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# Ductility loss of 7075 aluminum alloys affected by interaction of hydrogen, fatigue deformation, and strain rate



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#### ABSTRACT

The ductility loss phenomenon of the 7075 alloys affected by the interaction of the hydrogen, pre-fatigue deformation, and strain rate was elucidated. In order to investigate the tensile properties of pre-fatigued material in varying humidity environment, quasi-static and impact tensile tests on pre-fatigued materials were conducted, which were prepared in environments with different humidities. The flow stress obtained by tensile test was not affected by pre-fatigue deformation with varying humidity and frequency, regardless of the strain rate. In contrast, pre-fatigue deformation resulted in a loss of ductility. The ductility loss phenomenon, owing to pre-fatigue deformation, was most significant for a combination of long pre-fatigue time, high humidity, and low strain rate. Therefore, the microstructure of the materials was observed in order to determine the main factor. The microstructure of pre-fatigued materials using TEM indicated that dislocations accumulated in some regions inside the crystal grains near the Al<sub>7</sub>Cu<sub>2</sub>Fe (local dislocation field). Thus, the ductility loss phenomenon is believed to occur as this local dislocation field develops during tensile deformation, thereby promoting the formation and growth of voids, and in turn promoting ductile fractures. According to the hydrogen-enhanced localized plasticity theory, accumulated hydrogen promotes the formation and multiplication of dislocations near Al<sub>7</sub>Cu<sub>2</sub>Fe, and increases the density of the local dislocation field, which in turn results in the progression of the ductility loss. Furthermore, the hydrogen transport mechanism owing to dislocations operates at low strain rates; thus, hydrogen affects the dislocation activities during tensile deformation. This results in a faster density increase of the local dislocation field, which in turn promotes a loss of ductility. This phenomenon can therefore be described as a type of hydrogen-induced ductility loss.

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#### 1. Introduction

Many studies have been conducted on the negative effects of hydrogen on the mechanical properties of metals; hydrogen embrittlement, which occurs via hydrogen penetration of metals, results in a loss of ductility. In recent years, with the development of fuel cell vehicles, the connection between hydrogen and mechanical properties has been evaluated. This connection is especially important for lining materials, which can seal off hydrogen, and materials for hydrogen stations under various load conditions such as tensiton, compression and fatigue.

The mechanism of hydrogen embrittlement has been examined analytically, theoretically, and experimentally [1]. We believe that

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experimental examination of the process of hydrogen—main factor of hydrogen embrittlement-diffusion and accumulation within materials is essential to determining the mechanism, which governs hydrogen embrittlement. Therefore, by using a testing machine which is equipped with a quadrupole mass spectrometer installed in an ultrahigh vacuum (QMS-UHV) chamber, we have estimated the diffusion behavior of hydrogen in materials from the corresponding behavior at the tensile deformation and the fracture; this estimation was performed for T6-tempered 6061 and 7075 aluminum alloys (6061 alloy and 7075 alloy, respectively) [2,3]. First, we considered the relationship between deformation processes and the hydrogen diffusion behavior of the 6061 and 7075 alloys, and showed that the amount of hydrogen released was higher in the latter than in the former. Dislocation transport is believed to significantly influence the diffusion of hydrogen during plastic deformation; thus, the relationship between mobile dislocations and hydrogen plays an important role in hydrogen emission. According to Tien [4], maximum interaction between the mobile dislocations and hydrogen occurs at the strain rate  $(\dot{\varepsilon}_c)$  at which the dislocation velocity is maximum in a state where hydrogen is trapped. We showed empirically that hydrogen diffusion is higher at this strain rate than at other strain rates [3]. In the strain rates lower than  $\dot{\varepsilon}_c$ , the dislocation velocity decreases for the hydrogen diffusion rate; therefore, hydrogen diffuses into the material instead of being transported by dislocations. Furthermore, as the duration of diffusion increases, hydrogen becomes more easily trapped in the microstructure, as precipitates in the material. On the other hand, the dislocation velocity increases at strain rates higher than  $\dot{\epsilon}_{c}$ , and as such, dislocations move more rapidly than the rate at which hydrogen diffuses and hydrogen transport via dislocation motion is not favored. However, previous studies were mainly conducted at quasi-static strain rates, compared to the hydrogen diffusion rate, and high-speed phenomena (impact deformation) have been only scarcely studied.

In contrast, the tensile properties of pre-fatigued materials have been studied. For example, Itabashi et al. [5] conducted an impact tensile test on pre-fatigued 2219-T87 aluminum alloys, and reported that its strength was lowered owing to pre-fatigue deformation. There, the relationship between this phenomenon and the pre-fatigue deformation was examined via macro-observations of the damage to the surface and sides of the specimen; however, a definitive correlation was not found. In addition, Santana et al. conducted a study on the ductility of the impact tensile deformation for pre-fatigued steel (4140T) and 6061-T6 aluminum alloys [6-8]. The ductility stemming from the pre-fatigue deformation increased and decreased in the aluminum alloys and steel, respectively. Moreover, the fracture morphology of the surfaces of the aluminum alloys remained unchanged even with prefatigue deformation; however, the fracture on the surfaces of the steel transitioned from ductile to brittle owing to the effect of prefatigue deformation. Although the fracture surfaces were examined in that study, a detailed discussion of the basis of the ductility loss was not provided. Therefore, the ductility loss mechanism owing to pre-fatigue deformation has not been explicitly determined.

As such, in this study, we focused on the relationship among the pre-fatigue deformation, hydrogen, and strain rate. We performed quasi-static and impact tensile tests of 7075 alloys that were pre-fatigued in a humid environment.

#### 2. Experimental

#### 2.1. Specimens

The material used for this study was 7075 aluminum alloy, and its chemical composition is shown in Table 1. Test specimens were cut from rolled sheets of thickness of 1.0 mm to a gauge length of 8.0 mm, a width of 4.0 mm, a fillet radius of 2.0 mm. The specimens were solution treated at 493 °C for 20 min, quenched in water, and then aged at 120 °C for 24 h (T6-temper). All of the specimens were prepared by polishing both surfaces with emery paper (#2000).

**Table 1** Chemical composition of 7075 aluminum alloy (mass%).

| Alloy | Si   | Fe   | Cu   | Mn   | Mg   | Cr   | Zn   | Ti   | Al   |
|-------|------|------|------|------|------|------|------|------|------|
| 7075  | 0.09 | 0.27 | 1.82 | 0.00 | 2.55 | 0.21 | 5.79 | 0.01 | Bal. |

#### 2.2. Pre-fatigue conditions

To apply the effect of atmospheric hydrogen on 7075 alloy during fatigue deformation, we employed the hydrogen introduction method that is used in the slow strain rate tensile test (SSRT) [9,10]. The principle is that a newly formed aluminum surface is exposed to air when a surface oxide film of aluminum alloy is ruptured by tensile deformation in a high-humidity environment. Hydrogen then diffuses the alloy by a reaction at the aluminum surface as follows [9,11,12]:

$$2Al(s) + (3 + X)H_2O \rightarrow Al_2O_3 \cdot X(H_2O)(s) + 3H_2(g)$$
 (1)

To apply pre-fatigue deformation to the specimens in a high-humidity environment, the fatigue test was performed at 25 °C (room temperature) and at more than 90% relative humidity (RH90-PreF). The stress in the pre-fatigue deformation was fixed to 300 MPa, which was an applied maximum stress of 60% of the proof stress for 7075 alloy. The stress ratios were at 0.1 and 10; fatigue testing was performed under elastic pulsating tensile loading. The frequency was fixed at 0.1. The number of cycles was fixed at 6000 times. For comparison, specimens were prepared with pre-fatigue deformation at a relative humidity of less than 20% (RH20-PreF).

#### 2.3. Quasi-static test and impact test

A quasi-static tensile test was performed at initial strain rate of  $1.0 \times 10^{-3} \, \text{s}^{-1}$  at room temperature using a universal testing machine (Instron, 4206).

An impact tensile test was performed using the Split Hopkison Pressure Bar (SHPB) method [13,14]. The SHPB method has become a generally accepted test method for strain rates in the range of  $10^2-10^4\,\mathrm{s^{-1}}$ . Fig. 1 shows a schematic of the SHPB apparatus (Institute of Space Dynamics, ST-R-5000) used for the impact tensile test. This apparatus consists primarily of a striker tube, an input bar, an output bar, and an associated recording system, including a Wheatstone bridge box, a differential amplifier (NF Corporation, 5307), and a digital oscilloscope (Yokogawa Meters & Instruments Corporation, DL750). These bars made of stainless steel are 20 mm in diameter. After the specimen was fitted into the external-thread jig with a taper pin, it was threaded and attached by screw fixation between the input and output bars.

When the striker tube impacts the yoke on the end of the input bar, a tensile stress wave (input wave:  $\varepsilon_i$ ) propagates through the input bar. When the tensile stress wave reaches the specimen attached between the input and output bars, part of the stress wave propagates through the specimen (transmitted wave:  $\varepsilon_t$ ), while the remaining part is reflected at the specimen (reflected wave:  $\varepsilon_r$ ). These stress waves can be measured by a foil strain gauge (KYOWA, KFG-2-120-C1-16) and a semiconductor strain gauge (KYOWA, KSP-1-350-E4) attached to the input and output bars.

By applying the elementary one-dimensional elastic wave propagation theory, we can determine the nominal stress  $(\sigma)$ , the nominal strain  $(\varepsilon)$  and the strain rate  $(\dot{\varepsilon})$  in the specimen as follows:

$$\sigma(t) = \frac{AE}{A_{S}} \epsilon_{t}(t) \tag{2}$$

$$\varepsilon(t) = \frac{2c_0}{l_S} \int_0^t \left[ \varepsilon_i(t) - \varepsilon_t(t) \right] dt$$
(3)

$$\dot{\varepsilon}(t) = \frac{2c_0}{l_S} [\varepsilon_i(t) - \varepsilon_t(t)] \tag{4}$$

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