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Improved resistance to hydrogen embrittlement of friction stir welded high carbon steel plates

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ABSTRACT

The hydrogen embrittlement of the friction stir welded high carbon steel plates was evaluated by the cathodic hydrogen charging method. The welding was performed below Ac1 transformation point and therefore the stir zone of the welds contained refined ferrite matrix distributed with cementite particles. After hydrogen charging for 4 h, large quantities of irreversible dome-shaped blisters were formed on the surface of the base metal. However, no blisters were observed in the stir zone even after charging for 16 h. In addition, hydrogen-induced internal cracks were formed throughout the thickness of the base metal. But the development of the internal cracks in the base metal was restricted when getting close to the stir zone. The hydrogen charged stir zone showed less reduction of ductility than the base metal during tensile testing, which reveals that the friction stir welded steel joints showed higher resistance to hydrogen embrittlement than base metal. Copyright © 2015, Hydrogen Energy Publications, LLC. Published by Elsevier Ltd. All rights reserved.

Introduction

Medium and high carbon steels are widely used in many common applications. For example, with the development of nuclear energy, carbon steel has become a candidate container material for the disposal of used fuel in a deep geological repository in sedimentary host rock. Carbon steels are also used as pipes due to their high strength, high toughness to improve transportation efficiency under high pressure and high temperature conditions. However, carbon steels are very vulnerable to hydrogen embrittlement (HE) when exposed to a hydrogen containing environment, which leads to internal cracks in the materials and unpredictable failure of the materials under lower stress [1,2]. Therefore, prediction of the long term service behavior of the carbon steel products under hydrogen environment has become inevitably

important. It is generally believed that the metallurgical features of the steels such as the chemical composition and corresponding microstructure exert dominant influence on the hydrogen induced degradation of strength [3–8]. And the mechanism causing HE is the same when hydrogen has entered steel structure, regardless of the hydrogen sources. A special case of HE in steel is caused by arc welding, in which the hydrogen from moisture is dissolved in the steel melt and remains in the solid after cooling to room temperature [9,10]. In spite of decades of research effort, unfortunately, HE is still one of the most serious problems in steel welding.

Friction stir welding (FSW) was invented by the Welding Institute (TWI) of the UK in 1991 with the original purpose of joining Al and Al alloys [11]. Recently, this technique has been expanded to many high melting point materials including Cu, Ti, Fe and steels. High carbon steels, which are considered as unweldable materials by fusion welding methods due to the

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formation of the brittle martensitic phase, now can be successfully welded by FSW technique [12–16]. For example, Cui and Chung et al. successfully carried out the FSW of a series of high carbon steels at a welding temperature below and above the Ac1 transformation point [17–19]. It was found that due to the complex material flow in the stir zone (SZ) and the thermal-mechanical process imposed on the work-pieces, a very complicated and versatile microstructure can be formed in the welds and significantly depended on the welding conditions and the properties of the material. Because of the unique microstructure, the HE behavior of the friction stir welded steels must be different with those produced by other fusion welding methods. Recently, Khodir et al. first observed the suppression of hydrogen blistering in the friction stir welded SK4 steel after cathodic hydrogen charging process [20]. In Khodir's study, the suppression of the blister formation was proposed to be caused by the microstructural refinement and the prevention of the formation of hard carbide particles in the soft ferrite matrix. However, the authors only reported the changes of the surface appearance after hydrogen charging. The microstructural changes inside the material and mechanical properties affected by the introduced hydrogen were not illustrated. More recently, Sun et al. also found the suppression of hydrogen-induced damage in the friction stir welded low carbon steel joints [21]. However, no other studies about the HE behavior of the FSW processed steels have been published so far.

In this study, 2 mm thick high carbon steel plates were friction stir welded with a low heat input and at a peak welding temperature lower than Ac1 point. The sensitivity to HE in terms of hydrogen blistering and hydrogen-induced cracking of the welds was evaluated and compared with that of the base metal (BM) by the hydrogen cathodic charging method and subsequent tensile tests. The mechanism of improved resistance to HE of the welds was thus put forward and discussed.

Experimental procedure

Materials

The as-received 2-mm thick SK4 high carbon steel plates were subjected to FSW and stir-in-plate welding was performed under a load-controlled mode. The chemical composition of the steel plate is listed in Table 1. The rotating tool used for welding was made of a WC-based material, which had a shoulder diameter of 12 mm, probe diameter of 4 mm and probe length of 1.8 mm. The tool axis was tilted by 3° with respect to the normal direction of the sample surface. The friction stir welding process is shown in Fig. 1. Although the welding parameter can be varied in a wide range, the

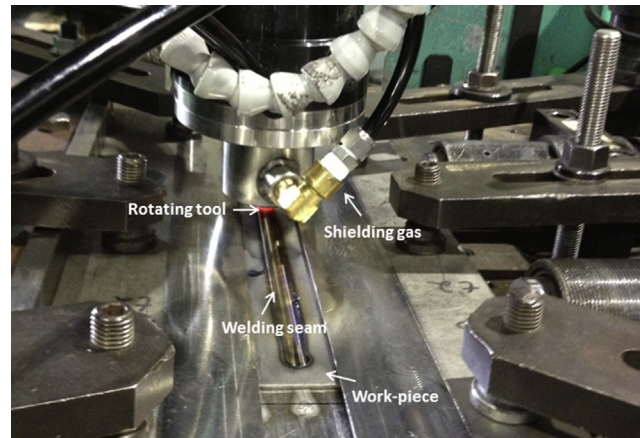


Fig. 1 – Photo showing the friction stir welding process.

travelling speed and rotation speed of the rotating tool in this study were kept constant of 100 mm/min and 120 rpm, respectively. Argon shielding gas was used during the welding process to protect the welds from oxidation. It was found that the welding temperature was below critical Ac1 transformation point and no phase transformation occurred during the welding process.

Cathodic hydrogen charging

After welding, the dog-bone like tensile specimens and rectangular specimens for microstructural characterization were cut by an electrical discharge machine from the welds transverse to the welding direction. The rectangular specimens comprised all the specific zones including the BM and SZ. The tensile specimens had a gauge length of 6 mm, width of 3 mm and thickness of about 2 mm. Therefore the test sections were completely within the area of the SZ. Prior to hydrogen charging, the entire surface of the specimens were mechanically ground using emery papers up to grade 4000 and polished with a colloidal silica oxide suspension, then rinsed and degreased with acetone. The specimen, which was electric resistance spot welded to a thin stainless wire, was placed into a charging cell filled with an aqueous solution of 0.5 M H₂SO₄ containing 1 g/L thiourea and surrounded by a Pt wire. Hydrogen was introduced into the specimens by cathodic charging at a current density of 500 A/m² using a regulated direct current power supply at room temperature. The rectangle and the tensile specimens were hydrogen charged for 4 and 16 h, individually. After hydrogen charging, the specimens were immediately washed with distilled water and acetone. The surfaces of the charged specimen were still very shiny and the hydrogen blisters could be visually observed.

Table 1 – Chemical composition of the as-received steel plate.

Steel type	Chemical composition (mass %)									
	C	Si	Mn	P	S	Cr	Ni	Cu	Al	Fe
SK4 steel	0.95	0.2	0.42	0.017	0.003	0.147	0.01	0.01	0.001	Bal.

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