

# Change in dislocation mobility of tempered martensitic steel through charged hydrogen

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**Abstract.** To clarify the relationship between dislocation slip and hydrogen for elucidation of hydrogen embrittlement mechanism, the effect of hydrogen on dislocation slip was examined by comparing changes in the stress-strain curve and stress relaxation behavior with/without hydrogen charging. Tempered martensitic steel with the tensile stress of 1480 MPa was used. Hydrogen charging was conducted by the electrolysis charging. Tensile tests were conducted for hydrogen-charged specimens containing hydrogen from 0 to 5.6 mass ppm at strain rates from  $1.83 \times 10^{-5}$  to  $1.83 \times 10^{-2} \text{ s}^{-1}$ . In the stress-strain curve, the elastic modulus did not change, but the upper and lower yield points increased through charged hydrogen. The tensile strength increased and the work hardening rate did not change through charged hydrogen. The yield points and tensile strengths increased with increasing the amount of hydrogen. It suggests that hydrogen suppressed dislocation slip, that is, hydrogen caused hardening. Stress relaxation tests were conducted for hydrogen-charged specimens containing hydrogen from 0 to 4.7 mass ppm. Even though the amount of hydrogen increased, the amount of stress relaxation remained constant. Therefore, hydrogen probably did not promote the increase in dislocation density. These results reveal that hydrogen causes not softening but hardening.



## 1 Introduction

Mechanical properties of steel include various indexes such as yield strength and work hardening rate. It is important to examine the influence of hydrogen on these indexes in detail in considering the hydrogen embrittlement mechanism from the viewpoint of the relationship between dislocation slip and hydrogen. It has been reported that dislocation slip is suppressed [1] or promoted [2] by hydrogen charging. Since these results were influenced by the concentration of impurities, hydrogen content, temperature, and strain rate, among other factors, a unified view has not been obtained. Several methods, for example, tensile tests and stress relaxation tests, are utilized for evaluating the effect of hydrogen on mechanical properties [1]. The relationship between dislocation slip and hydrogen can be evaluated in more detail by comparing the results obtained with these methods. Therefore, in this study, the influence of hydrogen on dislocation slip was investigated by several experimental methods for tempered martensitic steel, which is a practical steel containing a lot of impurities.

## 2 Experimental

Induction quenched and tempered martensitic steel with a tensile stress of 1480 MPa was used as the specimens. Table 1 gives the chemical composition of the specimens and figure 1 shows their shape. The specimens had a parallel part with a diameter of 3 mm and a length of 20 mm. The specimen shows quasi-cleavage fracture in hydrogen environment [3]. The specimen surface was polished using emery paper (800, 1000, and 2000 grit). They were electrochemically charged with hydrogen in an aqueous solution of 0.1 N NaOH and 5.0 g·L<sup>-1</sup> NH<sub>4</sub>SCN with a current density from 0 to 30 A·m<sup>-2</sup> at 30 °C for 72 h. Tensile tests were carried out at strain rates from 1.83×10<sup>-5</sup> to 1.83×10<sup>-2</sup> s<sup>-1</sup>. The stress relaxation test was carried out in the following procedure. Specimens were electrochemically charged with hydrogen in an aqueous solution of 0.1 N NaOH and 5.0 g·L<sup>-1</sup> NH<sub>4</sub>SCN with a current density from 0 to 15 A·m<sup>-2</sup> at 30 °C for 72 h. They were subjected to stress of 0.92  $\sigma_B$  (elastic region) or 0.98  $\sigma_B$  (plastic region) at a strain rate of 1.83×10<sup>-5</sup> s<sup>-1</sup> and locked at the gripper interval for 1800 s.  $\sigma_B$  is a tensile stress. The specimen was deformed up to 0.92  $\sigma_B$  or 0.98  $\sigma_B$  then the machine was stopped and the specimen allowed relaxing. The stress decrease with time was recorded. The relaxation stress was defined as the difference between the initial subjected stress and the stress after the test. The hydrogen content was measured by thermal desorption analysis (TDA) using a gas chromatograph at a heating rate of 100 °C·h<sup>-1</sup>. The hydrogen contents of specimens charged with hydrogen at current densities of 5, 10, 15 and 30 A·m<sup>-2</sup> were 1.1, 3.1, 4.7 and 5.6 mass ppm, respectively.

**Table 1.** Chemical composition of specimen material (mass %)

C	Si	Mn	P	S
0.32	1.60	0.75	0.014	0.005

### 3 Results

#### 3.1 Tensile test

Figure 2 shows the stress-strain curves of tempered martensitic steel specimens with various hydrogen contents at a strain rate of  $1.83 \times 10^{-5} \text{ s}^{-1}$ . Elongation decreased with increasing hydrogen content, whereas the upper and lower yield points and the tensile strength increased. The elastic modulus did not change with increasing hydrogen content.

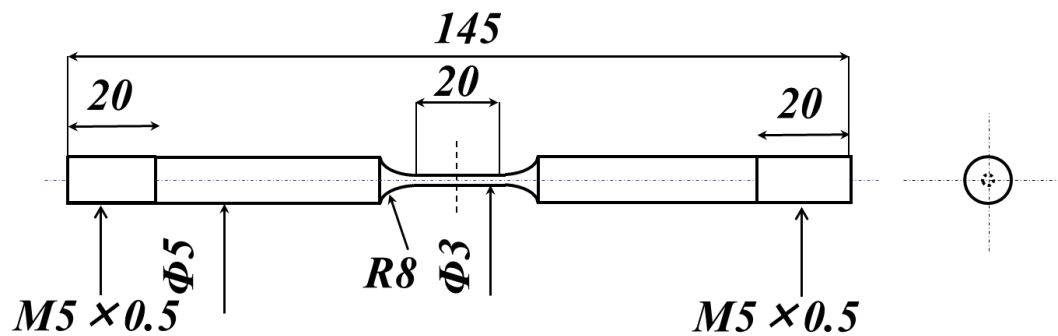


Figure 1. Shape of specimen

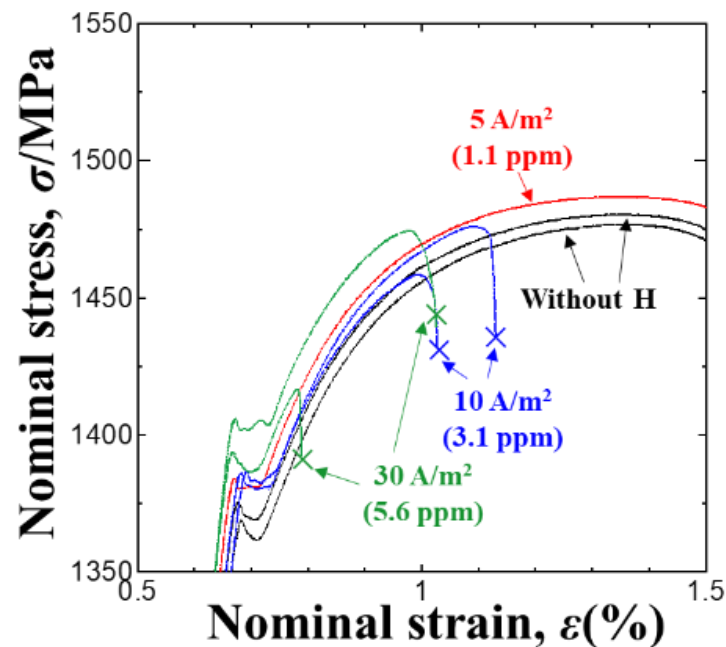
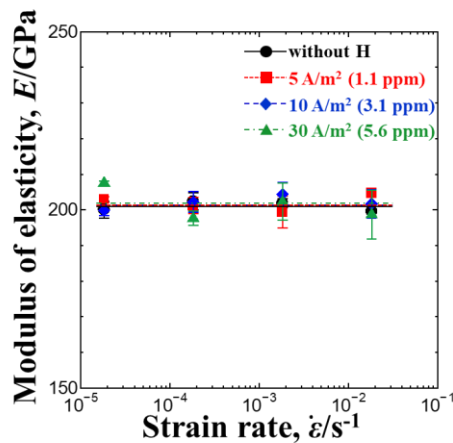
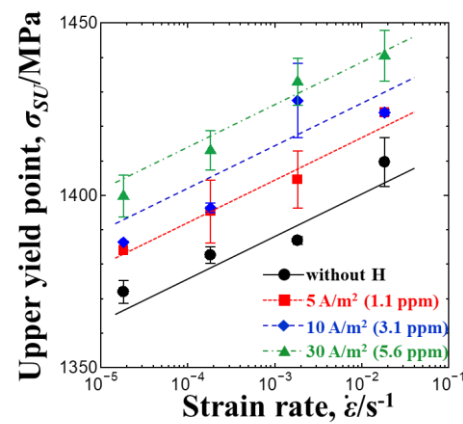


Figure 2. Stress-strain curves of tempered martensitic steel specimens with various hydrogen contents at a strain rate of  $1.83 \times 10^{-5} \text{ s}^{-1}$ .

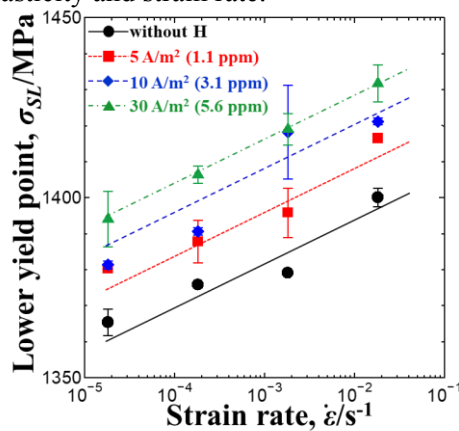
Figure 3 shows the relationship between the modulus of elasticity and strain rate. The modulus of elasticity was defined as the slope of straight line connecting the point at 0 MPa and 1000 MPa in the stress-strain curve. The modulus of elasticity did not change with increasing strain rate at any hydrogen content, nor did it change with increasing hydrogen content at any strain rate. Figure 4 shows the relationship between the upper yield point and strain rate. The upper yield point increased with increasing strain rate at any hydrogen content and it increased with increasing hydrogen content at any strain rate. Figure 5 shows the relationship between the lower yield point and strain rate. The lower yield point increased with increasing strain rate at any hydrogen content and it increased with increasing hydrogen content at any strain rate. Figure 6 shows the relationship between the tensile strength and strain rate. The tensile strength increased with increasing strain rate at any hydrogen content and it increased with increasing hydrogen content at any strain rate. Therefore, the upper and lower yield points and the tensile strength increased with increasing hydrogen content at any strain rate. The results suggest that hydrogen suppressed dislocation slip,



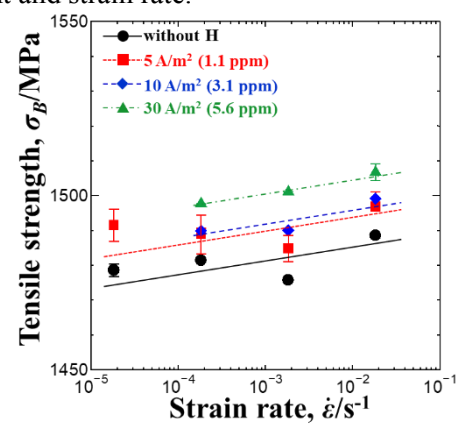
**Figure 3.** Relationship between modulus of elasticity and strain rate.



**Figure 4.** Relationship between upper yield point and strain rate.



**Figure 5.** Relationship between lower yield point and strain rate.



**Figure 6.** Relationship between tensile strength and strain rate.

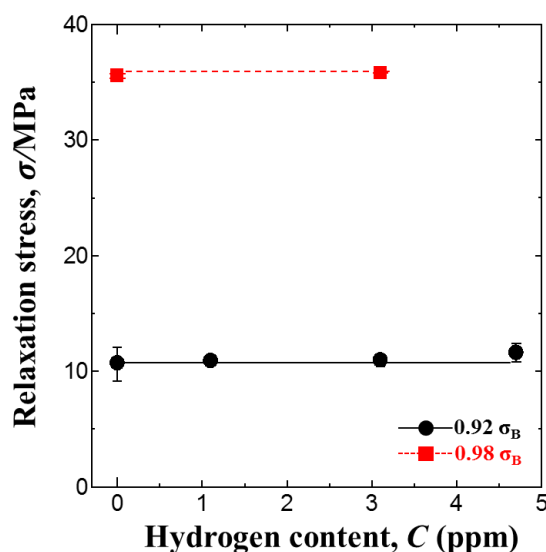
i.e., it caused hardening.

### 3.2 Stress relaxation test

Figure 7 shows the relationship between relaxation stress and hydrogen content. Even though the amount of hydrogen increased, the amount of stress relaxation remained constant under all stress conditions. Hence, hydrogen probably did not promote any increase in dislocation density.

## 4 Discussion

A previous study reported that the formation of lattice defects is accelerated by plastic strain in the presence of hydrogen in pure iron [4], Inconel 625 [4], SUS304 [5], and SUS316L [5]. It has been reported that vacancy defects such as vacancies or vacancy clusters form in cold-drawn pearlitic steel at the rupture location in the plastic region in the presence of hydrogen [6]. It has been reported that resistance to deformation increases through the introduction of vacancies by neutron irradiation in SUS304, SUS316 and SUS347 [7]. Therefore, the cause of the increase in the lower yield stress and tensile strength with increasing hydrogen content in the present study was probably due to hydrogen or vacancy-type defects. Presumably, such defects became resistance to dislocation slip. On the other hand, it has been reported that strain-induced lattice defects are not increased by hydrogen in the elastic region in cold-drawn pearlitic steel. Several models have been proposed for the relationship between the concentration of solute atom and solid solution strengthening [8, 9]. Hence, the cause of the increase in the upper yield stress with increasing hydrogen content was probably due to hydrogen. It implies that hydrogen promoted resistance to dislocation slip. However, it is a subject for future study whether strain-induced lattice defects are promoted or not by straining in the elastic/plastic region in the presence of hydrogen in tempered martensitic steel. A further subject for study is whether these defects affect dislocation slip or not.



**Figure 7.** Relationship between relaxation stress and hydrogen content.

In high strength steels which are frequently used in practice, few studies have been reported about hardening or softening by hydrogen. However, in the present study, it was revealed that in the tempered martensitic steel, softening phenomenon by hydrogen charging was not observed in a wide range from the elastic region to the plastic region, and hardening phenomenon was specifically observed in the plastic region. From these results, it was suggested that hydrogen did not promote the movement of dislocation but obstruct it. These are important results that can lead to elucidation of the mechanism of hydrogen embrittlement in the real environment.

**5 Conclusion**

The following findings were obtained concerning the influence of hydrogen on dislocation slip in tempered martensitic steel.

- (1) Tensile tests were conducted for hydrogen-charged specimens containing hydrogen from 0 to 5.6 mass ppm at strain rates from  $1.83 \times 10^{-5}$  to  $1.83 \times 10^{-2} \text{ s}^{-1}$ . The modulus of elasticity did not change with increasing strain rate at any hydrogen content. The upper and lower yield points and the tensile strength increased with increasing strain rate at any hydrogen content. The modulus of elasticity did not change with increasing hydrogen content at any strain rate. The upper and lower yield points and the tensile strength increased with increasing hydrogen content at any strain rate.
- (2) Stress relaxation tests were conducted for hydrogen-charged specimens containing hydrogen from 0 to 4.7 mass ppm. Even though the amount of hydrogen increased, the amount of stress relaxation remained constant at both stress conditions of  $0.92 \sigma_B$  and  $0.98 \sigma_B$ .
- (3) Hydrogen caused not softening but hardening. It was suggested that hydrogen did not promote the movement of dislocation but obstruct it.

**References**

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