

# Changes in mechanical properties following cyclic prestressing of martensitic steel containing vanadium carbide in presence of nondiffusible hydrogen

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## ARTICLE INFO

### Article history:

Received 2 June 2016

Received in revised form

25 July 2016

Accepted 4 August 2016

Available online 5 August 2016

### Keywords:

Mechanical property

Hydrogen embrittlement

Cyclic loading

Martensite

Lattice defects

Vanadium carbide

## ABSTRACT

Changes in the states of nondiffusible hydrogen and mechanical properties after cyclic prestressing in the presence of only nondiffusible hydrogen were examined for martensitic steel containing vanadium carbide. The relationship between the change in the state of nondiffusible hydrogen and mechanical properties was also investigated. The hydrogen desorption profile in the high-temperature range decreased and that in the low-temperature range increased with increasing stress amplitude during cyclic prestressing in the presence of only nondiffusible hydrogen. Thus, the application of cyclic prestressing changed the state of hydrogen from a stable to an unstable one because of vacancies and their clusters. Hydrogen embrittlement susceptibility after cyclic prestressing increased with increasing stress amplitude and number of prestressing cycles in the presence of only nondiffusible hydrogen. This relationship indicates that hydrogen embrittlement susceptibility increased with the increasing amount of hydrogen detrapped from trap sites of nondiffusible hydrogen during cyclic prestressing. These results revealed that nondiffusible hydrogen easily detrapped from vanadium carbide due to the application of cyclic prestress and probably interacted with vacancies and their clusters, thus increasing hydrogen embrittlement susceptibility. The change of nondiffusible hydrogen to diffusible hydrogen and accumulation of vacancies and their clusters during cyclic prestressing are concluded to be the dominant factors in hydrogen embrittlement after the application of cyclic prestress.

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## 1. Introduction

One of the authors has reported that not only the hydrogen content and applied stress, but also the state of hydrogen is closely involved in hydrogen embrittlement. For instance, nondiffusible hydrogen of 2.7 mass ppm does not cause ductility loss, but diffusible hydrogen of 0.8 mass ppm does [1]. Based on these results, formation of hydrogen trap sites where hydrogen is strongly trapped and strong trapping of harmful diffusible hydrogen at trap sites as nondiffusible hydrogen have been widely used as methods to reduce hydrogen embrittlement susceptibility of high-strength steel [1]. As examples of the reduction of hydrogen embrittlement susceptibility, the formation of hydrogen trap sites at the interface of TiC due to Ti addition [2–5] and at the interface of VC due to V addition [6,7] has been reported.

It has also been found that the state of hydrogen is changed

because of the interaction between hydrogen and dislocations. For instance, a hydrogen desorption peak newly appears in the high-temperature range in the desorption profile of diffusible hydrogen resulting from the application of plastic strain [8], constant elastic stress [9], cyclic elastic stress [10], and rotational bending [11,12] in the presence of diffusible hydrogen. This new hydrogen desorption peak corresponds to a hydrogen-induced lattice defect caused by the interaction between hydrogen and dislocations and is involved in hydrogen embrittlement. More specifically, not all hydrogen, but rather diffusible hydrogen in steel plays a role in hydrogen embrittlement.

In contrast to diffusible hydrogen, the hydrogen desorption profiles of cold-drawn pearlitic steel were not changed by the application of plastic strain in the presence of only nondiffusible hydrogen. One of the authors has reported that the formation of hydrogen-induced lattice defects is not enhanced because nondiffusible hydrogen does not interact with dislocations, even under the application of plastic strain [13]. In the case of cold-drawn pearlitic steel subjected to cyclic stress, it has been shown that when nondiffusible hydrogen alone is present, it does not enhance the formation and propagation of cracks in fatigue tests [14].

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In this context, the effects of diffusible hydrogen on the state of hydrogen and hydrogen embrittlement under the application of various stress types have been researched. However, few studies have attempted to investigate the effects of nondiffusible hydrogen on the state of hydrogen and hydrogen embrittlement under cyclic stress. Changes in the state of nondiffusible hydrogen due to the application of cyclic prestress and subsequent hydrogen embrittlement susceptibility of vanadium-added martensitic steel in the presence of nondiffusible hydrogen were examined in the present study. The relationship between the change in the state of nondiffusible hydrogen and hydrogen embrittlement susceptibility was also investigated. Moreover, reversibility of the changed state of hydrogen and hydrogen embrittlement susceptibility after the application of cyclic prestress with nondiffusible hydrogen was also clarified.

## 2. Experimental

### 2.1. Material

The chemical composition of the quenched and tempered martensitic steel examined in the present study was 0.41 mass% C, 0.20 mass% Si, 0.71 mass% Mn, 0.005 mass% P, 0.005 mass% S, and 0.30 mass% V. Plate specimens of 1 mm in thickness were quenched at 920 °C and tempered at 600 °C to precipitate vanadium carbide (VC). The specimens were machined to 2.5 mm in width, 1 mm in thickness and 20 mm in gage length. Their tensile strength ( $\sigma_B$ ) was 1420 MPa.

### 2.2. Hydrogen charging and hydrogen analysis

The specimen surface was polished with #1000 emery paper. Specimens were charged with hydrogen by immersion in a 20 mass%  $\text{NH}_4\text{SCN}$  solution kept at a temperature of 30 °C as specified by the FIP (Fédération Internationale de la Précontrainte) test procedure [15]. Hydrogen-charged specimens were cut to 20 mm in length from the hydrogen-charged area and their hydrogen content and states of hydrogen were analyzed by thermal desorption analysis (TDA) using a gas chromatograph at a heating rate of 100 °C  $\text{h}^{-1}$  from room temperature to 250 °C. The sample gas was analyzed every 5 min. A standard gas mixture of Ar + 50 vol ppm  $\text{H}_2$  was used for calibration of the hydrogen content.

### 2.3. Preparation of standard specimen with only nondiffusible hydrogen

The residual hydrogen content was measured by means of TDA after a plate specimen was immersed for a sufficient time for the hydrogen concentration to reach complete equilibrium at the center of the specimen, which was then kept at 30 °C for 0–25 d in a thermostatic chamber. The holding time to completely remove diffusible hydrogen from the specimen, i.e., only nondiffusible hydrogen remained, was determined based on the hydrogen content and state of hydrogen as measured by TDA. The specimen containing only nondiffusible hydrogen is defined as the standard specimen in the present study.

### 2.4. State of hydrogen and hydrogen embrittlement susceptibility after application of cyclic prestress

The state of hydrogen and hydrogen embrittlement susceptibility were evaluated by means of TDA and a tensile test, respectively, right after the application of cyclic prestress to the standard specimen in the presence of only nondiffusible hydrogen. Fig. 1 shows a schematic diagram of the applied cyclic prestress

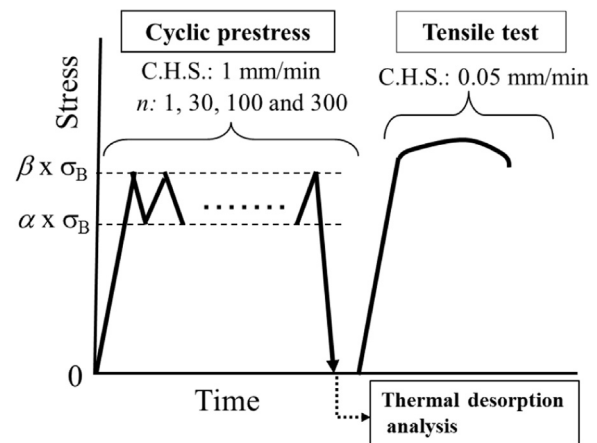


Fig. 1. Schematic diagram of applied cyclic prestress and subsequent tensile test and thermal desorption analysis.

and subsequent TDA and the tensile test.

The applied cyclic prestress levels were a triangular wave at A ( $\alpha$ : 0.4,  $\beta$ : 0.7), B ( $\alpha$ : 0.05,  $\beta$ : 0.7) and C ( $\alpha$ : 0.05,  $\beta$ : 0.9), where  $\alpha$  and  $\beta$  are the lower and upper limits of the applied stress ratio, respectively. The crosshead speed (C.H.S.) during cyclic prestressing was 1 mm/min. The numbers of cycles ( $n$ ) were 1, 30, 100 and 300. The C.H.S. in the subsequent tensile test was 0.05 mm/min, and hydrogen embrittlement susceptibility was then evaluated by the difference in fracture strain.

### 2.5. Reversibility of state of hydrogen and hydrogen embrittlement susceptibility

The reversibility of the change in the state of hydrogen during the application of cyclic prestress and hydrogen embrittlement susceptibility was also investigated. The state of hydrogen and hydrogen embrittlement susceptibility were evaluated by means of TDA and a tensile test, respectively, after the application of cyclic prestress to the standard specimen in the presence of only nondiffusible hydrogen and holding for 0, 1 and 7 d at 30 °C in the thermostatic chamber.

### 2.6. Comparison of hydrogen embrittlement susceptibility of specimens after application of constant prestress or cyclic prestress

The hydrogen embrittlement susceptibility of specimens after the application of constant prestress or cyclic prestress in the presence of only nondiffusible hydrogen was compared. Two types of specimens were prepared. The first type was subjected to 30 cycles of cyclic prestress at the prestress levels of B (0.05–0.7  $\sigma_B$ ). The second type was subjected to constant prestress at 0.7  $\sigma_B$ , i.e., the maximum stress of prestress level B, for 30 min. Constant prestress was applied for a longer time than the 30 cycles of cyclic prestress at prestress level B (20 min). In other words, the multiplication between applied prestress and its application time of the specimen to which constant stress was applied was higher than that of the specimen cyclically prestressed.

## 3. Results

### 3.1. State of hydrogen in vanadium-added martensitic steel

Fig. 2 shows the hydrogen desorption profiles of specimens immersed in a 20 mass%  $\text{NH}_4\text{SCN}$  solution at a temperature of 30 °C for various times. For the specimen hydrogen-charged for

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