

## Hydrogen embrittlement characteristics of hot-stamped 22MnB5 steel

Mitsuhiro Okayasu, Takafumi Fujiwara

Graduate School of Natural Science and Technology, Okayama University  
3-1-1 Tsushimanaka, Kita-ku, Okayama, 700-8530, Japan

### Abstract

Hydrogen embrittlement (HE) characteristics of 22MnB5 steel (U-bent specimen) manufactured using hot-stamping process at various temperatures were experimentally and numerically investigated. Steel resistance to HE was examined through delayed failure tests under static and cyclic loading during hydrogen charging. First, the low cyclic loading caused severe HE, in which a clear difference in the extent of HE was obtained depending on the hot-stamped sample, which directly affected the microstructural characteristics and stress–strain distribution. The hot-stamped samples with large martensite phase showed low resistance to HE compared with those with small martensite phase because of the high concentration of hydrogen trapped in the phase boundaries. Moreover, the dual phase (ferrite and martensite) of the hot-stamped samples reduced their resistance to HE, which is caused by the hydrogen trapped in the lamellar-shaped pearlite phase. The resistance to HE was improved by low-temperature heating at 200 °C for 1 h because of the generation of  $\epsilon$ -carbides as trap sites as they render the hydrogen non-diffusible. Furthermore, the internal strain in the U-bent sample could accelerate HE because of the high concentration of hydrogen. These results were verified by experimental and numerical analyses. Thus, the hydrogen trapping mechanism was proposed as a valid mechanism for HE in 22MnB5.

*Keywords:* 22MnB5; hot stamping; hydrogen embrittlement; hydrogen trapping; martensite

### 1. Introduction

Automobile bodies are fabricated using stamped frame structures. The body design should ensure high crash resistance and light weight to guarantee the safety of the people in the

automobile and improve fuel efficiency, respectively. Thus, high-strength steel has been employed in the manufacture of frame structures. During the 1990s, high-strength steel sheets (tensile strength  $>780$  MPa) were used in safety reinforcing parts, such as bumper-reinforcing materials and door impact beams. In the beginning of the 2000s, a 1,470-MPa class cold-rolled ultra-high-strength steel sheets were first used because of their high press formability and stability of their mechanical properties. These properties lead to a great improvement in the fuel economy and safety performance of the automobiles during impacts. Furthermore, high strength steel sheets (tensile strength  $>1,500$  MPa) have been increasingly used to manufacture car bodies to reduce weight and improve impact resistance. Hot stamping is one of the technologies used to produce components made of steel with a tensile strength of  $>1,500$  MPa. Hot stamping has more advantages than cold stamping, such as high press formability, ability to form high-strength steel, and capability of forming complex shapes. The high strength complex hot-stamped parts can be manufactured using steel sheets heated to more than its  $A_3$  line followed by a rapid cooling process, i.e., austenite-to-martensite transformation. 22MnB5, which has high strength of  $>1,500$  MPa, is an example of hot-stamped steels (also called press-hardened steels). Hot-stamped steels are manufactured at a cooling rate of approximately  $48\text{ }^\circ\text{C}\cdot\text{s}^{-1}$  to increase the tensile strength to  $>1,520$  MPa [1].

Currently, automobile door-frame structures made by TOYOTA MOTOR Corp. are assembled by combining ultra-high-tensile steel with hot-stamped steels; i.e., ultra-high-tensile steel with a tensile strength of 1,180 MPa is used for the front pillar and side sill, and 1,500-MPa-class hot-stamped steel is used for the door reinforcement beams (center pillar). Because  $>18\%$  of the automobile parts of Toyota Prius are produced by hot stamping, hot-stamped steels are expected to be increasingly employed for automobile parts in the future. However, hot-stamped steels face several challenges, e.g., hydrogen-induced delay fracture,

which should be eliminated before using the steel for automobile parts. This challenge is particularly present in hot-stamped steels with high tensile strength of  $>1,200$  MPa, as they are more sensitive to hydrogen embrittlement (HE).

During steel heating to reach the high temperature of austenitization, hydrogen atoms can diffuse from the atmosphere to the steel sheets, which can cause degradation of the mechanical properties, i.e., HE [2]. To prevent HE of the steel sheets, an Al–Si coating can be used to protect the steel from hydrogen penetration [3]. Hydrogen trapping is also a useful approach to improve the resistance of HE. Hydrogen atoms tend to concentrate in the area around vanadium carbides (VC) particles, as they have a strong ability to trap hydrogen atoms. Chen et al. [4] have examined the effect of vanadium-microalloying element on HE for 30MnBNb hot-stamped steel. Compared with the V-free experimental steels, the steels with 0.14 wt% V have many dispersive precipitates, which could enhance the hydrogen atom dispersion in the steels. In a study by Zhang et al. [5], a tempering treatment at 200 °C slightly enhanced the HE resistance of hot-stamped low-carbon boron-alloyed steel, which contained 0.20 C wt%, 0.85 Si wt%, 1.60 Mn wt%, 0.006 P wt%, 0.002 S wt%, and 0.0015 B wt%. Lee et al. [6] subjected a 22MnB5 steel to a tempering process at various temperatures (150 °C–520 °C) and found that the steel susceptibility to HE decreased with increasing tempering temperature. The hydrogen-induced failure in steels tempered between 150 °C and 350 °C was found to be related to the glide-plane decohesion, and the inclusion particles resulted in an HE-induced cleavage failure. The HE sensitivity of steel samples tempered at 460 °C and 520 °C decreased as a result of the reduction in dislocations.

These studies have experimentally examined the HE characteristics of press-hardened steels. However, there is still a lack of information on the HE mechanisms and delayed failure characteristics of press-hardened steels. In particular, information related to hot-stamped U-

bent specimens is important, as this specimen is the principal part in door impact beams and bumper reinforcements of automobile parts [7]. Thus, in this work, the material properties and HE characteristics of hot-stamped U-bent 22MnB5 prepared with different microstructural formation and different strain levels at various temperatures were investigated experimentally and numerically.

## **2. Experimental procedures**

### *2.1. Material preparation*

This study was conducted using uncoated quenchable 22MnB5 1.6-mm-thick steel sheets manufactured using a rolling process at 600 °C for automotive components. The chemical composition of the used 22MnB5 steel in mass% was 0.23C, 0.02Si, 1.3Mn, 0.2Cr, 0.02Ti, 0.0029B. The hot-stamped samples (HS) were made with U-bent shape at different temperatures, i.e., 810 °C, 920 °C, and 1,000 °C, to form various microstructures: dual-phases and martensite structures with different grain sizes. The hot workpiece was cooled rapidly via die-clumping using a hydraulic mechanical servo press at a load of 60 tons, where the quenched/tempered dies made of commercial SKD11 tool steel were employed. The 22MnB5 steel sheets were heated in an electric furnace before the hot-stamping process. Furthermore, to improve the steel resistance to HE, the HS were subjected to low-temperature heating for 1 h at 200 °C (HST). The aim of this approach is to reduce the internal strain and to form  $\epsilon$ -carbide precipitates. Figure 1(a) shows the shapes of the hot-stamped U-bent specimens, where the specimen in the bent region is strained to approximately 6.5%.

## 2.2. Material properties

Electron backscatter diffraction (EBSD) analysis was conducted to investigate the microstructural characteristics of the hot-stamped 22MnB5 steels using a high-resolution scanning electron microscopy (SEM) system (JSM-7000F, JEOL) with an acceleration voltage of 15 kV, beam current of 12  $\mu$ A and a step size of  $\sim$ 260 nm. The samples were prepared for this test by polishing their surfaces using 1- $\mu$ m alumina particles to obtain a mirror finish. To examine the precipitation characteristics of the hot-stamped 22MnB5 steels after low-temperature heating, a scanning transmission electron microscopy (STEM) test was performed using replica samples. The STEM samples were prepared by mechanical thinning followed by etching with 1% nitric acid and 99% methanol to a thickness of approximately 100 nm. The sample surface was coated by carbon before observation using STEM (Talos F200X) by an accelerating voltage of 200 kV.

## 2.3. Mechanical properties

Hardness and tensile tests were conducted at 25 °C. The hardness measurements were conducted using a Vickers hardness tester by applying a force of 98 N to the sample surfaces for 15 s. The tensile properties were investigated using the HS before and after hydrogen charging in  $\text{NH}_4\text{SCN}$  for 48 h. In this method, rectangular-dumbbell-shape specimens ( $1 \times 1 \times 1.6$  mm) were designed for the tensile test. The test specimens were cut from the non-deformed zones of the U-bent specimens. The tensile load was applied at  $1 \text{ mm} \cdot \text{min}^{-1}$  up to the fracture point using a screw-driven universal testing machine, whose capacity is 50 kN. Note that tensile tests were conducted immediately after hydrogen charging.

The resistance to delayed fracture was investigated using U-bent specimens under hydrogen charging. In this case, the delayed fracture tests were conducted under static and

cyclic loadings. Figure 1(b) shows the experimental setup for both loading systems. The fracture resistance under cyclic loading was evaluated on the basis of the relationship between the applied load ( $P$ ) and cycle number to final failure ( $N_f$ ). Tensile–tensile cyclic loading was applied to the U-bent specimens in an  $\text{NH}_4\text{SCN}$  solution under load control using a sinusoidal waveform at a frequency of 2 Hz and a load ratio of 0.2. Because the tested steel was required to become hydrogen embrittled in a limited time in  $\text{NH}_4\text{SCN}$ , a pre-hydrogen charging was conducted for 1 and 48 h before cyclic loading. On the other hand, the static loading was applied at 1,000 MPa under electric charging in 3 g/L  $\text{NH}_4\text{SCN}$  + 3% NaCl at  $10 \text{ A}\cdot\text{m}^{-2}$  without pre-hydrogen charging. The hydrogen content in the sample was measured by means of thermal desorption analysis using a gas chromatograph with a linear heating rate of  $200 \text{ }^\circ\text{C}/\text{h}$ . The sample gas was analyzed with a 5 min interval using argon as a carrier gas. In the present analysis, the hydrogen content detected around  $100 \text{ }^\circ\text{C}$  was considered to be the diffusible hydrogen content [8].

In addition, the U-bent specimens for the delayed fracture tests were set to the testing machine using two jointing methods (Fig. 1(b)): (i) tight jointing using bolts (for the static-loading test) and (ii) universal jointing through surface contact (for the cyclic loading test). After the mechanical tests, the fracture surface observations were conducted using SEM.

#### *2.4. Finite element (FE) analysis*

FE analysis was conducted to examine the stress and strain distributions in the U-bent specimens. In this analysis, a three-dimensional four-node tetrahedron solid finite element was employed. The FE model was designed on the basis of the U-bent specimen. As mentioned above, the U-bent specimens for the delayed fracture tests were jointed with two different methods. For the universal jointing, uniform contact loading and surface fixing were used, and,

for the tight jointing using bolts, the U-bent specimens were loaded by the contact of the bolt head with the static-loading as shown in Figure 2. The mesh sizes adjacent to the non-deformed and deformed areas were 70 and 1.5  $\mu\text{m}$ , respectively. Elastic modulus and Poisson's ratio for the steel used here were determined as  $E = 210 \text{ GPa}$  and  $\nu = 0.3$ , respectively. As the U-bent specimen was manufactured using the hot-stamping process, the material properties could be changed especially in the deformed areas due to severe strain [9,10]. In our pre-study, a slightly elevated hardness was obtained in the deformed zone, which is introduced in Section 3. This high hardness value indicates that the elastic modulus in the deformed zone could increase (approximately 10% higher than that in the unbent zone) [9,11]. In this case, plain strain conditions were selected because the brittle failure could occur as a result of hydrogen charging.

### **3. Results and discussion**

#### *3.1. Material properties of the hot-stamped 22MnB5 steel*

Figure 3 shows the image quality (IQ), inverse pole figure (IPF), and kernel average misorientation (KAM) maps recorded in the non-deformed and deformed zones of the U-bent samples prepared at 810 °C, 920 °C, and 1,000 °C. From the IPF maps, the grain size of the martensite structure significantly increased to approximately 25  $\mu\text{m}$  after stamping at 1,000 °C (HS 1,000 °C) compared with that of the other samples. Although the lath martensite-like structure was observed for HS 810 °C, weak martensite (or the dual phase of ferrite and martensite) could be formed because of the sample heating to less than its  $A_3$  line. The mean KAM value for the three samples in the bent regions was approximately 1.57°, which is nearly 5% higher than that in the non-deformed zone. This difference in the microstructural characteristics could form a change in the mechanical properties.

Figure 4 shows the Vickers hardness of the U-bent HS, in which the hardness measurement was conducted at the cross section of the sample in the non-deformed and deformed regions. The hardness level was found to differ depending on the sample, where a low hardness was observed in the HS 810 °C. This can be attributed to the dual phase of ferrite and martensite. Similarly, a high hardness value was observed for the martensite samples (i.e., HS 920 °C and HS 1,000 °C). It was also clear that the hardness values slightly increased in the deformed areas, especially for both martensite samples. This can be attributed to the work hardening with high dislocation density. Note that the increment rate of the hardness, because of the work hardening for the HS, was much lower than that of the cold-stamped samples. Tewary et al.[12] reported a variation in the hardness as a function of cold deformation of Twinning-Induced Plasticity (TWIP) steel, where the hardness value significantly increased even when a small strain was applied.

### *3.2. Tensile properties*

Figure 5(a) shows the representative tensile stress versus tensile strain curves of HS 810 °C, HS 920 °C, and HS 1,000 °C before and after their subjection to hydrogen charging for 48 h. The obtained tensile properties are summarized in Fig. 5(b). Before the charging process, high tensile strength and strain were obtained in case of HS 920 °C and HS 1,000 °C, e.g., the ultimate tensile strength (UTS) and fracture strain of HS 920 °C and HS 1,000 °C were >12% higher than those of HS 810 °C. This high UTS was affected by the high hardness of martensite structure for HS 920 °C and HS 1,000 °C, which is a similar trend to that for the results of the hardness illustrated in Fig. 4. On the other hand, the low tensile properties of HS 810 °C before charging are affected by the low quality of its dual phase steel. It is reported that deformed dual phase steel revealed that void nucleation occurs predominantly along the ferrite-martensite interface,



which could make reduction of the tensile strength and fracture strain; moreover, it is considered that increasing the martensite content raises both the tensile strength and the ductility [13].

Following hydrogen charging, the stress vs. strain curves show visible HE in the three specimens. In this case, severe HE occurred in HS 810 °C, as both the fracture strain and UTS decreased, i.e., the pre-yield failure occurs for HS 810 °C. In HS 920 °C and HS 1,000 °C, the fracture strain values decreased, but the reduction in UTS was not observed. These results show that dual-phase steel (HS 810 °C) was more sensitive to HE of martensitic steels. The approach has been conducted in a previous work [14], where the HE phenomenon in the related dual-phase steels caused a reduction in the fracture strength but no pre-yield failures. In this case, a change in the fracture mode from ductile dimpling to transgranular cleavage was observed. Takahashi et al. have reported that HE of the dual-phase steel showed some anomalous behavior [15].

To understand the failure characteristics of the HS after hydrogen charging, failure analysis was conducted. Figure 6 shows SEM images of the fracture surfaces after tensile tests of the HS 810 °C, HS 920 °C, and HS 1,000 °C before and after charging. Dimple-based ductile fracture, in which there were numerous occurrences of necking associated with dimple-based failures, was the dominant feature type in the three samples before charging. A change in the failure mode was visible after charging. Brittle-intergranular failure is obtained across a wide area of the fracture surface in HS 810 °C after charging, which results in a low UTS and low fracture strain. In contrast, the ductile and brittle mixed failure mode is observed in HS 920 °C and HS 1,000 °C, where cleavage and slip failures were detected in the center and near the surface of the specimen, respectively. This slip-based ductile failure mode could be attributed to the high resistance to HE.

In a previous study, Zhang et al. have attempted to improve the resistance of cold-rolled Al-containing medium-Mn steel to HE using an annealing process, in which the steel showed

excellent strength and ductility [16]. Similar low-temperature heating was studied to improve the resistance of high-strength steels to HE [17]. Thus, to improve the resistance of the HS to HE in this study, the HS sample was heated at 200 °C for 1 h before hydrogen charging for 48 h (HST). Figure 7 shows the resulting tensile properties of HST 920 °C. Although the tensile strength of HST 920 °C slightly decreased, the UTS and fracture strain of HST 920 °C apparently improved compared with those of HS 920 °C. Currently, there is no explanation for this phenomenon, but this could be a result of the hydrogen trapped within the precipitates.

A STEM analysis was conducted to examine the microstructural characteristics of HST 920 °C in detail. Figure 8 shows the STEM images of HS 920 °C before and after the heating process at 200 °C for 1 h. The lath martensite structures with grain size of a few micrometers were observed in both samples. In addition, several precipitates ( $\epsilon$ -carbide) with a diameter of approximately 100 nm were observed in the martensite phases of HST 920 °C but were not observed in HS 920 °C. These precipitates could act as trapping sites for hydrogen, leading to high HE resistance in HST 920 °C. Chen et al. reported that an effective trapping mechanism may be achieved through the incorporation of finely dispersed V–Mo–Nb carbides in bearing steels [18]. Figure 9 displays the SEM images of the fracture surfaces of HST 920 °C after tensile tests. As seen, brittle failure mode was detected in HST 920 °C after hydrogen charging. A comparison between HS 920 °C and HST 920 °C shows that a large area of ductile failure mode was noted in HST 920 °C. Such extended ductile failure region in HST 920 °C can be attributed to the improvement in HE.

Figure 10(a) shows the hydrogen desorption rate vs. heat temperature curves (hydrogen desorption curves) for HS 810 °C, HS 920 °C, HS 1,000 °C, and HST 920 °C. The hydrogen release was clearly detected in all samples upon heating to approximately 120 °C and did not further increase upon additional heating. This high desorption rate of hydrogen is related to the

diffusible hydrogen dissolved in the samples, which causes severe HE. Figure 10(b) shows the hydrogen content in the four samples. Low hydrogen content was detected in HST 920 °C compared with that of the other HS. However, high hydrogen content was observed in the dual phase of HS 810 °C. These differences in the hydrogen content could be attributed to the HE resistance, i.e., the high and low resistance of HST 920 °C and HS 810 °C to HE, respectively.

In this case, the low hydrogen content in HST 920 °C could be affected by the hydrogen trap in the precipitates, which renders the hydrogen non-diffusible. Ishizaki et al. [19] have examined the diffusibility of hydrogen in steels. They concluded that the main reason for hydrogen low diffusibility is the interaction forces between carbon and hydrogen. Here, this occurrence could be attributed to the interaction force between  $\epsilon$ -carbide and hydrogen in HST 920 °C.

### 3.3. Delayed fracture tests

Figure 11(a) shows the relationship between the load amplitude ( $P_a$ ) and the number of cycles to final fracture ( $N_f$ ) for HS 920 °C and HS 1,000 °C. In this approach, the U-bent specimens were immersed in an  $\text{NH}_4\text{SCN}$  solution for 1 and 48 h before cyclic loading, i.e., pre-hydrogen charging. The  $P_a$ - $N_f$  curves indicate that the resistance of the two test specimens to HE was similar at high load amplitudes, which can be attributed to their tensile properties (Fig. 5). However, a visible difference in the resistance to HE was observed at a low load amplitude ( $P = 135 \text{ N}$ ). This indicates that the extent of HE resistance depends on the loading conditions. In addition, the loading speed (loading time) changed the severity of HE as a slowly applied load increased HE [17]. This can be related to the time required to diffuse hydrogen to the trap site on the prior austenite ( $\gamma$ ) grain (PAG) boundary, i.e., the longer the time, the lower the resistance of HE. Resistance to HE also differs depending on the pre-hydrogen charging time (1 or 48 h)

before the cyclic loading; i.e., the longer the hydrogen pre-charging, the lower the resistance to HE, which is the reason why the resistance of HS 920 °C to HE was >1.5 times that of HS 1,000 °C at the low applied loading.

Figure 11(b) shows the results of the delayed fracture test under static loading, i.e., the applied load vs. time to failure for HS 920 °C and HS 1,000 °C. In this test, the U-bent specimens were loaded by bolt jointing. As seen, high and low fracture resistance was obtained for HS 920 °C and HS 1,000 °C, respectively, which is similar to the results of their  $P_a-N_f$  curves at low cyclic loading conditions. The difference in the HE extent can be attributed to the different microstructural characteristics between HS 920 °C and HS 1,000 °C, i.e., grain size, as previously mentioned. In this case, hydrogen could be trapped around PAG at different concentrations; e.g., the higher hydrogen concentration in HS 1,000 °C is due to small grain boundary area, arising from the large grain diameter of PAG. In a previous work [20], the susceptibility to HE was found to be related to the grain size in the martensitic medium carbon steel (C: 0.36%); e.g., large PAG exhibited high hydrogen concentrations at its grain boundaries.

To interpret this, the size of PAG in HS 920 °C and HS 1,000 °C was investigated. Figure 12 illustrates the microstructures of both samples, showing the boundary of the prior  $\gamma$  grain examined using EBSD. Different sizes of PAG boundaries were observed as indicated by the white lines. A large PAG was formed in HS 1,000 °C, which was approximately twice that of HS 920 °C. These differences in PAG can be attributed to the severity of HE because of the aforementioned different concentrations of hydrogen. Momotani et al. [21] reported that hydrogen accumulation leads to microcracks along the prior austenite grain boundaries and brittle fractures in their vicinity. Moreover, Singh et al. [22] reported that, in hydrogen-charged specimens, intergranular crack growth occurred along the large prior austenite grain boundaries

and transgranular crack propagation through small PAGs. This result is also observed in the failure analysis conducted here (Fig. 7).

### *3.4. Failure analysis of delayed fracture tests*

Figure 13(a) shows the U-bent specimens after the delayed fracture test, showing the fracture positions. In this case, the U-bent specimens were loaded using tight and universal jointing. The failure positions differed depending on the jointing method. In case of tight jointing, the U-bent samples were fractured around the corner between the non-deformed and deformed regions. In contrast, the specimen failure occurred on top of the U-bent specimen with the universal jointing. The different failure may be caused by different stress distribution patterns. To verify the different fracture modes, FE analysis was conducted.

Figure 14 shows the von Mises strain distribution of the U-bent specimen loaded by two jointing methods. Two strain distribution patterns were observed depending on the jointing system. A high strain concentration occurred on top of the U-bent specimen for the universal jointing, which corresponds to the fracture area shown in Fig. 13. In case of tight jointing, the strain non-uniformly spread out to the wide area in the specimen around the corner between the non-deformed and deformed areas, as indicated by the dashed circles. Moreover, a relatively high strain was detected in the specimen around the bolt and nut. The non-uniformly distributed strain causes hydrogen to diffuse to the U-bent specimen. Therefore, HE in the 22MnB5 steel is affected by the trap sites caused by the strain (stress) distribution and microstructural characteristics.

## **4. Conclusions**

The HE of 22MnB5 steel was investigated experimentally and numerically using HS manufactured at different temperatures. The obtained results can be summarized as follows:

- 1) The HS prepared at 810 °C was formed by the dual phases of ferrite and martensite, and the martensite structure was created because of hot stamping at 920 °C and 1,000 °C. The size of the martensite phase increased during heating to 1,000 °C compared with that at 920 °C. A slightly high hardness was obtained in the deformed zone for the HS because of work hardening.
- 2) Reductions in both UTS and fracture strain were detected for the dual-phase steel (HS 810 °C) after hydrogen charging, in which the pre-yield failures were observed. For the martensitic steels (HS 920 °C and HS 1,000 °C), no visible decrement in UTS was observed although there was a decrease in the fracture strain. The steel resistance to HE was improved for the martensitic steel (HS 920 °C) after low temperature heating, where a large number of precipitates ( $\epsilon$ -carbide) created a trapping site.
- 3) Dimple-based ductile fracture was a dominant feature in the three HS before the charging. After hydrogen charging, brittle-intergranular failure occurred across a wide area of the fracture surface in HS 810 °C. However, a ductile and brittle mixed failure mode was observed in HS 920 °C and HS 1,000 °C, in which cleavage and slip failure are detected in the center and near the surface of the specimen, respectively. These failure modes were attributed to the level of HE.
- 4) The steel resistance to HE was investigated by a delayed failure test under static and cyclic loadings after hydrogen charging. HS 920 °C and HS 1,000 °C exhibited different resistance to HE (HS 1,000 °C showed lower resistance) at low levels of cyclic loading. This was affected by the high concentration of hydrogen atoms around the prior austenite grain.

- 5) The failure in the U-bent samples was attributed to the stress–strain distribution, where the high-strain (or high stress) region is caused by the large hydrogen content, i.e., the fracture point. In this case, a high stress concentration occurred on top of the U-bent specimen when universal jointing was used. However, the strain (or stress) values were spread out in non-uniform patterns to the wide area in the U-bent specimen as a result of the tight jointing, causing a fracture to occur around the corner of the specimen between the non-deformed and deformed regions.

### **Acknowledgements**

This research was conducted in one of the projects of a commission business future exploitation research program, controlled by the New Energy and Industrial Technology Development Organization (NEDO), Japan.

### **Compliance with ethical standards**

**Conflicts of interest: The authors declare no conflict of interest.**

### **References**

- 1 S-W. Wang, P-K. Lee, Investigation of die quench properties of hot stamping steel 15B22, *China Steel Tech. Rep.* 26(2013)22–31.
- 2 S. Zhang, S. Liu, J. Wan, W. Liu, Effect of Nb–Ti multi-microalloying on the hydrogen trapping efficiency and hydrogen embrittlement susceptibility of hot-stamped boron steel, *Mater. Sci. Eng. A* 772(2020)138788.
- 3 C. Georges, T Sturel, P. Drillet, J.-M. Mategne, Absorption/Desorption of Diffusible Hydrogen in Aluminized Boron Steel, *ISIJ Int.* 53(2013)1295–1304.
- 4 W.-j. Chen, P.-f. Gao, S. Wang, H.-z. Lu, Z.-z. Zhao, Effect of vanadium on hydrogen embrittlement susceptibility of high-strength hot-stamped steel, *J. Iron Steel Res. Int.* In press.

- 5 Y. Zhang, W. Hui, X. Zhao, C. Wang, H. Dong, Effects of hot stamping and tempering on hydrogen embrittlement of a low-carbon boron-alloyed steel, *Materials* 11(2018)2507.
- 6 S.J. Lee, A.R. Joseph, K. George, K.M. David, Hydrogen embrittlement of hardened low-carbon sheet steel, *ISIJ Int.* 50(2010)294–301.
- 7 S. Takagi, Y. Toji, M. Yoshio, K. Hasegawa, Hydrogen Embrittlement Resistance Evaluation of Ultra High Strength Steel Sheets for Automobiles, *ISIJ Int.* 52(2012)316–322.
- 8 M. Okayasu, L. Yang, Influence of microstructure on the mechanical properties and hydrogen embrittlement characteristics of 1,800 MPa grade hot-stamped 22MnB5 steel, *J. Mater. Sci.* 54(2019)5061–5073.
- 9 A.I. Medved', A.E. Bryukhanov, The variation of Young's modulus and the hardness with tempering of some quenched chromium steels, *Met. Sci. Heat Treat.* 11(1969)706–708.
- 10 M. Ortiz, J. Ovejero-Garcia, Effect of hydrogen on Young's modulus of AISI 1005 and 1070 steels, *J. Mater. Sci.* 27(1992)6777–6781.
- 11 J. Musil, F. Kunc, H. Zeman, H. Polakova, Relationships between hardness, Young's modulus and elastic recovery in hard nanocomposite coatings, *Surf. Coat. Technol.* 154(2002)304–313.
- 12 N.K. Tewary, S.K. Ghosh, S. Bera, D. Chakrabarti, S. Chatterjee, Influence of cold rolling on microstructure, texture and mechanical properties of low carbon high Mn TWIP steel, *Mater. Sci. Eng. A* 615(2014)405–415.
- 13 M. Sarwar, R. Priestner, Influence of ferrite-martensite microstructural morphology on tensile properties of dual-phase steel, *J. Mater. Sci.* 31(1996)2091–2095.
- 14 R.G. Davies, Hydrogen embrittlement of dual-phase steels, *Metall. Trans. A* 12A(1981)1667–1672.
- 15 K. Takashima, Y. Yoshioka, K. Yokoyama, Y. Funakawa, Hydrogen embrittlement behavior of ultra-high strength dual phase steel sheet under sustained tensile-loading test, *ISIJ Inter.* 58(2018)173–178.
- 16 Y. Zhang, C. Shao, J. Wang, X. Zhao, W. Hui, Intercritical annealing temperature dependence of hydrogen embrittlement behavior of cold-rolled Al-containing medium-Mn steel, *Int. J. Hydrogen Energy* 44(2017)22355–22367.
- 17 M. Okayasu, J. Motojima, Microstructure-dependent hydrogen diffusion and trapping in high-tensile steel, *Mater. Sci. Eng. A* 790(2020)139418
- 18 Y.-S. Chen, D. Haley, S.S.A. Gerstl, A.J. London, F. Sweeney, R.A. Wepf, W.M. Rainforth, P.A.J. Bagot, M.P. Moody, Direct observation of individual hydrogen atoms at trapping sites in a ferritic steel, *Science* 355(2017)1196–1199.
- 19 T. Ishizaki, T. Takeda, K. Higashi, Hydrogen evolution from steels containing  $\epsilon$ -Cu precipitates, *J. Jpn. Inst. Metals* 46(1982)28–37.



- 20 H. Fuhigami, H. Minami, M. Nagumo, Effect of grain size on the susceptibility of martensitic steel to hydrogen-related failure, 86(2006)21–29.
- 21 Y. Momotani, A. Shibata, D. Terada, N. Tsuji, Effect of strain rate on hydrogen embrittlement in low-carbon martensitic steel, Int. J. Hydrogen Energy 42(2017)3371–3379.
- 22 R. Singh, A. Singh, P.K. Singh, D.K. Mahajan, Role of prior austenite grain boundaries in short fatigue crack growth in hydrogen charged RPV steel, Int. J. Press. Vessel. Pip. 171(2019)242–252.

### Figure captions

**Fig. 1.** Photographs of **(a)** U-bent specimen and **(b)** jointing methods for the delayed failure tests under static and cyclic loading

**Fig. 2.** Models of FE analysis showing the contact conditions: **(a)** universal jointing and **(b)** tight jointing using bolts.

**Fig. 3.** Microstructural characteristics of the U-bent samples prepared at 810 °C, 920 °C, and 1,000 °C, showing the image quality (IQ), inverse pole figure (IPF), and kernel average misorientation (KAM) maps examined using EBSD

**Fig. 4.** Vickers hardness test results for the hot-stamped U-bent samples heated at various temperatures: non- and deformed region

**Fig. 5 (a)** Representative stress–strain curves for the 22MnB5 steel prepared at 810 °C, 920 °C, and 1,000 °C before and after hydrogen charging for 48 h. **(b)** Ultimate tensile strength ( $\sigma_{UTS}$ ) and failure strain ( $\epsilon_f$ ) of the 22MnB5 steel prepared at 810 °C, 920 °C, and 1,000 °C before and after hydrogen charging for 48 h

**Fig. 6.** SEM images of the fracture surfaces after the tensile tests of HS 810 °C, HS 920 °C, and HS 1,000 °C **(a)** before and **(b)** after hydrogen charging for 48 h

**Fig. 7.** Tensile properties for the 22MnB5 steel prepared at 920 °C before and after a low temperature heating at 200 °C for 1 h

**Fig. 8.** STEM images for the HS 920 °C and HST 920 °C, showing martensite with and without  $\epsilon$ -carbides

**Fig. 9.** SEM images of the fracture surfaces after the tensile tests of HST 920 °C before and after hydrogen charging for 48 h

**Fig. 10 (a)** Hydrogen desorption rate versus heat temperature curves (hydrogen desorption curves) for the U-bent samples prepared at 810 °C, 920 °C, and 1,000 °C. **(b)** Hydrogen level versus heat temperature curves for the U-bent samples prepared at 810 °C, 920 °C, and 1,000 °C

**Fig. 11 (a)** The relationship between the load amplitude ( $P_a$ ) and the number of cycles to final fracture ( $N_f$ ) for HS 920 °C and HS 1,000 °C. **(b)** The relationship between the applied static stress and time to failure for HS 920 °C and HS 1,000 °C

**Fig. 12.** SEM images of the microstructures of the non-deformed and deformed areas in 22MnB5 prepared by hot stamping at 920 °C and 1,000 °C, showing prior austenite grain boundary

**Fig. 13.** Photographs of the U-bent specimens of HS 810 °C, HS 920 °C, and HS 1,000 °C after the delayed failure tests, showing fracture area in the specimens with **(a)** universal jointing and **(b)** tight bolt jointing

**Fig. 14.** The von Mises strain distribution of U-bent specimen loaded by **(a)** universal jointing and **(b)** tight bolt jointing