Toshan Prasad Chaturvedi, 2026, 14:6
ISSN (Online): 2348-4098
ISSN (Print): 2395-4752

International Journal of Science, Engineering and Technology
An Open Access Journal

Comparative Investigation of Zn, Ni and Zn–Ni Electroplated Coatings for Mitigating Hydrogen Embrittlement in DP980 Steel

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Abstract- Hydrogen embrittlement (HE) is a well-known problem in high-strength steels, particularly dual-phase (DP980) steel, as hydrogen absorption can significantly deteriorate its mechanical properties and compromise its structural integrity. In the present study, the effectiveness of zinc (Zn), nickel (Ni), and zinc–nickel (Zn–Ni) alloy coatings in mitigating hydrogen embrittlement was investigated by comparing their ability to restrict hydrogen penetration into the steel substrate. The coating thicknesses were measured using an Elcometer thickness gauge. The mechanical properties of uncharged, hydrogen-charged, and coated hydrogen-charged specimens were evaluated using a universal testing machine (UTM). Furthermore, scanning electron microscopy (SEM) was employed to examine the fracture surfaces and investigate the influence of hydrogen charging and coating application on the fracture characteristics. The results revealed that the Zn–Ni alloy coating exhibited superior resistance to hydrogen embrittlement compared with the individual Zn and Ni coatings under hydrogen-charging conditions, with improved mechanical properties (UTS and % elongation). The corresponding HEI value for the Zn-Ni alloy coating is 12.1%. This lower HEI indicates that the Zn-Ni alloy coating effectively reduces hydrogen permeation under hydrogen-charging conditions. This improved performance is attributed to the formation of a relatively dense Zn–Ni alloy coating, which acts as a barrier to hydrogen penetration and diffusion. The different electrochemical reactivities of Zn and Ni within the alloy coating may also contribute to modifying the interfacial electrochemical behaviour during hydrogen exposure, thereby limiting hydrogen ingress into the DP980 steel substrate. Overall, the findings demonstrate that Zn–Ni alloy coatings offer greater potential for mitigating hydrogen embrittlement in DP980 steel than individual Zn and Ni coatings.

Keywords- Hydrogen embrittlement; DP980 steel; Zn coating; Ni coating; Zn–Ni alloy coating; Hydrogen diffusion; Mechanical properties.

I. INTRODUCTION

Hydrogen Embrittlement (HE) is a phenomenon where the metal shows loss of ductility and premature failure due to the entry of hydrogen. This hydrogen can enter through various means, for example, through environmental exposure or during industrial machining processes [1]. This occurs with a combination of stress, which may be internal residual stress or externally applied. This phenomenon causes crack initiation, a reduction in load-bearing capacity, and sudden brittle failure below the yield stress [2,3]. This phenomenon is mostly seen in the automobile, oil and gas, and aviation industries. After the study, it was found that high-strength steels (> 1200 MPa) are most susceptible to HE. Materials susceptible to HE include high-strength steel, X60-90 hydrogen pipeline steel, and advanced high-strength steels such as dual-phase steel, TRIP steel, TWIP steel, and Martensitic steel [4,5]. Major factors responsible for HE in materials are stress either applied or residual, environment (must be hydrogen), material susceptibility, temperature, and pressure [6]. HE can occur through various mechanisms, including hydrogen-enhanced decohesion (HEDE), hydrogen-enhanced localized plasticity (HELP), adsorption-induced dislocation emission (AIDE), hydrogen-enhanced microvoid formation and coalescence (HEMP), and mixed-mode failure mechanisms [7-12]. However, the exact mechanism responsible for hydrogen-induced material failure is not always clearly identified. In

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many cases, multiple mechanisms may operate simultaneously or interact with one another, collectively contributing to the degradation of mechanical properties and ultimately leading to hydrogen-induced failure.

Various electroplating techniques, such as nickel (Ni), zinc (Zn), zinc–cobalt (Zn–Co), and nickel–chromium (Ni–Cr) coatings, have been investigated to develop protective barriers against hydrogen diffusion into metallic materials [13-18]. These coatings can reduce hydrogen permeation and consequently improve resistance to hydrogen embrittlement (HE). However, their effectiveness is not always consistent. While some coatings exhibit significant reductions in hydrogen permeation, others provide limited protection and may not effectively prevent HE. This variation depends on factors such as coating composition, microstructure, thickness, porosity, and the presence of defects, which influence hydrogen transport through the coating and its interaction with the underlying substrate [19-21].

In the present study, dual-phase DP980 steel was selected to investigate the effects of hydrogen embrittlement (HE) and evaluate the effectiveness of different surface coatings in mitigating hydrogen-induced degradation. To improve the resistance of the material to hydrogen ingress, zinc (Zn), nickel (Ni), and zinc–nickel (Zn–Ni) alloy coatings were deposited on the surface of the base material. The thickness of each coating was measured using an Elcometer coating thickness gauge to evaluate the deposited layer thickness. Furthermore, the mechanical properties of both uncoated (bare) and coated specimens were evaluated under uncharged and hydrogen-charged (HC) conditions using a universal testing machine (UTM). Scanning electron microscopy (SEM) was employed to examine the fracture surfaces and investigate the influence of hydrogen charging on fracture characteristics and microstructural features. These investigations aimed to assess the effectiveness of the different coatings in reducing hydrogen permeation, mitigating hydrogen-induced degradation of mechanical properties, and influencing the fracture behaviour of DP980 steel.

II. EXPERIMENTAL WORK

1. Material selection and sample preparation

The material used for the experimental inquiry is DP980 steel. It offers good machinability, high tensile strength, and wear resistance. DP980 steel is used in a variety of industrial components, including shafts, gears, and fasteners. Mechanical components may fail due to hydrogen embrittlement resulting from poor material selection. Minimum tensile strength of this material is 980MPa. The chemical composition of DP980steel is provided in Table 1. For the mechanical properties evaluation of the proposed steel, samples are prepared in accordance with ASTM E8. The geometric representation of UTM samples is shown in Fig. 1.

Table – 1 The Chemical Composition of DP980 (mass%)

Carbon	0.11
Silicon	1.0
Aluminum	0.015-1.0
Copper	0.2
Manganese	2.9
Chromium + Molydbenum	1.4
Phosphorus	0.05
Sulphur	0.01

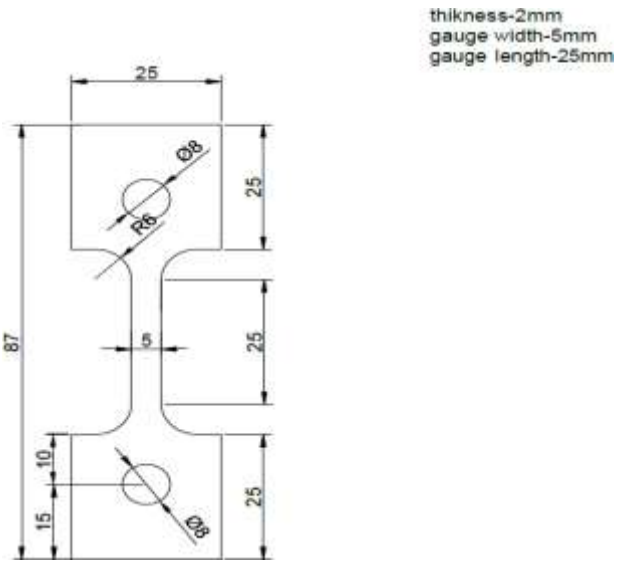

Fig. 1- DP980 sample dimension

2. Coatings and Thickness Measurement

Electroplating is a method in which materials are electrodeposited onto a substrate, usually a metal, by applying an electric current to a pair of electrodes immersed in an electrolyte. The anode was its positive terminal, whereas the cathode was its negative terminal. The electrolyte solution contains ions or electrically activating particles. When a DC voltage is applied between the two electrodes, the ions move toward the electrode with the opposite charge. Positive ions gravitate toward the cathode, while negative ions gravitate toward the anode, resulting in a current flow between the electrodes and the completion of an electrical circuit. Watts nickel plating, also referred to as Watts nickel plating, is primarily used for decoration and was invented by Professor Oliver P. Watts. Nickel sulfate ($NiSO4 \cdot 6H2O$) is used as an electrolyte in Watts plating. Zn plating is also used for to improve corrosive resistance. The composition of the nickel bath and Zn bath is represented in Table 2 and 3. The plating solution (alkaline) comprising Zn and Ni in a concentration ratio of 9-11:1 was used for the electroplating. The bath was composed of various constituents, with their concentrations as follows, Zn concentrate (9–12 g L-1), Ni concentrate (9–1.4 g L-1), $Na2CO3$ (56 g L-1), and NaOH (140 g L-1). The efficiency of plating bath was near to 80 %. The DP980 steel samples were subjected to various treatments before electroplating, such as degreasing, rinsing (water), abrasive grit blasting, and surface activation by acid.

Table 2 Chemical Composition of Nickel Electroplating

S.No.	Name of chemical component	Chemical formula	Quantity
1	Nickel sulphate	$NiSO4.6H2O$	150-300 gm/l
2	Nickel chloride	$NiCL2.6H2O$	60-150 gm/l
3	Boric acid	$H3BO3$	37-52 gm/l
4	Benzene sulphonic acid	$C6H6O3S$	0.75- gm/l

Table 3 Chemical Composition of Zinc Electroplating

s. no.	Name of chemical component	Chemical formula	quantity
1	Zinc sulphate	$ZnSO4$	115.0 gm/l
2	Sodium sulphate	$Na2SO4$	56.8 gm/l
3	Boric acid	$H3BO3$	24.7 gm/l
4	Sulphuric acid	$H2SO4$	1.13 gm/l

In this experiment, an ultrasonic-based device called the elcometer is used to measure coating thicknesses. An average coating thickness of 43 µm was observed in all coated specimens (Fig. 2).



Fig-2 Average coating thickness of Zn and Zn-Ni coated specimen by elcometer

3. Hydrogen Charging

Hydrogen charging is a technique for diffusing hydrogen into a material to study the effects of hydrogen embrittlement in steel. This procedure requires an electrolyte to carry out the

electrochemical reactions. Thiourea (NH2SCNH2) and distilled water, as well as concentrated sulphuric acid (H2SO4), are used to prepare the electrolyte solutions. The quantity of each component, as well as its chemical composition, is listed in the table 4 below.

Table 4 Chemical Composition of Hydrogen Charging Electrolyte

S.No.	Components in hydrogen charging solution	Quantity of component
1	Distilled water (H_2O)	1 liter
2	Thiourea (NH_2SCNH_2)	3-5 gram
3	Concentrated sulphuric acid (H_2SO_4)	27 ml

After making the electrolyte solution, pour it into the jar and provide 3.89 Volt and 0.35 Ampere DC current. Hydrogen charging was carried out for 12 hours in different coated and uncoated specimens. Fig. 3 represents the hydrogen charging setup for different HC condition

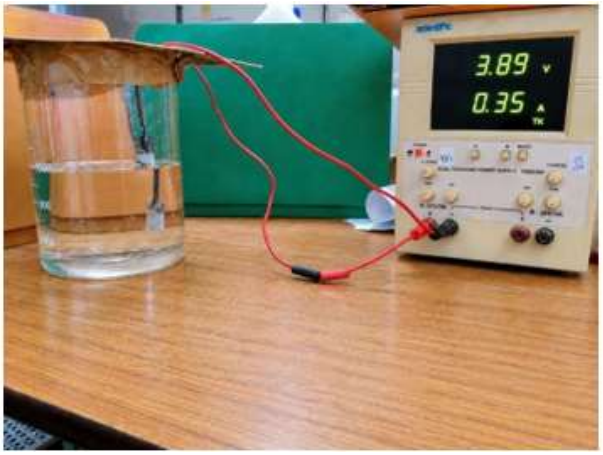

Fig. 3 hydrogen charging setup

4. UTM test

A universal testing machine (UTM) is a common method of evaluating materials, particularly metals, in which the specimen is stretched at a constant rate. The DP980 steel, a high-strength steel, was employed in the experimental inquiry because it is commonly used in the automotive sector and in other applications such as columns. It is more vulnerable to hydrogen embrittlement than other metals. For the tensile test, several types of electroplated specimens with hydrogen-charged and uncharged hydrogen are used.

5. SEM test

SEM is used to examine fractures. This study is performed to inspect the material's microstructure and characteristics. The investigation of material surface defects is also known as fractography. This approach is commonly used to determine the cause of engineering structural failure, especially in product failure and forensic or fault investigations. In materials science research, fractography is used to construct and evaluate theoretical models of fracture behavior. The goal of the fracture exam is to discover the reason for failure by examining the trend and modes of fracture surface.

III. RESULTS AND DISCUSSION

The tensile test is performed on the UTM system for the electroplated sample under two conditions, i.e., with hydrogen charge and without hydrogen charge, to examine the effect of hydrogen embrittlement.

Different electroplated specimens, namely Zinc, Nickel, and Zn-Ni DP980 specimens with hydrogen charge condition and uncharged specimens, were tested for tensile testing to determine the peak stress, yield stress, and percentage elongation till failure. Table 5 represents the comparison of test results in the mechanical properties (UTS, YS and % Elongation) of the hydrogen-charged and the uncharged sample to see the actual effect of hydrogen charging. The hydrogen embrittlement index (HEI) was calculated to assess the effect of hydrogen on the material's susceptibility to embrittlement. A low Hydrogen Embrittlement Index (HEI) signifies that the material is less susceptible to hydrogen embrittlement, whereas a high value of HEI indicates that the material is very susceptible to HE. Variation in mechanical properties in different operating conditions (uncharged, hydrogen charged and coated hydrogen charged) is shown in the stress-strain curve below in Fig. 4

$$\mathrm{HEI} = \frac{El(un) - El(HC)}{El\ (un)}$$

Where $El(un)$ represents the percentage elongation in uncharged condition (bare specimen) and $El(HC)$ represents the percentage elongation in the hydrogen-charged condition.

Table 5 UTM Test Results of Charged and Uncharged Sample

S. No.	DP980 steel sample condition	Ultimate Tensile Stress (UTS)	Yield Stress	Elongation at Break (%)	Hydrogen Embrittlement Index (HEI)
1	Bare uncharged sample	1052.7 MPa	927 MPa	15.7 %	
2	Bare HC sample	930.5 MPa	823 MPa	9.8 %	37.9
3	Zinc coated HC sample	948.2 MPa	811.4 MPa	10.7 %	31.8
4	Nickel coated HC sample	946.8 MPa	817.8 MPa	11.6 %	26.1
5	Zn-Ni coated HC sample	985.7 MPa	845.7 MPa	13.8 %	12.1

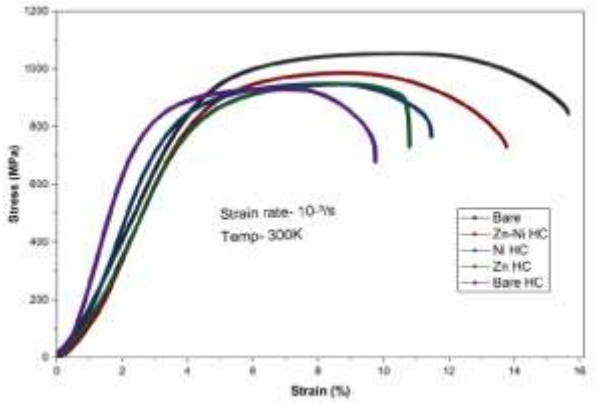

Fig.- 4 Comparison of Stress vs Strain Curve for Bare and Coated HC specimen

The influence of hydrogen on mechanical properties is investigated using a UTM test after hydrogen charging; we observe significant changes across the graphs. Hydrogen diffuses into the bare hydrogen-charged specimen when it is hydrogen-charged in a 0.5 M H2SO4 solution at a current density of 100mA/cm2 for 12 hours, and the mechanical properties (UTM and % elongation) are reduced, as mentioned in Table-5 and shown in Fig. 4. High value of HEI (37.9%) suggests the DP980 steel material's susceptibility to HE.

The mechanical properties of Zn and Ni-coated hydrogen-charged specimens were not significantly improved; only minor improvements were observed with HEI of 31.8% and 26.1%, respectively. This high value of HEI suggested that the Zn and Ni coatings were unable to create a barrier to hydrogen penetration during hydrogen charging. Fig. 4 also suggests a minor improvement in UTS and percentage elongation of Zn- and Ni-coated HC specimens. But in both Zn- and Ni-coated HC specimens, necking starts at a later stage of the specimen as compared to the early-stage necking just after the UTS in the bare HC specimen.

The effect of hydrogen on the mechanical properties of Zn-Ni-coated hydrogen-charged specimens was investigated by UTM and shown in Fig. 4. The improved necking behavior in the Zn-Ni-coated HC specimen was observed, with improved mechanical properties and lower HEI (12.1 %).

Thus, it was concluded from the experimental results that hydrogen charging diminishes the ductility of the material and increases the brittleness of the high-strength steel. Zn-Ni coating mitigates hydrogen penetration and creates resistance to hydrogen transport owing to the relatively low hydrogen diffusivity in nickel compared with ferritic steel.

Fracture surface examination of the bare (uncharged) specimen was characterized by SEM, confirming a classic ductile dimple rupture mode, traditionally identified as a cup-and-cone fracture when tested in air, as shown in Fig. 5(a). Fractography of the bare specimen suggested that the initial crack began from the center region and spread towards the outer surface.

SEM observation shows the structure of a broken tensile specimen after 12 hours of hydrogen charging. The microstructure is exceptionally smooth and transparent when viewed at high magnification. In all of these illustrations, two types of fractures occurred: ductile and brittle. The outer region of the bare HC specimen shows a brittle fracture mode with quasi-cleavage development, while the central region shows a deep central crack, which suggests that during hydrogen charging, hydrogen diffuses from the surface to the center of the specimen and embrittles the material (Fig. 5b).

Figures 6(a) represents the fractography of the zinc-coated hydrogen-charged specimen. From the fractography, it is clearly visible that the crack is at the corner of the specimen, suggesting that hydrogen diffuses from the Zn coating into the base DP steel. Cleavage development is also observed in the images, suggesting a change in fractography from ductile to brittle due to hydrogen diffusion. It was also concluded from the fracture images that hydrogen can penetrate from the Zn coatings and change the fracture mode of the material from ductile to brittle.

Figure 6(b) shows the fractography of the nickel-coated hydrogen-charged specimen. Although Ni has low hydrogen diffusivity, hydrogen still diffuses from the surface to the center of the specimen, thereby reducing mechanical properties and altering the material's fracture mode. Dimples were shallower due to the effect of hydrogen in the Ni-coated, hydrogen-charged specimen. Mixed-mode fracture was observed in Ni-coated, hydrogen-charged fracture images, suggesting that the fracture surface was altered and that hydrogen has penetrated from the Ni coating.

The fracture image of the Zn-Ni-coated sample shows a shallower dimple near the surface and a dimple rupture in the central region, suggesting that the Zn-Ni coating reduces hydrogen penetration and is effective in a hydrogen atmosphere, as shown in Fig. 6 (c). The Zn-Ni-coated HC fracture specimen represents a fracture characteristic similar to that of the bare uncharged specimen (Fig. 5a). Ni has a low hydrogen diffusion coefficient, and a dense Zn-Ni alloy coating creates a barrier for hydrogen diffusion due to different electrochemical reactivities when the Zn-Ni-coated specimen is exposed to a hydrogen environment. Zn-Ni coating acts as an entry barrier by modifying the surface kinetics of hydrogen adsorption, absorption and recombination.

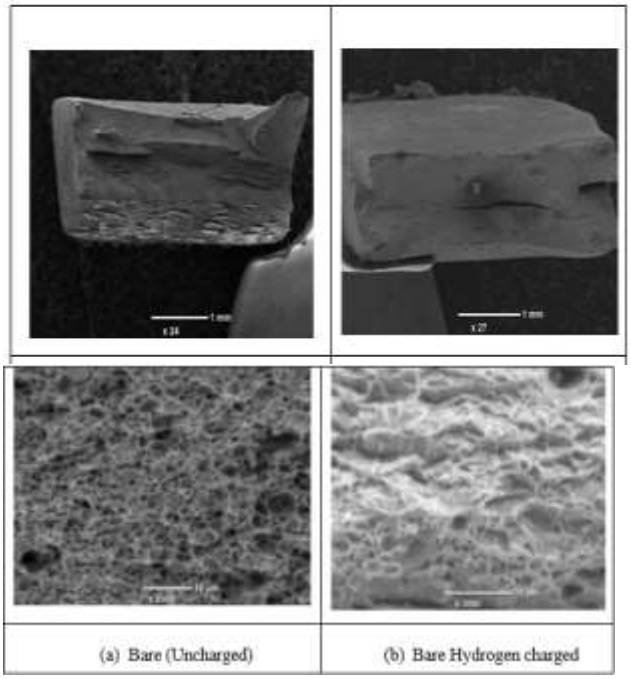

Fig. 5 Fracture surface examination of Bare and Bare HC specimens

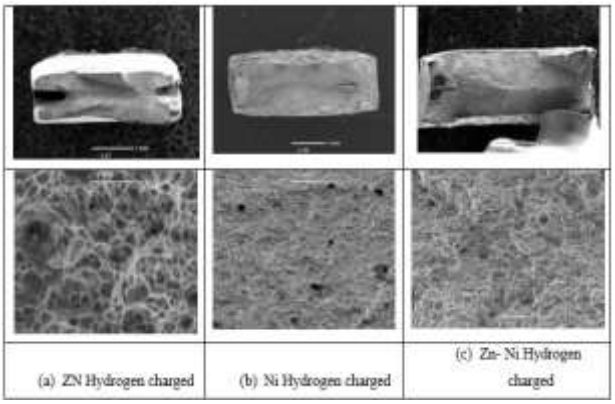

Fig. 6 Fracture surface examination of Zn, Ni, and Zn-Ni coated specimens

IV. Conclusion

The process of diffusing hydrogen into a material, resulting in hydrogen embrittlement. Tensile testing

before and after hydrogen charging was performed to investigate the effects of hydrogen charging on the mechanical characteristics of several types of electroplated DP980 samples. With electroplating, the mechanical properties improve compared to the bare hydrogen-charged sample. Zn and Ni electroplating are less effective than Zn-Ni alloy electroplating, which yields better mechanical properties (UTS and % elongation) and lower HEI compared to a hydrogen-charged bare sample. The stress-strain diagrams in the results and discussion section clearly show that the hydrogen-charged samples fail more quickly and at a lower UTS than the hydrogen-uncharged samples in the tensile test. Basically, hydrogen charging lowers the ductility of the bare HC high-strength steel and increases its brittleness. Several important findings are summarized below.

- Universal tensile strength (UTS) and percentage elongation of DP980 steel decreased from 1052.7 MPa to 930.5 MPa and from 15.7% to 9.8 % in the bare uncharged condition to the bare hydrogen-charged condition, respectively. It was also concluded that the susceptibility of DP980 steel towards HE was increased during hydrogen charging, with a corresponding hydrogen embrittlement index (HEI) of 37.9%.
- Zn and Ni coatings are electrodeposited on DP980 steel samples to prevent hydrogen infusion in the mentioned steel. The experimental results showed that mechanical properties (UTS and % elongation) were not significantly improved by these electroplating processes. Only small improvements were observed in mechanical properties. The hydrogen embrittlement index (HEI) values for Zn- and Ni-coated hydrogen-charged specimens were 31.8% and 26.1%, respectively. Although the HEI value decreased compared to the bare hydrogen-charged specimen (37.9%), but only a small reduction in HEI was observed.
- Zn-Ni alloy coating showed better results compared to Zn and Ni-coating under the same hydrogen-charging condition or operating condition. UTS and % elongation of Zn-Ni alloy coatings were 985.7 MPa and 13.8 %, which is higher than those of the Zn-coated and Ni-coated hydrogen-charged specimens. The corresponding HEI value for the Zn-Ni alloy coating is 12.1%. This lower HEI indicates that the Zn-Ni alloy coating is effective in reducing hydrogen permeation under hydrogen-charging conditions.
- Zn-Ni coating acts as an entry barrier by modifying the surface kinetics of hydrogen adsorption, absorption and recombination. This dense, continuous coating can reduce the effective pathways for hydrogen transport, diminish hydrogen-metal interactions, and create a barrier for hydrogen diffusion and embrittlement. Ni has a low hydrogen diffusion coefficient, and a dense Zn-Ni alloy coating creates a barrier for hydrogen diffusion due to different electrochemical reactivities when the Zn-Ni-coated specimen is exposed to a hydrogen environment.
- Fractography analysis also suggested that the bare HC specimen failed in a brittle manner, as hydrogen diffuses from the surface to the center of the specimen, and a center crack was seen in fractography, whereas the bare uncharged specimen showed a dimple rupture characteristic.
- Scanning electron microscopy (SEM) analysis of the fracture surface of Zn-coated and Ni-coated HC specimens shows cracking at the corner and shallower dimples, which suggests that hydrogen diffuses from these coatings and changes the fracture mode.
- Fracture characteristics of Zn-Ni-coated HC specimens show characteristics similar to those of bare, uncharged specimens. Dimples were uniformly distributed. Dimple rupture was observed in the central region of the Zn-Ni-coated hydrogen-charged fracture specimen, which suggested that hydrogen atoms could not permeate from the surface to the central region while hydrogen charging was performed. However, the Zn-Ni alloy coating cannot completely eliminate hydrogen diffusion.

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