THREE-DIMENSIONAL ANALYSIS ON HYDROGEN-RELATED INTERGRANULAR CRACK PROPAGATION IN MARTENSITIC STEEL

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Abstract
This study investigated three-dimensional propagation behavior of hydrogen-related intergranular crack in martensitic steel by X-ray computed tomography and FIB-SEM serial sectioning. Macroscopic analysis using X-ray computed tomography revealed that the crack morphology exhibited more continuous in the hydrogen-charged specimen. Through FIB-SEM serial sectioning, we found that the crack-tip blunting and ductile rupture of un-cracked ligaments were associated with a certain grain boundary segment in the uncharged specimen. In the case of hydrogen-related intergranular crack propagation, even very fine low-angle grain boundary segments (sub micro-meter size) could act as obstacles to crack propagation. Based on the results, we can propose that misorientation of each grain boundary segment has a large influence on local crack arrestability of intergranular crack propagation.

1. Introduction
From economic and environmental viewpoints, the demand for high-strength steels is increasing. As the susceptibility to hydrogen embrittlement of high-strength steels, particularly martensitic steels, is considerably high, hydrogen embrittlement is one of the serial issues hindering wide application of high-strength martensitic steels. To develop advanced high-strength steels with high resistance to hydrogen embrittlement, understanding the underlying mechanism of hydrogen embrittlement is very important. However, the detailed fracture sequence of hydrogen embrittlement has not yet been clarified. The typical modes of hydrogen-related fracture are classified into quasi-cleavage and intergranular. The hydrogen-related intergranular fractures are often linked with more severe brittle behaviors. Conventional two-dimensional (2D) observation is one of the general methods for statistical analysis on crack propagation behavior. However, it has some difficulties for analysis on crack arrestability in detail. By contrast, three-dimensional (3D) technique can analyze only limited areas so that it is not appropriate for statistical analysis at all. However, the controlling factors for crack arrestability can be effectively examined by 3D analysis. The present study utilized 3D analysis techniques, namely, X-ray computed tomography (X-ray CT) and focused ion beam-scanning electron microscopy (FIB-SEM) serial sectioning, and studied hydrogen-related intergranular fracture behavior in a martensitic steel.

2. Results
The present study used as-quenched martensitic steel (Fe-8Ni-0.1C). Hydrogen was introduced to the heat-treated specimen by electrochemical charging in an aqueous solution containing 3 % NaCl + 3 g L^{-1} NH_4SCN. The diffusible hydrogen content (H_D) was measured by thermal desorption analysis. We found from the fracture toughness tests (unloading compliance tests) that the crack-growth resistance decreased with increasing hydrogen content. However, the hydrogen-related intergranular cracks propagated in a stable manner even when the H_D was large (up to 4 wt. ppm).

The X-ray CT results shown in Figure 1 indicate that the fracture propagated discontinuously with a certain area of un-cracked ligaments. The positions of un-cracked ligament are indicted by arrows. The crack propagation tended to be more continuous with increasing hydrogen content. Moreover, the hydrogen-related intergranular crack can propagate with a small crack opening-displacement. Accordingly, we can propose that the macroscopic continuous crack propagation resulted in the decrease of crack-growth resistance with increasing hydrogen content.

The microscopic 3D analysis using FIB-SEM serial sectioning revealed that the crack-tip blunting and ductile rupture of un-cracked ligaments were associated with a certain grain boundary segment in the

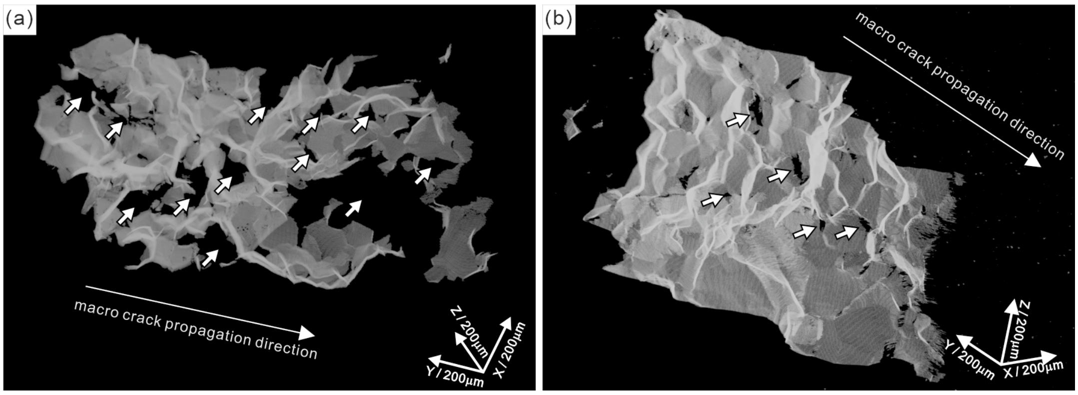

Figure 1 3D crack morphologies reconstructed by X-ray CT of the (a) uncharged specimen and (b) hydrogen-charged specimen (H_D = 4.00 wt. ppm).

uncharged specimen. As shown in Figure 2, the hydrogen-related intergranular crack propagation was arrested locally at fine grain boundary segments (sub micro-meter size). Detailed crystallographic analysis suggested that low-angle grain boundary plane segment acted as an obstacle to crack propagation. Based on the results, we concluded that local crack-arrestability of low-angle grain boundary plane segment could be the possible reason for the stable crack propagation even when the hydrogen content was large.

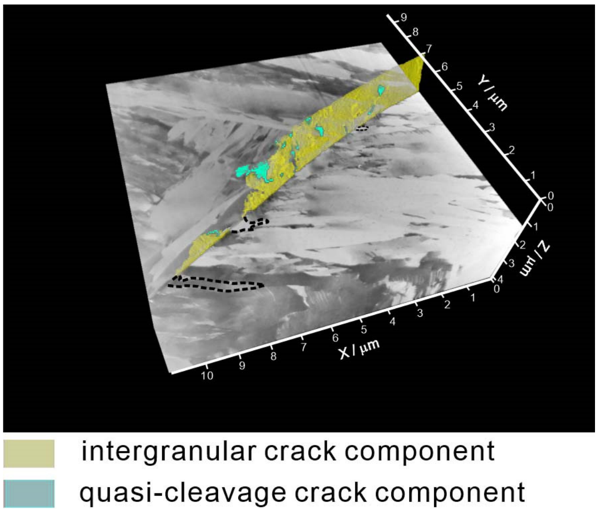

Figure 2 3D crack morphology reconstructed by FIB-SEM serial sectioning and microstructure on the section in the hydrogen-charged specimen (H_D = 4.00 wt. ppm).

3. Conclusions

We investigated the 3D morphology of intergranular crack in the uncharged and hydrogen-charged specimens. The macroscopic crack morphology exhibited more continuous in the hydrogen-charged specimen. The intergranular crack propagation was arrested locally at a certain grain boundary segment. In the case of hydrogen-related intergranular crack propagation, even very fine low-angle grain boundary segments (sub micro-meter size) also could act as obstacles to crack propagation. Based on the results, we can propose that the local crack-arrestability depends on crystallography of each grain boundary segment.

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