

# Investigation of Delayed Crack Propagation Associated with Hydrogen Effect in a Thin Sheet of Single-Crystalline BCC Iron-Based Alloy

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(単結晶 BCC 鉄基合金の薄板における水素効果に関連した遅れき裂伝ぱの研究)

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#### 論 文 内 容 の 要 旨

Most metallic materials always contain geometric discontinuities, which are crack-like defects. The crack then propagates and reaches a critical length for satisfying unstable fracture, which leads to sudden failure in steel structures. Furthermore, crack propagation associated with Hydrogen Embrittlement (HE) is the most concern for material engineering. Despite there are numerous studies related to HE in metallic materials, these efforts are still incomplete to fully understand the nature of HE. Especially, under the sustained load that the remote stress is well below the yielding strength, it is supposed that the crack does not propagate in the air environment; however, it will propagate in a hydrogen environment. This shows a strong effect of HE. Most of the previous studies, particularly those related to experiment analyses, were conducted with specimens which have relatively large thickness. Hence, HE process is typically predominated under plane strain conditions where the highest stress tri-axiality exhibits ahead of the crack tip. Significantly reducing the specimen thickness can decrease the stress tri-axiality ahead of the crack tip, then results in the increases of crack tip plastic strain and thereby reducing the amount of hydrogen enrichment near the crack tip. Takahashi et al. conducted a hydrogen-induced delayed crack propagation test using a relatively thin sheet of single-crystalline Fe-Si alloy and found that the crack growth mode was the same as that of thick specimens. They suggested that the plastic deformation around the crack tip affected the rate-limiting process. Again, this showed a strong effect of HE.

On the other hand, common metallic materials are polycrystalline structure. Thus, grain boundary causes some difficulties in clarifying the crack propagation mechanism. Related to HE, in some metallic materials, the formation of hydride is also an obstacle to investigating HE mechanism. Hence, using a single-crystalline Fe-Si alloy is suitable, in which this material has a relatively high yield strength and exhibits free from the potential effect of grain boundary as well as hydride formation. Consequently, the key objective of the present Ph.D. thesis is to investigate characteristics of crack propagation under the sustained load in a hydrogen environment and under monotonic load in air using a thin specimen of single-crystalline Fe-Si alloy based on practical issues in order to contribute to further understand the crack propagation and the nature of HE.

This work includes five chapters. The order of chapters is streamlined in order to clarify the objectives as well as the results of this research. The dissertation is structured as follows:

In Chapter 1, a general introduction was described. Several mechanisms of HE were reviewed, and ductile crack propagation was described. Also, characteristic and the use of single-crystalline materials was briefly discussed. Distribution of hydrogen at the crack tip and hydrogen sources were also pointed out. Finally, the motivations and goals of this work were exposed.

In Chapter 2, characteristics of ductile crack propagation of a center-cracked tension specimen in a hydrogen environment were investigated. A center-cracked specimen was subjected to sustained load in a

hydrogen atmosphere at room temperature. Hydrogen-induced delayed crack propagation was observed. Crack propagation accompanied discontinuously extensive plasticity. The crack tip plastic deformation associated with the effect of hydrogen during the crack propagation leaved three adjacent regions where different plastic strain gradients and dislocation densities were observed. A model that demonstrated the mechanism of hydrogen-induced crack propagation was proposed. The results revealed the effects of plastic deformation and hydrogen-dislocations interaction around the crack tip on the rate-limiting process of hydrogen-induced delayed crack propagation in thin specimens.

In Chapter 3, the effects of hydrogen on macroscopic and microscopic features of crack propagation were investigated. The centered-cracked specimens were tested under a sustained load in a hydrogen environment while under continuous stretching in an air environment. A comparison between these features was made to elucidate the role of hydrogen on the hydrogen-induced delayed crack propagation. The results showed that despite significantly reducing the specimen thickness, the crack growth mode of a thin specimen was the same as that of thick specimens. Surprisingly, the crack propagation process was identical irrespective of test environments and consisted of three stages: (1) crack tip blunting by dislocation emission from the crack tip; (2) micro-voids initiation ahead of the crack tip; (3) sub-crack initiation and opening process. The microstructures were composed of three distinct layers characterized by plastic strain gradients and dislocation densities. These layers were significantly affected by the environment in which Adsorption-Induced-Dislocation-Emission (AIDE) and Hydrogen-Enhanced-Localized-Plasticity (HELP) mechanisms were believed to be relevant. Also, reverse plastic strain in the regions behind the fracture surface due to unloading was observed, which contributed to not only enlarge Crack-Tip-Opening-Angle (CTOA) but also blunt the crack tip.

In Chapter 4, as mentioned above, the crack propagation was discontinuous and leaved striation on the fracture surfaces in both air and hydrogen. This chapter investigated the characterization of striations and slips traces of specimens presented in the above two chapters. Besides, a crack tip plastic deformation at a stationary crack and short crack propagation were investigated by Electron Channeling Contrast Imaging (ECCI) and Electron BackScatter Diffraction (EBSD). Scanning Electron Microscope (SEM) fractography was also aided. The possible mechanism of discontinuous crack propagation, as well as constant striation spacing, was discussed. The results explicitly revealed that the microscopic mechanism of hydrogen-induced delayed crack propagation was one of the probable mechanisms for ductile crack propagation in the present crystal orientation and material. Striation was formed by extensive slips emitted from the crack tip, which were mainly the contribution of specific  $(1\bar{1}2)[\bar{1}11]$  and  $(\bar{1}12)[\bar{1}\bar{1}\bar{1}]$  slip systems. Discontinuous crack propagation was mainly caused by the interaction of the crack and  $(1\bar{1}2)[\bar{1}11]$  and  $(\bar{1}12)[\bar{1}\bar{1}\bar{1}]$  slip bands formed ahead of the crack tip. These slip bands were characterized that the spacing between slip bands was constant and independent of crack length. Hence, striation spacing was the same as that of slip bands ahead of the crack tip.

In Chapter 5, conclusions and future work were presented.