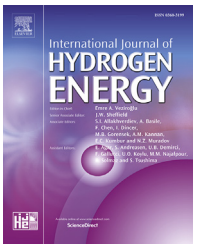
INTERNATIONAL JOURNAL OF HYDROGEN ENERGY XXX (2018) 1–9

Available online at www.sciencedirect.com

ScienceDirect

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



Crystallographic feature of hydrogen-related fracture in 2Mn-0.1C ferritic steel

Kazuho Okada [a,*], *Akinobu Shibata* [a,b], *Yasunari Takeda* [a], *Nobuhiro Tsuji* [a,b]

[a] *Department of Materials Science and Engineering, Kyoto University, Yoshida-honmachi, Sakyo-ku, Kyoto 606-8501, Japan*

[b] *Elements Strategy Initiative for Structural Materials (ESISM), Kyoto University, Yoshida-honmachi, Sakyo-ku, Kyoto 606-8501, Japan*

ARTICLE INFO

Article history:
Received 28 February 2018
Received in revised form
27 April 2018
Accepted 2 May 2018
Available online xxx

Keywords:
Hydrogen embrittlement
Ferritic steel
Electron backscattering diffraction
Crystallographic orientation analysis
Hydrogen micro-print technique

ABSTRACT

The present paper investigated crystallographic feature of hydrogen-related fracture in a 2Mn-0.1C steel having a simple ferritic microstructure. We found that the mechanical properties (in particular post-uniform elongation) were degraded by concurrent hydrogen-charging. Most of the fracture surfaces (over 90%) of the concurrently hydrogen-charged specimens showed quasi-cleavage morphologies with serrated markings, but no intergranular fracture surface was observed. Through a detailed crystallographic orientation analysis using EBSD, we have clarified that micro-cracks formed at ferrite grain boundaries and the micro-cracks propagated inside grains along crystallographic {011} planes of ferrite, leading to the quasi-cleavage fracture. Hydrogen micro-print technique revealed that hydrogen accumulated along ferrite grain boundaries under tensile-loading. On the basis of the obtained results, we propose that the fracture on {011} planes is an intrinsic characteristic of hydrogen-related quasi-cleavage fracture in steels having BCC phases.

Introduction

Catastrophic and premature fracture of metals and alloys due to hydrogen is well documented [1] and termed as 'hydrogen embrittlement', 'hydrogen-related fracture', or 'delayed fracture'. Several models have been proposed to explain reason for hydrogen embrittlement [2,3]. However, essential mechanism of hydrogen embrittlement has not yet been fully understood.

It is well known that high-strength steels, especially martensitic steels, are highly susceptible to hydrogen embrittlement. From viewpoints of fuel-efficiency and collision safety, demands for high-strength steels are increasing more and more in automobile industries, and thus hydrogen embrittlement has become one of the major issues in steel research nowadays. Lath martensitic structure is a typical microstructure in high-strength low- and medium-carbon steels. Lath martensite consists of several structural units with different length scales within a single prior austenite grain, i.e., lath, block, and packet [4–6]. In addition, martensitic structures contain high densities of lattice defects like dislocations introduced by lattice invariant deformation occurring during martensitic transformation [7].

* *Corresponding author.*
E-mail address: okada.kazuho.56z@st.kyoto-u.ac.jp (K. Okada).

https://doi.org/10.1016/j.ijhydene.2018.05.011
0360-3199/

Two typical fracture modes have been recognized in hydrogen embrittlement of martensitic steels: intergranular fracture and quasi-cleavage fracture. Hydrogen-related intergranular fracture always occurs at prior austenite grain boundaries [8–10] although lath martensite structures contain several kinds of boundaries. A first-principle calculations [11] and molecular statics simulations [12] suggested that segregation of hydrogen atoms leads to the reduction of cohesive energy of a grain boundary in iron. Quasi-cleavage fracture, on the other hand, is a transgranular fracture on non-typical cleavage planes in crystals. Resultant fracture surfaces usually show serrated markings, which are different from typical river patterns on typical cleavage fracture surfaces [13–16]. Kim and Morris [17] observed microstructures beneath quasi-cleavage fracture surfaces in martensitic steels by transmission electron microscopy (TEM) and reported that the hydrogen-related quasi-cleavage fracture surfaces were parallel to lath boundaries of martensite. Nagao et al. [18] also proposed using focused ion beam machining and TEM observations that the quasi-cleavage fracture in a low-carbon martensitic steel propagated along lath boundaries. Shibata et al. [19–21] investigated microstructural and crystallographic features of the hydrogen-related fracture in low- and medium-carbon martensitic steels using electron backscattering diffraction (EBSD) analysis and revealed that the quasi-cleavage fracture occurred along {011} planes of martensite.

Hydrogen-related fracture behaviors in martensitic steels have been extensively studied because of their high susceptibility to hydrogen embrittlement. However, it is still not understood whether the characteristics described above are intrinsic ones of hydrogen-related fracture or originated from the martensitic structures. In addition, the complicated microstructures of martensitic steels make it difficult to observe changes of microstructures during hydrogen-related deformation. To study ferritic steels with simple ferrite microstructures is suitable to understand the intrinsic nature of hydrogen-related fracture. Martin et al. [14] reported that voids initiated at and extended along intersections between slip bands led to the quasi-cleavage fracture in a pipeline steel. Wang et al. [22] confirmed hydrogen-related intergranular fracture in a stress relaxation test of pure iron, and observed organized dislocation cell structures just beneath the intergranular fracture surfaces. However, these previous studies about hydrogen-related fracture of ferritic steels concentrated on observing dislocation structures, and crystallographic features of hydrogen-related fracture in ferritic steels have not yet been understood, even though such features are very important for understanding underlying mechanisms of hydrogen-related fracture. Earlier studies suggested the occurrence of hydrogen-related fracture on {011} plane in ferrite structure [23–26]. For example, Takano et al. [24] studied hydrogen-related fracture behavior of iron single crystal by etch-pits technique and reported that the hydrogen-related fracture occurred on {011} plane. More precise and detailed analysis will be benefit for deep understanding the crystallographic features of hydrogen-related fracture in ferrite structure. In the present research, we systematically investigate crystallographic feature of hydrogen-related fracture in a low-carbon steel having a simple ferrite microstructure.

Experimental

A 2Mn-0.1C (mass %) steel was used in the present study. The detailed chemical composition of the steel is shown in Table 1. As-cast ingot of the 2Mn-0.1C steel was cold-rolled from 15 mm to 1.5 mm in thickness, and then austenitized at 1173 K for 1.8 ks in vacuum, followed by furnace cooling. After the heat treatment, the specimens were mechanically ground down to a thickness of 1 mm in order to remove decarburized layers formed during the heat treatment.

Sheet-type tensile test specimens with a gauge length of 10 mm, width of 5 mm, and thickness of 1 mm were cathodically pre-charged with hydrogen for 86.4 ks. The hydrogen charging solution was 3% NaCl + 3 g L^{-1} NH_4SCN, and the used current density was 5 A m^{-2}. Uniaxial tensile tests at a slow strain rate of 8.3×10^{-6} s^{-1} were performed at ambient temperature under two different conditions, i.e., in air and under hydrogen concurrent-charging condition. The solution and current density for the hydrogen concurrent-charging were the same as those in the pre-charging procedure described above. For the hydrogen concurrent-charging tensile test, the solution was exchanged with new one before the test started in order to keep the hydrogen-charging-ability of the solution. In the tensile test in air using the pre-charged specimen, the test was started always 2.4 ks after the completion of pre-charging. It is well known that diffusible hydrogen, which can move around room temperature, plays a crucial role on hydrogen embrittlement [27]. Diffusible hydrogen content (H_D) was measured using thermal desorption analysis (TDA) at a heating rate of 100 K/hr using J-SCIENCE LAB CO. Ltd.: JTF-20A. TDA was started always 2.4 ks after the completion of pre-charging or tensile test.

Fracture surfaces of tensile-tested specimens were observed by scanning electron microscopy (SEM) using JEOL: JSM-7800F. Sections perpendicular to the normal direction (ND) of the tensile-tested specimens were analyzed using EBSD in SEM (JEOL: JSM-7100F). A Ni layer with a thickness of approximately 100 μm was electrodeposited onto the tensile-tested specimens to preserve the fracture surfaces. The electrodeposition solution was an aqueous solution of 150 g L^{-1} Ni_2SO_4 + 15 g L^{-1} H_3BO_4. The deposition was performed at ambient temperature and current density of 30 A m^{-2} for 265.6 ks. The specimen thickness after the Ni electrodeposition was about 1.2 mm. One side of the electrodeposited specimen was mechanically polished until the specimen reached a final thickness of approximately 0.6 mm, and then electrolytically polished in the solution before the observation. In addition, microstructures around micro-voids and cracks were observed by back-scattering electron (BSE) image using SEM.

Hydrogen accumulation behavior during tensile-loading was evaluated using hydrogen micro-print technique. The hydrogen micro-print technique is a method to analyze local

Table 1 – Chemical composition of the steel used in the present study (mass %).

C	Mn	Si	P	S	Fe
0.103	2.03	0.01	<0.002	0.0010	Bal.

Please cite this article in press as: Okada K, et al., Crystallographic feature of hydrogen-related fracture in 2Mn-0.1C ferritic steel, International Journal of Hydrogen Energy (2018), https://doi.org/10.1016/j.ijhydene.2018.05.011