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Electroplating of reduced-graphene oxide on austenitic stainless steel to prevent hydrogen embrittlement

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ABSTRACT

In this study, reduced graphene oxide was deposited on stainless steel by an electroplating method and investigated for application as a protective barrier against hydrogen embrittlement. After electroplating, the reduced graphene oxide on stainless steel was evaluated via scanning electron microscopy and infrared spectroscopy. A thick and rough reduced graphene oxide layer was formed on the substrate, and removal of the oxidation product from the graphene oxide was confirmed by infrared spectroscopy. Electrochemical methods (i.e., potentiodynamic polarization tests and electrochemical impedance spectroscopy) and slow strain rate tests were performed to analyze the hydrogen embrittlement resistance. The hydrogen reduction reaction was decreased and a durable protective layer was formed after formation of the reduced graphene oxide. The decreased stress and strain under hydrogen conditions were ameliorated under hydrogen reduction conditions by the formation of reduced graphene oxide; this was caused by the formation of hydro-generated graphene and the increased hydrogen diffusion length.

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Introduction

Exposing metal surfaces to hydrogen conditions or reduction reactions can cause hydrogen embrittlement (HE), which can induce catastrophic failure [1]. HE has received a large amount of attention in various fields such as pipelines for natural gas and petroleum [2,3], nuclear power plants [4,5], and high-pressure hydrogen storage tanks [6,7]. The major mechanisms of HE in metals are related to a decrease in the fracture energy, caused by the presence of atomic hydrogen at the crack tip, and an increase in the dislocation mobility caused by the presence of hydrogen atoms, which increases the local

plasticity [8,9]. To prevent the latent risks of HE, various methods have been suggested; these include heat treatments [10], inhibition of HE [11], and protective barriers of zirconium dioxide [12]. However, the drawbacks of these methods include the usage of toxic substances, low efficiencies, and deleterious effects on the substrate material. Thus, development of advanced methods that can diminish hydrogen penetration is required.

Graphene is a 2D, atomically-thin film of carbon atoms that has unique characteristics [13–15]. Graphene has remarkable chemical inertness and is stable in ambient atmospheres up to 400 °C. Also, its membrane can be impermeable to gas molecules [16] and inhibits the oxidation of underlying metal

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substrates due to its hydrophobic nature, which is a result of its non-polar covalent double bonds, which prevent hydrogen bonding with water [17,18]. In this context, if graphene is used as a coating material on metals, it can provide an extremely lightweight coating and halt charge transfer at the metal-electrolyte interface (if the coating is continuous and ionically impermeable). Thus, many researchers have studied graphene as a coating material [19–23]. Additionally, graphene coatings formed by chemical vapor deposition (CVD) on copper have been shown to improve the HE resistance [24].

However, depositing graphene coatings via CVD can influence the metal properties due to the high temperature of the procedure. In particular, in the case of stainless steel, the corrosion properties are diminished after graphene coating via CVD due to the formation of metal carbides. Although this process can be improved by nickel plating stainless steel, these additional processing steps are not economical for real-world applications [25]. Recently, reduced graphene oxide (rGO) has been applied as an alternative to depositing graphene by CVD; rGO is appealing due to its low cost, high volume, and room-temperature processing capabilities. One of the methods used to generate rGO is the electrophoretic deposition (EPD) method, which is a well-developed and economical method that has been successfully applied for the deposition of carbon nanotubes to form transparent conducting films and phosphors for displays [26–28]. In a previous study, rGO was deposited on stainless steel by an EPD process [29]. There is no adhesion after the deposition and reduction of graphene oxide, and it is produced without harsh and toxic chemicals at room temperature. This method has a variety of potential applications due to its many advantages including its rapid, high-yield, large-area, low-cost, and environmentally-friendly process. Thus, this approach can be utilized as a graphene-coating process to improve the HE resistance of stainless steel in applications that require room-temperature processing and complex surface coatings.

In this study, the influence of rGO (as a protective barrier) on the HE resistance of the stainless steel was investigated. rGO was coated onto a stainless steel substrate by EPD. The electrochemical behavior of rGO on the stainless steel substrate was measured by potentiodynamic polarization tests in the cathodic region and by electrochemical impedance spectroscopy. Also, the reduction of graphene oxide was assessed by Fourier transform infrared (FT-IR) and X-ray photoelectron spectroscopy (XPS). The mechanical properties of the specimens were evaluated by slow strain rate tests (SSRTs) under tensile stress with simultaneous hydrogen charging to accelerate the hydrogen reduction reaction on the surface.

Materials and methods

Preparation of specimens

The graphene oxide (GO) used in this study was synthesized from purified graphite using the modified Hummers' method [30]. GO was dispersed in water and sonicated for 4 h at room temperature, and the concentration of the solution was 4 mg/mL. The 316 austenitic stainless steel was used in this study. After preparing the GO solution, the EPD process was

conducted under a direct current (DC) voltage of 10 V, and the deposition time needed to deposit a sufficient thickness of rGO was 8 min. Graphene oxide migrated toward the positive electrode due to the negative charge of the graphene oxide. After deposition, the specimens were dried in a vacuum chamber to prevent additional oxidation. A schematic showing the EPD process for the formation of rGO on the stainless steel substrate is illustrated in Fig. 1.

Characterization of reduced graphene oxide

To investigate the rGO on the stainless steel substrate, the surface and cross section were observed by scanning electron microscopy (SEM). Also, the removal of the oxidation domain from graphene oxide, including deprotonated carboxylate groups and carbon to oxygen atomic ration (C/O ration) were confirmed by FT-IR and XPS analyses and compared with filtered graphene oxide.

Electrochemical measurements

To analyze the electrochemical properties of rGO on the stainless steel substrate, cathodic polarization tests and electrochemical impedance spectroscopy (EIS) were carried out using a conventional three-electrode cell. The test specimen was used as the working electrode, a saturated calomel electrode was used as the reference electrode, and a graphite rod was used as an auxiliary electrode. All of the electrochemical measurements were conducted using a multi-potentiostat/galvanostat model VSP-300 after the samples were immersed for 2 h in a solution of 0.5 M sulfuric acid (H_2SO_4) + 250 mg/L arsenic trioxide (As_2O_3) (pH = 1.0) to allow for easy penetration and diffusion of hydrogen atoms into the specimens. As_2O_3 is an arsenic-based poison that diminishes H_2 formation and increases hydrogen adsorption on the surface, and is used to increase hydrogen reaction on the surface. Before measurements, nitrogen gas was put into the solution for 2 h to exclude the influence of oxygen; these conditions were maintained during the measurements. Cathodic polarization tests were performed at a scanning rate of 0.166 mV/s from +100 mV vs. open-circuit potential (OCP) to $-2000 \text{ mV}_{\text{SCE}}$. EIS measurements were conducted using a potentiostat under OCP with a sinusoidal amplitude of 10 mV in the frequency range from 100 kHz to 1 mHz. Impedance plots were interpreted on the basis of an equivalent circuit using a suitable fitting procedure using the ZsimpWin software.

Slow strain rate tests (SSRTs)

To evaluate the effect of rGO deposition on the resistance to HE of stainless steel, slow strain rate tests (SSRTs) were carried out at a constant strain rate of 1.0×10^{-6} /s. SSRTs have been widely applied to evaluate the properties of environmentally-induced cracking (i.e., stress corrosion cracking and HE) [31,32]. Fig. 2 shows a schematic of the SSRT equipment. A plate-type tensile specimen was machined according to ASTM standard E8; this had a gauge length of 25.4 mm and a thickness of 2.0 mm. Except for the gauge length portion, the tensile specimen was coated with insulating lacquer and rubber silicon to ensure that the immersed

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