

Electrochemical hydrogen permeation measurement through high-strength steel under uniaxial tensile stress in plastic range

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In the electrochemical hydrogen permeation test for high-strength steels under plastic loading, the failure of palladium coating has raised controversy over the reliability of the permeation data. To avoid rupture of a palladium layer under plastic stress, a new permeation technique was employed. This study clearly proves that the palladium film obtained by the new technique leads to a highly stable background current even under severe stress conditions, ensuring the reliability of the permeation data under both elastic and plastic stress.

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The hydrogen permeation behavior through steel has been electrochemically measured mainly by using the Devanathan–Stachurski cell [1]. This method has been successfully used by plating palladium (Pd) on the hydrogen exit side of the steel membrane. The thin layer of Pd coating on the steel surface improves the hydrogen oxidation, which ensures a stable hydrogen permeation current [2–6]. Therefore, the coating property of Pd is an essential factor in the success of the hydrogen permeation test for steel products. For a steel membrane with no externally applied stress, there is no difficulty in performing the hydrogen permeation test. However, for a steel membrane under externally applied stress, the reliability of the hydrogen permeation test depends greatly on the stability of the thin Pd coating layer, since the stability of that layer changes with the strength and elongation level of the steel substrate. When a Pd-coated steel sheet is subjected to externally applied stress above the yield strength (YS) of Pd (<205 MPa), the thin layer of Pd coating is plastically deformed and damaged, resulting in an unstable background current density in the permeation test.

Many studies have been undertaken to measure the hydrogen permeation current for various metallic materials under tensile stress within their plastic range [7–10]. For metallic materials that have yield strengths less than

that of Pd, such as pure iron and nickel of commercial purity, a stable and reliable hydrogen permeation current can be measured since the stability of the Pd coating layer can be maintained within such stress ranges. However, for high-strength steels with yield strengths much higher than that of Pd, the reliability of the permeation data cannot be guaranteed because of the failure of Pd coating layer. Kurkela et al. [11] measured the hydrogen permeation current for a Cr–Mo steel (YS = 496 MPa) within its plastic range by using the conventional Pd coating method and permeation test procedure. They reported current spikes due to the disruption of the Pd coating layer by the plastic strain. Although this study reported valuable information about the effect of applied stress on hydrogen transport, it did not explain clearly whether the decrease in the permeation current under plastic load was due to the trapping of hydrogen by dislocations or to the combined effect of the reduced rate of hydrogen oxidation by disruption of Pd layer and accelerated dissolution of the substrate steel and subsequent current decrease by passivation at a high anodic potential. In order to avoid any porosity associated with the rupture of a Pd film coated on to high-strength Cr–Mo steels (YS in the range of 580–660 MPa) during tensile straining, Brass and Chêne [12] conducted a permeation test without Pd deposition and reported an extremely low permeation current because of the low efficiency of the oxidation of hydrogen. Park et al. [13,14] developed an iron oxide film anodically grown in 0.1 M NaOH solution to replace the Pd coating.

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Although the application of such an iron oxide film for the hydrogen permeation test under applied stress provides consistent permeation results, the hydrogen oxidation current is much lower than what can be obtained using a Pd film due to the low efficiency of the hydrogen oxidation reaction ($\text{H} \rightarrow \text{H}^+ + \text{e}^-$) on the iron oxide film. This means that many of the H atoms that diffuse out of the steel cannot be oxidized and measured because of H_2 recombination.

In this study, particular attention is given to developing a Pd coating technique to ensure a stable background (passivation) current regardless of the level of stress. It is important for future research to understand the relationship between the applied stress level and the hydrogen permeation behavior for various high-strength steels. The test material used in this study is 64-mm-pressure vessel steel (ASTM A516) plate containing around 0.2 wt.% C. The test steels were austenitized by heating to 910 °C for 30 min and cooled to room temperature using a mixture of oil and water. A tempering heat treatment was then carried out at 650 °C for 100 min to toughen the test specimen, which has a YS of 338 MPa.

The modified hydrogen permeation technique was performed in reference to ISO17081 [15]. Instead of coating the Pd layer on the hydrogen exit side of the specimen before loading, however, the loading was performed prior to the Pd coating. By changing the sequence of the Pd coating and loading, the deformation problem of the soft Pd coating layer can be completely eliminated. The prepared steel membrane, with 1 mm thickness, was initially installed in a constant load test (CLT) frame and argon gas was continuously applied to both sides of the steel sheet to suppress surface oxidation. Then, contrary to the conventional test procedure, the desired constant load was applied prior to the Pd plating on the steel. The designed plating cell was then assembled with the steel membrane in the hydrogen detection side. The thin Pd film was electrochemically deposited to the steel membrane using the prepared coating bath (a mixture of 2.54 g of PdCl_2 and 500 ml of 28% aqueous ammonia solution). A galvanostatic current density of 2.83 mA cm^2 was cathodically applied for 2 min. After the electroplating, a modified Devanathan cell was then assembled with the steel membrane. The detection side was filled with 0.1 M NaOH solution purged with Ar gas. The Pd-plated surface exposed to the solution was maintained at a potential of 250 mV_{SCE}, at which the dominant reaction was oxidation of H atoms which diffused through the steel membrane. Once the background current reached a steady state and became stabilized, the H_2S -saturated NACE TM0284-96A [16] solution chosen as the hydrogen charging solution was poured into the charging side of the cell.

In order to clarify the influence of the applied tensile stress on the background passive current and the morphology of the Pd coating layer, an experiment was carried out to measure the effect of tensile stress on the background current in the absence of hydrogen. A stable background current density (below 0.1 $\mu\text{A cm}^2$) was initially obtained from the Pd coated steel membrane in deaerated NaOH solution without any applied stress, after which the steel membrane was deformed by

applying the constant load. The changes in the background current that were dependent on the constant load level were clearly identified, and the results are illustrated in Figure 1. There was no discernible change in the background current until the applied stress level was within the elastic range of the Pd film, whereas a change in the background current in the form of a current spike was observed upon exceeding the elastic limit of the film. A peak appeared whenever the constant load level was increased, probably due to disruption of the Pd film, leading to local detachment of Pd particles from the plastically deformed Pd coating layer. A more significant change in the background current was clearly observed when the deformation condition for the steel substrate changed from the elastic to the plastic range. As soon as the applied load level exceeded the elastic limit of the steel substrate, a drastic increase in the current was observed. In addition, the value of the current increase was much higher than that observed when the load level was in between the elastic limit of the Pd film and that of the steel substrate, indicating that there were more significant changes in the surface state as well as severe rupture of Pd film within the plastic deformation range of the steel. Figure 2 supports these findings.

Figure 2(a) presents the morphology of the Pd coating layer without any applied stress. There are no pores or voids in the coating layer, and the steel surface is fully and uniformly covered with small Pd particles of around 100 nm. In contrast, Figure 2(b) and (c) presents the morphology of the coating layer observed after applying a constant load of 120% of the yield strength (120% YS)

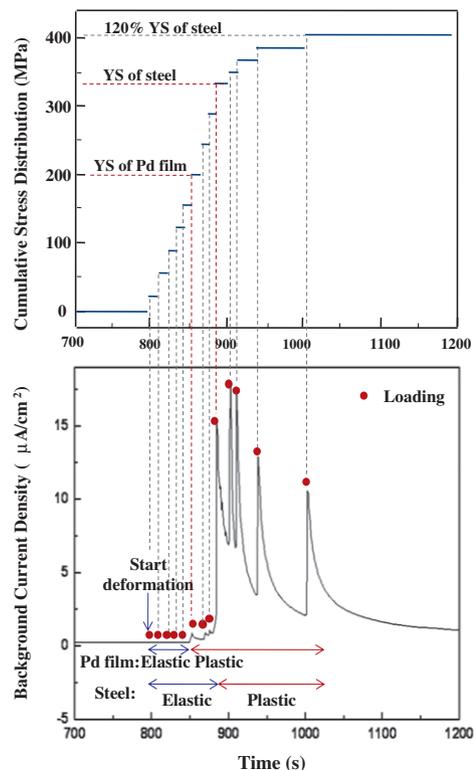


Figure 1. Change in background current density with constant tensile stress up to 405 MPa (120% YS of the steel substrate).

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