

Hydrogen Embrittlement Prevention in High Strength Steels by Application of Various Surface Coatings-A Review



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Abstract The applicability of various coatings such as graphene, reduced graphene, Al, Cu, Zn, carbon, nitrogen, oxygen, niobium, boron nitride, and TiAlN as a protective barrier to high strength steel for hydrogen embrittlement was studied. These all coatings were applied by different coating techniques such as (chemical vapor deposition (CVD), electroplating discharge (EPD), electrolysis, gas diffusion, plasma diffusion, high velocity oxygen fuel (HVOF), magnetically enhanced plasma ion plating system, plasma vapor deposition, and ion beam sputter.) on the high strength steel substrate followed by characterization of applied coating and mechanical testing. Reduction in life cycle due to the hydrogen embrittlement was analyzed.

Keywords Hydrogen embrittlement · Graphene coating · Scanning electron microscopy

1 Introduction

The phenomenon of hydrogen embrittlement (HE) will occur when metal surfaces are exposed to hydrogen environment which leads to catastrophic failure. It was found that HE considered as an important problem in several application areas, such as nuclear plant reactor vessel [1, 2], high-pressure gaseous hydrogen storage tanks [3, 4], petroleum and natural gas pipelines [5, 6].

Generally, when metal comes to contact with the hydrogen or working in hydrogen atmosphere, then some common ways by metals failed are hydrogen embrittlement (HE), hydrogen-induced-blistering, hydrogen attack, cracking due to precipitation of internal hydrogen, and hydride-formation cracking [7]. Generally, there are two mechanisms which responsible for the HE in steels. First one is that at cracks or crack tip, hydrogen atoms are accumulated, and decreases the fracture energy while inspiring cleavage-like failure [8, 9]. The second mechanism includes mobility of

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dislocations increased due to effect of hydrogen at crack tip through shielding effect, reducing the shear strength, and ultimately responsible for local plasticity enhancement [10]. In order to protect the equipment from the hydrogen diffusion, it is necessary to measure how much concentration of hydrogen has been diffused into the material at the time of failure, but threshold concentration of hydrogen which is causing the equipment failure is not found clearly. And the basic mechanism which is responsible for the HE is also not found clearly still some discussion is going on this [11].

It was found that hydrogen diffusion causes mechanical properties broadly tensile strength and fatigue strength are reduced with and without changes in yield strength. Temperature, source of hydrogen, and surface condition are responsible for HE [12]. Various methods which were responsible for hydrogen entrance in the material are cathodic reaction or charging, electroplating, during welding, etc. [13]. Literature said that the cathodic charging plays a very significant role for hydrogen diffusion and current density considered as an important factor for hydrogen absorption and propagation in steel. For hydrogen diffusion and embrittlement occurrence in steel, current density must be in range of 0.02–40 mA/cm² [14, 15]. It was concluded that at higher current density, hydrogen absorption and propagation are faster than the lower current density, so more hydrogen is diffused at higher current density.

In this paper, the prevention of HE in high strength steels by application of different surface coatings such as graphene, reduced graphene, Al, Cu, Zn, carbon, nitrogen, oxygen, niobium, boron nitride, and TiAlN, etc., is discussed and selection of suitable coatings is given to reduce the HE phenomena.

2 Hydrogen Embrittlement in Steel

High strength steels are mostly subjected to hydrogen embrittlement effect. The embrittlement refers to change from ductile phase to brittle phase. Hydrogen caused the embrittlement is termed as “hydrogen embrittlement.” HE can be noticeable in different ways. HE causes reduction in the following

- Ultimate-tensile strength and braking strain or ductility
- Fracture strength and toughness.

Furthermore, there is a case where unstressed components are subjected to failure. However, there is a case in which ductility will decrease with no change in tensile strength [16]. Examples of internal hydrogen embrittlement is solutions used during fabrication like acid cleaning, electroplating, pickling, and providing protective coatings, phosphating, etching, paint stripping, etc. Second way of HE is external hydrogen embrittlement which occurs during the working condition of equipment. The following mechanisms by which hydrogen is entering and trapped in the lattice are

- Hydrogen-enhanced decohesion mechanism (HEDE),

- Hydrogen-enhanced local plasticity model (HELP), and
- Adsorption-induced dislocation emission (AIDE) [7, 17, 18].

After entering into the metal lattice hydrogen will be trapped in different regions. Examples of hydrogen traps are dislocations, grain boundaries, voids, and phase boundaries, micro cracks, precipitates, interfaces, solutes, surface cracks, surface oxides, and 0D, 1D, 2D lattice defects. In last 50 years, various authors focus on research related to hydrogen diffusion and hydrogen embrittlement (HE) [19–21]. Hydrogen causes slip localization [22–24], softening and hardening [24–32], interaction between hydrogen–dislocation [32–35] and creep [36] have been also reported, and apart from all these effects, HE has severely effect fatigue crack rate behavior as well as tensile properties.

3 Hydrogen Embrittlement Prevention

Complete prevention of HE in high strength steels is laborious but we can prevent some extent so that we can save the life of the equipment. To reduce the hydrogen diffusion from metal surface, sharp and very steady variation, notches must be prevented or avoided and removal of residual stresses are performed before the processing step [37, 38]. Sometimes, baking operation is performed to remove hydrogen which was available in lattice or surface during the processing. In broad view, baking is considered as a heat-treatment-process and processing temperature is depending on the process material [39]. Prevention from HE can also be achieved by adding specific alloying material to the base or parent material [7].

Our main focus on prevention of HE by surface coatings, surface coatings can be done by different ways. Very well-known methods are chemical vapor deposition, physical vapor deposition, electroplating, plasma vapor deposition, gas diffusion, etc.

3.1 HE Prevention by Reduced Graphene Oxide (RGO) Coating

Graphene is most suitable element to reduce the HE from high strength steel materials. It has a thin film of carbon atom which has some unique properties [40–42]. Its chemical inertness properties make him to use at various operating condition. It is also stable in atmosphere condition temperature to 400 °C and reduce the oxidation of the substrate material because of hydrophobic characteristic or property [43], and that is due to its non-polar covalent double bonds, which is responsible for prevention of hydrogen atom bonding with the water [44, 45]. That is the reason, we are selected the graphene material as a coating due to its light weight and halt charge transmit at the metal–electrolyte interface. Thus, most of the researchers have studied and selected

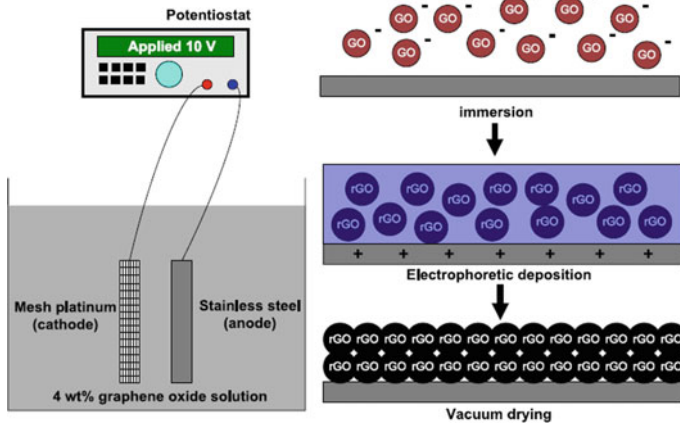


Fig. 1 rGO deposition in the stainless-steel substrate by using EPD technique [47]

the graphene as the coating material [46]. However, graphene coating can diminish corrosion properties of stainless steel as it is coated by CVD. Graphene has been replaced by reduced graphene oxide (rGO) because of its high-volume, low in cost, and processing capabilities at room temperature. One of the economical methods to develop rGO on stainless steel is electrophoretic-deposition method (EPD).

Kim and Kim [47] explained the effect of reduced graphene oxide coating to reduce HE in stainless-steel material. They used the electrophoretic-decomposition technique to coat the rGO above the substrate material. Evaluation of mechanical properties was done by slow strain rate test (SSRT) during hydrogen charging condition [47].

Synthesization of purified graphene to graphene oxide (GO) was done by modified-hammers method [48]. After this synthesization, GO was spreaded out in water and sonicated at room temperature for 4 h in 4 mg/ml concentration solution. After preparation of GO solution, electrophoretic-deposition technique was used at 10 V DC current supply to deposit rGO. For achieving required thickness of rGO above the substrate material time required to be 8 min. After completion of deposition process, samples were taken in vacuum for drying to prevent from oxidation [47].

A below representation showing electrophoretic-deposition process for the establishment of reduced graphene oxide on the stainless-steel substrate material is illustrated in Fig. 1.

3.1.1 Characterization of Sample

The investigation of the rGO in surface and cross-section of substrate material (stainless steel) was done by scanning electron microscopy (SEM).

3.1.2 Electro Chemical Measurements

Electro chemical behavior of rGO on stainless-steel specimen was analyzed using the conventional three electrode cell. Cathodic-polarization experiment or examination and electrochemical-impedance spectroscopy were conducted on rGO. Initially, 0.5 M sulfuric acid solution (H_2SO_4) with 250 mg/L arsenic trioxide (As_2O_3) (pH = 1.0) was prepared and samples were kept in that solution for 2 h. So that hydrogen diffusion and penetration were taken place easily. Nitrogen gas flows through the solution to remove the dissolved oxygen for 2 h and then measurement was done [47].

3.1.3 Slow Strain Rate Tests (SSRT)

SSRT have been widely applied to environmental induced cracking like corrosion cracking and HE. In this case, SSRT testing was performed at strain rate of $1.0 \times 10^{-6}/s$ to evaluate the effect of rGO deposit or coating to reduce the hydrogen embrittlement effect in stainless-steel material. Here, HC was done at a negative current ($-0.5 A/cm^2$) for investigation of behavior or effect of HE in coated and bare samples by SSRT testing.

During the testing, it was found that the mechanical properties like the yield, tensile stresses, and strain, of stainless-steel specimen in hydrogen charging condition are reduced. After applying the rGO coating to stainless steel specimen, it was found that the mechanical properties of coated sample return back to initial value of uncharged specimen. Elongation behavior of hydrogen-charged specimen was also affected and reduced from percentage elongation of 40.1% to the value of 23.5% for uncharged to hydrogen-charged specimen. While elongation percentage of rGO-coated hydrogen-charged specimen comes around 35.6%. So, effect of HE in coated sample was reduced and these coating acts a barrier for hydrogen diffusion in stainless steel [47].

3.2 *Coating of Zn, Ni, Cu, Al, PVD-Ti-DLC, Carbon, Nitrogen, and Oxygen Diffusion Layers*

Electroplating of Zinc (Zn), Nickel (Ni), Copper (Cu), Aluminum (Al), PVD-Ti-DLC, various diffusion layers such as oxygen, nitrogen, and carbon, and electroless NiP coatings are examined and evaluated to reduce the effect of HE in 304 austenitic stainless-steel material. SSRT testing was performed for mechanical properties estimation [17].

Metastable grades like AISI 304 material rigorously experience the HE effects because of austenite to ferrite phase transformation at the time of operation [49–51].

Table 1 Overview of various coatings [62]

Coating material	Coating type	Deposition technique	Thickness μm	Hardness	Adhesion acc [HF]	Remarks
Al	On top	Electroplating	22	15–25 HB	4	Globular structure
Cu	On top	Electroplating	2	50–110 HB	1	Thin coating with pin load
Ni	On top	Electroplating	12	510 HV	1	Ni adhesion coating
Zn	On top	Electroplating	11	92 HV	2	Ductile zinc coating
NiP	On top	Electroless	10	550 HV	2	Amorphous NIP type coating
Ti-DLC	On top	PVD	3	1440 HV	1	Ti adhesion coating
Carbon	Diffusion	Gas diffusion	25	1225 HV	1	Long term gas diffusion
Nitrogen	Diffusion	Gas diffusion	500	300 HV	1	Small term gas diffusion
Oxygen	Diffusion	Plasma diffusion	1	1200 HV	2	Oxidizing at -550°C

Calculation of HV was done by some formula $\text{HB} = 0.95 \text{ HV}$

Zn, Ti-DLC, Al, Cu, and the oxygen diffusion layers as well as amorphous Nip coatings were examined because of their unique quality of low hydrogen diffusion coefficient [52–61].

Two AISI 304 stainless steel of internal heat number 40, 4 were used as a substrate material for testing various coatings and diffusion layers. The coatings properties were illustrated by metallo-graphic cross sections, Vickers and Rockwell testing, and X-ray diffraction (XRD) technique was used for internal stresses and phase analysis [62]. A simple coating and some diffusion layer properties are given in Table 1.

Cylindrical specimen is having an outer diameter 8 mm, notch diameter 6 mm, radius on notch is 0.2 mm, and angle of notch is 35° . Stress concentration factor is 3.4. Fatigue testing was performed in that specimen and operated in atmosphere of hydrogen (10 MPa) until the specimen has broken or failure occurs.

3.3 Niobium Coating on API 5CT P110 Steel

In gas and oil industry, the components are subjected to extreme conditions and aggressive environment. The corrosion phenomena in API 5CT P110 steels is very

Table 2 Niobium coatings spraying parameter [83]

Parameters used	
Flow rate of oxygen (L/min)	164
Flow of propane (L/min)	144
Compressed air flow (L/min)	181
Nitrogen flow—carrier gas (L/min)	290
Gun to substrate distance (mm)	300
Power feed rate (g/min)	41
Speed of Gun transverse (m/s)	0.2
Number of passes	5

fast [63] and HE occurs in that steel due to working in aggressive condition [64]. By application of coating in P110 steel, they exhibit a good corrosion resistance and also work effectively in hydrogen environment as they show resistance for, HE [65].

Thermal spray coatings have received very good attention for protection of oil and gas industry components against HE [66–69]. These kinds of coatings have growing and expanding application in protection against fatigue operation [70] and oxidation process [71–73] and economical and very efficient choice to meet all requirement [74]. Recently, various authors are working in development of nanostructured coating which have better and superior properties [75, 76]. Other coating techniques such as high velocity oxygen fuel (HVOF) is also very efficient in case of corrosion in API 5CT P110 steel [77].

Niobium has very good corrosion resistant in various media [78, 79], and refractory properties [80, 81]. HVOF coating technique was used to deposit niobium in API 5CT P110 steel as it acts as a protective film over the base material against the corrosion.

The parameters which are responsible for spraying process are depicted in Table 2. The surface roughness of coating substrate was measured by contact profilometer in which by Ry values were obtained. The microhardness of both materials was obtained by microhardness tests and this hardness test was performed at the 3 N load and 15 s holding or processing time, as specified and given in the ASTM standard [82].

4 Conclusions

- i. Hydrogen embrittlement resistance of rGO coating in stainless steel was examined and investigated by Kim and Kim. Their results showed that rGO-coated samples have less tendency to absorb the hydrogen than the uncoated sample.
- ii. Resistance for hydrogen was increased by rGO coating because of C-H bond formation and increase the diffusion length, during the charging condition. So

it is clear that rGO can act as a barrier for hydrogen penetration and acts as a resistance for, HE.

- iii. During hydrogen charging condition in coated samples, some surface irregularities, defects such as pores, coating and surface interface act as a hydrogen accumulator and hydrogen adsorbed sites. So, these sides inhibiting the entry of hydrogen from coated sample APT CT P110 steel. It is concluded that hydrogen-trapping capacity of coated sample was 7.5 time more than the normal steel specimen.
- iv. Thermal spraying coating of niobium using HVOF also reduces the susceptibility of hydrogen embrittlement and act as barrier for hydrogen diffusion.
- v. Electroplating of Zn, Cu, Ni, Al, PVD-Ti-DLC and electrolysis NiP coatings along with oxygen, nitrogen, and carbon diffusion layers was examined for reducing the susceptibility of HE. It was found that carbon and nitrogen diffusion layers work effectively to reduce hydrogen diffusion.
- vi. It was found that very little work is carried out for tensile and ductility improvement when worked on hydrogen atmosphere. The Ni and C diffusion layers reduced crack propagation in hydrogen atmosphere.

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