

Delayed Fracture Mechanism of 1 700 MPa-Class Quenched and Tempered Bolt under Atmospheric Corrosion Environment

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Overcoming delayed fracture under atmospheric corrosion environment is a critical challenge for the practical application of ultra-high-strength bolts. This study investigated the delayed fracture behavior of 1 700 MPa-class quenched and tempered bolts fabricated from a 0.4%C-2%Si-1%Cr-1%Mo steel (mass%) by means of outdoor exposure tests, accelerated tests, and thermal desorption spectrometry. In a 10-year outdoor exposure test, the delayed fracture behavior of the ultra-high-strength bolts was categorized into two types: rupture at the thread runout and the first thread root in the nut, and fracture at the thread root in the nut and the under-head fillet. Outdoor exposure tests using notched bolts could reproduce the delayed fracture at the thread runout. By measuring the strain change for the shank of notched bolt during the outdoor exposure test, we successfully observed the delayed fracture process: the initiation of intergranular fracture along the boundaries of prior-austenite grains, followed by unstable fracture, and culminating in the formation of a shear lip. The delayed fracture of the bolts was attributed to hydrogen embrittlement. Slow-strain-rate testing for hydrogen-pre-charged notched specimens showed a linear relationship between the fraction of intergranular fracture (IGF) area and the peak value of the maximum principal stress σ_{max}^* at the notch root. The delayed fracture mechanism of the ultra-high-strength bolts was discussed based on the σ_{max}^* and the IGF area fraction at the fracture locations.

KEY WORDS: low-alloy steel; ultra-high-strength bolt; outdoor exposure test; delayed fracture; hydrogen embrittlement.

1. Introduction

Increasing the strength of bolts reduces the number and size of bolts required, leading to more compact and lightweight joints. This provides advantages such as labor-saving construction and enhanced performance of structures. For example, in the automotive field, a 1 600 MPa-class quenched and tempered bolt for plastic region tightening was put into practical use in 2018, contributing to the realization of a variable compression ratio engine that achieves both fuel efficiency and power output.¹⁾

The primary bottleneck restricting the strengthening of bolts is delayed fracture. In 1964, when an industrial standard was established for high-strength hexagon bolts for friction grip joints (JIS B 1186), high-strength bolts were classified up to a strength level of F13T (tensile strength $\sigma_B \geq 1\,300$ MPa).^{2,3)} However, because of the delayed fracture, F13T was excluded from the industrial

standard in 1967, and the production of F11T bolts ($\sigma_B = 1\,100$ – $1\,300$ MPa) ceased in 1981. Until the practical application of a 1 400 MPa-class super-high-strength bolt⁴⁾ in 1996, the strength level of high-strength bolts was limited to F10T ($\sigma_B = 1\,000$ – $1\,200$ MPa). In the inspection and repair of bridges using F11T bolts, delayed fracture of the bolts has become a significant issue. It is believed that delayed fracture of the bolts in an atmospheric environment is caused by hydrogen embrittlement. Hydrogen generated by a corrosion reaction penetrates the interior of the bolt, diffuses, and accumulates at stress concentration regions such as the thread root, making the bolt brittle.

To accelerate the development of high-strength bolts, it is necessary to establish a standard evaluation method to ensure delayed fracture resistance under actual usage conditions. Various evaluation methods for delayed fracture properties such as the JIS draft (the method of delayed fracture testing of high-strength steels)⁵⁾ have been proposed.^{6–8)} In 2010, the Japan Society of Steel Construction (JSSC) proposed the H_C^*/H_E^* method. As parameters, this

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method uses local critical hydrogen concentration for hydrogen embrittlement H_C^* and local hydrogen concentration from the service environment H_E^* at the peak value of the maximum principal stress in a stress concentration region.⁹⁾ In 2014, JSSC presented a guideline for evaluating delayed fractures, which stochastically estimates the values of H_C^* and H_E^* ¹⁰⁾ and it was determined that a delayed fracture can occur under the condition of $H_C^* \leq H_E^*$.¹¹⁾ The H_C^*/H_E^* method is currently considered the most rational and optimal approach for evaluating the delayed fracture of high-strength bolts. However, this evaluation method must be scrutinized through verification tests using various bolts to enhance its reliability.

Kimura *et al.*^{12–14)} investigated the delayed fracture behavior of prototype ultra-high-strength bolts through exposure tests as part of research and development aimed at realizing 2 000 MPa-class ultra-high-strength bolts. The exposure tests were conducted at the Miyakojima test site of the Japan Weathering Test Center (JWTC). In 1 700 MPa-class quenched and tempered bolts (nominal designation of thread: M12) fabricated from a 0.4%C-2%Si-1%Cr-1%Mo steel (mass%), a delayed fracture was observed during an exposure test at an applied stress σ_a of 1 050 MPa where $H_C^* < H_E^*$.^{12,13)} Furthermore, a 1 700 MPa-class quenched and tempered bolt with an improved shape (nominal designation of thread: M22) was fabricated from a 0.5%C-2%Si-1%Cr-1%Mo steel.¹⁴⁾ Its thread shape was confirmed to be effective for improving delayed fracture resistance using the H_C^*/H_E^* method.

As described above, progress has been made in understanding the delayed fracture of high-strength bolts in atmospheric corrosion environments and in developing evaluation methods. However, many aspects of the delayed fracture, including its occurrence mechanism, remain unclear. One reason for this is that the delayed fracture of bolts occurs suddenly after a period ranging from several months to several years.^{2,3,9,12–16)} This makes it difficult to directly observe the process from crack initiation to final rupture. For the 0.4%C-2%Si-1%Cr-1%Mo steel,^{12,13,17)} this study first investigated the delayed fracture behavior of 1 700 MPa-class quenched and tempered bolts during the exposure test conducted over a 10-year period at the JWTC Miyakojima test site.^{12,13)} Next, to reproduce the delayed fracture of the ultra-high-strength bolts, exposure tests were performed at the National Institute for Materials Science (NIMS) in Tsukuba, using bolts with a notch machined into the shank (notched bolts). The amount of hydrogen intrusion and the fracture morphology were studied in the retrieved bolts, and the strain changes in the shanks of the notched bolts were measured to clarify the delayed fracture process. Considering the results of the accelerated tests for hydrogen embrittlement, the delayed fracture mechanism was comprehensively discussed for the 1 700 MPa-class quenched and tempered bolts.

2. Experimental

2.1. Materials and Bolts

Table 1 lists the chemical compositions of 0.4%C-2%Si-1%Cr-1%Mo steels. As previously reported,¹²⁾ M12 hexagon bolts with a nominal length of 60 mm were fabricated

Table 1. Chemical compositions of 0.4%C-2%Si-1%Cr-1%Mo steels (mass%).

Lot.	C	Si	Cr	Mn	Mo	P	S	Al	T-N	O
1	0.39	1.98	1.00	0.20	0.99	<0.001	<0.001	0.020	0.0012	0.0009
2	0.43	2.04	1.05	0.21	1.02	0.002	0.001	0.019	0.001	0.0008

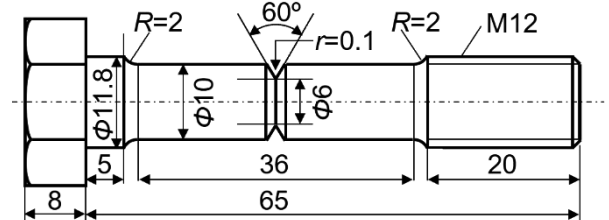


Fig. 1. Shape and dimensions (mm) of M12 notched bolt.

Table 2. Tensile properties of M12 bolts (F1, 2) and QT sample (M2), that were quenched and tempered at 500°C: 0.2% proof stress $\sigma_{0.2}$, tensile strength σ_B , total elongation (EL), and reduction in area (RA).

Lot.	$\sigma_{0.2}$ (MPa)	σ_B (MPa)	EL (%)	RA (%)
F1	1 550	1 679	—	—
F2	1 526	1 699	—	—
M2	1 506	1 764	9.4	30

through the following process. Warm tempformed (TF) bars were formed into bolts through warm forging, then austenitized at 950°C for 0.5 h, oil quenched, and tempered at 500°C for 1.5 h. Of the 45 bolts, 15 were coated with geomet, which offers superior corrosion resistance, and 15 bolts were electrogalvanized, to make clear the relationship between the corrosion and the delayed fracture of the bolts. Nuts (height: 14 mm, hardness: 480 HV) were fabricated from a 0.5%C-2%Si-1%Cr-1%Mo steel and coated with geomet. **Figure 1** shows the shape and dimensions of an M12 notched bolt with a stress concentration factor of 4.9. The notched bolts were fabricated by warm forging the TF bars of Lot 2 steel to form a hexagon head, followed by quenching and tempering. Tempering time of the notched bolts was 1 h, and the shank and the threaded portion were machined. In addition, the quenched and tempered (QT) samples were prepared by normalizing the TF bars at 990°C for 10 min, followed by quenching and tempering under the same conditions as those applied to the notched bolts. **Table 2** shows the tensile properties of the bolts and QT samples. The tensile properties of the bolts¹²⁾ were evaluated by wedge tensile testing (wedge angle: 4°). The 0.2% proof stress $\sigma_{0.2}$ and σ_B were calculated by dividing the respective loads by the tensile stress area at the threaded portion (84.3 mm²).

2.2. Accelerated Tests for Hydrogen Embrittlement and Hydrogen Analysis

Hydrogen embrittlement susceptibility was evaluated by the conventional-strain-rate test (CSRT)⁷⁾ with a crosshead speed of 1 mm min⁻¹ and the slow-strain-rate test (SSRT)^{12,18)} with a crosshead speed of 0.005 mm min⁻¹.

thickness of 44 mm (SCM440, hardness: 300 HV) using the turn-of-the-nut method. A dry lubricant was sprayed onto the threaded portions, and the bolts were pre-tightened to a torque of 20 Nm before marking them. The marking portion was set to 0°, and the bolts were finally tightened by rotating the nuts to the target rotation angle of 40°. Equations (5) and (6) represent the relationship between the axial force F and the strain ε (Fig. S1(a) (Supporting Information)) and the relationship between ε and the nut rotation angle θ (Fig. S1(b)), respectively.

$$F(\text{kN}) = 3.12 + 0.0144 \varepsilon (\mu\text{e}) \dots\dots\dots (5)$$

$$\varepsilon (\mu\text{e}) = 500 + 46.5 \theta (^\circ) \dots\dots\dots (6)$$

Strain gauges (2-gauge 3-wire wiring method) were inserted through holes with a diameter of 1.1 mm that were drilled into the head and attached to the shank. The strain gauges were located 15 mm from the under-head. After attaching the strain gauges, the holes in the head were filled with a filler. In the 1st period, twenty sets of bolts were tightened, and the average value of σ_a was 1 290 MPa (standard deviation: 70 MPa). In the 2nd period, the average value of σ_a was 1 320 MPa (standard deviation: 40 MPa) for twelve sets of bolts. Note that for six of the notched bolts in the 2nd period, the notched shanks were spray-painted with an organic zinc-rich coating and wrapped with waterproof tape to prevent corrosion. Additionally, an exposure test was carried out for four sets of notched bolts attached with strain gauges (average value of σ_a : 1 440 MPa, standard deviation: 60 MPa).

3. Experimental Results

3.1. Delayed Fracture Behavior

Figure 3 shows the appearance of untreated bolts that

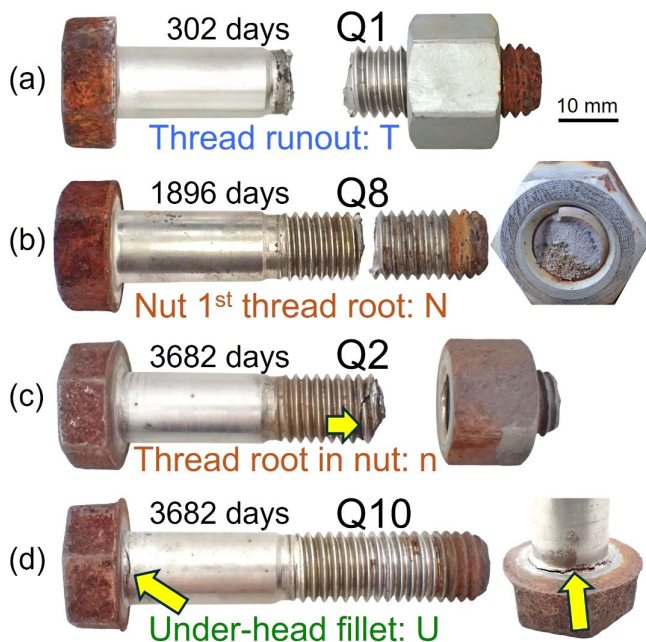


Fig. 3. Appearance of non-surface-treated M12 bolts (F1_Q) failed at (a) thread runout, (b) nut 1st thread root, (c) thread root in nut, and (d) under-head fillet during outdoor exposure test in Miyakojima, Okinawa. Arrows in (c, d) indicate cracks. (Online version in color.)

failed during the exposure test (see Fig. S2 for the other bolts). The fracture locations of these bolts can be broadly classified into the following: thread runout, nut 1st thread root, thread root in the nut, and under-head fillet. Although partial rust is visible on the shank and the free-threaded portion, which were exposed in a semi-closed environment, the degree of corrosion remains minimal even after 3 682 days, compared to the head and the tip of the threaded portion. There are multiple cracks in the bolts that failed at the thread root in nut (Fig. 3(c)). **Figures 4 and 5** summarize the occurrence of delayed fracture of the bolts. In Fig. 5, “NA” indicates the bolts that failed within the 28-day period from the bolt tightening to the start of the exposure test; twelve electrogalvanized bolts (E) and three untreated bolts (Q) failed at the under-head fillet (Fig. S2(c)). It was inferred that this premature failure of bolts was caused by hydrogen embrittlement resulting from hydrogen intrusion during the bolt manufacturing process.¹²⁾ Ninety-seven days after the start of the exposure test, the first rupture was observed at the under-head fillet of an untreated bolt; however, since its shank was also completely corroded (Fig. S2(a)), it is highly likely that this bolt failed prematurely. Among the untreated bolts, three ruptured after 302 to 377 days at the thread runout, and one ruptured after 1 896 days at the nut 1st thread root. Seven bolts with geomet coating (G) ruptured at the nut 1st thread root after 555 to 3 053 days. When the bolts were removed after 3 685 days, it was found that three of the untreated bolts had failed at the thread root in nut, and one had failed at the under-head fillet (Fig. S2(a)). For the bolts with geomet coating, three had failed at the nut 1st thread root, and five had failed at the thread root in nut (Fig. S2(b)). Furthermore, three electrogalvanized bolts had failed at the nut 1st thread root (Fig. S2(c)). Therefore, in the 10-year exposure test, the rupture frequency of the bolts was high at the nut 1st thread root and the thread runout. This result is largely consistent with the results of previous exposure tests conducted on 1 300–1 500 MPa-class high-strength bolts^{2,3,9)} and the 1 700 MPa-class ultra-high-strength bolts fabricated from a 0.5%C-2%Si-1%Cr-1%Mo steel.¹⁴⁾

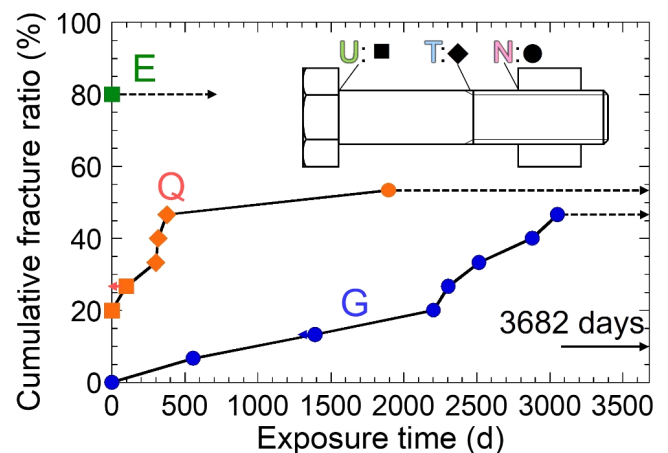


Fig. 4. Cumulative fracture ratio as a function of exposure time for the bolts (F1) with geomet coating (G), electrogalvanizing (E), and non-surface-treatment (Q). U, T, and N denote the fracture locations in Fig. 3. The bolts with unknown fracture dates are indicated by left-pointing arrows. (Online version in color.)

G1 ←1391 N	G2 ←3682 n	G3 ←3682 N	G4 ←3682 n	G5 555 N
G6	G7 2304 N	G8 ←3682 N	G9 ←3682 n	G10 2204 N
G11 ←3682 n	G12 3053 N	G13 ←3682 n	G14 2878 N	G15 2515 N
E1 ←3682 N	E2 NA U	E3 NA U	E4 NA U	E5 NA U
E6 NA U	E7 NA U	E8 NA U	E9 NA U	E10 NA U

E11 NA U	E12 NA U	E13 ←3682 N	E14 ←3682 N	E15 NA U
Q1 302 T	Q2 ←3682 n	Q3	Q4	Q5 NA U
Q6 ←3682 n	Q7	Q8 1896 N	Q9 NA U	Q10 ←3682 U
Q11 ←3682 n	Q12 317 T	Q13 NA U	Q14 ←97 U	Q15 377 T

U: Under-head fillet, T: Thread runout,
 N: Nut 1st thread root, n: Thread root in nut

Fig. 5. Summary of delayed fracture of the bolts (F1) with the exposure test in Miyakojima, Okinawa. The bolts with geomet coating (G1-15), electrogalvanizing (E1-15), and non-surface-treatment (Q1-15) were fastened in two steel plates, and exposed from May 21, 2013, to Jun. 20, 2023 (3 682 days). The bolts that failed before the exposure test are denoted by NA. The bolts with unknown fracture dates are indicated by left-pointing arrows. (Online version in color.)

Figure 6 shows the appearance of the notched bolts after exposure testing. Although it can be seen that the shank of the notched bolts is partially corroded, the corrosion is minor. Compared to the bolts shown in Fig. 3, the degree of corrosion on the head and the tip of the threaded portion is less severe in the notched bolts because of the shorter exposure time and milder corrosion environment. Regarding this, it is well recognized that the primary environmental factors governing the corrosion rate are the temperature, relative humidity, and airborne salt deposition. The respective average values for these factors from 2002 to 2012²¹⁾ were 24.0°C, 78%, and 48.7 mg NaCl m⁻² d⁻¹ at the JWTC Miyakojima test site, and 15.0°C, 73%, and 2.4 mg NaCl m⁻² d⁻¹ at the NIMS outdoor corrosion test site. Furthermore, as shown in Fig. 6(b), there were unfractured bolts with water droplets adhering to their shank immediately after removal; four such bolts were identified during the 1st period, and five during the 2nd period. Note that the bolt shown in Fig. 6(b) was retrieved six days after a rainfall. It has also been reported, in the case of F11T and F13T bolts used in railway bridges, that the shanks were often found to be wet when the bolts were extracted from

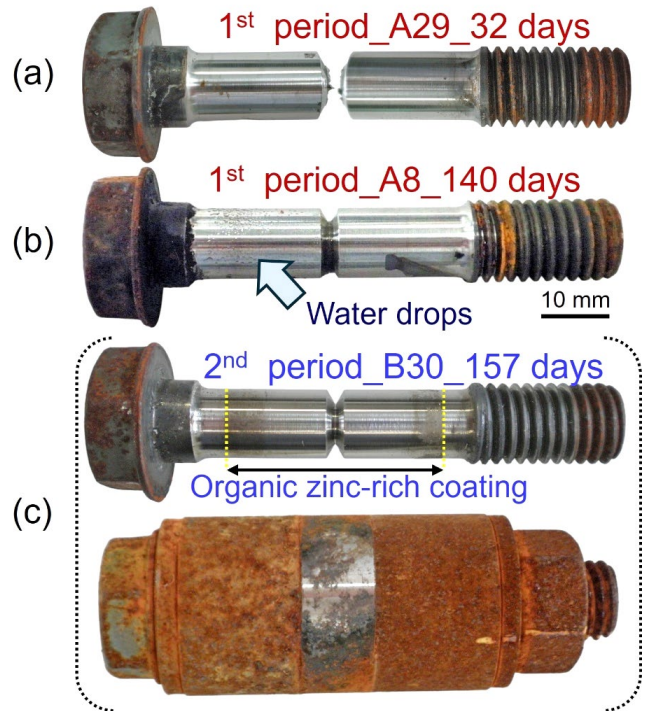


Fig. 6. Appearance of the notched bolts (F2) with outdoor exposure test in Tsukuba, Ibaraki: (a, b) 1st period from Mar. 23, 2022, to Dec. 19 (271 days), and (c) 2nd period from Mar. 27, 2023, to Aug. 31 (157 days). In 2nd period, six bolts were coated with organic zinc-rich paint to suppress hydrogen intrusion from the surfaces of the shank with a notch. (Online version in color.)

the bolted joints where delayed fracture had occurred.³⁾ **Figure 7** summarizes the occurrence of delayed fracture of the notched bolts. For reference, daily precipitation data²²⁾ measured at the Meteorological Research Institute, located approximately 2 km from NIMS, is also shown. Of the eleven bolts retrieved within 4–24 h of rupture during the 1st and 2nd period exposure tests, ten had been exposed to rain within three days prior to the rupture, and the daily precipitation ranged from 2 to 46 mm. These suggest the influence of rainwater on the occurrence of delayed fracture of the bolts. In the 1st period, delayed fracture began on the 15th day, and the cumulative fracture number of the bolts increased abruptly to nine over the following 71 days (Fig. 7(a)). When the bolts were removed after the 271-day exposure test, two bolts had failed at the notched portion and one had failed at the under-head fillet. The total number of unfractured bolts was six: four of these were retrieved on days 25, 27, 140, and 203, and two were retrieved after the 271-day exposure test was completed. Therefore, the cumulative fracture ratio of the bolts in the 271-day exposure test is 70–90%. The period when the delayed fracture of the notched bolts frequently occurred was approximately 300 days earlier than that at the thread runout of untreated bolts shown in Fig. 4. The cumulative fracture ratio in that period was also higher for the notched bolts. The reasons for this are considered to be: 1) the H_C^*/H_E^* ratio for the notched bolts was set lower, and 2) the axial force for the notched bolts was approximately 40% of that for the bolts shown in Fig. 4 and consequently, water was likely to penetrate more easily through the gaps in the bolted specimens using the notched bolts, although the corrosion environment was

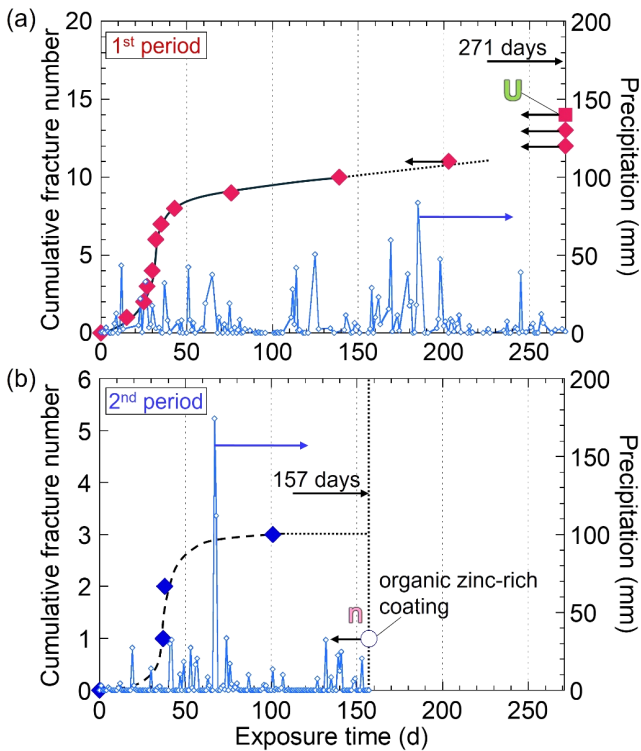


Fig. 7. Cumulative fracture number of the notched bolts (F2) as a function of exposure time. Daily precipitation in Tsukuba²²⁾ is shown for reference. U and n denote the fracture locations in Fig. 3. The bolts with unknown fracture dates are indicated by left-pointing arrows. (Online version in color.)

milder for the notched bolts. The stress concentration factor of 4.9 simulates the stress state in the thread runout. Hence, in the exposure test using the notched bolts, it was possible to reproduce the delayed fracture that occurred at the thread runout of the untreated bolts in a way that accelerated its occurrence (Fig. 7(b)). This result could be reproduced in the 2nd period exposure test. Furthermore, the rust prevention treatment using organic zinc-rich paint could suppress the occurrence of a delayed fracture at the notched portion. Note that one of the six bolts with the rust prevention treatment (notched bolt B20) failed at the thread root in nut.

3.2. Hydrogen Intrusion Behavior

Figure 8 shows the representative hydrogen desorption curves. Note that unfractured bolts were ruptured by the CSRT to confirm the damage and then were cut at the positions shown in Fig. 9. The fracture location of the bolts subjected to the CSRT was at the nut 1st thread root. The rust on the bolts was removed by sandblasting before performing the thermal desorption spectrometry. Hydrogen desorption peaks appear at around 140–200°C and 400°C in the bolts subjected to exposure testing. Similar hydrogen desorption curves were observed in the F11T bolts (nominal designation of thread: M22) that had been tightened in plastic regions and exposed for nine years.²³⁾ Since the hydrogen desorption peak at around 400°C was also detected at the tip of the threaded portion (position 7) of the notched bolts prior to exposure testing, it is believed to correspond to the release of non-diffusible hydrogen. On the other hand, hydrogen release at around 140–200°C was also observed in the samples with cathodic hydrogen charging or immer-

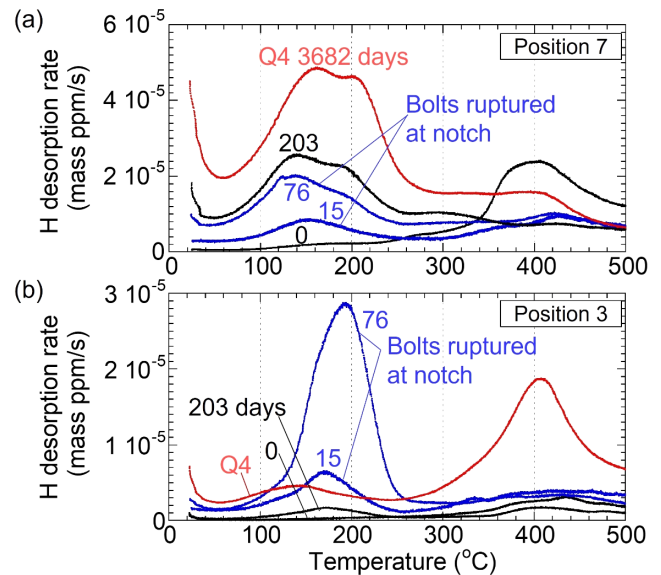


Fig. 8. Variation in hydrogen desorption rate during thermal desorption spectrometry analysis with a heating rate of $100^{\circ}\text{C h}^{-1}$ for the notched bolts (F2) exposed in 1st period: (a) position 7 and (b) position 3 in Fig. 9. Data for the non-surface-treated bolt (F1_Q4) in Fig. 5 are also shown. (Online version in color.)

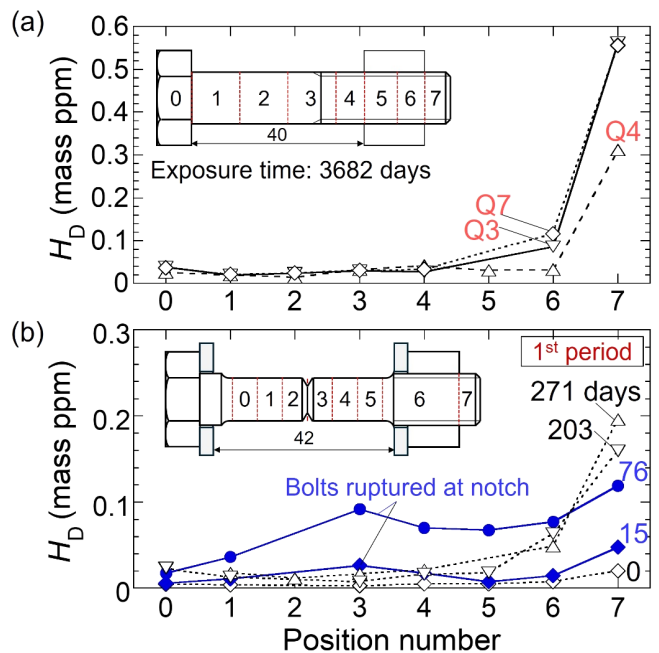


Fig. 9. Relation between diffusible hydrogen content H_D and sampling position: (a) the non-surface-treated bolts (F1_Q) in Fig. 5, and (b) the notched bolts (F2) in 1st period. (Online version in color.)

sion testing, and it corresponds to the release of diffusible hydrogen, which can cause hydrogen embrittlement. It has been reported that for the F11T bolts tightened in a plastic region, H_D was highest in the free-threaded portion (corresponding to position 4 in Fig. 9(a)), which had been plastically deformed across its entire cross section.²³⁾ As shown in Fig. 9(a), even in the present bolts tightened in an elastic region, H_D tended to be slightly higher in position 3 including the thread runout and position 4 for the free-threaded portion ($H_D \approx 0.03$ mass ppm), compared to positions 1 and

2 in the shank ($H_D \approx 0.02$ mass ppm). However, the highest H_D value for these unfractured bolts (Q3, 4, 7) is observed at the tip of the threaded portion (position 7) that had been exposed to an outdoor environment and is much higher than that (≈ 0.03 mass ppm) at the head (position 0) that had also been exposed to an outdoor environment. Of the seven untreated bolts retrieved after the 3 682-day exposure test, the average H_D value at the tips of the threaded portions for the six bolts was 0.49 mass ppm (minimum: 0.31 mass ppm, maximum: 0.56 mass ppm). In addition, for the remaining bolt Q2 (Fig. 3(c)), H_D was measured to be 0.40 mass ppm at the tip of the threaded portion. This portion was cut off from the bolt, sandblasted to remove the rust, and then was held for 72 h at room temperature. In contrast, when a plate 15 mm long, 5 mm wide, and 1 mm thick was cathodically charged with hydrogen at a current density of 10 A m^{-2} for 48 h in 0.1 N NaOH aqueous solution, H_D increased to 1.0 mass ppm. When the plate was subsequently held at room temperature, H_D decreased to 0.27 mass ppm after 24 h, and then to 0 mass ppm after 72 h.¹⁷⁾ Thus, these results indicate that the hydrogen absorbed into the bolts through the lengthy exposure test is in a state where it is difficult to release at room temperature. On Miyakojima Island, the airborne salt deposition is higher than that on the main island of Okinawa, and the corrosion environment is more severe.²⁴⁾ Hence, it is reasonable that H_D at the tip of the threaded portion of the untreated bolts tended to be higher than H_E ($= 0.36$ mass ppm) measured by the immersion test simulating the corrosion environment on the main island of Okinawa.¹²⁾ We believe that during the exposure test, hydrogen repeatedly intruded from the bolt surface with the highest hydrogen concentration, diffused into the interior of the bolt, and then was absorbed. In addition, during the 3-day period between the retrieval and removal of the bolts, the hydrogen concentration on the surface may have decreased, resulting in the release of part of the hydrogen. Therefore, H_E in the bolts for the exposure test is estimated to be 0.31–0.56 mass ppm or higher. A similar distribution of the absorbed hydrogen was observed in the unfractured notched bolts that were retrieved on days 203 and 271 (Fig. 9(b)). Regarding the notched bolts that failed during the exposure test, although high H_D values were observed at the notched portion of some of the bolts (such as those retrieved on days 15 and 76), the maximum values were still observed at the tip of the threaded portion. **Figure 10** shows the variations in H_D at the notched portion (position 3) and the tip of the threaded portion (position 7) as a function of the exposure time. Regardless of whether or not a delayed fracture occurred, H_D at the tip of the threaded portion increased rapidly up to 100 days, and then levelled off at 0.1–0.4 mass ppm. Considering that the saturation time of H_D was 15 days in the immersion test using round rod specimens with a diameter of 7 mm and a length of 20 mm,¹²⁾ the H_D distribution within the tip of the threaded portion can be considered nearly uniform in the exposure test for 100 days or longer. On the other hand, at the notched portion of the bolts retrieved within 4 to 24 h after the delayed fracture, H_D ranged from 0.02 to 0.09 mass ppm; although this was lower than H_C in Fig. 10, it was higher than H_D for the unfractured bolts (≤ 0.02 mass ppm). Li *et al.*²⁵⁾ exposed the notched specimens machined from 1 700 MPa-class quenched and

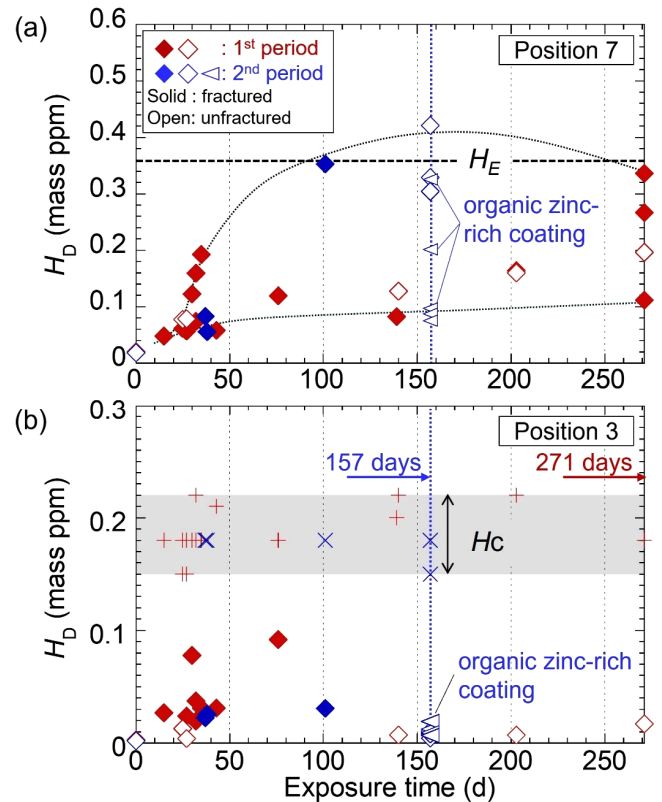


Fig. 10. Change in H_D as a function of exposure time for the notched bolt (F2): (a) position 7, and (b) position 3 in Fig. 9. The H_E value in Fig. 2(b) is presented in (a). The H_C values in (b) were calculated from the power law relationship between σ_{NB} and H_C in Fig. 2(a). (Online version in color.)

tempered 0.6%C-2%Si-1%Cr-1%Mo steel to an outdoor environment and then performed a SSRT, demonstrating that σ_{NB} decreased as H_D increased. As mentioned earlier, given that the corrosion in the notched portion was minor and pitting corrosion was not observed, it is reasonable to conclude that the delayed fracture occurred through hydrogen intrusion. In the 2nd period, the results of the exposure test for the notched bolts with rust-proofed shanks demonstrated that hydrogen, which contributed to the delayed fracture, penetrated from the surface of the notched portion rather than the tips of the threaded portion. Furthermore, the delayed fracture under conditions where H_E was lower than H_C has also been confirmed in previous exposure tests of M22 high-strength bolts.⁹⁾ In this regard, Omura²⁶⁾ pointed out that for M22 bolts exposed to an outdoor environment, the hydrogen concentration on the surface is higher than the average hydrogen concentration. Therefore, the delayed fracture must be discussed using the local hydrogen concentration in the stress concentration region.

3.3. Fracture Morphology

Figure 11 shows macroscopic fracture surfaces for the bolts that failed during the exposure tests and the notched specimens that ruptured during the SSRT (see Fig. S3 for the macroscopic fracture surfaces of the other bolts and notched specimens). The distinct steps observed in Figs. 11(e), 11(g) indicate that the cracks propagated from multiple origins. When the crack area containing the origin is defined as the “fracture origin zone”, intergranular

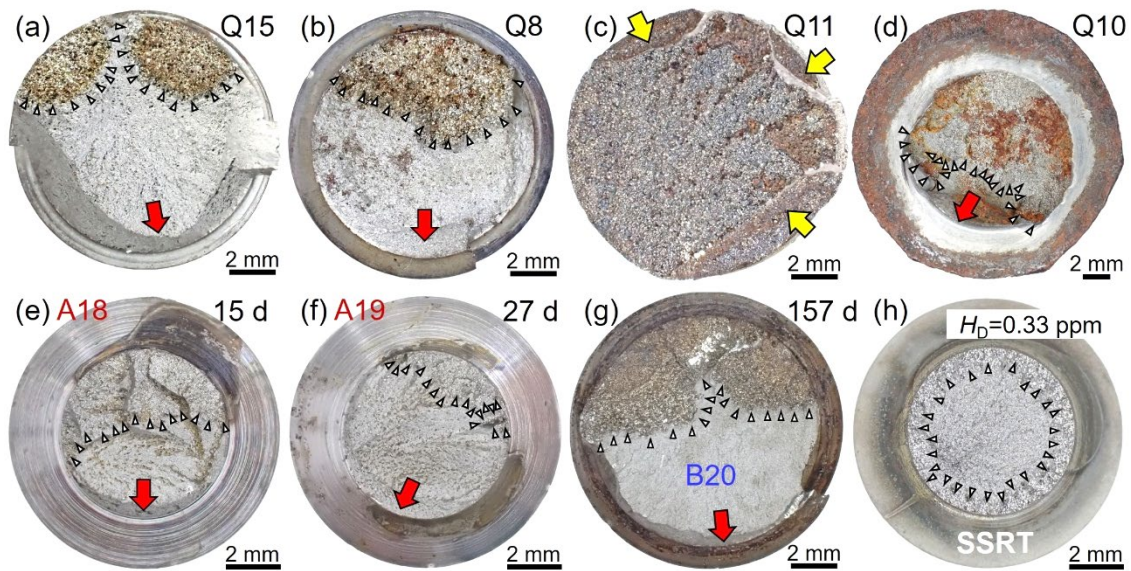


Fig. 11. Macroscopic fracture surfaces for the non-surface-treated bolts (F1_Q) failed at (a) thread runout, (b) nut 1st thread root, (c) thread root in nut and (d) under-head fillet, and for the notched bolts (F2) failed at (e, f) notch root and (g) thread root in nut. (h) shows a macroscopic fracture surface of the notched specimen with SSRT. Red arrows and triangles indicate shear lips and intergranular fracture (IGF) areas, respectively. The fraction of IGF area in Q11 bolt (c) is almost 100%, and yellow arrows indicate cracks. (Online version in color.)

cracks along the boundaries of prior-austenite grains were observed in the fracture origin zones, as shown in Fig. 12. Intergranular cracking is commonly observed in hydrogen embrittlement of high-strength steels^{6,8,12,17,19,20,27–31} and the delayed fracture of bolts.^{3,16,32} In the SSRTs conducted under the condition where the H_C^* is nearly identical to H_C^* of the bolts in Fig. 2(b), the fracture origin zones tended to uniformly develop at multiple locations around the notch root (Figs. 11(h), S3(c)). In contrast, for the untreated bolts (Figs. 11(a), 11(b)) and the notched bolts (Figs. 11(e), 11(f)), the formation of the fracture origin zone was localized to one, two, or three locations around the thread root and the notch root. A similar macroscopic fracture surface has been reported in the delayed fracture of high-strength bolts.³ This indicates that the hydrogen intrusion was localized. Furthermore, dimple patterns can be seen on the final fracture surface of the untreated bolt (Fig. 12(b)), and a shear lip is formed along its outer periphery. Such delayed fracture behavior was also observed in the 1 700 MPa-class ultra-high-strength bolts fabricated from the 0.5%C-2%Si-1%Cr-1%Mo steel.¹⁴ In the final fracture surfaces of the notched specimens ruptured during the SSRT, quasi-cleavage fracture surfaces were occasionally observed (Fig. 12(f)). Quasi-cleavage patterns were also observed in the notched specimen subjected to the SSRT without hydrogen charging. Hence, the fracture morphology of the notched bolts was macroscopically identical to that of the untreated bolts. However, the fracture mode on the final fracture surface of the notched bolts (Fig. 12(d)) was similar to that of the notched specimens with SSRT (Fig. 12(f)). From these findings, it is inferred that unstable fracture occurred in the bolts once the area of the fracture origin zone reached a certain size. On the other hand, the bolts in which cracks were found after the exposure test (Figs. 11(c), 11(d), 11(g)) were ruptured by the CSRT. In bolt Q10 (Fig. 11(d)) that failed at the under-head fillet, the fracture origin zone was wider than the final fracture zone. In bolt Q11 (Fig. 11(c))

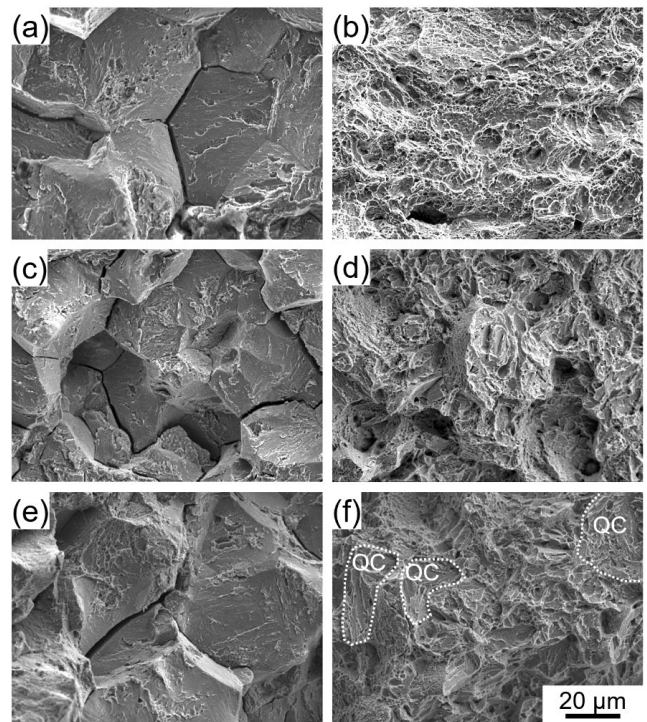


Fig. 12. Fracture surfaces of (a, b) Q8 bolt in Figs. 11(b), (c, d) the notched bolt in Fig. 11(f), and (e, f) the notched specimen with SSRT in Fig. 11(h). (a, c, e) show intergranular fracture cracks located approximately 0.2 mm from the thread root or notch roots. (b, d, f) were taken from the central sections. QC in (f) denotes quasi-cleavage fracture.

that failed at the thread root in nut, cracks were found at several thread roots in addition to the main crack as shown for bolt Q2 in Fig. 3(c), and the intergranular fracture surface was observed across nearly the entire macroscopic fracture surface. It is possible to clearly distinguish between the intergranular fracture area where corrosion had progressed

and the ductile fracture area in notched bolt B20 (Fig. 11(g)) that failed at the thread root in nut. Its fracture strength at the threaded portion as measured by the CSRT was also high at 601 MPa, and thus the crack propagation was determined to be arrested. **Figure 13** shows the relationship between the fraction of the intergranular fracture area and the exposure time. The fraction of the intergranular fracture area was measured from the macroscopic fracture surface of the bolt. Here, the intergranular fracture area was defined as an area containing intergranular cracks. In Fig. 13(b), the notched bolts ($\blacklozenge$) in which the intergranular fracture area was located within the minimum cross section at the notch root are distinguished from the notched bolts ($\diamond$) in which the intergranular fracture area extended beyond the minimum cross section (Fig. S3(b)). The intergranular fracture area corresponds to the fracture origin zone in the bolts that ruptured at the nut 1st thread root or thread runout, and the fraction of the intergranular fracture area was 20–40% regardless of the surface treatment or the exposure time. On the other hand, for the untreated bolts in which the fracture was confirmed after the 3 682-day exposure test, the fraction of the intergranular fracture area was approximately 70% at the under-head fillet (U) and 100% at the thread roots in nut (n). Even among the bolts coated with geomet, there was one (G11) in which the fraction of the intergranular fracture area at the thread root in nut (n) was nearly 100% (Fig. S3(a)). The fraction of the intergranular fracture area in all the notched bolts ranged from 20% to 80%, and it was 20% to 40% for the notched bolts ($\blacklozenge$) in which the intergranular fracture area was located within the minimum cross section at the notch root as shown in Figs. 11(e), 11(f). Hence, the fraction of the intergranular fracture area was not influenced by the exposure time. Furthermore, the fraction

of the non-propagating crack area was approximately 60% for notched bolt B20 that failed at the thread root in nut (n) shown in Fig. 11(g), and the fraction of the intergranular fracture area for the notched bolts that failed at the under-head fillet (U) was nearly 100%. Therefore, the fracture morphology of the bolts varies depending on the location where the delayed fracture occurs, rather than the surface treatment or the exposure time.

4. Discussion

4.1. Damage of Bolts Caused by Long-term Outdoor Exposure

The bolts subjected to the 10-year exposure test may have accumulated damage that cannot be detected by visual inspection. Homma *et al.*³²⁾ performed a detailed fractography analysis on the fracture surface of a 1 300 MPa-class high-strength bolt (SCM435) that ruptured 16 years after the start of an outdoor exposure test. They pointed out that a deteriorated zone, where microcracks propagated along the boundaries of prior-austenite grains (Frail zone), formed just below the thread root through the accumulation of localized plastic deformation damage from repeated hydrogen intrusion during long-term outdoor exposure. Here, we discuss the influence of the damage accumulation with the long-term outdoor exposure on the occurrence of delayed fracture in the thread runout and the nut 1st thread root⁹⁾ where the maximum equivalent strain at the stress concentration region is high, as is σ_{max}^* .

Figure 14 shows the CSRT results for the untreated bolts and the notched bolts that were subjected to the exposure tests. The distance between the bearing surfaces of the bolt and nut was set to 40 mm. This figure also shows the CSRT results for the cathodically hydrogen-pre-charged bolts and notched specimens (shape and dimensions are presented in Fig. 2(a)). The fracture location of the bolts was at the nut 1st thread root. When H_D was 1 mass ppm or higher, the hydrogen-pre-charged bolts ruptured at a tensile stress below $\sigma_{0.2}$ as shown in Table 2, and there was no difference between σ_B of the bolts and σ_{NB} of the notched specimens. This indicates that the σ_{max}^* values for the nut 1st thread

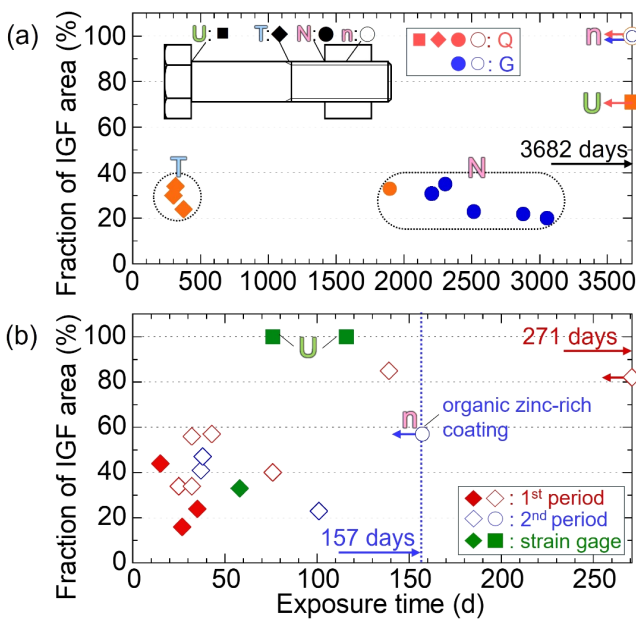


Fig. 13. Fraction of intergranular fracture (IGF) area as a function of exposure time: (a) the bolts (F1) with geomet coating (G), non-surface-treatment (Q), and (b) the notched bolts (F2). U, T, N, and n denote the fracture locations. The notched bolts in which the IGF area is beyond the minimum cross-section at the notch are denoted by $\diamond$ in (b). The bolts with unknown fracture dates are indicated by left-pointing arrows. (Online version in color.)

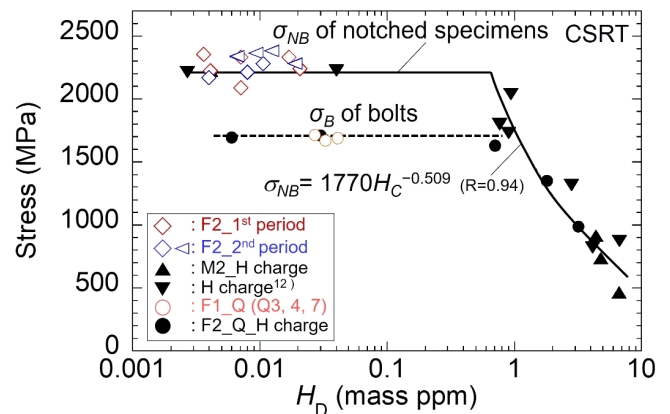


Fig. 14. Changes in σ_B of the non-surface-treated bolts (F1, 2_Q), σ_{NB} of the notched bolts (F2) in Fig. 10 and σ_{NB} of the notched specimens as a function of H_D with CSRT at a crosshead speed of 1 mm min⁻¹. Solid symbol indicates the bolts and the notched specimens charged with hydrogen before CSRT. (Online version in color.)

root and the thread runout are considered identical within the σ_a range of this exposure test. From the FEM analysis of the M22 bolts, σ_{max}^* in the thread runout portion was estimated to be slightly more than 90% of σ_{max}^* at the nut 1st thread root.⁹⁾

In the 10-year exposure test, H_D increased to 0.03 mass ppm (Fig. 9(a)) in the free-thread portion including the nut 1st thread root for the untreated bolts. This increase in H_D suggests that hydrogen was repeatedly intruded and released from the bolt surface. The reason for this is that hydrogen intrusion proceeds through fluctuations in the surface hydrogen concentration caused by the repeated cycles of drying and wetting.²⁶⁾ Regardless of the exposure time, the σ_B values for the untreated bolts were identical in the CSRT, and the rupture stress was 1–3% lower than the σ_B . Although H_D could not be measured in a geomet-treated bolt (G6), its σ_B was 1 710 MPa and its rupture stress was 1 700 MPa, which are identical to those of the untreated bolts. Furthermore, damaged areas such as the intergranular fracture area were not observed on the fracture surfaces of these bolts (Fig. S3(a)). Similarly, there were no differences in σ_{NB} and the fracture surface morphology among the notched bolts with H_D of 0.02 mass ppm or less (Fig. S3(b)). As described earlier, no significant difference was observed in the fracture morphology between the bolts that failed at the thread runout and the nut 1st thread root during the exposure test (Figs. 11, 13). Based on these findings, we conclude that although hydrogen repeatedly intruded during the 10-year outdoor exposure test, the resulting damage did not accumulate to a critical level that would affect the mechanical properties of these bolts.

In CSRT using a specimen with uniform hydrogen distribution, H_D corresponds to H_C^* .⁷⁾ H_C^* for the notched bolts can be estimated to be 1.8 mass ppm at σ_a of 1 300 MPa from the relationship between σ_{NB} and H_C shown in Fig. 14. For the SSRT results shown in Fig. 2(b), H_C^* for the notched bolts is 0.7 mass ppm. For the notched bolts shown in Fig. 10(a), assuming that the hydrogen distribution within the tip of the threaded portion is uniform and H_D (0.1–0.4 mass ppm) corresponds to H_E , Eq. (4) thus yields H_E^* of 0.5–2.2 mass ppm. These estimates indicate the possibility of $H_C^* \leq H_E^*$ in either CSRT or SSRT. Similarly, in the bolt exposure test performed at the JWTC Miyakojima test site, H_C^* is estimated to be 1.2 mass ppm for the SSRT and 2.8 mass ppm for the CSRT, whereas H_E^* is estimated to be 1.3–2.3 mass ppm for H_E of 0.31–0.56 mass ppm. This also indicates that the $H_C^* \leq H_E^*$ condition can be obtained through a corrosion reaction. Therefore, in this exposure test, we believe that the delayed fracture of the bolts at the thread runout or the nut 1st thread root occurred because $H_C^* \leq H_E^*$ arose through a localized corrosion reaction on the bolt surface.

4.2. Delayed Fracture Mechanism

In the outdoor exposure test, when the hydrogen intrusion pathways were restricted to the bolt head and nut sections, the delayed fracture behavior of the bolts could be broadly classified into two types. One involved a rupture at the thread runout or the nut 1st thread root, while the other involved a fracture at the under-head fillet or the thread root in nut.

Figure 15 shows the changes in the shank strain (axial force) during the exposure test for the notched bolts. Here, the strain was expressed as a relative value obtained by subtracting the strain value at the finish point of the delayed fracture (F_f) from the measured strain value. The strain value at F_f was set to 0. There was also no difference in the strain change among the bolts until the delayed fracture occurred. Focusing on this point, the strain change for the fractured bolt was superimposed onto the strain change for the unfractured bolt by adjusting the difference in the strain between these two bolts. Therefore, as shown in the figures, using the strain for the unfractured bolts as a reference, the time at which the strain started to decrease was defined as the delayed fracture starting point (F_s).

Bolt C1 ruptured at the notch root after a slight decrease in the strain. The time from F_s to F_f was 30 min. The

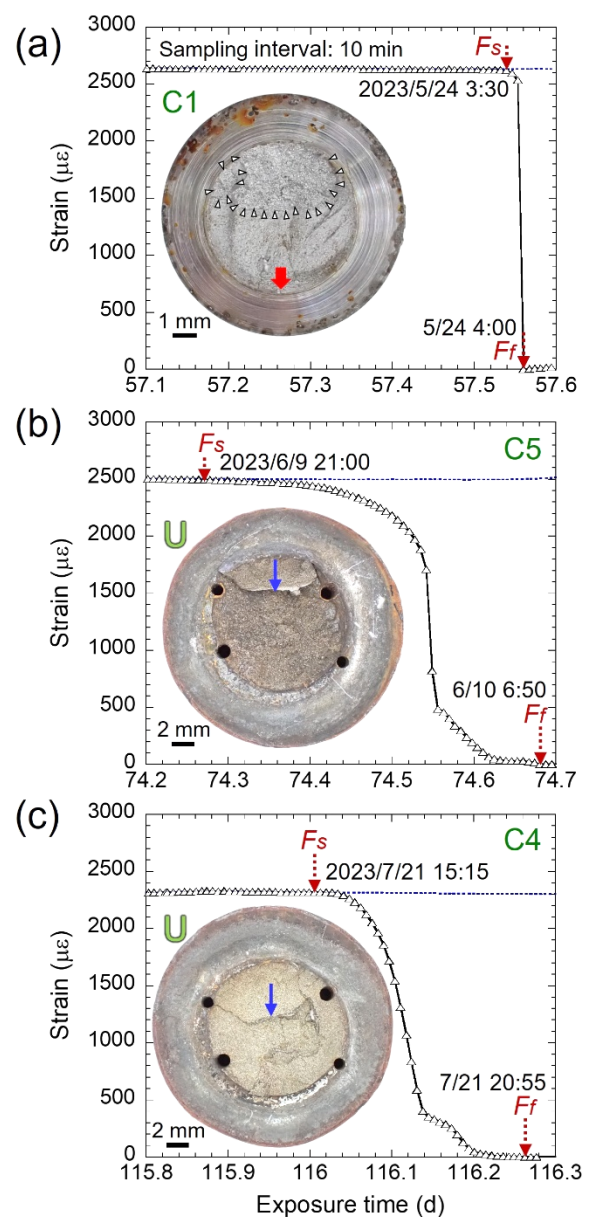


Fig. 15. Change in strain for the shank as a function of exposure time for the notched bolts (F2) failed at (a) notch root, and (b, c) under-head fillets. The macroscopic fracture surfaces are shown. Strain change of unfractured bolt is shown as a blue dashed line for reference. Blue arrows in (b, c) indicate steps. (Online version in color.)

decrease in nominal stress at the notch root was approximately 100 MPa from F_s to the onset of an unstable fracture, which presumably corresponds to the formation of the fracture origin zone with an area fraction of 33% (Fig. 13(b)). Yamasaki and Takahashi²⁷⁾ investigated the correlation between H_D and the crack initiation and propagation in constant load testing using SCM440 steel with σ_B of 1 330 MPa by acoustic emission (AE) source wave analysis. This constant load test was conducted at σ_a of $0.9\sigma_B$ ($= 1 197$ MPa) for a notched specimen with a stress concentration factor of 3.5 designed to simulate stress distribution in the threaded portion. They reported that the time from initial crack initiation to rupture increased with a decrease in H_D and was approximately several hundred minutes at around H_C ($= 0.41$ mass ppm).

For bolts C4 and C5, which failed at the under-head fillet, the strain (axial force) continuously decreased, and after the rate of strain decrease slowed down at around $500 \mu\epsilon$, it began to decrease again. On the day that the delayed fracture occurred in bolt C5, 33.5 mm²²⁾ of rain fell, and the fracture surface of bolt C5 exhibited the most advanced corrosion among the bolts. The fraction of the intergranular fracture area was nearly 100% in bolts C4 and C5 (Fig. 13(b)), and steps were observed on the fracture surfaces of the bolts. It is thus inferred that the change in the strain corresponds to such discontinuous crack propagation. The time from F_s to F_f for bolts C5 and C4 was respectively 590 and 340 min, and the decrease in nominal stress during this period was approximately 350 MPa in the shank with a cross-sectional area of 109.4 mm². Hence, in the under-head fillet portion, crack propagation occurred slowly under a low stress, accompanied by intergranular cracking. Shibata *et al.*³¹⁾ quantitatively evaluated the crack propagation of hydrogen-charged as-quenched martensitic steel by fracture toughness testing and 3D X-ray computed tomography. The results demonstrated that even at a high hydrogen content of 4 mass ppm, the steel exhibited a certain degree of crack growth resistance, and unstable premature fracture did not occur immediately.

Figure 16 shows the relationship between the fraction of the intergranular fracture area and σ_{max}^* for the notched specimens (the relationship between σ_{max}^* and H_C^* is shown in Fig. 2(b)), as well as the untreated bolts and the notched bolts that were subjected to the exposure tests. Considering a reduction in axial force of up to 10% due to relaxation,^{3,9)} σ_{max}^* for the bolts was calculated using $0.95\sigma_a$, and the error bars indicate the values of σ_{max}^* within the range of $(0.9-1.0)\sigma_a$. The σ_{max}^* values in the nut 1st thread root and the thread runout were calculated using Eq. (2). Furthermore, based on the FEM analysis for JIS M22 bolts, it has been estimated that at σ_a of $0.75\sigma_{0.2}$, σ_{max}^* at the thread root in nut and the under-head fillet are identical and this value is approximately 80% of σ_{max}^* in the nut 1st thread root.⁹⁾ The σ_{max}^* values at the thread root in nut and the under-head fillet were thus calculated using Eq. (7),^{9,11)} which accounts for the stress concentration in the threaded portion. For the notched bolts, the σ_{max}^* values at the thread root in nut were calculated using Eq. (7), and the σ_{max}^* values at the under-head fillet were set to three times the value of σ_a at the shank by assuming that the stress concentration factor was 3.³³⁾

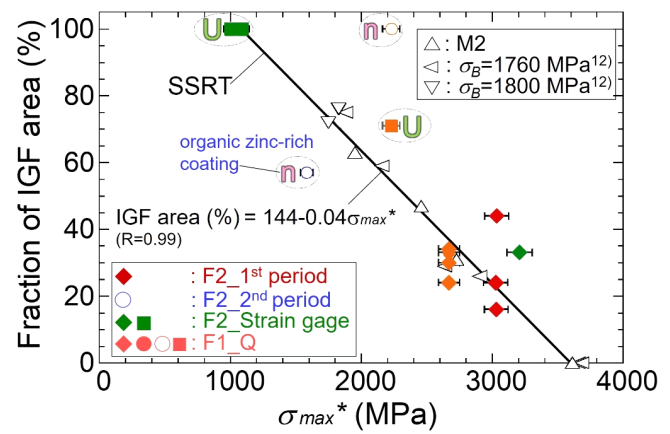


Fig. 16. Relationship between the fraction of IGF area and σ_{max}^* for the non-surface-treated bolts (F1_Q) and the notched bolts (F2) in Fig. 13. The regression line was calculated from the SSRT data of the QT samples in Fig. 2. The σ_{max}^* of bolts were calculated at 0.95 times the σ_a using Eq. (2) or (7), considering stress relaxation, and the error bars indicate the σ_{max}^* at $(0.90-1.0)\sigma_a$. The σ_{max}^* of the notched bolts failed at under-head fillet (U) in Fig. 15(b), (c) were calculated to be 3 times the σ_a at the shank. (Online version in color.)

$$\left(\frac{\sigma_{max}^*}{\sigma_B}\right)_{r=0.25 \text{ mm}} = 0.564 + 1.511\left(\frac{\sigma_{NB}}{\sigma_B}\right) - 0.380\left(\frac{\sigma_{NB}}{\sigma_B}\right)^2 \quad \dots (7)$$

A linear relationship is observed between σ_{max}^* and the fraction of the intergranular fracture area for the notched specimens with the SSRT; the lower the σ_{max}^* , the higher the fraction of the intergranular fracture area. A similar correlation was also confirmed in other medium-carbon low-alloy quenched and tempered steels^{19,29)} (Fig. S4).

The data points for the bolts that ruptured at the nut 1st thread root or the thread runout are plotted near the regression line, similar to those for the notched bolts that ruptured at the notch root. For the 1 700 MPa-class ultra-high-strength bolts fabricated from the 0.5%C-2%Si-1%Cr-1%Mo steel, σ_{max}^* could not be estimated due to the special shape; however, the average area fraction of the fracture origin zone at axial forces of 391 and 445 kN was 2.0% and 1.3%, respectively, and decreased with larger axial force.¹⁴⁾ These findings demonstrate that the delayed fracture of the bolts can be explained by a hydrogen embrittlement mechanism in which an unstable fracture occurs when intergranular cracking initiates and the area fraction of the fracture origin zone reaches a critical value. For the nut 1st thread root and the thread runout in which the σ_{max}^* values were high, it was interpreted that once a condition of $H_C^* \leq H_E^*$ arose due to hydrogen intrusion through a localized corrosion reaction, the fracture origin zone formed rapidly as shown in Fig. 15(a), and the bolt ruptured.

On the other hand, for the thread root in nut and the under-head fillet, the σ_{max}^* was lower than that in the nut 1st thread root and the thread runout, and thus the critical area fraction of the fracture origin zone required for the onset of an unstable fracture was larger according to the regression line in the figure. Additionally, as shown in Figs. 15(b), 15(c), the axial force decreased as the crack propagated. This decrease in axial force reduced the stress in the vicinity

of the crack tip, thereby inhibiting crack propagation. Under the condition where crack propagation is inhibited, the crack tip may become blunted due to plastic accommodation. If the supply of hydrogen to the vicinity of the crack tip is interrupted, the crack blunting will likely be further accelerated. The decrease in the axial force reduction rate observed at low axial forces (around 500 μE) for bolts C5 and C4 in Figs. 15(b), 15(c) suggests the possibility of crack tip blunting. In particular, in the thread root in nut, cracks were less likely to open due to the mechanical restraint created by the contact between the bolt–nut connection threads. For example, H_C^* at the thread root ($\sigma_{\max}^* = 1\,586\text{ MPa}$) for notched bolt B20 that failed at the thread root in nut can be estimated to be 9.3 mass ppm based on the relationship between $\sigma_{\max}^*$ and H_C^* shown in Fig. 2(b). This H_C^* value is significantly higher than the H_E^* value ($= 2.2\text{ mass ppm}$) that was estimated at the notch root with a stress concentration factor of 4.9 in the previous section. Therefore, for notched bolt B20, the arrest of crack propagation observed at the thread root in nut (Fig. 11(g)) is believed to be the result of the combined effects of these factors. Furthermore, the results obtained by the constant load test and the AE source wave analysis described earlier have shown that once a crack was initiated, it could propagate even under H_D values equivalent to a fraction of H_C .²⁷⁾ Kawamori and Yuse³⁴⁾ performed exposure tests using U-bend specimens for SCM435 steel plate with a σ_B value of 1 549 MPa in atmospheric corrosion environments, and conducted *in situ* observations of hydrogen intrusion and hydrogen embrittlement behavior. They demonstrated that crack initiation, arrest, and propagation were repeated in conjunction with the entry and release of hydrogen from the environment. Therefore, it is inferred that the axial force decreased while hydrogen intrusion from the crack surface accelerated the crack propagation, and consequently, the fraction of the intergranular area became larger than the critical area fraction of the fracture origin zone. Based on the corrosion condition observed on the fracture surface, we discussed the delayed fracture process where a newly initiated crack propagated along the grain boundaries after the arrest of cracks. Examples are the bolts that failed at the thread root in nut ($\sigma_{\max}^* = 2\,230\text{ MPa}$, $H_C^* = 2.5\text{ mass ppm}$) as follows. In bolt Q11 (Fig. 11(c)), the fracture surfaces for the cracks indicated by arrows exhibited more advanced corrosion than that for the main crack, suggesting that these cracks were formed earlier than the main crack. In bolt G11 (Fig. S3(a)), it is considered that following the formation of a fracture origin zone covering 20–30% of the fracture surface area, the cracks were initiated and then they propagated along the grain boundaries due to hydrogen intrusion. This resulted in a fracture surface where the intergranular fracture area covered nearly 100% of the surface area.

Although $\sigma_{\max}^*$ at the thread runout was nearly identical to that at the nut 1st thread root, the difference in the fracture time between these locations for the untreated bolts (Fig. 4) suggests a difference in the hydrogen intrusion behavior. For the bolts with geomet coating, no delayed fracture occurred at the thread runout. This is considered to be due to the anti-rust effect of the geomet coating, which prevented hydrogen intrusion. However, the occurrence of delayed fracture was concentrated at the nut fitting portion

including the nut 1st thread root and the thread root in nut. One possible reason for this is that the geomet coating may have been damaged during the bolt tightening or exposure testing, allowing the hydrogen to enter locally through the damaged areas. Furthermore, in railway bridges, it has been reported that high-strength bolts frequently ruptured at the under-head fillets in the joints where the bolts had been tightened with their heads facing downward. One reason cited is that water tended to accumulate at under-head fillet portions.³⁾ From this perspective, in the present exposure test with the heads facing upward, four untreated bolts, fourteen bolts with geomet coating, and three bolts with electrogalvanizing failed at the nut fitting portion (Fig. 5). In contrast, delayed fracture at the under-head fillet occurred only in the two untreated bolts. Although there was limited data on the exposure tests for the notched bolts with the heads facing downward, the number of bolts that failed was larger at the under-head fillet than at the nut fitting portion; three bolts failed at the under-head fillet and one bolt failed at the nut fitting portion (Figs. 7, 13(b)). Thus, it was suggested that the ingress and accumulation of rainwater are closely related to the occurrence of delayed fracture of the bolts. In actual bolted joints, rainwater may penetrate the gaps between the bolted plates, further complicating the delayed fracture behavior.

The rupture of the bolts at the nut 1st thread or the thread runout could be roughly evaluated using the H_C^*/H_E^* method with the hydrogen intrusion environment set to pH 2.¹⁰⁾ On the other hand, from the relationship between H_C^* and H_E^* in Fig. 2(b), the H_C^* values at the under-head fillet and the thread root in nut are estimated to become significantly larger than the H_E^* values as $\sigma_{\max}^*$ decreases. If there is no damage such as surface scratches, it is necessary to consider a hydrogen intrusion environment that is more severe than pH 2. Delayed fractures at these locations can sometimes be difficult to detect by visual and tactile inspection, which poses a problem for the practical application of bolts. Hence, for ultra-high-strength bolts, we determined that it is a critical challenge to clarify the mechanism of hydrogen intrusion associated with localized corrosion at locations where rainwater tends to accumulate and $\sigma_{\max}^*$ is low, and to improve the accuracy of H_E^* estimates.

5. Conclusions

Delayed fracture behavior of 1 700 MPa-class quenched and tempered bolts fabricated from a 0.4%C-2%Si-1%Cr-1%Mo steel was investigated by conducting outdoor exposure tests in which water ingress was restricted to the head and nut sections. Furthermore, we observed the process of delayed fracture by measuring the strain change on the bolt shank and conducted accelerated tests for hydrogen embrittlement. The delayed fracture mechanism of the ultra-high-strength bolts was then comprehensively discussed. The following findings were obtained.

(1) Delayed fracture behavior of the bolts over the 10-year exposure test performed at the JWTC Miyakojima test site was broadly classified into two types: rupture at the thread runout and the nut 1st thread root, and fracture at the under-head fillet and the thread root in the nut. The exposure tests using the notched bolts at the NIMS outdoor corrosion

test site could reproduce the delayed fracture occurring at the thread runout of the non-surface treated bolts in a way that accelerated the fracture occurrence. Furthermore, it was confirmed that the delayed fracture at the thread runout and the notched portion of the bolts during the exposure tests could be protected by rust prevention treatment.

(2) It was suggested that the delayed fracture of the bolts was caused by the intrusion of hydrogen generated through localized corrosion reactions on the surfaces at the fracture locations, and the ingress and accumulation of rainwater influenced the occurrence of the delayed fracture.

(3) The fracture morphology of the bolts varied depending on the location of the delayed fracture rather than on the surface treatment or exposure time. The macroscopic fracture surfaces of the bolts that ruptured at the thread runout or the nut 1st thread root consisted of the fracture origin zone containing intergranular cracks along prior-austenite grain boundaries, the final fracture zone, and the shear lip. In contrast, the majority of the macroscopic fracture surfaces of the bolts that failed at the under-head fillet or the thread root in nut were covered by intergranular fracture areas, and some bolts were even observed to have an intergranular fracture area fraction of nearly 100%.

(4) In the SSRT, the fraction of the intergranular fracture area was proportional to the peak value of the maximum principal stress near the notch root σ_{max}^* . The delayed fracture occurring at the thread runout or the nut 1st thread root at which the σ_{max}^* is the highest could be explained by a hydrogen embrittlement mechanism in which an unstable fracture occurs when intergranular cracking is initiated and the area fraction of the fracture origin zone reaches a critical value. It could be interpreted that the fracture origin zone was formed when the local hydrogen concentration from the service environment H_E^* exceeded the local critical hydrogen concentration for hydrogen embrittlement H_C^* near the maximum stress point, leading to the rupture of the bolt.

(5) Regarding the delayed fracture occurring at the under-head fillet or the thread root in nut at which the σ_{max}^* was low, we suggested a mechanism in which not only does the axial force decrease with the initiation and propagation of cracks, but the propagation of cracks is also accelerated by the intrusion of hydrogen from the surfaces of the cracks.

Supporting Information

[Figs. S1–S4] This material is available on the Journal website at <https://doi.org/10.2355/isijinternational.ISIJINT-2026-159>.

Conflict of Interest Statement

The authors declare no conflicts of interest in this study.

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