HYDROGEN ENVIRONMENT EMBRITTLEMENT OF LOW ALLOY STEEL AT ROOM TEMPERATURE

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ABSTRACT

To verify the safety application and use of Cr-Mo steel for the high-pressure hydrogen equipment, tensile, fatigue and crack growth tests of JIS-G4105-1979 SCM435 and 440 steel under 45MPa high-pressure hydrogen at room temperature were conducted. There were no significant differences in tensile deformation behaviors between air environment and 45MPa hydrogen until the maximum load point. However, hydrogen tested specimen broke with less ductility accompanied by many surface crack on the specimen surface which are caused by the specimen machining. The scatter of ductility was observed due to the specimen surface preparation conditions. As a result, reduction of ductility in gaseous hydrogen environment is caused not only by specimen machining effect but also by the presence of non-metallic inclusion at surface.

1 INTRODUCTION

For the safe and wide spread use of fuel cell cars, Japanese government and industries now studies regulations and recommended practices of the vast utilization of hydrogen gas. For the future planning, fuel cell car is to be pressurized up to 75MPa by hydrogen fuel gas station. In order for the commercial use of such ultra-high pressure hydrogen equipment, it is necessary to establish the appropriate design, manufacturing practice and inspection planning considering material performance under ultra high-pressure hydrogen environment. In this regard, hydrogen environment embrittlement (H.E.E.) of high strength steels, which are conventionally used for ultra high pressure component, is of great interest since a few literatures1) concerning H.E.E. is available at this stage (ex. Ohnishi [1]). In this study, 45MPa fatigue testing machine and autoclave are facilitated and JIS-G4105-1979 SCM435 and 440 steels are tested in 45MPa gaseous hydrogen environment at room temperature.

2 EXPERIMENTAL PROCEDURES

Pressure vessel with hydraulically actuated, closed-loop-controlled material testing machine has been developed. Figure 1 shows schematic drawing of the vessel. The pressure vessel has an inside diameter of 240mm and the depth of 500mm which is capable of standard cylindrical tension specimen (ϕ 8mm G.L.=50mm), fatigue specimen (ϕ8mm G.L.=15mm) and compact tension specimen (1T-CT) which are utilized in this experiments. The actuator capacity is 240kN in tension and 100kN in compression. Load calculator cancels load component raised from the pressure so that the external load cell can measure the specimen load only. Strain was measured by clip-on type extensometer or measured through the rod mounted to the specimen so that the transducers outside of the top projection of the autoclave detected the rod position. It is well known that the hydrogen environment embrittlement is affected by the purity of surrounding hydrogen gas since hydrogen environment embrittlement by gaseous hydrogen occurs by gas phase diffusion of molecular hydrogen to some critical location where metal is stressed and/or stressed crack surface. Hoffmann and Rauls [2] demonstrated that only 0.1ppm oxygen inhibits the hydrogen environment embrittlement. Fukuyama [3] reported CO and SO_2 gas inhibits the crack growth while H_2S promotes crack growth. For such reason, to minimize the experimental uncertainty caused by impurity gases, hydrogen gas used for material testing is 99.99999% purity by volume with the following impurity levels: O_2<0.02ppm, CO<0.01ppm with a dew point of –80 ℃. Pressurization and system purging is conducted before testing:(1) pressurization with dry N_2 gas, (2) evacuation and (3) pressurization/ depressurization with the pure hydrogen gas. Test materials are JIS-SCM low alloy series: SCM435 and SCM440 which are the popular materials for the ultra high-pressure equipment. The chemical compositions are exhibited in table 1. Since specimen surface conditions are known as the factors affecting the hydrogen tension test results (ASTM-G142-98[4]), specimens are prepared by 1) Highly polished by abrasive papers, 2) Electro polished by 0.2mm in

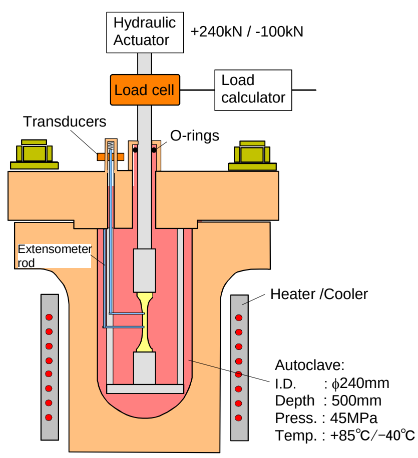


Figure 1 Fatigue testing machine

thickness in order to eliminate the effect of cold work on specimen surfaces 3) As machined conditions. Surface roughnesses were measured on 600x600μm in length area by laser displacement devices. The averaged surface roughnesses(Ra) for each conditions were about 1)0.5μm, 2) 0.6μm and 3)1.2μm respectively.

Table 1 Chemical compositions of the steel tested (mass.%)

Steel	Heat	C	Si	Mn	P	S	Cu	Cr	Ni	Mo
SCM435	-	0.36	0.23	0.76	0.014	0.010	0.020	1.06	0.03	0.19
SCM440	HeatA	0.42	0.22	0.76	0.008	0.003	0.02	1.10	0.02	0.24
	HeatB	0.42	0.23	0.77	0.012	0.002	0.02	1.00	0.02	0.18

3 RESULTS AND DISCUSSIONS

Figure 2 shows stress versus strain curves tested in various cross-head speeds using highly polished specimen (0.5μm) in 45MPa gaseous hydrogen at room temperature. There were no significant differences in tensile deformation behaviors between air environment and 45MPa hydrogen until the maximum load point. However, hydrogen tested specimen ruptured with less ductility and characterized by many cracks originated from the surface (Figure 2). Figure 3 shows surface cracks of tension specimen observed after tested in 45MPa gaseous hydrogen. The two circumferential cracks seem to have originated from the specimen machining itself or possibly by

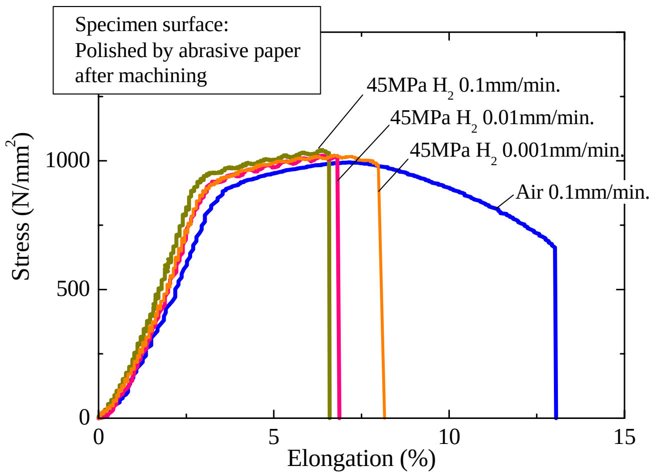


Figure 2 Tensile curves of SCM440-heat A under 45MPa gaseous hydrogen and 0.1MPa air

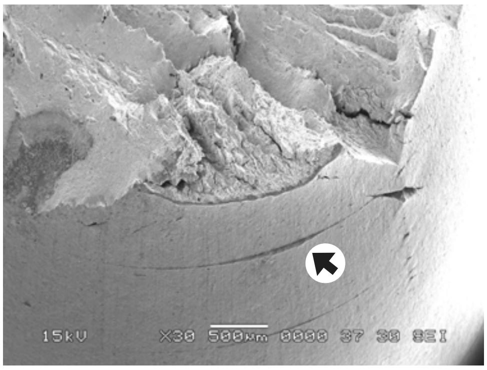


Figure 3 Surface cracks observed for hydrogen tested specimen of SCM435 (Specimen surface is polished by abrasive paper after machining)

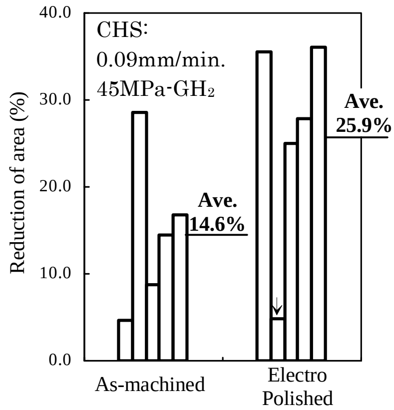
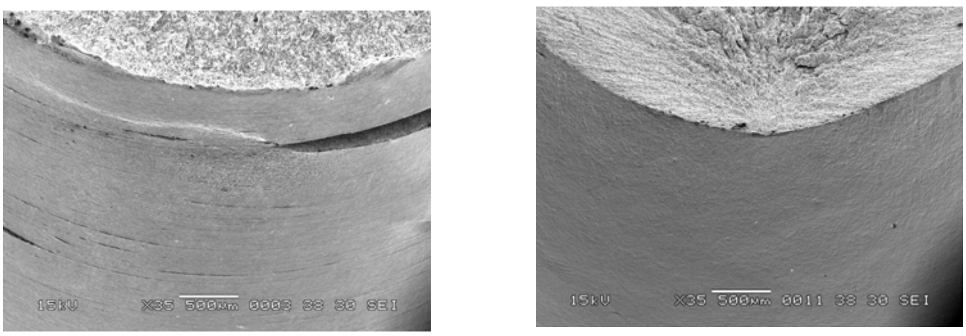


Figure 4 Duplicated tests results of tensile tests on differently surface prepared conditions of SCM435

the effect of cold work caused from specimen machining. Small cracks in line are found to be the dispersed non-metallic inclusions from EDS analysis. Five numbers of tests are duplicated on both electro polished condition and as-machined surface condition and ductility data are compared in Figure 4. Though the average ductility is improved by polishing effect, scatter in the reduction of area was observed. Figure 5 shows the fracture surface of electro polished specimen which ruptured with the poor ductility in comparison with the as-machined specimen. The examination of the fracture surface demonstrates that surface cracks disappears by polishing the surface, however the fracture origin was turned out to be the non-metallic inclusion (MnS) at the surface which is the cause of scatter in ductility. The strain rate controlled (0.1%/s) tension/compression low cycle



(a) (b)

Figure 5 Fracture origin for as-machined (a) and electro polished (b) specimen of SCM435 fatigue test on SCM440 steel results are shown in Figure 6. The specimen surfaces are polished by abrasive paper conditions so that the specimen machining effect would be eliminated. We reached the preliminary results that the number of cycles to the crack initiation is less in 45MPa hydrogen than that in air. We plan to investigate the surface influence on crack initiation and crack growth behavior under gaseous hydrogen and present in final conclusions.

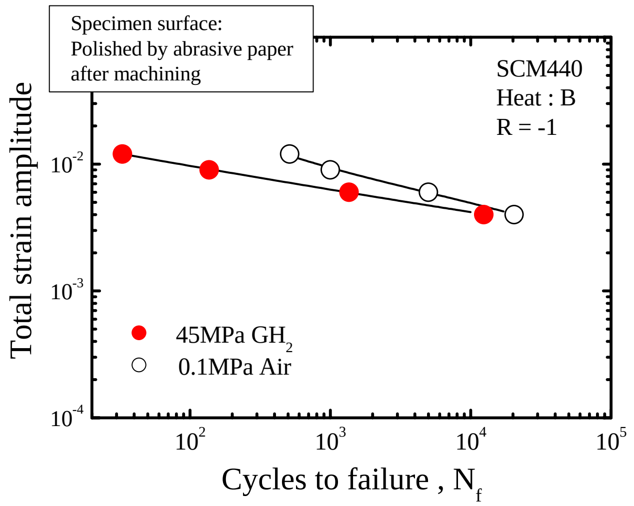


Figure 6 Effect of low-cycle fatigue life

4 CONCLUSIONS

1. From the hydrogen tension test results on SCM435 and SCM440 steels, they ruptured with less ductility than in air and showed hydrogen embrittlement though the yield strength and maximum tensile strength were almost the same values as tested in air condition.

2. Though the specimen surfaces were electropolished, hydrogen tensile ductilities were scattered and this was found to be caused by the non mettallic inclusions dispersed on the specimen surfaces.

3. Low cycle fatigue test results shows that the number of cycles to the crack initiation is less in 45MPa hydrogen than that in air.

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