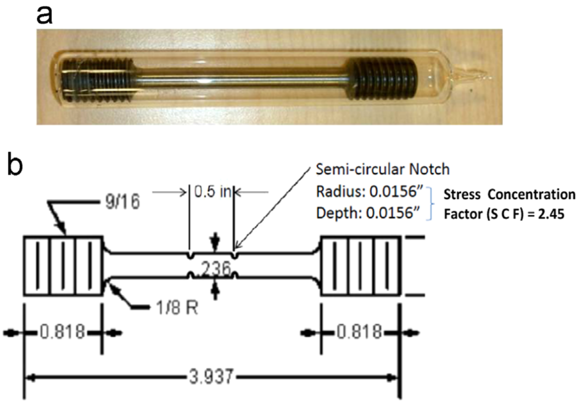

Fig. 1. (a) Quartz encapsulated (sealed after argon impregnation at 2–3 atm) sample used for austenitization at 1175 °C (1 h). (b) Double notched round bar tensile sample (measurements are given in inch).

Table 3
Average size of the martensitic packets and their constituent blocks as function of PAGS.

PAGS (ASTM)	10.5 ± 0.5	6.5 ± 0.5	3.5 ± 0.3
PAGS (μm)	10 ± 3	40 ± 10	100 ± 20
Number of lath packets per grain	1	2–3	2–4
Packet size (μm)	10 ± 3	20 ± 5	40 ± 10
Lath width (μm)	0.5–1	1–2	1–3

a better understanding and stronger interpretation of a hydrogen induced fracture process.

Generally, hydrogen induced brittle fracture is microscopically manifested as intergranular fracture as well as transgranular fracture. Another interesting fractographic feature often associated with hydrogen embrittlement is termed quasi-cleavage which displays characteristics often associated with macroscopically brittle fracture with local plasticity. Recent results [12–14] discussed some mechanistic aspects associated with quasi-cleavage but the precise details and applicable models remain the subject of controversy [15]. Martin et al. [12,14] stated that quasi-cleavage can result from the growth and coalescence of microvoids that initiate and grow within intense slip band intersections and mechanistically they rendered the HELP mechanism precedence over

Table 4
Hydrogen concentration (wt. ppm) as function of Hydrogen charging time found by Shim et al. [34]. The electrochemical hydrogenation process followed in this work closely resembles Shim et al. approach.

Charging time (min)	5	10	20	30	40
Hydrogen concentration (wt. ppm)	1.92	2.61	3.93	4.7	5.45

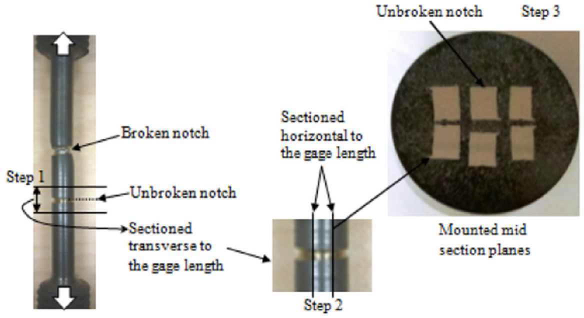

Fig. 3. Sectioning of the unbroken notches for the metallographic investigation.

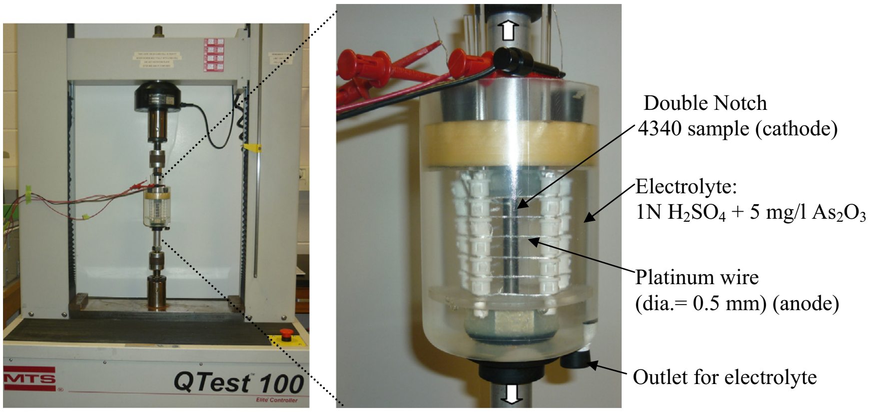

Fig. 2. Hydrogen charging apparatus was mounted on the tensile instrument (MTS Q-Test). Samples were charged with hydrogen and immediately loaded under tension at cross head speed of 0.01 mm/min and the electrolyte was simultaneously removed.

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