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Hydrogen Embrittlement of 316L Stainless Steels Exposed in 1.0M Hydrochloric Acid Solution

Muhammad Shahmy Hadi[1], Safaa N. Saud[2], Esah Hamzah[1*], Mohd Fauzi Mamat[1,3]

[1] School of Mechanical Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
[2] Faculty of Information Sciences and Engineering, Management & Science University, Shah Alam, Selangor, Malaysia
[3] Faculty of Engineering Technology Mechanical and Manufacturing, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia

Corresponding Author Email: esah@mail.fkm.utm.my

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ABSTRACT

This paper aims to determine the different sources of hydrogen that cause hydrogen embrittlement in ferrous materials. The 316L stainless steel was selected as the research object, and subjected to immersion and electrochemical tests in 1.0M HCl solution. The hardness and tensile strength of the test samples were measured before and after hydrogen embrittlement. The hydrogen, which exists in the form of hydrides, was detected through X-ray diffractometry (XRD), while the fracture profile and microstructure of the samples were observed through optical microscopy and scanning electron microscopy (SEM), respectively. The results show that the hydrogen ions diffused into the surface of the steel, forming iron hydrides. In this way, the hardness of the steel was enhanced, and brittle failure occurred under tensile load; the amount of hydrides and the probability of hydrogen embrittlement increase with the immersion time in the acid solution; the sample immersed in 1.0M HCl for one day presented the most prominent hydrogen embrittlement, i.e. the highest hardness and lowest ductility. The research results provide insights into the occurrence of hydrogen embrittlement in ferrous materials.

1. INTRODUCTION

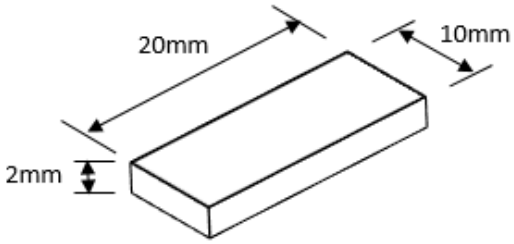
Hydrogen embrittlement (HE) is a type of deterioration of materials and is linked to corrosion [1]. HE results in a material loss of ductility, which implies increased brittleness [2]. The loss of ductility and toughness of material can cause cracks and disastrous brittle failures at stress under the yield stress of a material. The hydrogen attack and/or induced cracking (HIC) are also two phenomena denoting the hydrogen embrittlement. Hereafter, resistance to hydrogen embrittlement is one of the major structural characteristics of the stainless steels 316L [2]. Hydrogen embrittlement mechanism in a metal is through the ingression of hydrogen into the metal at a high temperature or ambient temperature [3]. Once the hydrogen is absorbed, it may combine with carbon and form methane or present as in the state of atomic or molecular hydrogen [2]. Due to the large size of the methane molecules, the diffusion process of these molecules will be rather difficult, and thus resulting in enormous stress from crystallographic defects (dislocations and vacancies) or discontinuities (inclusion and/or matrix interfaces, voids) inside the metals. This pressure will initiate cracks if there is no action taken to diffuse out the hydrogen [3, 4]. Hydrogen enters the material by hydrolysis and separation of the H2 molecules followed by H proton absorption, while the electron is released into the metal's permitted electron gas. The H protons spread into regions of elevated stress where they accumulate and transferring the materials to behave brittle-like [5]. In latest high-pressure hydrogen parts such as oil transportation pipelines, reactor vessels, hydrogen storage tanks and fuel cell vehicle valves, austenitic stainless steels such as type 316L are commonly used as parts due to their superior resistance to hydrogen embrittlement. On the other hands, the hydrogen induced degradation may also be resulted due to the low stability of austenite phase and therefore, the mechanical performance tends to be reduced under the plastic deformation condition corresponding to the strain-induced of the α′ martensite transformation [4-6]. Michler et al. [6] found that the nickel-containing austenitic stainless steels is a key parameter for HE resistance to produce the semi-finished product, particularly between 10 and 12.5 wt.% of nickel. The hydrogen effect on local plastic strain growth during the deformation or fracture phase was shown in the study by Rosenberg et al. [7]. In addition, two features of ductile failures are noted in accordance with sample hydrogen content: lower density of microevils for greater levels of hydrogen and brittle secondary cracking in relation to lower hydrogen content ductile fractured surfaces [8]. As there is very limited research of the phenomenon of hydrogen embrittlement in 316L stainless steel and little evidence in the literature of the impact of corrosive formation layers, which affected the mechanical characteristics of stainless steel in the acidic working environment. The form of corrosion known as hydrogen embrittlement in type 316L of stainless-steel plate was therefore investigated using two methods, namely by immersion and electrochemical tests in 1.0M HCl solution. Mechanical experiments and analysis of the components were carried out on the steel samples in order to comprehend and define the hydrogen embrittlement mechanism that influenced the microstructure and their corresponding mechanical

characteristics of the 316L stainless-steel.

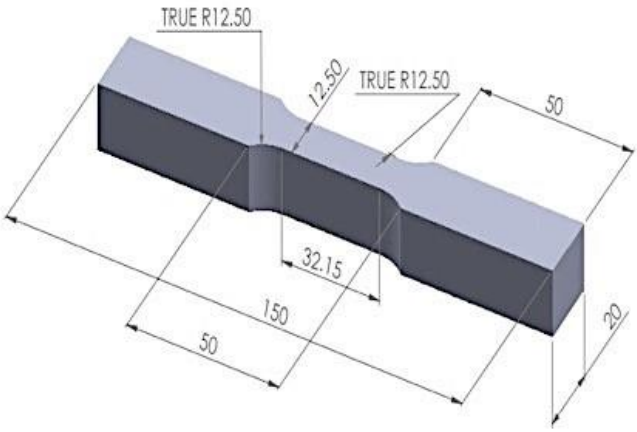
2. EXPERIMENTAL PROCEDURE

2.1 Sample preparation

The materials used for the research was type 316L stainless steel. The stainless-steel samples were prepared in accordance to ASTM E8/ E8M-13a [9] standard for tensile test before and after both immersion and electrochemical tests. Rectangular samples were also prepared for the hardness test and materials analysis. The samples were cut using EDM wire cutting machine. The dimensions of the samples are given in Figure 1a and b.



(a)



(b)

Figure 1. Dimensions of (a) hardness and materials analysis and (b) tensile test sample (unit: mm)

The rectangular sample for materials analysis was mounted in acrylic material using hot mounting machine for easier handling during subsequent materials preparation steps. After mounting, the steel sample was ground on grinding machine using silicon carbide papers with grit no 80, 100, 300, 800 and 1000 and 2000 and rinsed in distilled water. This was followed by polishing using rotary polishing machine equipped with polishing cloth and polishing liquid until a mirror-like surface was obtained. The sample was then rinsed in water and dried using a mini blower. The last step of the sample preparation was chemical etching which is normally done on the samples after polishing prior to microstructural study to reveal the structure. Stainless steels rectangular samples were immersed into a Vilella's reagent (45ml Glycerol + 30ml HCl + 15ml HNO3) in accordance to ASTM E407-07 [10] for 5 minutes etching time. The samples were then cleaned and dried before they were observed under optical and scanning electron microscopes.

2.2 Hydrochloric acid immersion test

Corrosion test to determine the hydrogen embrittlement effect on stainless steel 316L were performed. There were two types of hydrogen embrittlement tests carried out in this study, namely, immersion test and electrochemical test. The electrolyte used for both tests were 1.0M hydrochloric acid which acted as a source of hydrogen ions. During the immersion test, nitrogen gas of about 3-5 bubbles per second controlled by the gas regulator, was constantly supplied to the acid solution in the beaker to prevent oxidation on the steel samples during the test. The schematic diagram of immersion test is shown in Figure 2. In this test, the hydrogen ions present in the 1.0M hydrochloric acid electrolyte would diffuse into the sample surface and formed hydride with the metallic ions. Three prepared samples both for mechanical tests and materials analysis were immersed in the electrolyte for 10 minutes, one hour and one day respectively. The samples were then removed from the solution, cleaned in water and dried.

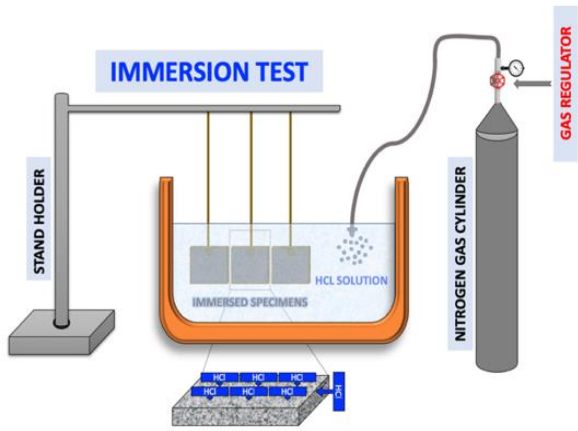


Figure 2. Schematic diagram of the immersion test set-up

In electrochemical test, the samples were partially immersed in 1.0M hydrochloric acid solution and electrically connected to a voltage supplier as shown in Figure 3. The 316L stainless steel test samples were connected to a negative terminal which then acted as the cathode. It was also connected to another similar steel metal which acted as the anode. The immersion times were 10 minutes, one hour and one day for three different samples. The samples were removed after completion of the test and dried using a mini blower.

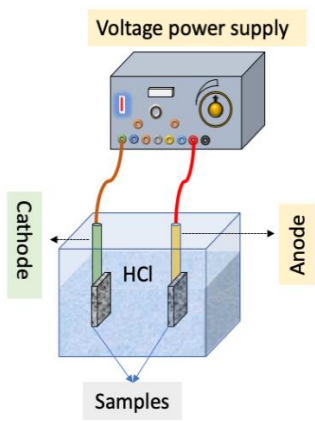


Figure 3. Schematic drawing of an electrochemical test set-up

2.3 Microstructure and Elemental Analysis

Materials analysis of the samples before and after hydrogen embrittlement tests were carried out using optical microscope (ZEISS Axiotech, Germany), variable scanning electron microscope (VPSEM S-3400N, Hitachi, Japan) attached with and energy dispersive x-ray spectrometer (EDS), whereas x-ray diffractometer (D5000 Siemens XRD) was used to analyze the phases and compounds of the sample after hydrogen embrittlement tests.

2.4 Mechanical tests

Mechanical tests, namely, tensile test using Instron 5980-type tensile test machine and hardness test using Matsuzawa Vickers (SN: DV 6213 test equipment were performed on samples before and after the hydrogen embrittlement tests in order to observe its effect on the mechanical properties of the steel samples.

3. RESULTS ANALYSIS

3.1 Microstructural analysis

Figure 4a and b represents the microstructure of as-received stainless steel 316L observed under optical microscope. Deformation twins at higher magnification can be seen in Figure 4a. The microstructure of the as-received samples observed by using SEM is shown in Figure 4b. Both optical and SEM micrographs act as a reference and are compared to the results obtained after hydrogen embrittlement test. Twin is usually formed and can be observed after the activation of multiple slip systems. Another possible factor for grain refinement will be the intersection of twin borders and newly produced sub-grain boundaries [11-15].

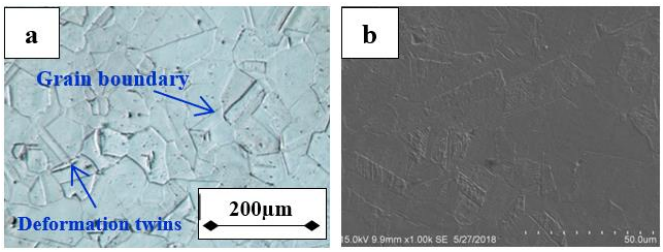


Figure 4. (a) Optical micrograph and (b) SEM image of as-received sample of stainless steel 316L

Figure 5a displays the microstructure on the surface of the steel samples before hydrogen embrittlement by immersion test. It shows the austenite grains littered with twins, typical of structure for type 316L stainless steel. Similar surface morphology was observed in steel sample after 10 minutes immersion in acid solution, indicating no hydrogen embrittlement occurred in the sample, as shown Figure 5b. However, a few pits appeared as dark spots along the grain boundaries were observed for steel samples which was immersed for one hour in acid solution as presented in Figure 5c. As expected, more pits were observed for sample as shown in Figure 5d, which was immersed for 24 hours indicating more metal hydrides were formed due to the diffusion of hydrogen ions into the steel surface and resulted in the break off metal-metal ion bonds and replaced by the more brittle metal hydrides. This led to the cracks initiation and propagation when stress was applied and result in the formation of pits. Generally, it is well-known for its distinct segregation effects in type 316L stainless steel and other similar steels, nickel has high-and low-content band-like structures [11]. Hydrogen can also penetrate materials by surface defects, such as micro-cracks and manufacturing micropores. The penetrated hydrogen atoms spread across the grain boundaries and combined to be form as a methane gas with iron alloys [11, 12].

Figure 6(a-d) shows the morphology before and after ten minutes, one hour, and one day of electrochemical tests. No changes on the surface before hydrogen embrittlement test through electrochemical method as shown in Figure 6a. On the other hands, Figure 6b shows possible small hydride, layer formed at the surface of the sample after 10 minutes electrochemical test in acid solution. Further observation on sample that had undergone one-hour electrochemical test in acid solution, pits were found on scattered area of the surface of the samples as the result of hydrogen attack on the stainless-steel samples, as shown in Figure 6c. This may be due to formation of more metal hydrides from the diffusion of the hydrogen ions into the steel surface when the metal was immersed in the acid solution. Another possible mechanism of hydrogen embrittlement is the pressure build up occurs from within the metal due to the continuous diffusion of hydrogen atom into the stainless steel. When the two hydrogen atoms meet, they will combine to form molecular hydrogen which act like a gas and creates pressure within the steel. Pressure buildup will lead to rupture and pits are formed. After one day hydrogen embrittlement test was carried out, Figure 6d shows that the sample suffered severe corrosion attack due to hydrogen embrittlement after going through electrochemical test as the pits propagated and resulting in cracks formation. The mechanism is similar to sample immersed for one hour except that more hydrides were formed due to prolong immersion.

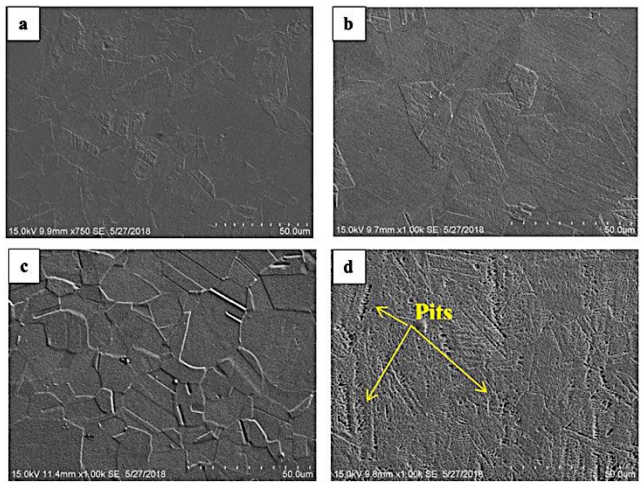


Figure 5. SEM images of stainless steel 316L samples after been immersed for; (a) 0 min, (b) 10 minutes, (c) one hour and (d) after one day

Figure 7 shows the amount of iron obtained from EDS analysis for each sample versus immersion time. The results show that the amount of iron is almost the same for all samples indicating that there was no oxidation of iron took place. No corrosion product observed on the samples and therefore, no loss of material on the metal surface because it acted as a

cathode. No hydrogen element was detected using EDS due to the limitation of the EDS equipment which could not detect light and small element such as hydrogen. Thus, further investigation was required using XRD to observe any formation of hydride.

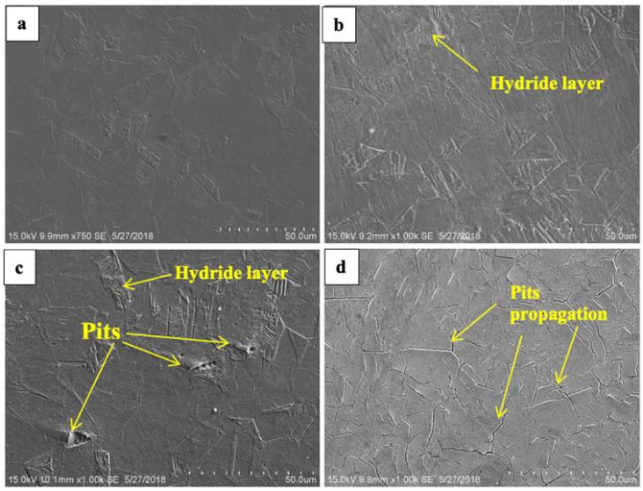


Figure 6. SEM images of stainless steel 316L after electrochemical test after (a) 0 min, (b) 10 minutes test, (c) one-hour test and (d) one day

The XRD analysis was carried out to detect the presence of hydride in the sample. Glazing angle was set at 5° because hydrogen atom or hydride was mostly present at the surface of the sample after it went through hydrogen embrittlement test. Figure 8 represents the XRD results of stainless steel 316L samples before and after immersion test. It is found that there are three peaks were obtained from the diffractograms and all peaks belong to austenite (pure γ structure) which is the main structure of stainless steel 316L namely, $(111)_\gamma$, $(200)_\gamma$ and $(220)_\gamma$ [16]. Remarkably, there was no metal hydride detected in the samples even though many pits were observed on the surface of the samples especially after one day (24 hours) immersion as shown in Figure 6d. This may attribute to the peaks for γ phase and hydrides are so close to each other that it is difficult to identify them. However, XRD spectrum of stainless steel 316L sample before and after 10 minutes, one hour, and one day for electrochemical test as shown in Figure 9, the metal hydride, mainly iron hydride (FeH), peaks were detected in the samples. Iron hydride is formed by the combination of hydrogen atom with metal atom once hydrogen ion is absorbed into the metal.

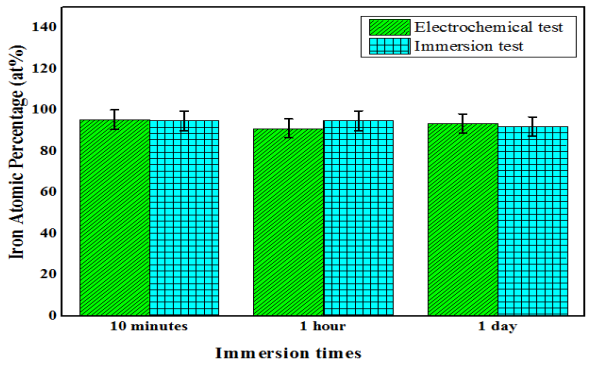


Figure 7. Amount of iron (in at. %) versus immersion time based on the EDS analysis

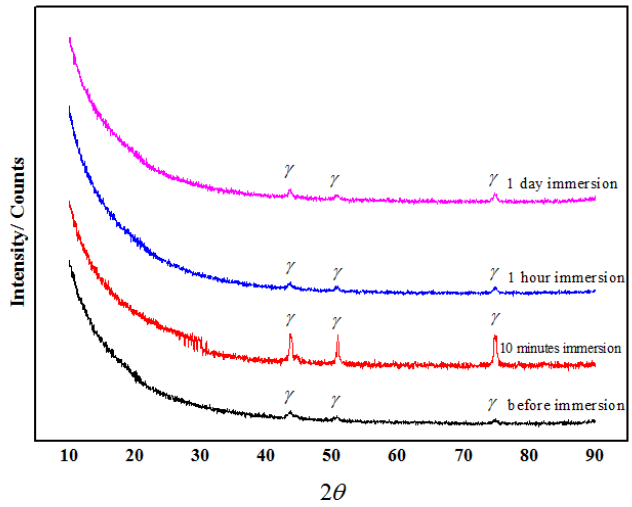


Figure 8. XRD result for stainless steel 316L sample before and after 10 minutes, one hour and one day immersion test

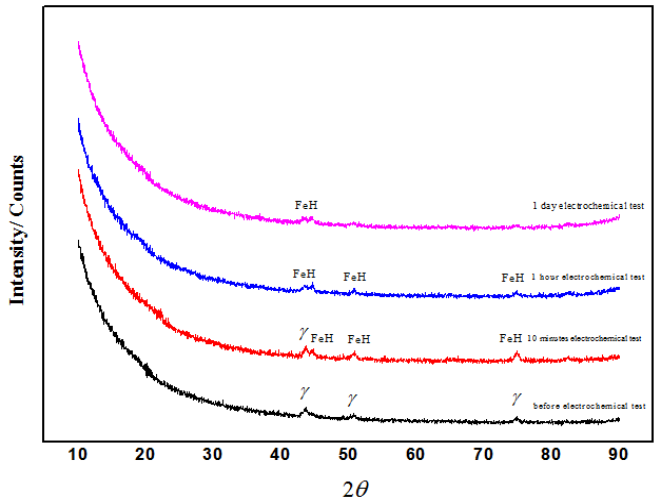


Figure 9. XRD result for stainless steel 316L sample before and after 10 minutes, one hour and one day electrochemical test

3.2 Mechanical test analysis

3.2.1 Hardness test

Vickers Hardness test of stainless steel 316L samples after hydrogen embrittlement test are shown in Figures 10. It shows that hardness increases as the immersion time increases for both immersion and electrochemical tests. The hardness of the initial material was ~280Hv. After the hydrogen embrittlement tests the hardness values increased, as the time of exposing increased [17-19]. The hardness value increased by 10.8% and 7.97% for electrochemical and immersion tests after one day exposure, respectively. It is known that absorption of hydrogen ion into the surface of steel sample forming iron hydride harden the surface of stainless steel [19, 20]. Presence of hydrogen at the surface layer increases the hardness of a stainless steel 316L [18, 19]. The diffusion of hydrogen atoms is much higher in the martensitic phase than in the austenite phase, thus strengthening deformation used to improve the martensitic phase [19]. Huang et al. [11] indicated that the increase in micro hardness after hydrogen charging was probably due to hydrogen playing a role of solid solution hardening, just as it happened in the case with carbon and

nitrogen atoms. The highest hardness recorded was for the samples after being immersed for one day in the electrochemical test.

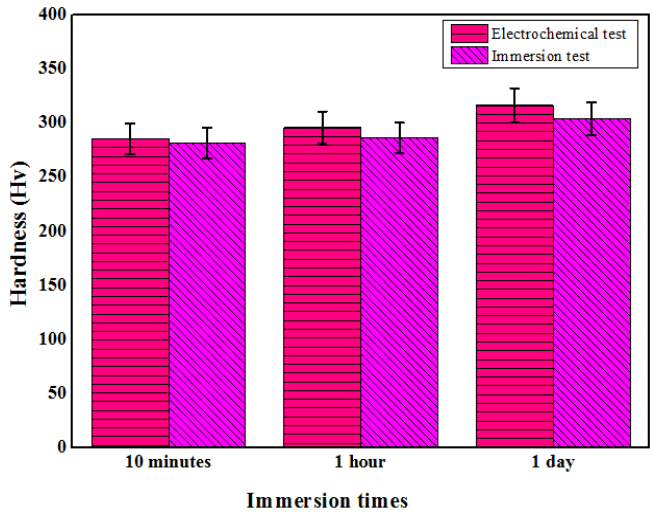


Figure 10. Hardness versus immersion time of stainless steel 316L samples for both hydrogen embrittlement tests

3.2.2 Tensile Test and fracture behavior

Figure 11 (a-e) shows the stress-strain curves and optical macrographs after different immersion duration for stainless steel 316L. The effect of hydrogen embrittlement on the stainless steel implied the ductility loss. Although the samples have been immersed at different immersion times starting from 10 minutes to one day, there was small difference on the ductility between the samples. Sample immersed for 10 minutes is the most ductile followed by one-hour immersion and one day immersion with the tensile strain at maximum load is 26.32%, 25.73% and 16.81% respectively. The macrostructure analysis was made on tensile test samples (gauge section) of stainless steel 316L after they were subjected to tensile test to determine its fracture mode. This analysis was done to determine whether hydrogen decrease the ductility of the samples after hydrogen embrittlement test. The fracture behaviour of stainless-steel sample before the HE test is shown in Figure 11b. The macrostructure reveals that the sample exhibits the features of ductile fracture. This type of fracture creates a cup and cone shape fracture which is caused by the cracking of the sample by shear deformation. Ductile fracture could still be observed on the samples after 10 minutes and one-hour immersion. However, changes could be seen as the fracture mode transforms from ductile to brittle fracture. This can be observed in Figure 11e, after the samples were immersed for one day in immersion test. The fracture was due to the fast propagation of crack nearly perpendicular to the direction of the applied stress.

Figure 12 (a-e) displays the comparison between different immersion times of electrochemical test for stainless steel samples along with their optical macrographs of the fracture surfaces. Sample immersed for one day is the least ductile with tensile strain of 19.78% if compared to sample immersed for 10 minutes, 49.03%. Tensile stress of the most ductile and the least ductile sample has the difference of almost 60%. Based on these stress-strain curves, the main difference between samples before and after hydrogen embrittlement (HE) test was the type of failure. Sample which did not undergo hydrogen embrittlement test experienced ductile failure, while the HE samples experienced brittle fracture, as deduced by the sudden, drastic decrease in stress after a strain similar with previous studies [21-26]. The tensile strain at maximum load of stainless steel before hydrogen embrittlement test (most ductile) is 50.29% while the tensile strain of stainless steel after one day immersion test (most brittle) is 19.78%. Reduction of tensile stress at maximum load of stainless steel after hydrogen embrittlement test could also be observed with the increment of immersion time. The ductility is also affected by the immersion test as it is inversely proportional with immersion time. As the immersion time increases, the ductility decreases because as the samples were exposed to an environment which contains hydrogen, more hydrogen ions are able to diffuse into the surface of the metal which forms hydride. The presence of hydride will reduce the ductility of the metal. The primary type of corrosion of stainless steel 316L is hydrogen embrittlement, which allows hydrogen ions to spread easier in the specimens, producing brittle carbide and hydride precipitates that reduce mechanical characteristics [26-28].

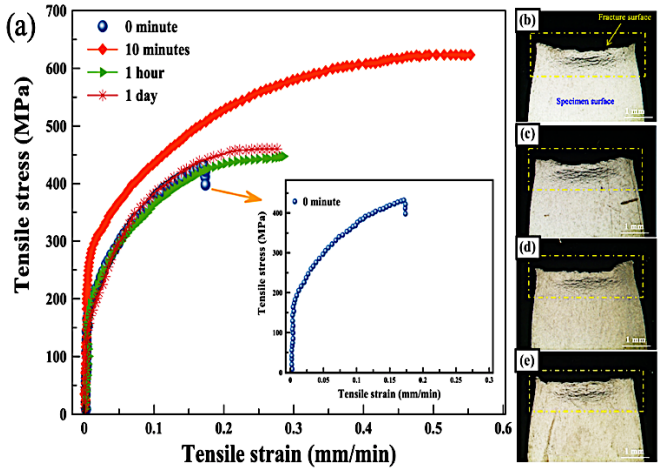


Figure 11. (a) Tensile stress - strain curves and (b-e) Optical macrographs of stainless steel 316L under the immersion test: (b) 0 minute, (c) 10 minutes, (d) one-hour, and (e) one day immersion test

The fracture mode of stainless steel 316L sample before electrochemical test is shown in Figure 12a. The micrograph reveals that the sample exhibits the features of ductile fracture. This type of fracture creates a cup and cone shape fracture. Changes on the fracture mode could be seen as the fracture mode transforms from ductile to brittle fracture. This can be observed in Figure 12 (b-d) after the samples undergone 10 minutes, one hour and one day electrochemical test with brittle fracture characteristics. Earlier research indicates the shear fracture area is widespread in the periphery of the fracture surface and in the fibrous region micronized dimples were coated by inclusions and dimples ascribed to grain-boundary sliding, before hydrogen fracture was breached [22-26]. While analysis after hydrogen embrittlement demonstrates mixed modes of brittle and ductile fracture, the fracture morphology recognized the distribution of tiny and large dimples. Matsuo et al. [22] stated that the hydrogen had reduced the average dimple size in stainless steel type 316L, which is distinct from the average dimple size [27]. Both tests show similar fracture features, clearly shows that the cracks started in the ferrite phase as confirmed by Zucchi et al. [28]. The ferrite grains showed a cleavage mode, characteristic of HE, while a rough morphology of crack formed in the austenite grains.

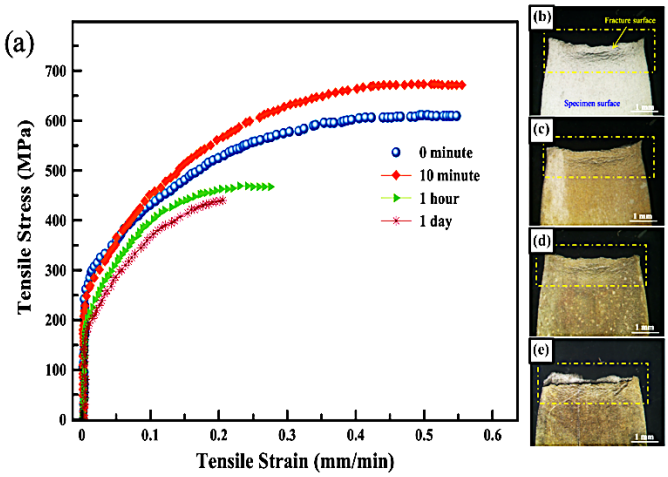


Figure 12. (a) Tensile stress - strain curves and (b-e) optical macrographs of the surface fracture of stainless steel 316L of the electrochemical test: (b) 0 minute (c) 10 minutes (d) one-hour and (e) one day immersion test

4. CONCLUSIONS

Following immersion in 0.1 M HCl for 10 min, 1 hour and 1 day at room temperature, the hydrogen embrittlement of 316L stainless steel was studied. The results showed that hydrogen exposure caused a significant decrease in the ductility and elongation of the material to failure. The results are due to the diffusion of H_2 to the metal's surface following long exposure (1 day) forming a hydride, thus increasing the ductility and rising steel hardness. A detailed investigation of the surfaces of the fracture allowed a relationship to be established between the depth of embrittlement and plastic strain. Further analysis of fracture features demonstrated that after the exposure to acid solution the transformation from ductile to fracture could occur. The study concludes that the long period of acid exposure significantly reduced the durability of 316L stainless steel to hydrogen corrosion and must therefore be taken into account when 316L stainless steel is recommended in harsh environments.

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