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# Unified evaluation of hydrogen-induced crack growth in fatigue tests and fracture toughness tests of a carbon steel

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#### 1. Introduction

Hydrogen energy is often spotlighted as an alternative energy source to fossil fuels. Recently, commercial fuel cell vehicles (FCVs) have been launched on the market, and a number of hydrogen refueling stations are under construction. In such hydrogen utilization systems, various metallic components such as hydrogen tanks, pipes and valves are directly exposed to a high-pressure hydrogen gas environment. Hydrogen easily diffuses into metals and deteriorates the mechanical properties of the metal such as tensile strength, ductility, fracture toughness and fatigue strength [1-12].

Current regulations and standards highly restrict the component materials that can be exposed to high-pressure hydrogen. For example, according to the JARI (Japan Automobile Research Institute) standards [13,14], only two types of metallic materials, 316L stainless steel and 6061-T6 aluminum alloy, which have excellent resistance to hydrogen, are allowed. In contrast, the use of bcc steels, such as carbon steels or low alloy steels, is restricted for those high-pressure hydrogen components because of their high susceptibility to hydrogen [15]. However, for the widespread

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# ABSTRACT

To investigate the effect of hydrogen on fatigue life characteristics and crack growth behaviors through the entire fatigue life of a carbon steel, tension-compression fatigue tests and elasto-plastic fracture toughness tests were conducted in a hydrogen gas environment under the pressures of 0.7 and 115 MPa. The fatigue tests revealed that the fatigue life and fracture morphology vary drastically with the hydrogen gas pressure. This study demonstrates that such differences can be explained by the combination of fatigue crack growth properties and fracture toughness properties in hydrogen gas at each pressure.

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commercialization of hydrogen systems, carbon steels and low alloy steels are likely to be used to reduce production costs. To enable such hydrogen-sensitive steels to be used in FCVs and hydrogen refueling stations, it is necessary to understand the mechanism of hydrogen-induced degradation properly. The degradation of fatigue properties is of primary importance because the hydrogen components undergo cyclic stress from the fluctuation of internal gas pressure.

One of the fundamental properties for evaluating the fatigue strength of metallic components is the *S*-*N* curve. Researchers have previously studied the *S*-*N* properties of low alloy steels and carbon steels under the influence of hydrogen [16–18]. However, most of the data were obtained in hydrogen gas at low pressures or in the test using hydrogen-charged specimens, which cannot be applied to the practical design of the ongoing 70 MPa-class FCVs and hydrogen stations. For such high-pressure hydrogen components, it is necessary to clarify the *S*-*N* properties of materials in a hydrogen gas environment under a pressure of more than 100 MPa.

In this study, fully reversed tension-compression fatigue tests using round-bar smooth specimens of low carbon steel JIS-SM490B were conducted in air and in hydrogen gas under pressures of 0.7 and 115 MPa at room temperature. These fatigue tests revealed that the fatigue life properties and fracture morphologies vary drastically depending on the hydrogen gas pressure. In







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Nomenclature			
COD da/dN E f HV J J <sub>1c</sub> K N N f P	crack-mouth opening displacement crack growth rate Young's modulus test frequency Vickers hardness J integral elasto-plastic fracture toughness stress intensity factor number of cycles number of cycles to failure load	$R$ $SZW_{c}$ $\Delta a$ $\Delta J$ $\Delta K$ $v$ $\sigma_{a}$ $\sigma_{B}$ $\sigma_{LY}$ $\varphi$	stress ratio stretch zone width critical stretch zone width crack extension length J integral range stress intensity factor range Poisson's ratio stress amplitude tensile strength lower yield stress reduction of area

addition, elasto-plastic fracture toughness tests using compacttension specimens were performed in the same environments. Considering all of the test results and fractographic observations together, crack growth properties in both the fatigue life tests and the fracture toughness tests are described based on the unified mechanism, hydrogen-induced successive crack growth (HISCG) [5].

# 2. Experimental

# 2.1. Material

The material used in this study was a 17 mm-thick hot-rolled plate of low carbon steel JIS-SM490B, with the chemical composition of 0.16% C, 0.44% Si, 1.43% Mn, 0.017% P and 0.004% S. The mechanical properties of this material obtained in the laboratory air were as follows:  $\sigma_{LY}$  = 360 MPa,  $\sigma_{B}$  = 540 MPa,  $\varphi$  = 78% and HV = 153, where  $\sigma_{LY}$  is the lower yield stress,  $\sigma_{B}$  is the ultimate tensile strength,  $\varphi$  is the reduction of area, and HV is the average Vickers hardness measured with a load of 9.8 N. Fig. 1 shows optical micrographs of polished and nital-etched microstructure. The material has a banded ferrite-pearlite structure that is parallel to the rolling direction.

## 2.2. Method of fatigue tests

The tension-compression fatigue tests were carried out in laboratory air and hydrogen gas under pressures of 0.7 MPa and 115 MPa at room temperature. The tests were conducted under constant stress amplitudes at a stress ratio R of -1 and a test frequency f of 1 Hz. Fig. 2 shows the shape and dimensions of the



Fig. 1. Optical micrograph of the nital-etched microstructure of JIS-SM490B steel.

round-bar specimens used in the fatigue tests. The axis of the specimen coincides with the rolling direction of the hot-rolled plate. The specimen surface was finished by polishing with emery paper and then buffing with a diamond paste. To complete a series of the test programs within a limited time frame, the frequency of the fatigue tests in air was changed from 1 Hz to 10 Hz at an N of  $2 \times 10^6$  cycles and ran out at an *N* of  $1 \times 10^7$  cycles. The tests in hydrogen gas were terminated if the specimen did not fail at an *N* of  $2 \times 10^6$  cycles. The purity of the hydrogen gas used for the experiments was higher than 99.999%. In a tension-compression fatigue test, bending misalignment of the specimen can easily lead to under-estimation of the fatigue limit. To avoid this difficulty, four strain gages were applied to the smooth section near the gripping fixture and the alignment was carefully adjusted in each test. After the fatigue tests, the fracture surfaces were observed with a scanning electron microscope (SEM).

## 2.3. Method of fracture toughness tests

The elasto-plastic fracture toughness ( $J_{\rm ic}$ ) tests were conducted in the same environmental conditions as the fatigue tests. The  $J_{\rm ic}$ tests provide the relationship between the energy for crack extension, *J* integral, and corresponding crack extension length,  $\Delta a$  (*J*- $\Delta a$ curve). In this study, the tests were conducted with strict adherence to ASTM E1820 [19]. This standard offers two alternative procedures for measuring crack extension, i.e., the (i) basic procedure and the (ii) resistance curve procedure. In the basic procedure, various displacements are applied to multiple specimens. Then, for each specimen, the *J* integral value is calculated from the area below the load–displacement curve and  $\Delta a$  is measured from the observation of the fracture surface. On the other hand, in the resistance curve procedure, a single specimen is used for obtaining the *J*- $\Delta a$  data, and crack extension during the test is monitored by the



Fig. 2. Shape and dimensions of the round-bar specimen for fatigue tests. The axis of specimen is parallel to the rolling direction of the hot-rolled plate.

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