Assessment of hydrogen embrittlement susceptibility of an Al-Cu-Mg alloy in humid air

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Abstract. In the present study, we investigated the hydrogen embrittlement susceptibility of Al-4%Cu-1.5%Mg alloys subjected to several heat treatments by means of SSRT tensile test and humid gas stress corrosion cracking(HG-SCC) test. For SSRT tensile test, the tensile test pieces were cut from cold-rolled sheets of 1mm thickness. The test pieces were solution-treated at 500 ^{o}C for 1h, water-quenched and aged at 140oC for 72h or 360h. SSRT tensile test was performed in two environments, humid air (HA) and dry nitrogen gas (DNG) at a strain rate of $1.39\times10^{-6}s^{-1}$. Fracture surfaces were observed with a scanning electron microscopy(SEM). For HG-SCC test, compact tension(CT) test pieces were cut from hot-rolled plate of 6mm thickness. The CT test pieces were solution-treated, water-quenched and aged at 190 ^{o}C for 9h, 50^{o}C for 96h or 140^{o}C for 72h. HG-SCC test was carried out based on High Pressure Institute of Japan standards; HPIS E103:2018. The pre-cracked CT specimens with stress loading were kept for 90 days in two environments, HA and DNG. After 90 days, in order to observe whether cracks propagated due to HG-SCC, the specimens were loaded up to fracture rapidly, followed by SEM observation. Tensile properties obtained by SSRT tensile tests were almost the same in two environments. Also, fracture surfaces were not affected by test environments. Moreover, in HG-SCC tests, crack propagation was not observed at each test conditions. Therefore, Al-4%Cu-1.5%Mg alloy had high-resistance to hydrogen embrittlement.

1 Introduction

Al-Zn-Mg and Al-Cu-Mg alloys have been used as structural components due to their high strength. It is well known that these alloys are susceptible to stress corrosion cracking (SCC), which is based on intergranular cracking. The crack propagation mechanism of SCC depends on the alloy system. In Al-Zn-Mg alloys, hydrogen embrittlement dominates the crack propagation. In Al-Cu-Mg alloys, on the other hand, the crack propagates by anodic dissolution along grain boundaries [1]. It has been reported that the peak-aged Al-Zn-Mg ternary alloy showed degradation of ductility when a slow strain rate technique (SSRT) test was performed in humid air [2], which was attributed to hydrogen embrittlement [2-5]. On the other hand, the peak-aged Al-Cu-Mg ternary alloy did not show such a degradation in humid air [2].

The reason why the Al-Cu-Mg alloy shows resistance to hydrogen embrittlement is still unclear. We need to reveal this in order to suppress hydrogen embrittlement in high strength aluminium alloys. We have investigated the behaviour of hydrogen in Al-Cu-Mg alloys using hydrogen microprint technique and SSRT tensile test [6,7].

The hydrogen embrittlement susceptibility of Al-Zn-Mg alloys depends on heat treatment [8,9]. As far, effect of heat treatment on hydrogen embrittlement of Al-Cu-Mg alloys has not been reported. In the present study, we investigated the hydrogen embrittlement susceptibility of Al-4%Cu-1.5%Mg alloys subjected to several heat treatments by means of SSRT tensile test and humid gas stress corrosion cracking(HG-SCC) test.

2 Experimental procedure

2.1 Specimen

The material used in this study was an Al-Cu-Mg ternary alloy. The chemical composition is shown in Table 1. In order to investigate the origin of high-resistance to hydrogen embrittlement in this alloy system, the amount of impurity elements such as iron and silicon, and manganese that is recrystallization inhibitor element have been minimized. Optical microscopic image after

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heat treatment is shown in **Fig.1**. Recrystallized grains were observed and some of second phase particles remained.

Table 1 Chemical composition of the specimen in mass%.

Si	Fe	Cu	Mn	Mg	Cr	Ti	Al
<0.01	0.01	4.0	<0.01	1.5	<0.01	0.02	Bal.

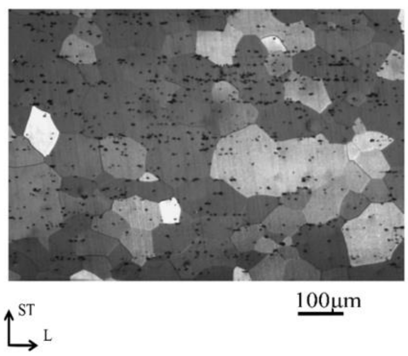

Fig.1 Optical microscopic image of the Al-4%Cu-1.5%Mg alloy after solution treatment.

2.2 SSRT tensile test

Sheet-type tensile test pieces with a gauge length of 12mm along rolling direction, width of 4mm and thickness of 1mm were cut from cold-rolled sheets. The test pieces were solution-treated at 500 °C for 1h and then water-quenched. The solution-treated specimens were aged at 140°C for 72h or 360h. The specimens were polished with abrasive papers to #2000 before SSRT tensile tests.

SSRT tensile tests were conducted at an initial strain rate of 1.39×10^{-6} s^{-1} at 25 °C in two environments: humid air (HA) with relative humidity (RH) of 90 % and dry nitrogen gas (DNG) atmosphere at ambient pressure with RH of 5% or less. When tensile testing is carried out in humid air, hydrogen uptake occurs resulting from the breakdown of the surface oxide and the reaction between the bare aluminum surface and water vapor. After tensile tests, fracture surfaces of the specimens were examined with a scanning electron microscopy (SEM).

2.3 HG-SCC test

HG-SCC test has been proposed to evaluate hydrogen embrittlement of aluminium alloys in humid air [10, 11]. In this method, hydrogen embrittlement susceptibility is judged by observing crack propagation of the pre-cracked specimens with stress loading in humid air.

From the hot-rolled plate, compact tension test pieces with a thickness of 6mm were cut by electric discharge machining. They were solution-treated at 500 °C for 1h, water-quenched and then aged at 190°C for 9h, 140°C for 72h or 50°C for 96h. After heat treatment, specimen surfaces were mirror-finished by buffing.

Pre-crack was introduced by fatigue machine and a constant stress intensity factor K_{IApp}, equivalent to 0.2% proof stress at a crack tip of 1mm length, was applied by a constant displacement method. Then, the test pieces were kept in two environments: HA and dry air with relative humidity of 10% or less. After holding the specimens for 30d, crack propagation on the surface was observed with an optical microscope, and then test pieces were subjected to fatigue loading in order to introduce post-crack with length longer than 1 mm, followed by rapid loading up to fracture. Fracture surfaces of the specimens were examined with SEM, and the region between the two fatigue crack surfaces regarded as HG-SCC surface.

3 Results and discussion

Figure 2 shows stress-strain curves of the specimens at a strain rate of 1.39×10^{-6} s^{-1}. Degradation of tensile properties was not observed in both aging conditions. Figure 3 shows SEM images of fracture surfaces of the specimens aged at 140 °C for 72h. In both test environments, fracture surfaces were covered with numerous dimples, which indicates the fracture resulted from ductile manner. In the specimen aged at 140 °C for 360h, intergranular fracture with fine dimples and ledges was observed as shown in Fig.4.

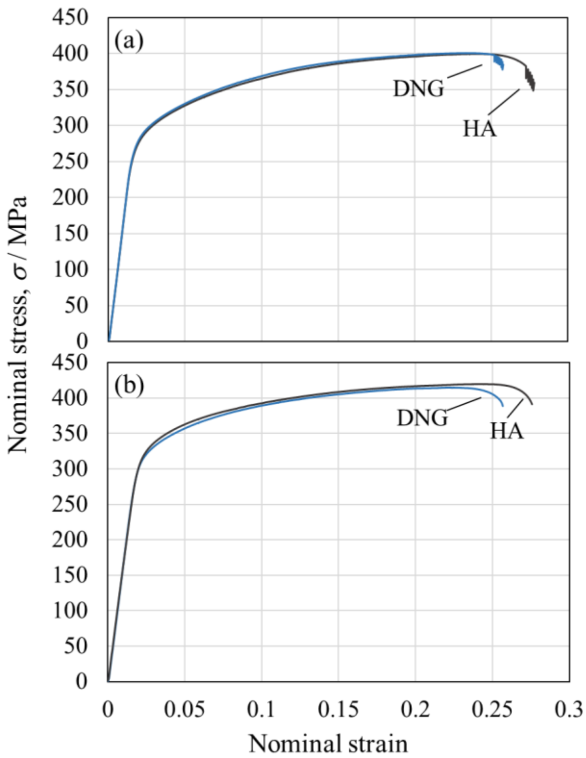

Fig.2 Stress-strain curves of the Al-4%Cu-1.5%Mg alloy tested in humid air (HA) and dry nitrogen gas (DNG) at a strain rate of 1.39×10^{-6} s^{-1}. The specimens were aged at 140°C for (a) 72h and (b) 360h, respectively.

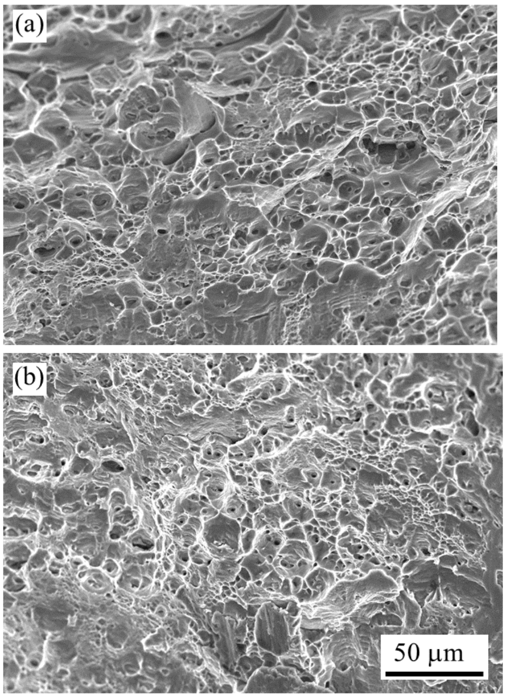

Fig.3 SEM images showing fracture surfaces of the Al-4%Cu-1.5% Mg alloy aged at 140 ºC for 72h after SSRT tensile test in (a) HA and (b) DNG, respectively.

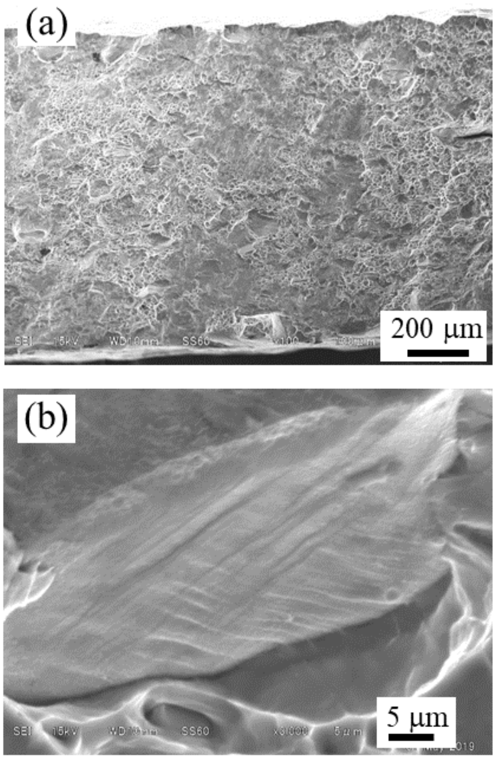

Fig.4 SEM images showing fracture surfaces of the Al-4%Cu-1.5% Mg alloy aged at 140 ºC for 360h after SSRT tensile test in HA. (a): low magnification, (b)enlargement of intergranular fracture surface.

Intergranular fracture surfaces with ledges were observed in the specimens tested in DNG as well as in HA. When fine precipitates on grain boundaries and narrow precipitate free zones are present, large stress concentration at the matrix slip band termination at the grain boundaries causes intergranular fracture with ledges [12]. Therefore, the results of SSRT tensile test and fracture surface observation indicated that the Al-Cu-Mg alloy aged at 140 ºC had high-resistance to hydrogen embrittlement.

Figure 5 shows SEM images of fracture surfaces of the specimen aged at 190ºC for 9h after HG-SCC test in HA. Crack propagation was not observed on any thickness position. Although the SEM images are not shown here, the fracture surface observation confirmed the crack did not propagate in other test conditions. Thus, the Al-Cu-Mg alloy had high-resistance to hydrogen embrittlement examined by HG-SCC test as well as SSRT tensile test.

The origin of high-resistance to hydrogen embrittlement of Al-Cu-Mg alloy has not been realized in the present study. To reveal this, hydrogen behavior in the Al-Cu-Mg alloy is need to be investigated by means of thermal desorption analysis, which is a method to measure hydrogen content and trapping site.

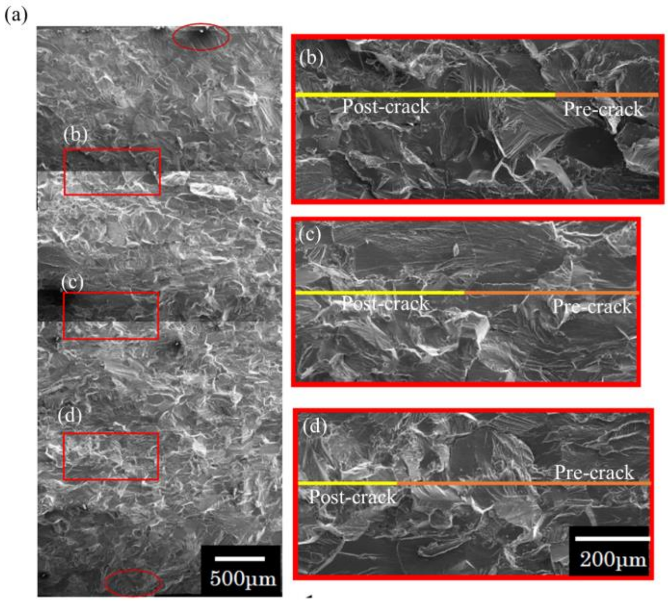

Fig.5 SEM images showing fracture surfaces of the Al-4%Cu-1.5% Mg alloy aged at 190 ºC for 9h after HG-SCC test in HA. (a): low magnification image near boundary between pre-crack and post-crack regions, (b)~(d): enlargement of squares in (a).

4 Conclusions

In the present study, hydrogen embrittlement susceptibility of Al-4%Cu-1.5%Mg alloy was examined by means of SSRT tensile test and HG-SCC test. In SSRT tensile test, degradation of tensile properties was not observed in any aging conditions and fracture surfaces were not affected by test environments. Also, in

HG-SCC test, the crack propagation was not observed in any test conditions. Therefore, the Al-Cu-Mg alloy had high-resistance to hydrogen embrittlement.

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