

Delayed Fracture Evaluation Methods in Ultra-High Strength Steel Sheets for Automotive Components

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Abstract:

JFE Steel has developed delayed fracture evaluation methods in ultrahigh-strength steel sheets intended for automotive component applications. For TS 1 470 MPa grade steel, the effects of shearing conditions, baking treatment, and secondary forming at the sheared edge during manufacturing processes of automotive components were investigated by delayed fracture tests based on four-point bending. By combining these evaluation methods with forming analysis by CAE, we demonstrated their capability of predicting delayed fracture using model components. To simulate hydrogen absorption behavior under actual environmental conditions, JFE Steel developed a cyclic test method, HeTsACETM. This method was utilized to examine the relationship between hydrogen absorption and delayed fracture.

1. Introduction

One challenge for the application of ultra-high strength steel sheets to automotive components is delayed fracture, which is caused by hydrogen absorption in steel sheets. The general automotive component production process includes a shearing process during blanking, a press forming process, and a painting and baking treatment process. Therefore, in delayed fracture evaluations of ultra-high strength steel sheets, it is necessary to consider not only material factors such as the material composition and microstructure, but also factors in the production process, that is, the effects of stress and strain generated in the shearing process¹⁾, the effect of strain applied in the press forming process²⁾, and changes in the microstructure in baking treatment³⁾. Furthermore, since hydrogen is absorbed as a result of corrosion even after the vehicle is produced, depending on the actual use environment, it is also necessary to judge the applicability of materials to auto-

motive components, taking into account the effect of hydrogen absorption.

For the first issue, that is, the consideration of factors related to the production process, the authors have proposed a delayed fracture evaluation method using a four-point bending test piece¹⁻³⁾. In Chapter 2, the respective effects of the shearing process, press forming process, and painting and baking treatment on delayed fracture were investigated in a simple manner using various types of immersion tests. In addition, model components simulating actual automotive components were fabricated, and the possibility of predicting the risk of delayed fracture of the sheared edge of automotive components by CAE based on the above-mentioned knowledge was verified.

For the second issue, consideration of the hydrogen absorption environment, we studied the hydrogen absorption behavior in the actual use environments of automobiles. The hydrogen absorbed in steel sheets, which is the cause of delayed fracture, is mainly generated by the corrosion reaction that proceeds at the steel sheet surface. Generally speaking, once corrosion is initiated, it does not cease completely, but continues at an increasing or decreasing rate, depending on changes in the surrounding environmental conditions. Since the hydrogen formed by the corrosion reaction also increases or decreases depending on the environmental conditions, the absorption rate of hydrogen in steel sheets changes with time. However, many points concerning the detailed mechanism of these changes are still unclear and have not been standardized in a unified evaluation standard. Therefore, as a method that reproduces the delayed fracture resistance during actual use, we developed and verified the effectiveness of a cyclical test for evaluation of delayed fracture which simulates the hydrogen absorption behavior in actual environments. The developed method, called HeTsACETM

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(Hydrogen embrittlement Test to simulate Actual Corrosion Environment), is described in Chapter 3 of this paper.

2. Delayed Fracture Evaluation Method for Thin Steel Sheets Considering Automotive Component Production Processes

Focusing on the production processes used in the manufacture of automotive components, this chapter examines the effects of shearing conditions in blanking in Section 2.1, forming strain in press forming in Section 2.2, and baking treatment in Section 2.3 as factors that affect delayed fracture resistance. In addition, as described in Section 2.4, model components simulating actual automotive components were prepared, and the possibility of predicting delayed fracture risk using model components by CAE of the sheared edge of automotive components was verified.

2.1 Effect of Shearing Conditions on Delayed Fracture¹⁾

This section reports the effects of residual stress and lattice defects at the edge, which occur as a result of shearing forming of 1 470 MPa grade cold-rolled steel sheets, on delayed fracture resistance.

As shown in **Fig. 1**, rectangular four-point bending test specimens with a sheared edge were prepared by performing shearing by lowering the upper blade with the steel sheet fixed by a blank holder at the position of the lower blade. The evaluation target was the sheared edge of the steel sheet remaining on the lower blade after shearing (hereinafter, lower-blade-side sheared edge), while the other edges were cut and removed. To investigate the effects of the shearing conditions, the ratio of the clearance between the upper and lower blades relative to the sheet thickness (shear clearance) was varied in the range from 5% to 20%, and the angle between the upper and lower blades (shear angle) was set between 0° and 2°. Stress loading of the rectangular test specimens was applied by four-point bending with the fracture surface side of the sheared edge as the

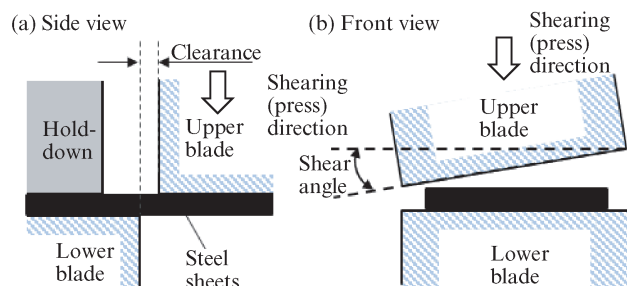


Fig. 1 Schematic diagram of shear method with clearance and shear angle

outer side of bending. In the delayed fracture test, the specimens were immersed for 96 h in 0.1% NH_4SCN + McIlvaine buffer solution adjusted to pH 7.0.

Figure 2 shows the results of the delayed fracture test evaluation of the steel sheared with a constant shear angle of 0° and various shear clearances and the results with a constant shear clearance of 15% and various shear angles. At shear clearances up to 15%, the maximum unfractured stress increased as the shear clearance increased, and at the shear clearance of 15%, cracking was not observed even at an applied stress of 1 400 MPa. On the other hand, at shear clearances larger than 15%, the maximum unfractured stress decreased. When the shear angle was changed, the maximum unfractured stress decreased as the shear angle increased. It is known that residual stress and lattice defects of the edge caused by shearing affect delayed fracture resistance. **Figure 3** shows the effects of the shear clearance and shear angle on the residual stress generated by shearing. When the shear angle is 0° and the shear clearance was increased in the range from 5% to 15%, the residual stress of the sheared edge decreased, but when the clearance was greater than 15%, almost no change was observed in the residual

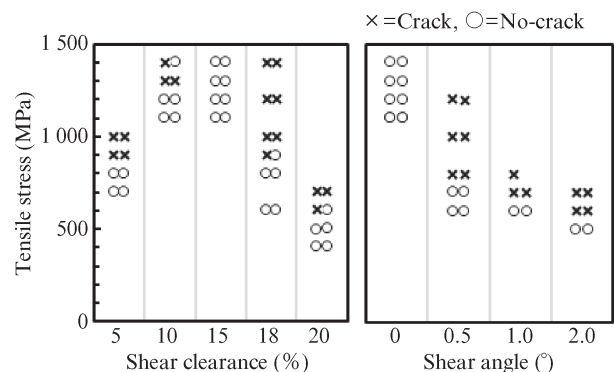


Fig. 2 Delayed fracture test results with various shear clearances and shear angles

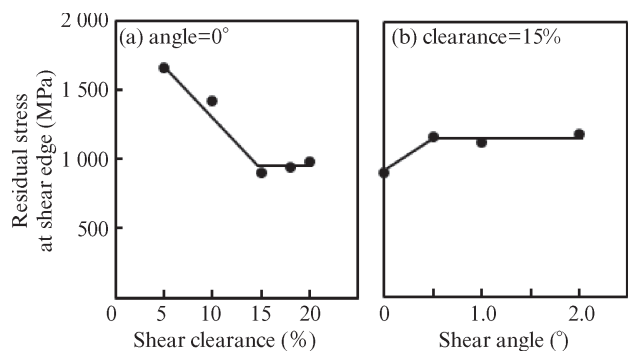


Fig. 3 Residual stress at shear edge; (a) with 0° shear angle and varied shear clearances and (b) with 15% shear clearance and varied shear angles

stress of the sheared edge. When the shear clearance was constant at 15% and the shear angle was increased from 0° to 0.5°, the residual stress of the sheared edge increased slightly. However, when the shear angle was larger than 0.5°, there was no significant change in the residual stress of the sheared edge.

Figure 4 shows the effects of the shear clearance and shear angle on the diffusible hydrogen content in the region within 1 mm from the edge. Since changes in the hydrogen content show a correlation with the amount of lattice defects, an increase in lattice defects of the sheared edge with increases in the shear clearance and shear angle was confirmed.

Figure 5 is a schematic illustration summarizing the changes in the maximum unfractured stress of the steel sheets accompanying changes in the shearing conditions and their main factors. Relative to the shearing conditions of a shear clearance of 15% and shear angle of 0°, the maximum unfractured stress of the steel sheets decreases as the shear clearance and shear angle increase.

The main factor in these changes is considered to be the increase in the amounts of dislocations and atomic vacancies induced by shearing. On the other hand,

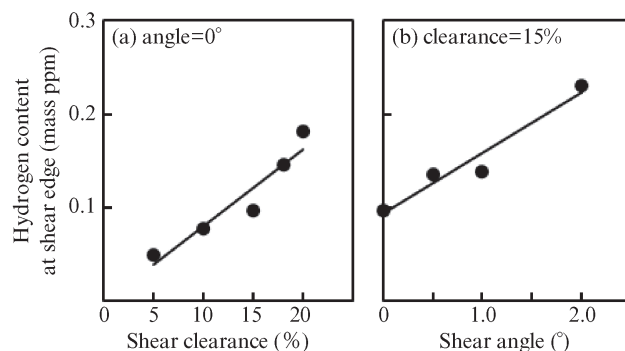


Fig. 4 Hydrogen content at shear edge; (a) with 0° shear angle and varied shear clearances and (b) with 15% shear clearance and varied shear angles

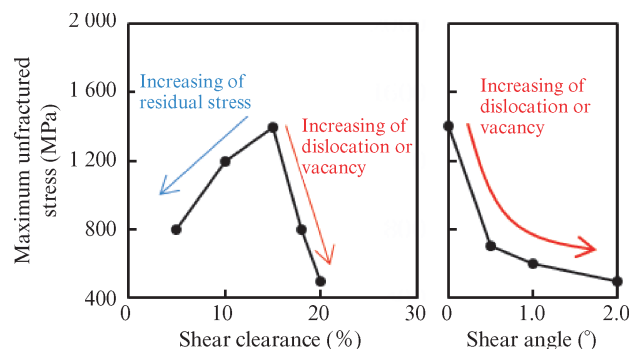


Fig. 5 Schematic illustration summarizing effects of shear angle and shear clearance on maximum unfractured stress

although the maximum unfractured stress of the steel sheets sheared under the condition of a shear angle of 0° decreased even when the shear clearance was reduced to less than 15%, the main cause in this case is considered to be an increase in the residual stress introduced by shearing.

2.2 Effects of Sheared Edge in Press Forming on Delayed Fracture²⁾

This section reports the effect on delayed fracture resistance characteristics when press forming strain was applied to sheared edge²⁾ caused by shearing of an ultra-high strength steel (UHSS) model alloy thin steel sheet to simulate the strain due to press forming.

Here, a steel sheet with a thickness of 1.4 mm was sheared by the same method as in Section 2.1, and a rectangular four-point bending test specimen with only the lower-blade-side sheared edge and the octagonal blank shown at the left in **Fig. 6** were prepared. For a strict evaluation, the shearing conditions were set to a shear angle of 0.5° and a shear clearance of 10%, referring to the knowledge obtained in Section 2.1.

The stress loading in the delayed fracture test was applied by four-point bending in the same manner as in the previous section. The delayed fracture tests of the four-point bending specimens and model components were carried out by immersion for 96 h in a hydrochloric acid solution.

The press forming and strain introduction methods for the sheared edge were as follows²⁾. Tensile forming strain was given by applying tensile deformation to rectangular specimens having a sheared edge. Using sheared samples with dimensions of 18 mm × 100 mm, 15 mm at each edge was fixed in the tensile testing machine by using clamps, and uniaxial tensile deforma-

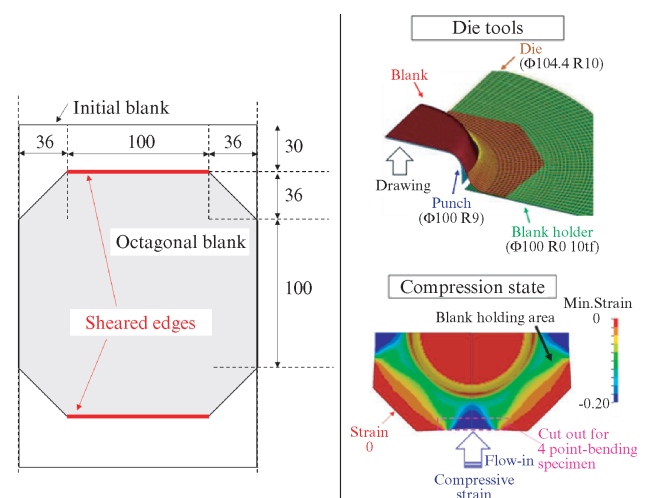


Fig. 6 Octagonal blank manufactured by shearing process (left), and compressive secondary forming by deep drawing (right)

tion was applied to the rectangular samples while measuring the strain with an extensometer with a gauge length of 50 mm. Compressive forming strain was applied to the octagonal blank by drawing. Octagonal blanks, in which the edge to be evaluated was the lower-blade-side sheared edge, were prepared by preliminary machining of the test steel to a rectangular shape with dimensions of 172 mm × 232 mm, followed by shearing as shown at the left in Fig. 6, with the shear angle and shear clearance set to 0.5° and 10%, respectively. Uniaxial compressive forming strain was then applied to the 100 mm long sides by deep drawing of the blank with the cylindrical deep drawing die shown on the right in Fig. 6. The blank holding force was set to 10 ton f, which is sufficient to suppress flange wrinkles. After deep drawing, a test specimen measuring 18 mm × 74 mm was cut out from the flange part so that the compression area of the long-side shear edge was the stress loading area at the center of four-point bending. The edges not subject to evaluation were ground to prevent delayed fracture.

Figure 7 shows the relationship between the amount of secondary forming strain and loading stress, indicating the boundary between the presence/absence of delayed fracture, as a schematic delayed fracture limit curve. Delayed fracture occurred in two regions: one in which the forming strain is small and the loading strain is high, and the other in which the forming strain is large and the loading strain is also high. From this diagram, it is possible to arrange the OK area where delayed fracture does not occur and the NG area where delayed fracture can occur in terms of the forming strain and loading stress.

Although delayed fracture is improved by a small amount of forming strain, on the other hand, delayed fracture risk increases if the amount of forming strain is excessive. As the reason for this, as described in the previous report²⁾ by some of the present authors, stress relaxation of the sheared edge occurs in the low strain region, but in the high strain region, damage accumulates as a result of the irregular surface of the sheared edge and forming strain.

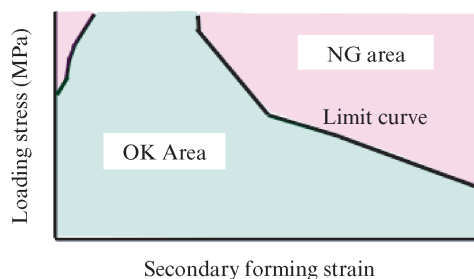


Fig. 7 Delayed fracture limit curve with secondary forming strain on sheared edge and loading stress

By using the limit curve of forming strain and residual stress shown in Fig. 7, it is considered possible to predict the risk of delayed fracture of press-formed components. Section 2.4 presents the results of a verification of this conjecture using model components.

2.3 Effect of Baking Treatment on Delayed Fracture³⁾

This section reports the effect of baking treatment of 1 470 MPa cold-rolled steel sheets on delayed fracture resistance characteristics. To eliminate the effects of shearing, grinding finishing was applied to all the edges of the rectangular four-point bending specimens. Stress loading was applied to the test specimens by four-point bending, and the delayed fracture test (N = 2) was conducted by immersing the test specimens for 96 h in a 0.1% NH₄SCN + McIlvaine buffer solution adjusted the pH to 5.0. Baking treatment, in which the specimens were held for 20 min after reaching 170°C, was performed after four-point stress loading.

Figure 8 shows the results of the delayed fracture test of four-point bending specimens (a) without and (b) with baking treatment. In comparison with the specimens without baking treatment, the maximum unfractured stress (fracture limit) of the specimens with baking treatment increased, indicating that delayed fracture resistance was improved by baking treatment.

From the hydrogen desorption curves obtained by TDA (Thermal Desorption Analysis) of the specimens with and without baking treatment, hydrogen desorption curves with a peak temperature of 80°C were confirmed for all specimens, and there were almost no differences in the hydrogen distribution position. On the other hand, hydrogen absorption was decreased by baking treatment, as the amount of hydrogen desorption was 0.79 ppm without baking treatment but 0.59 ppm with baking treatment.

Figure 9 shows the results of an investigation of the amount of carbon segregation to the prior austenite grain boundaries in the specimens (a) without and (b) with baking treatment by STEM-EDS. An increase in carbon segregation to the prior austenite grain bound-

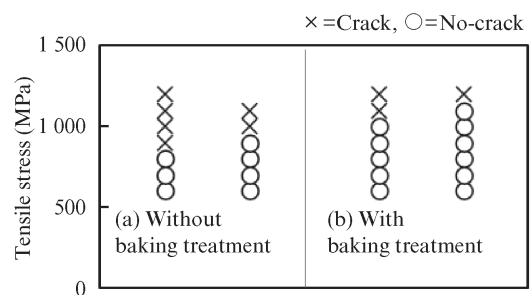


Fig. 8 Delayed fracture test results of bending specimens without (a) and with (b) baking treatment

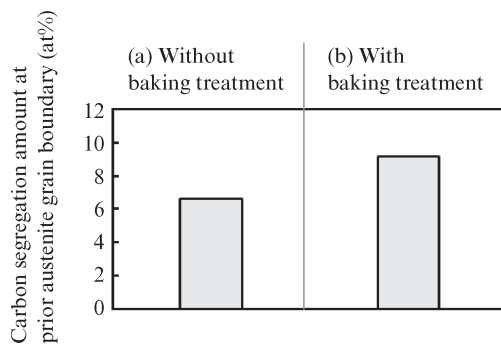


Fig. 9 Carbon segregation amount at prior austenite grain boundary of specimens without (a) and with (b) baking treatment

ary as a result of baking treatment was observed. The mechanism of improvement of delayed fracture resistance by baking treatment is considered to involve reduction of hydrogen absorption in the steel sheet and suppression of hydrogen absorption to the prior austenite grain boundary by the increase in carbon segregation to the grain boundary, which tends to suppress fracture at those grain boundaries.

2.4 Study of Delayed Fracture Risk Prediction Using Model Components

Advance prediction of positions with a risk of delayed fracture in automotive components is extremely useful for material selection and shortening the preparations for production. This section describes a method for prediction of the risk of delayed fracture by CAE using the knowledge gained up to the previous section.

Using the same thin steel sheet as in Section 2.2, the model component shown in **Fig. 10** was prepared by a first process of drawing and second process of form forming. This component includes the shapes assumed in actual components, namely, ① Concaved, ② Compressed, ③ Stretched (simulating a spot bearing surface) and ④ Bent.

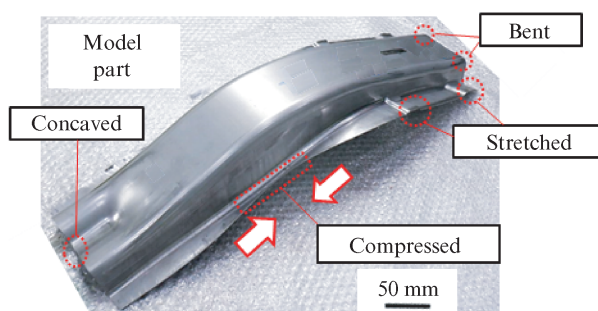


Fig. 10 Ultra-high strength steel model (part A) simulating automobile under parts for evaluation of delayed fracture

The outer periphery of this component is the lower-blade-side sheared surface resulting from shearing of the blank. As the shearing conditions, the shear angle and shear clearance were set to 0.5° and 10%, as in Section 2.2, and for a strict evaluation, baking treatment was not performed, referring to Section 2.3. In addition, two types of test specimens were also prepared by changing the width of the short side of the blank, part A with comparatively high stress in the strongly compressed area, and part B, with comparatively low stress in that area (part A is shown in **Fig. 10**).

In order to verify the delayed fracture risk, parts A and B were subjected to the same delayed fracture test by immersion in hydrochloric acid as in Section 2.2. The test results are shown **Fig. 11**. In part A, delayed fracture occurred only in ② the drawing compression (Compressed) area, whereas in part B, delayed fracture did not occur from any of the outer edges.

To verify the validity of these delayed fracture test results and the possibility of prediction, CAE calculations by FEM analysis of the amount of secondary forming strain and the residual stress of forming of the sheared edge were carried out for parts A and B.

The possibility of predicting the delayed fracture risk of the sheared edge at various parts of the model components was examined by comparing parts A and B by the CAE calculations using FEM analysis of the forming strain and the residual stress after forming of the sheared edge. It was investigated that whether it was possible to predict the delayed fracture risk of the sheared edge at each part of the model components by comparing this calculation results with the boundary of the occurrence of delayed fracture in **Fig. 7**. It is conjectured that a high risk of delayed fracture exists at positions where the amount of secondary forming strain and residual stress of forming of the model component in the CAE calculation are in the risk region (NG area) in **Fig. 7**, and the risk of delayed fracture is low in the safe region (OK area).

Figure 12 shows a comparison of the amount of

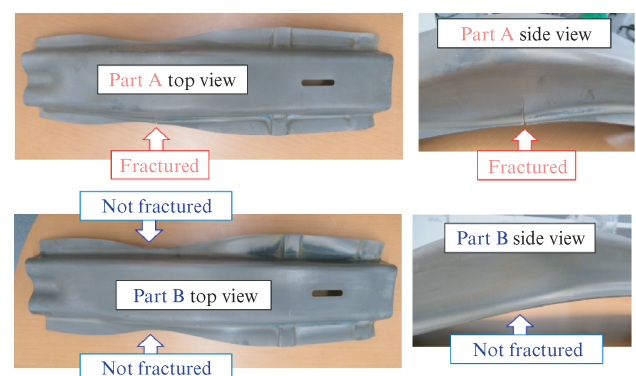


Fig. 11 Delayed fracture test results of model part A & B

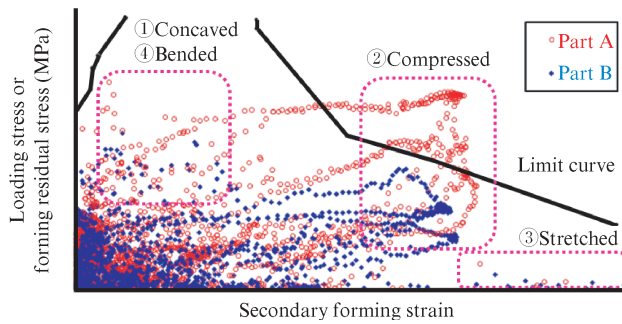


Fig. 12 Comparison between delayed fracture limit curve and forming CAE results of model part A & B for fracture prediction

secondary forming strain and residual stress of forming in the CAE calculation and the delayed fracture limit curve in Fig. 7. Figure 12 also shows the plotted areas containing the locations of the representative shapes of the model components. According to this comparison, it was predicted that delayed fracture would occur only at ② drawing compression (Compressed) area of part A, and delayed fracture would not occur in part B. These predictions are consistent with the results of the delayed fracture evaluation test in Fig. 11. Therefore, in the case of these model components, it is considered possible to predict the risk of delayed fracture and the location of fracture occurrence by combining a delayed fracture evaluation and forming CAE.

From the results described in this Chapter 2, it was suggested that prediction and reduction of the risk of delayed fracture in automotive components are possible by performing a delayed fracture evaluation which considers the automotive component production process, while also adopting forming CAE.

3. Delayed Fracture Evaluation of Materials by Cyclic Testing Simulating Actual Environments

Up to the previous chapter, this paper has examined the effects of the mass production process of automotive components on delayed fracture. Next, this chapter investigates the hydrogen absorption behavior of vehicles under actual environments.

This chapter reports the development of a cyclic test for evaluation of delayed fracture simulating the hydrogen absorption behavior in actual environments (HeTsACETM: Hydrogen embrittlement Test to simulate Actual Corrosion Environment) as a method that reproduces delayed fracture resistance during actual use and the verification results of its effectiveness.

As the test specimen, a cold-rolled steel sheet with tensile strength of 1 470 MPa grade was sheared under the conditions of a shear clearance of 5% and shear

angle of 0.8°, after which specimen was ground to a size of 30 mm × 100 mm, while leaving the lower-blade-side shear edge. Holes for mounting a jig were then drilled, the specimen was press formed under the conditions of a bending angle of 90° and bending radius of 7 mm. Tensile stress was applied to the surface layer on the convex side of the V-bending part of the specimen by narrowing the maximum opening by the prescribed amount by bolt-tightening.

A hydrogen absorption monitoring system⁴⁾ employing an electrochemical hydrogen permeation method was used to measure the absorbed hydrogen amount of the steel sheet. Referring to the strain introduced at the apex of the specimen by V-bending, an approximately equivalent amount of strain was applied to the steel sheet by cold rolling. The amount of absorbed hydrogen was determined by continuously measuring the hydrogen permeation current of this steel sheet. For details, please refer to reference⁴⁾.

Using the V-bending test specimen and hydrogen absorption monitoring system were used in the above-mentioned cyclic test for evaluation of delayed fracture simulating actual environments (HeTsACETM), the delayed fracture resistance and hydrogen absorption behavior of the test material were evaluated.

The HeTsACETM test was conducted for 9 weeks in a test chamber under cyclic humidity conditions, as shown in Fig. 13. The test temperature was set to one of three constant conditions of 10°C, 30°C or 50°C, and 10 g/m² of chloride was deposited on the specimens by spraying an aqueous solution of NaCl under these respective temperature conditions. At the test temperature of 30°C, additional chloride deposition conditions of 0.3 g/m² and 3 g/m² were added. Two times each week, water-washing was performed to remove the chlorides from the steel sheet surface in a process at a constant humidity of 35%, after which the test was resumed by spraying the NaCl solution again under the same conditions.

Figure 14 shows the results of the delayed fracture evaluation of the V-bending specimens when the loading stress was varied in the HeTsACE tests with the different test temperatures. The open circle (○) plots in

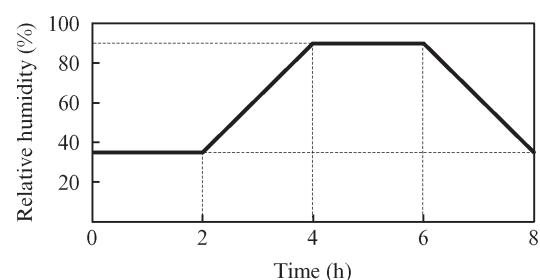


Fig. 13 Dry-wet cycle of HeTsACETM

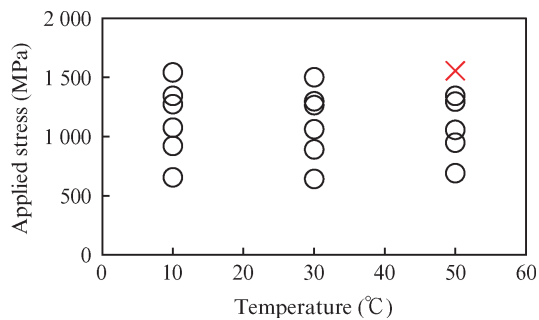


Fig. 14 Relationship between temperature and delayed fracture susceptibility in HeTsACE™ with 10 g/m² NaCl deposition

the figure indicate levels at which delayed fracture did not occur within the test period, and the × plots are levels at which delayed fracture occurred during the test period. The stress level at which delayed fracture can occur changed depending on the test temperature.

Figure 15 shows the change over time in the hydrogen permeation current measured by the hydrogen absorption monitoring system in the HeTsACE test conducted under the conditions of a test temperature of 30°C and deposited chloride amount of 3 g/m², indicating that the amount of hydrogen absorption in the steel increases as the hydrogen permeation current becomes larger.

From the viewpoint of short-term change over time, the hydrogen permeation current displayed periodic repeated increase and decrease corresponding to changes in the humidity, in a pattern that showed its maximum value when the relative humidity was approximately 60%. When NaCl is deposited on the surface of a steel sheet, a water film containing deliquesced NaCl forms when the relative humidity reaches about 60%⁵⁾. It is known that the thickness of the formed film becomes thicker as the relative humidity increases, and becomes thinner as the relative humidity decreases. In atmospheric corrosion, concentration of Cl⁻ ions at the corrosion interface accelerates the oxidation reaction of Fe²⁺ ions, causing a decrease in the

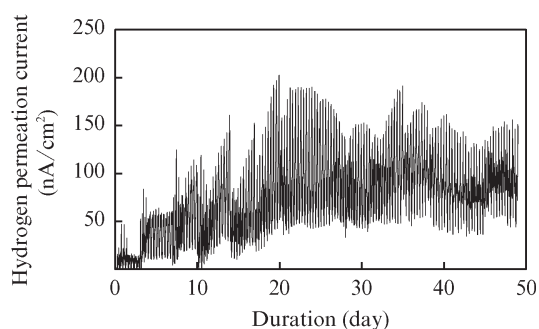


Fig. 15 Hydrogen absorption behavior in HeTsACE with 30°C and 3 g/m² NaCl deposition

local pH, and this pH decrease promotes hydrogen absorption⁶⁾. When the relative humidity is about 60%, the water film on the steel sheet surface is thinnest, resulting in enrichment of the Cl⁻ ions. It is thought that the hydrogen permeation current periodically shows its maximum value when the relative humidity is approximately 60% for this reason.

Furthermore, from the viewpoint of long-term change over time, the hydrogen permeation current increased with the passage of the test period, but after a certain period, it showed the behavior of a maximum value and then decreased slightly and stagnated. Under the condition of 10 g/m² salt deposition, the maximum value of the hydrogen permeation current during the test period increased with the test temperature, from 40 nA/cm² at 10°C to 270 nA/cm² at 30°C and 3 121 nA/cm² at 50°C. At the test temperature of 30°C, the hydrogen permeation current increased as the amount of salt deposition became larger, being 180 nA/cm² with salt deposition of 0.3 g/m² and 202 nA/cm² with salt deposition of 3 g/m². In the period from the start of the test until the hydrogen permeation showed its maximum value, it is considered that hydrogen absorption increases as a result of the above-mentioned enrichment of Cl⁻ ions. However, in the later part of the test, enrichment of Cl⁻ ions is suppressed by the formation of stable rust with the progress of corrosion, and the hydrogen absorption amount decreases and stagnates.

The hydrogen permeation current of cold-rolled steel sheets under an atmospheric corrosion environment shows a maximum value corresponding to the conditions during the test period, and then shows a tendency to stagnate thereafter, as can be seen in Fig. 15. This suggests that it may be possible to judge whether delayed fracture of a cold-rolled steel sheet under an atmospheric corrosion environment will occur or not based on whether the maximum hydrogen uptake exceeds the limit amount of absorbed hydrogen at which delayed fracture will occur, corresponding to the stress state of the material.

Figure 16 shows the maximum hydrogen uptake ratio dependence of the maximum unfractured stress (delayed fracture limit stress) obtained from a delayed fracture evaluation of a 1 470 MPa grade cold-rolled steel sheet in the HeTsACE test at different test temperatures and the results of measurements of the amount of hydrogen absorption. Here, the maximum hydrogen uptake ratio is calculated by the ratio of the maximum value of the absorbed hydrogen amount under each environmental condition and the maximum value of the absorbed hydrogen amount for a test temperature of 30°C and chloride deposition of 3 g/m². Assuming the use of this material in an actual environ-

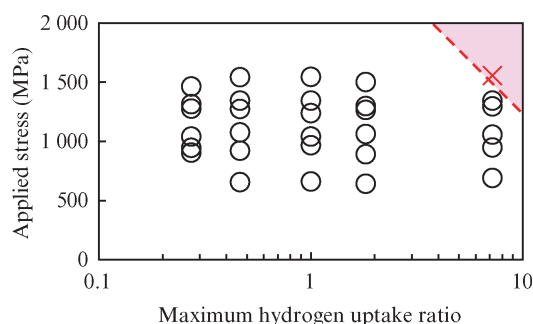


Fig. 16 Relationship between maximum hydrogen uptake ratio and delayed fracture of 1470 MPa grade cold rolled steel sheet

ment, the margin until the material use limit can be evaluated quantitatively by comparing the maximum values of the loading stress and hydrogen absorption amount with this diagram.

4. Conclusion

- In order to apply ultra-high strength steel sheets to automotive components, a delayed fracture evaluation technique that considers the automotive component production process and the actual use environment was developed.
- A delayed fracture evaluation was conducted, considering the effects of the shearing conditions and the forming strain on the sheared edge and the effect of heat in paint baking treatment.
- In model components, it was possible to predict the risk of delayed fracture by using a combination of the above-mentioned delayed fracture evaluation and

forming CAE.

- To simulate corrosion of vehicles in actual use environments, a cyclic test for evaluation of delayed fracture (HeTsACETM: Hydrogen embrittlement Test to simulate Actual Corrosion Environment) was proposed.
- Utilizing these evaluation techniques, it is expected to be possible to predict and reduce the risk of delayed fracture by more realistically evaluating the delayed fracture risk when applying ultra-high strength steel sheets to an actual vehicle.

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