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Study on the hydrogen-induced delayed fracture behavior of Q-P980 and MS980

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Abstract

In this paper, the hydrogen diffusion behavior and hydrogen induced delayed fracture (HIDF) of Q-P980 (Q-P: Quenching and Partitioning) and MS980 (MS: Martensitic steel) steels were investigated using hydrogen penetration, slow strain rate tensile (SSRT) tests, thermal desorption spectroscopy (TDS) tests, fracture analysis, and microstructural examination in this paper. The austenite in Q-P980 is massive retained-austenite (RA) with low stability. The TRIP (Transformation Induced Plasticity) effect will occur in the process of strain and change into high carbon martensite. HIDF is caused by a substantial amount of surplus hydrogen being enriched at the border and flaws. The fracture has a broad cleavage surface and is a typical quasi-cleavage fracture. MS980 has been sufficiently tempered, resulting in a substantial quantity of distributed spherical cementite (150nm) precipitating around the lath martensite. This size and form of cementite may successfully trap hydrogen while maintaining the material’s mechanical characteristics. And tempering can effectively reduce the local stress level of steel, so MS980 has a very low HE susceptibility. HIDF is related to local stress and hydrogen accumulation. We suppose that \( Z \) is a constant and \( Z_C \) is a critical value which associated to \( \sigma \) and \( C_{15} \) (the local stress and local hydrogen concentration), rising as \( \sigma \) and \( C_{15} \) rises. The atomic bonds at the crack tip, lattice position and the phase interface will fracture when \( Z_C \) reaches a particular value \( Z \). Tempering to minimize local stress and carbide precipitation to capture hydrogen are two strategies for reducing hydrogen embrittlement (HE) susceptibility, particularly for dislocation strengthened steel. Microalloying elements can generate precipitates that function as hydrogen traps and obstruct the HELP (Hydrogen Enhanced Localized Plasticity) process, lowering local stress and hydrogen accumulation.

1. Introduction

Hydrogen will dramatically deteriorate the mechanical properties (especially toughness and elongation) of steel materials, and unexpected delayed fracture occurs when it is far lower than the designed strength limit (even below the yield strength) [1]. Hydrogen-induced delayed fracture (HIDF) is frequently fatal due to its unpredictability, speed, and difficulty in detection [2]. Hydrogen embrittlement (HE) has emerged as a possible danger to advanced high-strength steel applications [3, 4].

The research and development of the third-generation advanced automotive steel mainly relies on the TRIP effect of retained austenite (RA) during deformation. One of the most representative is Q-P steel [5]. The strength and plasticity of Q-P steel are a suitable complement. However, there are currently few research on the HE properties of this type of steel, and no consensus on the impact of RA in Q-P steel on its HE. According to certain studies [6, 7], the RA can act as hydrogen traps and boost HE resistance. Yang et al [8] treated Fe-0.4C-1.5Mn-1.5Si (wt%) steel with Q-P and Q-T (Quenching & Tempering) and found that the HE of Q-P steel is milder than that of Q-T steel. Because the RA in Q-P steel can act as a hydrogen trap to prevent hydrogen...
diffusion, increase the solubility of hydrogen, and improve the HE resistance of Q-P steel. At the same time, the hydrogen content per unit area is low after refining the microstructure, which can further improve the plasticity. Zhu et al.\textsuperscript{[9]} treated Fe-0.22C-1.40Si-1.80Mn steel after Q-P with hydrogen charging, the total elongation decreased sharply from 19.5\% to 2.5\%, but the strength was hardly affected. Similarly, Lovicu et al.\textsuperscript{[10]} compared Q-P specimens and Q-T specimens with the same chemical composition and tensile strength. They discovered that Q-P specimens containing 17.5\% RA had higher HE than Q-T specimens without RA. The reason was that RA captured more hydrogen than martensite (M) as a hydrogen trap, and RA transformed into M during deformation, resulting in HIDF.

As a result, the interaction between hydrogen and microstructure in Q-P steel is still used to explain experimental phenomena including the relationship between the origin and propagation of hydrogen-induced cracks with microstructure. Hydrogen-induced cracks nucleate mainly along the strain-induced fresh M or M/A interface. It doesn’t go into detail on how different microstructures interact with hydrogen. Clarifying hydrogen diffusion and enrichment in diverse microstructures, as well as redistribution and enrichment under stress conditions, will aid understanding of the relationship between HE and the microstructure, and will encourage Q-P steel research into the mechanism of HE.

Because of its high strength and numerous structural defects, such as the prior austenite grain boundary, lath interface, dislocation, and phase interface, martensite is known as the most vulnerable structure to HE. A very little amount of hydrogen can induce considerable HE, especially when the strength level is greater than 1000MPa \textsuperscript{[11–13]}. Lee et al.\textsuperscript{[14]} quenched and tempered 22MnB5 steel for hot-forming and charged with hydrogen. The quenched specimen had significant HIDF, cleavage fracture occurred under low-stress conditions, and the specimens tempered at 460 °C and 520 °C did not have HIDF. The reduction in HE with increasing tempering temperature is due to a decrease in dislocation density and an increase in cementite as a hydrogen trap, lowering the hydrogen content at the grain boundary. Zhang Y et al.\textsuperscript{[15]} investigated the HE of ultra-high-strength hot stamping steel plate. They discovered that tempering or hot forming at 200 °C can significantly reduce its HE susceptibility, which is due to carbide precipitation in the tempering process of experimental steel and self-tempering of steel plate. Intercrystalline carbides can limit hydrogen enrichment at grain borders by acting as a hydrogen trap. According to the hydrogen trap theory, uniformly dispersed carbides can capture diffusible hydrogen (DH), reduce the DH diffusion coefficient, and reduce the HE susceptibility of advanced high-strength steel. Carbides, on the other hand, may diminish material elongation, and the pinning effect of carbides decreases as interfacial coherence decreases. As a result, the focus of attention is on selecting acceptable carbides and controlling carbide size, shape, and distribution in a reasonable manner.

We prepared two types of high-strength steel with a 980MPa grade in this paper (Q-P980 and MS980). Transmission electron microscopy (TEM) and field-emission scanning electron microscopy (FESEM) were used to analyze the microstructure of the experiment steels. Electron backscatter diffraction (EBSD) was used to investigate grain boundary information. X-ray diffraction (XRD) was used to determine the amount of retained austenite. SSRTs and constant load tensile tests (CLTs) of hydrogen pre-charged specimens were used to assess the steels’ HE susceptibilities. FESEM was used to analyze the characteristics of the fracture surfaces as well as the failure causes. Hydrogen permeation and thermal desorption spectroscopy (TDS) tests were used to determine the apparent diffusion coefficient ($D_{\text{app}}$) and hydrogen concentration, respectively. The HIDF behavior of Q-P980 and MS980 is widely known based on the above investigations. We also went into the impact of massive RA and cementite on steels. Finally, we expressed the effect of hydrogen on material delayed fracture.

2. Materials and methods

2.1. Materials

The experimental steels were two types of high-strength steels with a yield strength of 980 MPa (Q-P980 and MS980), with chemical compositions listed in table 1. Steel plates were used to make specimens for SSRTs, CLTs, and hydrogen permeation tests. Figures 1(a)–(c). The thickness of the specimens were approximately 1 mm.

2.2. Methods

2.2.1. Microstructure and fractography feature observations

The metallographic specimens were ground to 2000 grit using sandpaper, physically polished, and then etched with a 3 percent nitric acid alcohol solution to investigate the microstructure of the experimental steels. The Quanta FEG 450 FESEM was then used to observe the results. The TEM foils were ground to about 50 m and then electropolished with 10\% perchloric acid and 90\% ethanol at a low temperature using a twinjet electropolishing apparatus. The F2100 TEM was used to look for RA. After the SSRTs, the fracture morphologies of hydrogen-charged and hydrogen-free specimens were investigated using a Quanta FEG 450 FESEM. To remove the stress layer, EBSD and XRD specimens were ground to 2000 grit and electrolytically polished.
TESCAN MAIA3 SEM was used to scan EBSD specimens. The phase compositions were investigated using Cu-K radiation and a 0.02° step size in XRD analysis. 10 percent perchloric acid solution + 90% absolute ethanol electrolytic polishing solution.

2.2.2. Hydrogen permeation experiment
The hydrogen penetration specimens were manually polished after being ground to 5000 grit on both sides using sandpaper. To prevent corrosion or passivation of the anode surface, the anode side must be examined on the hydrogen permeation device (Figure 2) [16] after nickel plating (the nickel layer thickness is around 100 nm). This thickness not only ensures that the anode side is not oxidized, but it also ensures that measurement precision is not compromised. The hydrogen charging slot (Slot A) solution was 0.2 mol l\(^{-1}\) NaOH + 0.2 g l\(^{-1}\) thiourea solution (thiourea, being a toxic agent, acts as a negative catalyst for hydrogen recombination), whereas the hydrogen expansion slot (Slot B) solution was 0.2 mol l\(^{-1}\) NaOH solution. During the experiment, the specimens served as both anode and cathode. To ensure a constant cathode hydrogen charging current value, use one side of the specimen as the cathode and connect it to the constant current potentiostat in Slot A. The anode side of the specimen was connected to the electrochemical workstation towards Slot B to maintain a

![Figure 1](image1.png)

![Figure 2](image2.png)

Table 1. Chemical composition of MS980 and Q-P980.

| Steel    | C     | Si    | Mn    | P     | S     | Nb + Ti |
|----------|-------|-------|-------|-------|-------|---------|
| MS980    | 0.25  | 0.26  | 1.69  | 0.012 | 0.001 | 0.032   |
| Q-P980   | 0.22  | 1.58  | 2.01  | 0.013 | 0.002 | 0.08    |

TSCREEN MAIA3 SEM was used to scan EBSD specimens. The phase compositions were investigated using Cu-K radiation and a 0.02° step size in XRD analysis. 10 percent perchloric acid solution + 90% absolute ethanol electrolytic polishing solution.

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consistent potential value on the anode side’s surface, ensuring that hydrogen diffused from the cathode side was ionized immediately ($H \rightarrow H^+ + e^-$). The diffusion rate of hydrogen in the specimen is directly assessed by the transient curve of the anode current recorded on the anode side with respect to time. [17] As supplementary electrodes for the cathode chamber and anode chamber, two platinum electrodes were employed. The reference electrode should be chosen based on the demands of the experiment. In this experiment, the Hg/HgO reference electrodes were employed. After obtaining the anodic polarization curve, the apparent diffusion coefficient of hydrogen ($D_{ap}$) and the diffusible hydrogen concentration in the lattice ($C_0$) could be obtained by Fick diffusion law [18, 19]:

$$D_{ap} = \frac{I^2}{6I_{0.63}}$$

$$C_0 = 1.31 \times 10^{-6} \frac{I_{\infty} L}{D_{ap} S}$$

Where, $L$ is the thickness of the specimen, cm; $I_{\infty}$ is the saturated anode current, mA; $S$ is the anode area of the specimen, cm$^2$; $t_{0.63}$ is the lag time, which is the time corresponding to $I/I_{\infty} = 0.63$, $D_{ap}$ is the apparent diffusion coefficient of hydrogen in the material, cm$^2$ s$^{-1}$. $C_0$ is the diffusible hydrogen concentration in the lattice, mA·s·cm$^{-3}$.

2.2.3. SSRT test

The gauge section of the SSRT testing specimens was ground to 2000 grit on both sides with sandpaper, and the non-gauge half was sealed with silicon rubber. The specimens were precharged with 0.1 mol/L NaOH aqueous solution for 24 hours. The charging current densities for hydrogen were 0, 3, and 5 mA·cm$^{-2}$, respectively. The cathode was specimen, and the anode was platinum wire. All tests were carried out at room temperature with a strain rate of $1 \times 10^{-5}$ s$^{-1}$. The relative reduction in fracture strength and displacement was used to describe the HE susceptibilities $R$ [20].

$$R = \frac{R_0 - R_{H}}{R_0} \times 100\%$$

Where, $R_0$ and $R_{H}$ represent the tensile breaking strength and elongations of the specimens with and without hydrogen pre-charging, respectively.

2.2.4. CLT test

The CLT tests specimens were ground to 2000 grit on both sides with sandpaper to ensure that the gauge portion was smooth, and the non-gauge section was sealed with silicon rubber. Considering the experimental steels’ original structure has M and is sensitive to HE, the hydrogen charging solution was chosen to be 0.1 mol/L NaOH aqueous solution, the hydrogen charging current density was set to 1 mA·cm$^{-2}$, the anode was platinum wire, and the cathode was a specimen that was stretched while charging hydrogen. All of the experiments were carried out at room temperature. Figure 3 shows a schematic of the CLT test instrument.

Finally, the CLT test periods were acquired under various lag fracture strengths to determine its HE susceptibilities[21].
2.2.5. Thermal desorption spectroscopy (TDS) test

The specimens were cut to 10 mm × 30 mm × plate thickness and ground to 2000 grit on both sides with sandpaper. Following that, an electrochemical hydrogen charging test is performed. Figure 4 shows a schematic depiction of hydrogen charging. The hydrogen charging solution was 0.1 mol/l NaOH aqueous solution, and the hydrogen charging current density was 20 mA cm⁻². The specimens was continuously charged with hydrogen for 24h at room temperature, and then blown dry and transferred to TDS equipment for test. The heating rate is 100 °C·h⁻¹ and heated to 500 °C [21].

3. Results

3.1. Microstructural characterizations

Figure 5 shows the microstructure of the two experimental steels. The SEM images of the experimental steels are shown in figure 5 (a) and (b). Massive fresh martensite (FM) and strip tempered martensite made up the MS980 (TM). A substantial amount of spherical cementite (Fe₃C) is scattered around the TM, which has a diameter of around 150 nm, due to the sufficient tempering of MS980. On the ferrite matrix (F), the Q-P980 was made up of bainite (B) and M/A Island (M/A) structures, which were made up of high carbon martensite and RA.

The inverse pole figures (IPF) of MS980 and Q-P980 are shown in figure 5 (c) and (d), respectively. MS980 and Q-P980 have prior austenite grain sizes (PAGS) of 41.03 μm² and 118.56 μm², respectively, according to the IPF diagram and Image J.

The phase distribution diagrams of MS980 and Q-P980 are shown in figure 5 (e) (f). MS980 has very little austenite. In Q-P980, there was higher RA, and the results show that the RA in this micro zone was 5.9%. The Q-P980 also contained 90.86% body-centred cubic (BCC) iron phase and 9.14% RA phase, as shown by the XRD data in figure 11.

Figure 6 shows TEM images of the phases in the Q-P980, it can be found that there is massive martensite (M) (average diameter of ~3 μm) in the microstructure of Q-P980, which substructure is high-density dislocation (figure 6(a), and M/A Island with massive RA (average diameter of ~3 μm) was observed, as shown in figure 6(b).

3.2. Hydrogen diffusion behavior

The hydrogen permeation curves of the steels are shown in figure 7 when the hydrogen charging current density is 10 mA cm⁻². The lag time and penetration time of the specimens are t₀,63 and t₀, respectively. The steels’ $D_{ap}$
and $C_0$ were determined and presented in Table 2. MS980 and Q-P980 have $D_{ap}$ values of $4.87 \times 10^{-7}$ cm$^2$s$^{-1}$ and $2.103 \times 10^{-7}$ cm$^2$s$^{-1}$, respectively, and $C_0$ values of $1790.558 \times 10^{-6}$ mA·sm$^{-3}$ and $755.688 \times 10^{-6}$ mA·sm$^{-3}$, respectively. Q-P980 has a lower $D_{ap}$ value, as well as a lower $C_0$. The penetration time ($t_b$) of the

Figure 5. Microstructure of experimental steel. SEM: (a) MS980; (b) QP980. IPF: (c) MS980; (d) QP980. Phase: (e) MS980; (f) QP980.
steels, on the other hand, was in the reverse order. The results indicated that hydrogen diffusion through Q-P980 is more difficult.

This is mostly due to the presence of greater RA in Q-P980, which has a high hydrogen solubility and a very low hydrogen diffusion coefficient, according to the microstructural characterizations (section 3.1).

The hydrogen escape curves (the curve of the hydrogen escape rate with heating temperature) of the hydrogen-filled specimens of steels recorded by TDS are shown in figure 8. At 0–500 °C, MS980 and Q-P980 showed two distinct hydrogen escape peaks, as seen in the figure. However, the Q-P980 escape peak at 100 °C was visible, and the MS980 escape peak at 350 °C is more visible than the low-temperature side. At room temperature, hydrogen traps in steels may be categorized into irreversible and reversible trap sites based on the hydrogen-trapping energy. Low-energy traps, such as solute atoms, dislocations, voids, and low-angle grain boundaries, are considered reversible, but high-energy traps, such as inclusions and precipitates (TiC, Fe3C), and high-angle grain boundaries, are considered irreversible [22, 23].

Similarly, at room temperature, hydrogen in steel may be split into diffusible hydrogen (DH) and non-diffusible hydrogen (NDH). The hydrogen escaping from the low-temperature side is typically thought to be DH (hydrogen escaping at a temperature below around 300 °C), and the DH is the hydrogen captured by the reversible trap. The hydrogen escaping from the high-temperature side is mostly trapped by an irreversible trap and is NDH at normal temperature [24]. It’s worth noting that DH had the greatest impact on the HE susceptibilities of high-strength steel. Table 3 shows the hydrogen content of the experimental steels as measured by TDS after hydrogen charging in advance.

The result shows that the DH concentration of the Q-P980 specimen was 0.6037 ppm, which was higher than the total hydrogen content of MS980. This was due to the presence of a substantial quantity of RA in its microstructure, demonstrating that austenite is far more soluble in hydrogen than BCC and any hydrogen traps.
MS980 has a higher NDH concentration (0.1847 ppm) than Q-P980, which can be attributed to a substantial quantity of spherical cementite (Fe₃C) scattered around the TM.

3.3. Hydrogen embrittlement (HE) susceptibility evaluation

To investigate the experimental steels’ HE susceptibilities, SSRT tests were performed on tensile specimens from Q-P980 and MS980 steels with varying hydrogen charging current densities. The Stress-Displacement curves

Table 2. Hydrogen permeation experiment results of experimental steels.

| number | t₀,0.5(s) | t₀(s) | Dₘₚ (10⁻⁷ cm²/s) | C₀ (10⁻⁵ mA·s cm⁻²) |
|--------|-----------|-------|-------------------|---------------------|
| MS980  | 4589      | 1164  | 4.870             | 1790.558            |
| Q-P980 | 7944      | 3703  | 2.103             | 755.688             |

Table 3. Hydrogen content of MS980 and Q-P980.

| Specimen | DH (PPM) | NDH (PPM) | Total hydrogen (PPM) |
|----------|----------|-----------|----------------------|
| MS980    | 0.2365   | 0.1847    | 0.4212               |
| Q-P980   | 0.6037   | 0.1654    | 0.7691               |

MS980 has a higher NDH concentration (0.1847 ppm) than Q-P980, which can be attributed to a substantial quantity of spherical cementite (Fe₃C) scattered around the TM.
from the SSRT testing of the experimental steels are shown in figure 9, and the relevant mechanical data are given in table 4. The results reveal that the strength and plastic loss rates of MS980 are substantially lower than those of Q-P980 under the identical hydrogen charging conditions. The strength and plastic loss rates of Q-P980 were 19.3 percent and 79.8%, respectively, when the hydrogen charging current density was 5 mA·cm\(^{-2}\). The TRIP effect of RA in Q-P980 during SSRT testing might explain the high strength and plastic loss rates of Q-P980. CLT tests and XRD were performed on the Q-P980 for further verification.

In the CLT tests, the delayed fracture strength ratio \(\sigma_{\text{HIC}}/R_m\) is used to evaluate the HE susceptibilities of steels, where \(\sigma_{\text{HIC}}\) is the threshold stress and \(R_m\) is the tensile strength. The CLT test results of Q-P980 when the current density was 1 mA·cm\(^{-2}\) are presented in figure 10. The experimental steel’s threshold stress is 894 MPa at 1 mA·cm\(^{-2}\) current density (TRIP effect may not be activated), and the delayed fracture strength ratio \(\sigma_{\text{HIC}}/R_m\) is 0.827 (table 5). XRD experiments were used to examine the fractured samples in order to better understand the concept of HIDF in steels.

3.4. Results of x-ray diffraction (XRD)

Figure 11 and table 6 demonstrate the RA volume fraction of Q-P980 with various hydrogen charging current densities or constant loads. The volume proportion of RA reduced to varied degrees (mainly (220)\(\gamma\) and (311)\(\gamma\)), demonstrating that the RA had a TRIP impact with the strain during the SSRT process. Stress concentration and hydrogen diffusion enrichment are both linked to the HIDF of high-strength steel. When the hydrogen concentration and stress value reach a threshold level, HIDF will occur \([25]\). The DH concentration in steels rose as the hydrogen charging current density increased. The hydrogen concentration and tension in the steel reached critical levels when the RA was not entirely converted. So, when the final fracture occurred, the RA volume fraction showed an upward trend.

The RA volume percent of the fractured samples similarly showed a clear decreasing trend during the CLT testing, demonstrating that the samples all broke following the TRIP effect. The fracture took just 10 min for a stress of 1024Mpa (less than the tensile strength of Q-P980), suggesting that following the TRIP effect, only a little quantity of hydrogen may cause the steel to fracture.

Table 4. Results of SSRT tests of experimental steels.

| Hydrogen charging current density (mA·cm\(^{-2}\)) | Breaking strength (MPa) | Strength loss rate (%) | Displacement (mm) | Plastic loss rate (%) |
|-------------------------------------------------|-------------------------|------------------------|-------------------|----------------------|
| MS980                                            | 0                       | 1134                   | —                 | 2.91                 | —                    |
|                                                 | 3                       | 1124                   | 0.8               | 2.83                 | 2.7                  |
|                                                 | 5                       | 1124                   | 0.8               | 2.78                 | 4.5                  |
| Q-P980                                          | 0                       | 1097                   | —                 | 7.26                 | —                    |
|                                                 | 3                       | 925                    | 15.7              | 1.79                 | 75.3                 |
|                                                 | 5                       | 885                    | 19.3              | 1.47                 | 79.8                 |

Figure 9. SSRT curves of experimental steels.
Figure 10. CLT tests results of Q-P980.

Table 5. The $\sigma_{\text{HIC}}/R_m$ of Q-P980 under 1 mA cm$^{-2}$ current density.

| $i$/mA cm$^{-2}$ | Q-P980 | $\sigma_{\text{HIC}}$ (MPa) | $\sigma_{\text{HIC}}/R_m$ |
|------------------|--------|-----------------------------|--------------------------|
| 1.0              | 894    | 0.827                       |

Figure 11. XRD patterns of Q-P980 under different experimental conditions.

Table 6. RA volume fraction of Q-P980.

| Experiment | Experimental condition | Volume fraction of RA (%) |
|------------|------------------------|--------------------------|
| SSRT       | 0 mA cm$^{-2}$         | 3.47                     |
|            | 3 mA cm$^{-2}$         | 7.49                     |
|            | 5 mA cm$^{-2}$         | 8.36                     |
| CLT        | 924MPa/8h              | 5.11                     |
|            | 1024MPa/10 min         | 5.96                     |
|            | 894MPa/100h+           | 9.14                     |
4. Discussion

4.1. Fracture mechanism analysis

Figure 12 depicts the fracture morphologies of Q-P980. The overall fracture morphologies are shown in figures 12(a)–(c) when the hydrogen charging current density was 0, 3, and 5 mA·cm⁻², respectively. When the hydrogen charging current density of Q-P980 experimental steels was 0 and 3 mA·cm⁻², the outer layer of the fracture was an evident whole dimple structure, as illustrated in figures 12(d)–(g). The dimple has a diameter of 2–4 μm. It was a typical ductile microvoid coalescence fracture (MVC); The inner layer was constituted of a dimple, tiny cleavage surface, and secondary crack, which was a typical quasi-cleavage fracture (QC), and the small cleavage surface showed lath-like features. The existence of secondary cracks showed that its plasticity is excellent [26]. The secondary fractures in the inner layer of the fracture vanished when the hydrogen charging current density was 3 mA·cm⁻², and the area of the cleavage surface grew greater. When the hydrogen charging current density was 5 mA·cm⁻², the core portion of the fracture exhibited a flat cleavage surface with lath-like features, as illustrated in figures 12(h)–(i). The fracture had a dimple and tongue-like cleavage surface on the perimeter, and the entire fracture was an evident quasi-cleavage fracture (QC).

On the one hand, hydrogen promotes dislocation mobility, according to the HELP hypothesis [27]. Hydrogen, on the other hand, tends to congregate around dislocations and move with them. The local hydrogen concentration will be quite high in places with a high dislocation density. As a result of the dislocation’s action, hydrogen is concentrated near the crack tip, resulting in severe plastic deformation. Due to the influence of hydrogen, hydrogen-induced cracks will not be passivated, and on the contrary, they will merge and expand to form the main crack. The primary crack propagates ahead by merging the microcracks and holes that form near the crack tip on a regular basis. Due to plastic instability, once the crack has propagated to a certain extent, the material will undergo ductile ripping until it fractures.

MVC near the surface might be caused by low stress triaxiality, whereas QC in the middle could be caused by high stress triaxiality [28, 29]. The area of the QC zone grows as the hydrogen charging current density increases, notably when the hydrogen charging current density is 5 mA·cm⁻², when the entire section is QC fractured. This is mostly due to hydrogen-induced softening as a result of the HELP effect. As a result, hydrogen accumulates at the crack tip. When a certain value is attained, the HEDE (Hydrogen-Enhanced Decohesion) [30, 31] effect occurs, causing the QC area to expand [32–35].

Zhang S et al. [36] carried out hydrogen-charged SSRT tests and non-hydrogen-charged SSRT tests on 22MnB5, respectively. The results revealed that the fracture was dimples without hydrogen, with considerable changes in the fracture surfaces of the different hydrogen-charged specimens. While the majority of the surface fractured in a mixed mode of intergranular (IG) and quasi-cleavage (QC), only a minor region of the surface fractured in QC.

The difference between the two experimental steels is that Q-P980 requires strain to initiate the TRIP effect, therefore dislocation slip, and plastic deformation are required, resulting in dimples. When the dislocation had not entirely migrated in the apparent intergranular fracture of 22MnB5 [36], the local hydrogen concentration and stress value had approached the critical threshold. The presence of hydrogen weakened the bonding force between grains, allowing microcracks to form, expand, and combine under stress, eventually leading to fracture.

Figure 13 depicts the fracture morphologies of MS980. The overall fracture morphologies are shown in figures 13(a)–(c) when the hydrogen charging current density was 0, 3, and 5 mA·cm⁻², respectively. When the hydrogen charging current density was 0 mA·cm⁻², the outer layer and inner layer of the MS980 fracture formed an apparent complete dimple structure, as illustrated in figures 13(d)–(f). The diameter of the dimple was less than 2 μm. It was a typical ductile microvoid coalescence fracture (MVC). In the most core portion, there were a few minor cleavage surfaces. As shown in figures 13(h)–(m), the inner layer of the quasi-cleavage fracture was constituted of dimples and tiny cleavage surfaces when the hydrogen charging current density was 3 mA·cm⁻² (QC). There are fewer cleavage planes and no secondary cracks in this material than in Q-P980. The complete dimple structure was still visible on the outer layer of the fracture, and the dimple diameter was less than 2 μm. The fracture, however, had smooth cleavage fracture surfaces at both ends, indicating that it was a cleavage fracture (C). This might be due to a high hydrogen concentration in the sample’s outer layer. And it was discovered that the cleavage surface in figure 13(m) had evident river patterns.

In conclusion, the dimple and quasi-cleavage fracture morphology corresponded to the nucleation and propagation location of the main crack in MS980 experimental steel. Because of the low concentration of diffusible hydrogen in steel, there is minimal variation in fracture morphology under different hydrogen charging current densities.
Figure 12. Fracture morphology of Q-P980.
4.2. Effect of microstructure on HE susceptibilities

4.2.1. Effect of martensite on HE susceptibilities

As stated in the introduction, martensite is a major element impacting the HE in martensitic steel or hot stamping steel due to its high strength level and huge number of microstructure flaws. Under the same load, the hydrostatic pressure concentration around the fracture tip or notch increases due to its high strength level [37]. Figures 14(a), (b) shows the KAM (Kernel Average Misorientation: reflects crystal lattice distortion degree, represents local stress magnitude and dislocation density [38]) diagram and data for the two experimental steels.

The results indicate that the KAM average values of MS980 and Q-P980 are 0.72° and 0.38°, respectively. It can be concluded that martensite has a larger dislocation density and local stress than ferrite matrix. In addition, high-density dislocations in martensite, as well as other reversible hydrogen traps, catch a significant amount of
DH. At high hydrostatic pressure, high local stress, and hydrogen attraction, Martensite becomes the phase most susceptible to HE.

According to the hydrogen trap theory, evenly dispersed carbides can capture DH, lower DH diffusion coefficients, and diminish high-strength steels’ HE susceptibilities [22, 23]. MS980 was adequately tempered, and several scattered carbides-Fe₃C precipitated (150 nm). These carbides may be employed as irreversible hydrogen traps to catch a considerable quantity of hydrogen, lowering the steel’s DH content, and the cementite of this size and shape will not degrade the material’s mechanical qualities. Furthermore, several studies have shown that nanosized carbides (such as VC and NbC) can directly pin dislocations [39]. Zhang S et al [20] performed KAM analyses at the fracture of Nb-bearing and non-Nb steels and discovered that the Nb-bearing steels had lower average KAM values. The results indicated that the dislocation density in the region around the fracture surfaces of the hydrogen-charged specimens with NbC was lower than that for the non-Nb steel. During HIDF, the dislocation density of the steels was increased by HELP. Thus, the dislocation density increments resulting from HELP in the Nb-bearing steels were smaller than those in the non-Nb steel, i.e. the NbC.
precipitates hindered the HELP process. So that the accumulation of hydrogen and the increase of local stress were hindered.

4.2.2. Effect of RA on HE susceptibilities

For Q-P steel, TRIP steel, and other steels containing austenite, its role is determined by its stability. Austenite has a very high hydrogen solubility and a very low hydrogen diffusion coefficient when compared to other structures. The Q-P980 contains RA (>9%), as well as martensite and RA for M/A Island. As a result, the hydrogen permeation experiment revealed a very low apparent diffusion coefficient of hydrogen ($D_{ap}$), but the TDS test revealed a very high DH concentration. The morphology was mostly massive RA, according to the TEM results. Massive RA is highly unstable and easily transforms into high carbon martensite under strain, releasing a significant quantity of hydrogen. Furthermore, because Q-P980 has few additional hydrogen traps, it is unable to capture a substantial amount of hydrogen in a short period of time, and the change from austenite to martensite is a crystal lattice rebuilding process. A huge number of dislocations and twins were created as a result of this process, and the enhanced lattice distortion raised local stress, making the microstructure very susceptible to hydrogen. HIDF resulted from the surplus hydrogen being skewed and enriched near the boundary and flaws. As a result, if the RA is not stable enough in advanced high-strength steels including RA, such as Q-P steel and TRIP steel, the TRIP effect will occur under strain, and quasi-cleavage (QC) fracture is a common feature of HIDF in this steel. As a result, austenite stability is critical for advanced high-strength steels including austenite.

4.2.3. Effect of interface on HE susceptibilities

The influence factors of the interface on HE susceptibilities of high-strength steels are mainly divided into two categories: diffusion and aggregation of hydrogen at the interface. The second is the initiation and propagation of cracks in the steel. It mainly depends on the stress value and interface energy, and the specific impact degree shall be judged according to the actual situation. In the process of HIDF, the arrangement order of low-angle grain boundaries and special grain (LOW-\(\Sigma\)) boundaries is high, and the interface energy is low, which has been confirmed in its resistance to intergranular corrosion and stress corrosion [40].

The grain boundary distributions of MS980 and Q-P980 are shown in figures 15(a), (b), respectively. Figure 15(c) is a statistical diagram of grain boundaries of two steels. It is obvious that the grain boundary area of

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**Figure 14.** KAM patterns of experimental steels: (a)(c) MS980; (b)(d) QP980.
MS980 is much larger than that of Q-P980, which is conducive to the uniform distribution of hydrogen. The results indicate that MS980 has a higher proportion of LOW-∑ boundaries and low-angle grain boundaries, which are 31.30% and 26.14%, respectively. These two kinds of grain boundaries have relatively more minor grain boundary energy, which is conducive to hindering crack propagation. The Q-P980 has a higher proportion of high-angle and general grain boundaries, making fracture propagation more difficult to inhibit. Under the influence of external force, cracks form and grow early, eventually leading to fracture.

4.3. Effect of hydrogen on fracture process

All types of fracture processes, according to in situ TEM observations, are dependent on dislocation emission and movement, i.e. the degree of stress concentration [41]. The atomic link is broken when \( \sigma > \sigma_C \), leading in fracture. In situ observation by TEM showed that all kinds of fracture processes were based on dislocation emission and movement, i.e. the degree of stress concentration [42]. When \( \sigma > \sigma_C \), the atomic bond is destroyed, resulting in fracture. If hydrogen is present, on the one hand, it can reduce the atomic bonding force (Hydrogen-Enhanced Decohesion, HEDE) [34, 35], and on the other hand, the shielding effect of hydrogen can promote the emission and movement of dislocations (HELP) [28], and when the emission and movement of dislocations reach the critical state, the local stress will be equal to the atomic bonding force reduced by hydrogen, resulting in the nucleation and propagation.

As a result, high-strength steel HIDF is linked to stress concentration and hydrogen diffusion enrichment. When the hydrogen concentration and stress value reach a threshold level, HIDF will occur [25]. \( C_{HH} \) and \( \sigma \) represent the local hydrogen concentration and local stress levels, respectively. We suppose that \( Z_C \) is a value connected to \( \sigma \) and \( C_{HH} \), and that it will rise as \( \sigma \) and \( C_{HH} \) rises. The material will fracture when \( Z_C \) reaches a particular value \( Z \). When \( Z \geq Z_C \), the atomic bonds at the crack tip, lattice position, and phase interface will fracture, promoting crack initiation and propagation.

With increasing steel strength, the concentration of hydrostatic pressure around the fracture tip or notch rises (yield strength) [43]. The local tension near the fracture lowers as the hydrogen concentration rises. Martensite has a high strength level and a lot of microstructure flaws, which causes a lot of lattice deformation and local stress. When \( Z \) is constant, the value of \( \sigma \) is excessively high, leading in a reduction in critical hydrogen content (CHC), making martensite extremely sensitive to hydrogen. And Q-P980 fracture occurred in only 10 min when the constant stress was 1024 MPa. It can also be judged that the CHC becomes very low under high
local stress. If there is not enough irreversible hydrogen trap to capture hydrogen, \( Z_c \) reached the critical value \( Z \) prematurely.

As previously stated, RA stability is intimately linked to the HE of steels. Thin film RA or fine RA between martensite laths had high stability [44]. It may be utilized as hydrogen traps to catch a significant quantity of hydrogen and efficiently lower the DH content, which helps to improve HIDF resistance. When the stability of RA is low, however, a considerable quantity of RA will strain into high-carbon martensite. High-carbon martensite, on the one hand, has a high HE sensitivity while having a limited capacity to dissolve hydrogen. The transformation from austenite to martensite, on the other hand, is a crystal lattice rebuilding process. A high number of dislocations and twins are formed in this process, and the amplified lattice distortion raises local stress. The combination of the above two aspects making \( C_H \) and \( \sigma \) both grow rapidly and reach the critical value prematurely, resulting in the initiation and propagation of cracks, and finally lead to HIDF.

5. Conclusions

1. The Q-P980 contains more than 9% RA, which has a very high hydrogen solubility and a very low hydrogen diffusion coefficient for hydrogen, it is more difficult for hydrogen to pass through the Q-P980.

2. The austenite in Q-P980 is massive RA with low stability. The TRIP effect will occur in the process of strain and change into high carbon martensite. HIDF is caused by a substantial amount of surplus hydrogen being enriched at the border and flaws. Under certain stress situations, Q-P980 has an extremely high HE susceptibility. The fracture has a broad cleavage surface and is a typical quasi-cleavage fracture.

3. MS980 has been sufficiently tempered, resulting in a substantial quantity of distributed spherical cementite (150nm) precipitating around the lath martensite. This size and form of cementite may successfully trap hydrogen while maintaining the material’s mechanical characteristics. MS980 has a very low HE susceptibility because tempering significantly reduces the local stress level of steel.

4. HIDF is related to local stress and hydrogen accumulation. Especially for dislocation strengthened steel, tempering to reduce the local stress and precipitation of carbides to capture hydrogen are the methods to reduce the susceptibility of HE. Adding microalloying elements can form precipitates that can act as hydrogen traps and hinder the HELP process, so as to reduce the local stress and the accumulation of hydrogen.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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