Materials Science and Engineering A 528 (2011) 8090–8099

Contents lists available at ScienceDirect

Materials Science and Engineering A

journal homepage: www.elsevier.com/locate/msea

Hydrogen effect on cyclic plasticity and crack growth in coarse-grained iron

Yoji Mine[a,*], Jean-Marc Olive[a,b], Koichi Nagata[a,1], Tetsuro Mitsutani[a,2]

[a] Department of Mechanical Engineering, Kyushu University, Moto-oka, Nishi-ku, Fukuoka 819-0395, Japan
[b] CNRS, Institute of Mechanics and Engineering, UMR 5295, University of Bordeaux-ENSAM-ParisTech, 351 Cours de la Libération, Talence 33405, France

ARTICLE INFO

Article history:
Received 10 March 2011
Received in revised form 6 June 2011
Accepted 20 July 2011
Available online 2 August 2011

Keywords:
Mechanical characterization
Ferrous alloy
Hydrogen embrittlement
Fatigue
Crystal plasticity

ABSTRACT

The effects of hydrogen with a concentration of ~0.1 mass ppm on cyclic plasticity and crack growth were investigated on a Fe–0.01 mass% C alloy. In polycrystalline specimens, the grains covered with multiple gliding were decreased but those including discrete slip bands were increased in the presence of hydrogen. The hydrogen effect on cyclic plasticity of the coarse-grained specimens is characterized by the increased spacing and the decreased height of slip bands. Crystallographic crack growth was retained in the hydrogen-charged specimen unlike the uncharged specimen. This suggests that dissolved hydrogen restricts the number of the activated slip systems at the crack tip.

1. Introduction

Dissolved hydrogen was reported to increase the macroscopic flow stress of various metals and alloys [1–4]. By contrast, nano-scale examinations, e.g., in situ transmission electron microscopy (TEM) [5] and nano-indentation studies [6–8], indicated hydrogen-induced softening, which is seemingly contradictory to the macroscopic response of the bulk materials, i.e., hardening. Uyama et al. [9] inspected the effects of hydrogen precharge on the morphology of slip bands developed by cyclic loading and the fatigue crack initiation in annealed carbon steels with a ferrite–pearlite microstructure. In the macroscopic cyclic stress–strain hysteresis, hardening caused by hydrogen precharging was observed. Close examination of slip band characteristics in the ferrite grains showed that discrete and localized slip bands were formed only in the hydrogen-charged specimen, although cyclic loading without hydrogen caused uniformly deformed grains with multiple slips. This finding suggests that the macroscopic hardening reflects the ununiformity in microstructural scale as a result of the interaction between hydrogen and dislocation, as demonstrated by Birnbaum [10]. In the near-threshold fatigue region, this higher localization of slips also led to a drastic increase of transgranular crack initiation along the slip bands compared to the uncharged specimen, in which the crack initiation is mostly intergranular.

Fatigue process is fundamentally based on the development of persistent slip bands (PSBs), the crack initiation from them, and the cyclic plastic deformation in the vicinity of the crack tip. The effect of hydrogen on the plasticity of metals and alloys was reviewed in Ref. [11]. As characterized by PSBs [12], however, dislocation structures developed during cyclic loading differ from that during monotonic loading. The works on the hydrogen effects on the cyclic plasticity [13] and the development of dislocation structures around the fatigue crack [14] have successfully obtained useful data. Meanwhile, such fatigued dislocation structures can be correlated to the morphologies of slip bands at the specimen surface [15]. For this reason, the quantitative analysis of the hydrogen effect on the slip bands developed in a grain must provide essential information for the mechanistic understanding of the hydrogen-fatigue interactions. This study was initiated to quantify the characteristics of slip bands developed in coarse-grained specimens, with and without hydrogen, of a Fe–0.01 mass% C alloy under cyclic loading. The hydrogen-induced fatigue crack growth was also examined from the crystallographic viewpoint.

2. Material and experimental methods

The material used in this study was a 22-mm diameter rod of a commercially available Fe–0.01 mass% C alloy. The chemical composition in mass% was 0.01 C, 0.01 Si, 0.23 Mn, 0.012 P, and 0.015 S with the balance of Fe. In the as-received alloy, the average grain

* Corresponding author. Tel.: +81 92 802 3286; fax: +81 92 802 3065.
E-mail address: mine@mech.kyushu-u.ac.jp (Y. Mine).
[1] Present address: Future Architect, Inc.
[2] Present address: Denso Corporation.

0921-5093/$ – see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.msea.2011.07.036

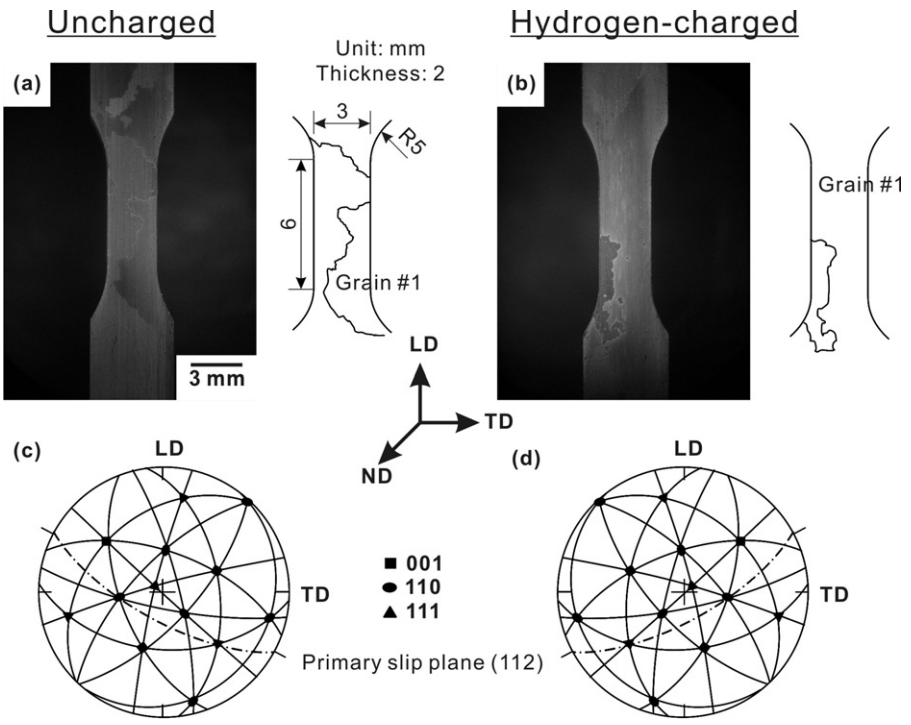

Fig. 1. (a and b) Grain shapes and (c and d) crystallographic orientations of grain #1 in coarse-grained specimens. LD denotes the loading direction, ND the normal direction and TD the transverse direction.

size was ~25 μm and the Vickers hardness, *HV*, was 87 ± 7 on average which was determined from 20 measurements with an applied force of 1.98 N for duration of 30 s, where the error range represents the 95% confidence interval.

Hydrogen release behaviour after hydrogen charging was studied on the polycrystalline iron. Cylinder-shaped samples with a diameter of 8 mm were charged with hydrogen by immersion at open circuit potential in an aqueous solution of 20 mass% NH_4SCN at a temperature of 313 K for a holding time of 48 h. Hydrogen content was measured after holding for the several times by a thermal desorption spectrometer equipped with a quadruple mass spectrometer at a heating rate of 0.5 K s^{-1}. The errors in the thermal desorption spectrometry (TDS) measurement are within ±5% of the measured values. To clarify the effect of plastic deformation on the hydrogen release behaviour, some samples were subjected to tensile and cyclic straining immediately after the hydrogen charge. The tensile straining at a nominal strain rate of 10^{-3} s^{-1} provided a plastic strain of 0.056. The cyclic plastic strain was introduced by tension-compression loading of 1000 cycles at a total strain range of 0.015 and at a frequency of 1 Hz. Neither necking nor cracking were observed in the tensile- and cyclic-strained specimens.

Grains were coarsened by a strain-annealing technique as follows. Sample with a diameter of 14 mm and a length of 160 mm was turned from the rod and then subjected to a nominal strain of approximately 4% by tension. The sample capsulated in a silica tube under a vacuum of 9×10^{-3} Pa was annealed for a holding time of 24 h at a temperature of 1173 K followed by furnace cooling to room temperature. The ~10-mm grain sizes were attained by this method. The coarse-grained sample was cut into a pair of fatigue specimens. A distorted surface layer by machining was electrochemically removed. Fig. 1 shows the shapes of coarse grains in the specimens and their crystallographic orientations determined by the back-reflection X-ray method. One specimen was charged with hydrogen by immersion in an aqueous solution of 20 mass% NH_4SCN at a temperature of 313 K for a holding time of 24 h. The surface of the specimen was re-polished with emery paper #2000 immediately after the hydrogen charging and was finished by electrochemical polishing at a voltage of 11 V in an electrolyte of 2000-ml phosphoric acid with 40-g gelatine and 40-g oxalic acid at 308 K. The surface of the other specimen without hydrogen charging that is the uncharged specimen, was processed in a similar way to the hydrogen-charged specimen. For comparison, a pair of polycrystalline iron specimens was also prepared.

Fatigue tests were conducted in ex-plane bending of plate at room temperature in laboratory air. The stress amplitude at the specimen surface was 260 MPa at a stress ratio of −1 and at a test

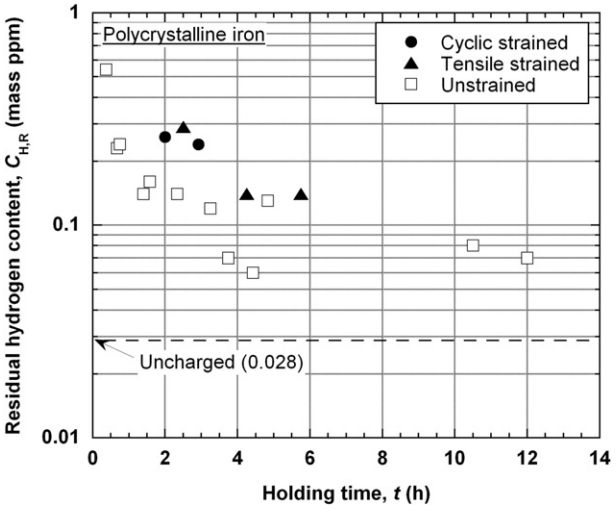

Fig. 2. Residual hydrogen content vs. room-temperature holding time after hydrogen charging in polycrystalline iron.