Improved Resistance to Hydrogen Embrittlement of Friction Stir Welded High Carbon Steel Plates

Yufeng Sun *, Hidetoshi Fujii

Joining and Welding Research Institute, Osaka University, 11-1 Mihongaoka, Ibaraki, 5670047, Japan

* Corresponding author: Dr. Y.F. Sun (Yufeng Sun),

Tel: 81-6-68798663; Fax: 81-6-68798653; Email: yfsun@jwri.osaka-u.ac.jp

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 hours, 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 hours. 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.

Keywords: Carbon steel; Friction stir welding; Microstructure; Hydrogen Embrittlement; Hydrogen-induced cracking;

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 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 12mm, probe diameter of 4mm 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 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.

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.

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 6mm, 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 1g/L thiourea and surrounded by a Pt wire. Hydrogen was introduced into the specimens by cathodic charging at a current density of 500A/m² using a regulated direct current power supply at room temperature. The rectangle and the tensile specimens were hydrogen charged for 4 and 16 hours, 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.

In order to determine the hydrogen content charged into the specimen, a Horiba EMGA-830 hydrogen content analyzer was employed for melt extraction. At least three specimens weighing about 0.2 g each were tested immediately after the hydrogen charging process. The specimens were heated up to their melting points temperature quickly for evaporating all gases, which were then taken by a nitrogen carrier gas through chemical reaction tubes to analyzing units and the thermal conductivity of the carrier gas was measured. The software thus calculated the hydrogen concentration of the sample based on the thermal conductivity variation.

Microstructure and mechanical properties evaluation

The microstructures of the FSWed joints as well as the BM before and after hydrogen charging were observed by optical microscopes (OM), scanning electron microscope (SEM). For OM and SEM observation, the samples were mechanically polished followed by chemical etching using 3% Nital solution. For electron backscattering diffraction (EBSD) measurement, the cross-sectional plane of the hydrogen charged BM and the welds were electro-polished using a solution of CH₃COOH:HClO₄=9:1. Tensile tests were carried out using an Instron tensile machine at a cross-head speed of 0.5 mm/min. For the charged specimen, the tensile tests were started within 20 minutes after hydrogen charging in order to minimize the hydrogen release from the specimen. After the tensile tests, the morphology of the fractured plane was characterized by SEM.

Experimental Results

Microstructural characterization before hydrogen charging

Fig. 2 (a) shows the cross-sectional macrostructure of the weld, in which the SZ and the BM are easily discernible. However, the thermo-mechanically affected zone (TMAZ) and heat affected zone (HAZ) were very narrow due to the low heat input and cannot be easily distinguished. Fig. 2 (b) and (c) show the microstructure of the BM and the SZ, respectively. The microstructures of the two specific zones were quite similar, indicating that the phase transformation did not occur in the SZ due to the welding

temperature below the critical A_{c1} transformation point. However, the different contrast between the two specific zones implied that the ferrite matrix in the SZ was significantly refined after the welding process. The details of the microstructure were further studied and shown in Fig. 3. Fig. 3 (a) and (b) are the SEM images showing the microstructures of the BM and SZ, respectively. The microstructure of BM consisted of pearlite structure and cementite particles distributed mainly on the grain boundary in the ferrite matrix. However, the SZ only shows a microstructure consisting of a high density of cementite particles distributed in the ferrite matrix. The original pearlite structure was fractured by the stirring of the rotating tool and could not be observed any more in the SZ. In addition, the cementite phase consisted of two types of particles. One type of the particle was a short rod-like, which was formed from the segment of the pearlite structure in the BM during the welding process. Another type of cementite particle was slightly larger and had an irregular shape as indicated by white arrows in the figure, which were the cementite particles originally distributed in the BM. The microstructural characterization again confirmed that the welding process was carried out below the A_{c1} phase transformation point. Fig. 3 (c) and (d) show the EBSD phase maps of the BM and the SZ, respectively. In the phase maps, only high angle boundary larger than 15° was indicated by black lines. Simultaneously, the ferrite phase was indicated with red color and the cementite phase was indicated with green color. In Fig. 3(c), the ferrite phase in the BM has an average grain size of $8\text{ }\mu\text{m}$. Some large cementite particles with a size of $3\text{ }\mu\text{m}$ were embedded in the ferrite matrix. In Fig. 3(d), the average size of the ferrite phase in the SZ was about $3\text{ }\mu\text{m}$, much smaller than that of the BM. The two kinds of cementite particles could also be distinguished in the map, which had a smaller mean particle size in SZ than that in the BM.

Microstructural characterization after hydrogen charging

The amount of the hydrogen absorbed into the specimens by cathodic hydrogen charging was measured immediately after charging in order to decrease the hydrogen release. The hydrogen contents of the BM and SZ before hydrogen charging were about 0.6 ppm, indicating that hydrogen could not be introduced into the material during the welding process. This is also regarded as one of the most important advantages of FSW technique over the fusion welding methods. After cathodic hydrogen charging, different amounts of hydrogen were introduced into the SZ of the welds depending on the different microstructure. After charging for 4 hours, the BM and the SZ had similar hydrogen content of about 7.0 ppm. After hydrogen charging for 16 hours, the hydrogen content in the BM increased to about 18 ppm. However, the hydrogen content was only about 13 ppm in the SZ.

After cathodic hydrogen charging, a typical example of the sample appearance is shown in Fig. 4(a), in which an obvious boundary was observed between the BM and SZ due to their very different resistance to the blister formation. The irreversible dome-shaped blisters with a high density were found on the surface

of the BM. The blisters showed different sizes and many large blisters were visually observed. On the contrary, the blisters were hardly observed on the surface of the SZ. Fig. 4(b) shows the magnified blisters formed on the surface of the BM after hydrogen charging for 4 hours. It was found that the dome-shaped blisters with various diameters ranging from several micrometers to several hundreds of micrometers were formed on the surface of the BM. In addition, the smaller blisters were formed not only on the original surface of the BM, but also on the bulging surface of the larger blisters. Some cracks propagated along the periphery of the larger blisters. In the SZs after hydrogen charging for 4 hours as shown in Fig. 4(c), a couple of short cracks can be observed on the sample surface near the BM. After hydrogen charging for 16 hours, the blisters were not observed on the surface of the SZ. However, only a few more short cracks were observed near the boundary between the BM and the SZ, as indicated in Fig. 4(d). This phenomenon reveals that the hydrogen induced blistering can be suppressed by the friction stir welding process.

Fig. 5 shows the SEM images of the blisters formed on the surface of the BM. In order to observe the bulging of the blisters clearly, the sample normal was tilted with a high angle of 70° to the electron beam. In Fig. 5(a), a large blister with a diameter of about $250\text{ }\mu\text{m}$ and a small blister with a diameter of $50\text{ }\mu\text{m}$ were observed on the surface. A curving crack along the periphery of the blister and was indicated by an arrow in the figure. Fig. 5(b) shows the carving crack at higher magnification, which continuously propagated along the sample surface. Due to the formation of the crack, a deep gap with a width of about $2\text{ }\mu\text{m}$ was generated inside the sample. The formation of the crack will certainly tear up the continuity of the material and therefore deteriorate its mechanical properties.

After hydrogen charging for 4 hours, the specimens were cut perpendicular to the welding direction and the cross-sectional planes were mechanically polished and chemical etched for microstructural characterization. For comparison, the hydrogen charged BM was performed in the same way. Fig. 6(a) shows the cross-sectional microstructure of the BM beneath the sample surface. Some cracks were found inside the specimen and propagated to the surface. The material beneath the sample surface was lifted up due to the gradually increased pressure, which was caused by the accumulation of hydrogen atoms resulting in molecular hydrogen formation. As a result, a dome-shaped blister was formed on the sample surface and surrounded by the exposed cracks. Fig. 6(b) shows a small crack formed inside the BM, which had not developed into larger size due to the limitation of hydrogen content. Fig. 6(c) shows the cross-sectional microstructure of the boundary between the SZ and the BM. The sample normal was titled with an angle of 70° to the electron beam. Thus the boundary was easily discerned due to the refined microstructure in the SZ and was indicated by a dotted line in the figure. On the BM side, some internal cracks indicated by white arrows were observed, which were distributed nearly parallel to the sample surface. However, no obvious cracks were found in the SZ. From the microstructure of SZ at high magnification as shown in Fig. 6(d), only a few small cracks at the interface of the cementite/ferrite matrix

were observed in some places. This phenomenon agrees well with the concept that the hydrogen induced cracking usually initiates near inclusions in the steel matrix [22-24].

After hydrogen charging for 16 hours, the hydrogen-induced cracks inside the BM were shown in Fig. 7. Fig. 7(a) shows the general distribution of the cracks throughout the sample thickness, in which backscattering electron (BSE) mode was used in order to display the cracks clearly. It was found that the stepwise cracks were formed from the surface to the center of the sample. Comparing with the cracks formed in the center part of the BM after hydrogen charging for 4 hours, the gap width of the crack became larger after hydrogen charging for 16 hours. Fig. 7(b) shows the interaction of two close cracks when propagating toward each other. In order to relax the internal pressure caused by the hydrogen accumulation, considerable plastic deformation occurred near the tip of the crack. Fig. 7 (c) shows the enlarged area between the two close cracks as indicated in Fig. 7 (b), a lot of micro-cracks were found along the cementite particles and some small cementite particles were broken into two smaller parts. However, in the area far from the large internal cracks, such kind of small cracks could not be observed. It thus revealed that the micro-cracks were formed due to the stress field around the crack tips. Conversely, the micro-cracks formed between the two close cracks made the tips of close cracks deflect toward each other, which finally led in some cases to the coalescence of the interacting cracks and the stepwise of the crack propagation [25-26].

The development of hydrogen-induced cracks has been studied widely and it was proposed that the cracks might propagate along the grain boundary or along some preferential orientations. In this study, EBSD measurement was carried out to reveal the development of the crack in the BM after hydrogen charging for 16 hours. Fig. 8 shows the EBSD maps of the charged specimen, in which only high angle boundary larger than 15° indicated as black lines was shown. Fig. 8 (a) shows the example of two hydrogen-induced cracks. Two pairs of the white arrows shown in the figure had very similar color code, indicating that the materials on the two sides of the crack had the same orientation and can be regarded as originally one grain [27]. It reveals that the development of the cracks was not completely along the grain boundary. The cracks may also penetrate into the ferrite matrix and the crack propagation sometimes terminated at the grain boundary of the ferrite matrix. Fig. 8(b) and (c) show the IPF map and the corresponding kernel average misorientation (KAM) map of two close cracks. The tips of the two separated cracks started to deflect toward each other when the stress fields near the tips of the two large cracks were overlapped with each other. In the local area between the tips of the two cracks, the average misorientation was obviously higher, indicating the high strain distribution caused by the promotion of the cracks.

After hydrogen charging for 16 hours, the internal cracks formed in the friction stir welds were also investigated. In Fig. 9 (a) and (b), the microstructure of the boundary between the BM and the SZ was shown. It was found that some nearly straight cracks were formed close to the boundary of the two

specific zones. However, the development of the cracks was blocked by the boundary and terminated therein. In the SZ close to the boundary, a few short straight cracks were observed parallel to the boundary, as indicated by white arrow in Fig. 9(b).

Fig. 10(a) shows the hydrogen-induced cracks in the SZ near the boundary. The short cracks **were** formed around the cementite particles and distributed parallel with each other along the boundary, namely, the material flow direction caused by the stirring of the rotating tools during the welding process. Fig. 10(b) and (c) showing the IPF and corresponding KAM map of the cracks in the SZ, in which only high angle boundaries larger than 15° indicated by black lines were shown. Comparing with that formed in the BM, the hydrogen-induced cracks in the SZ were much smaller and relatively straight. In addition, neither stepwise cracks nor deflected cracks were observed **in the stir zone**. However, the crack propagation was also found not only along the grain boundary, but also can penetrate into the ferrite matrix. From the KAM map, the average misorientation angle was much lower than that of the BM, indicating the less stress field formed around the tip of the cracks.

Tensile tests

The SZ and the BM were subjected to tensile tests before and after cathodic hydrogen charging. The tensile strain-stress curves are shown in Fig. 11, in which the original points of the curves were shifted along the horizontal axial for **clarity**. Generally, both the welds and the BM showed an obvious reduction in the ultimate tensile strength and ductility before failure after hydrogen charging. For the BM after hydrogen charging for 4 hours, the plastic strain was sharply reduced from 36% to 4%. For the SZ after charging for 4 hours, the plasticity decreased from 17% to 3%. After hydrogen charging for 16 hours, the BM showed **premature failure** under very low stress during the tensile tests due to the formation of large cracks throughout the sample thickness. However, the SZ **showed no** further deterioration of the mechanical properties. This phenomenon further confirmed that the HE sensitivity of steel **was** related to the microstructure of the material and can be improved by the FSW technique.

Fig. 12 shows the SEM images of the morphologies of the fractured plane of BM and the welds after hydrogen charging for different time. Fig. 12(a), (b) and (c) show the fractured plane of the BM before and after the hydrogen charging. The uncharged specimen showed **a ductile fracture surface with** considerable dimples resulting from microvoid coalescence (MVC). However, after hydrogen charging for 4 hours as shown in Fig. 12(b), a sharp and smooth fractured plane **was** observed, indicative of the cleavage fracture mode of the specimen. Moreover, micro-cracks **were** found around the cleavage fracture plane and indicated by white arrows in the figure. After hydrogen charging for 16 hours, the fractured plane only showed **cleavage plane** and a lot of hydrogen-induced cracks **were** found as indicated in Fig. 12(c). The fractures surfaces of the welds after hydrogen charging for 0, 4 and 16 hours were shown in Fig.

12 (d), (e) and (f), respectively. The fractured planes were all characterized by a very fine dimple pattern resulting from MVC. However, the carbide particles were embedded in the dimple on the fractured surface of the uncharged specimen, which indicated the strong interfacial bonding between the cementite particles and ferrite matrix. According to the microstructural characterization as shown in Fig. 6(d), the accumulation of hydrogen atoms around the carbide particles accelerated the movement of the dislocations and caused the formation of voids therein. On the other hand, the voids formed around the cementite particles led to the weak bonding between the cementite and the ferrite matrix. As a result, the cementite particles were stripped off from the ferrite matrix during the tensile tests. Almost no cementite particles were seen on the fractured surface of the hydrogen charged specimen and some short micro-cracks were observed as indicated by arrows in the figure. Along the edge of the dimple pattern, very small voids could be found and indicated by a small arrow in Fig. 12(f).

Discussion

As shown above, the friction stir welded SK4 high carbon steel joints showed much improved resistance to the HE than the BM. After hydrogen charging, the SZ of the weld showed less hydrogen contents and less hydrogen induced blisters or internal cracks than the BM. Because no phase transformation occurred during the welding process, the final welds and the BM comprised same phase constituents. However, much refined ferrite matrix and different distribution of the cementite particles were formed in the SZ. It was well known that the inclusions like cementite particles in this study can act as an obstacle to trap the hydrogen atoms [28-31]. Both calculation and permeation experiments showed that the binding energy E_b between hydrogen and Fe_3C particles is about 84 kJ/mol, much higher than the E_b between hydrogen and dislocation or grain boundary, which has a value of about 59 kJ/mol [32-34]. The distribution of hydrogen atom around the cementite particles had been detected by means of hydrogen microprint technique and imaging method [35-37]. Therefore, the hydrogen was mainly distributed around the cementite particles after hydrogen charging for short time, namely, 4 hours in this study. However, the hydrogen content was much higher in the BM than that in the SZ after hydrogen charging for 16 hours. Although the fragment of cementite particles in the SZ significantly increased the length of interface between the particles and the ferrite matrix and thereby increased the total number of trap site, the BM had a higher capacity for the hydrogen absorption. Because a lot of hydrogen-induced cracks formed throughout the thickness of the BM and thus new surface can be generated inside the cracks. No obviously cracks were formed in the stir zone. Therefore, more hydrogen diffused and stored inside both the cracks and the interface due to the high E_b between the hydrogen and the free surface, which is ranged from 70 to 95 kJ/mol. As a result, more trap sites for the hydrogen were increased in the BM.

In addition, the hydrogen induced cracks were more likely to occur in BM than in SZ. It is also considered to be caused by the refinement of the microstructure, including both the grain size of the ferrite matrix and the cementite particles. According to the high pressure theory [38-39], the material with lower strength is prone to the occurrence of failure. The BM had a coarse grain structure and low critical yield stress for the fracture to occur. Secondly, the smaller cementite particles in the SZ also increased the difficulties for the occurrence of crack initialization, comparing with the large irregular cementite particles in BM. The cracks usually initiated at the interfaces between the particle and the ferrite matrix and the critical condition for the crack initiation was the local stress distributions. It is well known that the hydrogen present in the steel readily diffuses towards potential crack nucleation sites and reduces the critical interfacial stress for crack initiation. The SZ had much larger interface area between the matrix and the smaller particles and generally reduced the local stress distributions. In addition, the smaller particles were more uniformly distributed within the ferrite grains in the stir zone rather than the distribution of large particles on the grain boundary in BM, which was more prone to the void initialization. Although the total hydrogen amount may be similar after charging for a certain time, a higher hydrogen concentration was generated around the hydrogen traps like grain boundary or cementite particles in BM than in SZ. Moreover, the refined grain structure in the SZ could reduce the local density of the harmful impurities [40]. As a result, higher pressure was exerted on the surrounding materials, which caused the BM to fail after the shortest exposure periods. It is worthy to note that the texture formed in the SZ due to the shear stress of the rotating probe may also exert some influence on the initialization of the hydrogen induced cracks, which needs to be further investigated. In consideration of the very tough problem that the HE usually occurs in the coarse grain structure in HAZ or nuggets in the arc welded steel products, the friction stir welding may be a promising substitute when the joints have to be exposed in the hydrogen environment. In addition, the surface of the steel materials can also be modified to improve its resistance to hydrogen embrittlement by friction stir processing methods, which was developed based on the friction stir welding technique.

Conclusions

In this study, the microstructure and mechanical properties of the friction stir welded high carbon steel joints obtained at a peak temperature below Ac1 transformation temperature were investigated before and after cathodic hydrogen charging. Based on the results shown above, the following conclusions can be drawn.

- 1) No phase transformation occurred in the weld and the weld had a microstructure consisting of refined ferrite matrix with homogeneous distribution of smaller cementite particles.

2) After hydrogen charging, the formation of hydrogen blister was suppressed in the SZ. However, large quantity of hydrogen blisters with various sizes was observed on the surface of BM.

3) After hydrogen charging, the stepwise hydrogen-induced cracks were formed beneath the sample surface and propagate throughout the thickness of the materials with the increase of charging time. However, only small cracks were observed around the cementite particles in the SZ and the crack propagation was blocked near the boundary between SZ and BM.

4) After hydrogen charging, the welds showed less reduction of ductility during tensile tests than the BM, indicating the improved resistance to HE of the welds.

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Captions of Tables and Figures

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

Fig. 1 Photo showing the friction stir welding process

Fig. 2 (a) OM image showing the cross-sectional macrostructure of the weld; and microstructure of (b) BM and (c) SZ. (F: ferrite; C: Cementite)

Fig. 3 SEM images showing the microstructure of (a) BM and (b) SZ; EBSD phase map of the (c) BM and (d) SZ

Fig. 4 (a) Typical appearance of the hydrogen charged specimen; OM images showing (b) blisters formed on the surface of the BM and (c) small cracks formed on the surface of the SZ after hydrogen charging for 4 hours; (d) surface morphology of the BM/SZ boundary after charging for 16 hours.

Fig. 5 (a) SEM image showing the hydrogen-induced blister formed on the surface of the BM; (b) the crack formed around the blister

Fig. 6 Cross-sectional microstructures showing the internal cracks formed after hydrogen charging for 4 hours. (a) and (b) inside the BM; (c) and (d) inside the SZ.

Fig. 7 SEM images showing the hydrogen-induced cracks formed in the BM after hydrogen charging for 16 hours. (a) BSE image showing the cracks formed throughout the sample thickness; (b) SEM images showing the two close internal cracks; and (c) the micro-crack in the area between the tips of two close cracks

Fig. 8 OIM maps derived from the EBSD measurement of the BM after hydrogen charging for 16 hours. (a) IPF map showing the development of the cracks; (b) IPF map and (c) corresponding KAM map showing the coalescence of the close cracks.

Fig. 9 Cracks formed in the SZ after hydrogen charging for 16 hours (a) and (b) boundary between SZ and the BM.

Fig. 10 (a) SEM images showing the hydrogen-induced cracks formed in the SZ; (b) IPF and (c) KAM map derived from the EBSD measurement of the SZ after hydrogen charging for 16 hours

Fig. 11 Tensile strain-stress curves of the BM and the welds after hydrogen charged for different time

Fig. 12 SEM images showing the fracture morphology of the specimens after tensile tests. The BM after hydrogen charging for (a) 0; (b) 4; and (c) 16 hours; the SZ after hydrogen charging for (d) 0; (e) 4 and (f) 16 hours.

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.

Fig. 1

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6

Fig. 7

Fig. 8

Fig. 9

Fig. 10

Fig. 11

Fig. 12