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In situ small-scale hydrogen embrittlement testing made easy: An electrolyte for preserving surface integrity at nano-scale during hydrogen charging

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ABSTRACT

An electrolyte for electrochemical hydrogen charging of corrosion-susceptible alloys is developed, which preserves the surface integrity at nano-scale by minimizing the surface roughness alternation. To assure the formation and adsorption of the hydrogen from the electrolyte, permeation tests were performed on Fe 3 wt%Si ferritic steel. X-ray photoelectron spectroscopy method was used to check the effect of the glycerol-based solution on the chemical composition of the sample surface. The surface analysis revealed minimal chemical and topography alteration on the surface after different electrochemical treatments. Various types of in situ small-scale mechanical tests such as nano-indentation, micro-pillar compression, and micro-cantilever bending tests were performed inside this electrolyte while the samples being charged with hydrogen under cathodic potential. These small-scale mechanical tests showed that the solution facilitates studying hydrogen embrittlement in nano- or micro-scale.

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Introduction

Most high strength and high performance alloys will experience a premature rupture when exposed to hydrogen (H) [1,2]. This phenomenon is called hydrogen embrittlement (HE) which is caused by the presence of H in metals [3]. H typically gets into the metal by surface adsorption followed by absorption and the diffusion/trapping phenomenon [4]. A vast amount of scientific research has been carried out on HE since 1874 when the very first report about the deleterious effect of H on the mechanical properties of steel was published by Johnson [5]. These researches led to several proposed

mechanisms [6–11]. Interaction of the H with microstructure and crystal defects, such as dislocations, vacancies, phase boundaries, grain boundaries, or precipitates (e.g. carbides) is determinant factor toward understanding the HE phenomenon [12–15]. In a typical macroscopic test, these defects that are distributed within the sample will start to interact with H at different incidents within the course of the testing [16–18]. Due to the large scale of the test and the large number of the defects involved, it is very hard to get mechanistic insight from the interaction of the defects with H. Such a mechanistic knowledge of H effect on defects is extremely valuable for modeling the HE phenomena and development of predictive tools which are highly demanded by the industry. One

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possible approach to study the mechanism of the H interaction with different microstructural features in detail and gain mechanistic information is to perform micromechanical [19–21] or nanomechanical [22–24] tests. Investigation of HE phenomenon in small scales imposes some experimental complications. As an example, in situ H charging is mandatory versus ex situ H charging for most metals [25,26]. Because, out gassing of H from small-scale samples leads to the rapid H out-gassing and depletion. The necessity of in situ testing becomes more pronounced for the alloys with higher H diffusion rate [27]. Electrochemical charging of H is one of the extensively utilized approaches in the literature [28,29]. However, a major challenge during small-scale testing combined with electrochemical charging of the sample is to preserve the surface roughness and the integrity of the micron-sized samples at the nanometer scale. Usually, in situ electrochemical small-scale testing takes several hours (including charging and testing time under slow strain rates) to accomplish. As a result, preserving integrity of the nano- or micro-scale samples turns to a challenging issue. Post-mortem analysis such as electron backscatter diffraction (EBSD) or electron channeling contrast imaging (ECCI) of the in situ electrochemically H charged and tested surface or micron-sized samples also requires an intact surface quality [30,31]. Under cathodic electrochemical potentials, although the overall surface would be preserved from corrosion, local variations in electrochemical potentials and chemistry of the electrolyte such as dissolved oxygen content and pH will result in small local areas corrosion and alteration of the surface which is unacceptable for small-scale testing while in a macroscopic test can be easily disregarded. The authors experienced the destruction of the small-scale testing samples (1 μm in diameter pillars) inside the borate buffer solution which is known to be a non-corrosive solution for iron base alloys [32]. These pillars were under cathodic polarization for several hours (Fig. 1). Experienced failure was the motivation for developing the solution introduced in this paper.

To overcome this problem, we used a similar strategy as proposed by Macák et al. [33,34]. They increased the viscosity of the electrolyte by replacing water with glycerol to control

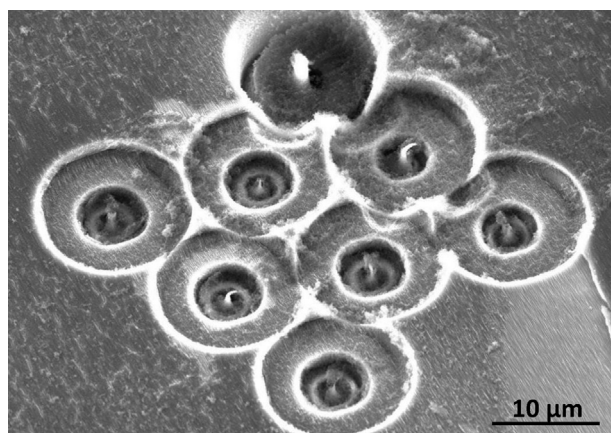


Fig. 1 – Scanning electron microscopic image of the Fe–3wt % Si, micro-pillars after in situ electrochemical pillar compression test inside an aqueous borate-buffer solution.

local concentration fluctuations and pH bursts in the electrolyte for growing nanotubes longer than 500 nm with higher aspect ratio inside acidic based solutions. Here, we report the application of such glycerol-based electrolyte for H charging during in situ small-scale mechanical testing to assure the sample surface integrity in nanometer scale. To show the applicability of this new electrolyte as a medium for electrochemical H charging, we performed electrochemical H permeation tests to confirm the H generation and uptake on the surface and to estimate subsurface H concentration (Permeation test). Further, the sample surface composition was analyzed using the X-ray photoelectron spectroscopy (XPS) method after electrochemical charging under both cathodic and anodic current densities and the results were compared to the electropolished sample (Surface analysis). XPS analyses were carried out in order to investigate the probable effect of the new solution on the surface chemical composition during electrochemical charging. Several types of in situ small-scale testing such as in situ electrochemical nano-indentation, in situ electrochemical micro-pillar compression, and in situ electrochemical cantilever bending tests were successfully performed inside the glycerol-based solution to show the capability of using this electrolyte in the small-scale testing of steels (In situ small-scale testing).

Experimental procedure

Material

Sodium Tetraborate, Decahydrate, ACS Grade with a 99.5% purity ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) from VWR chemical company and glycerol anhydrous for synthesis $(\text{HOCH}_2)_2\text{CHOH}$ from Merck company with a purity higher than 99% were used to make the electrolyte. The composition of the solution was 1.3 mol/L borax in glycerol and diluted with double distilled water of 20 vol percent to obtain the adequate conductivity. A Consort K911[®] instrument was used for resistivity measurements. The solution had a resistivity of 380 Ωcm at 25 $^\circ\text{C}$. This number decreased to 290 and 160 Ωcm at 30 and 50 $^\circ\text{C}$, respectively. A PHM210 Standard pH Meter[®] was used for pH measurement. The pH of this solution was ~ 7 and the boiling point was measured to be ~ 106 $^\circ\text{C}$. Differential scanning calorimetry (DSC) technique was used to measure the freezing point of the solution. DSC 250[®] from TA Instruments was used to perform the test. According to the heat flow-temperature curve presented in Fig. 2, there is a distinct change in the heat flow slope around ~ -58 $^\circ\text{C}$ which is considered as the start point for the freezing peak. The reported curve was obtained at 10 $^\circ\text{C}/\text{min}$ scan rate measurements from 40 $^\circ\text{C}$ to -80 $^\circ\text{C}$. Since decreasing the cooling rate gives a narrower and sharper freezing peak [35], no detectable change was seen in the DSC measured thermogram by 2 $^\circ\text{C}/\text{min}$ cooling rate down to -80 $^\circ\text{C}$.

Fe–3wt% Si alloy, which is a simple ferritic steel, was chosen as the testing material in this study. The chemical composition of the alloy is provided in Table 1.

This material has a high diffusivity and low solubility for H that is typical for ferritic steels [6,36]. Fe–3wt% Si, similar to other low alloy steels, has low corrosion resistance [37] and

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