

Effect of strain rate on hydrogen embrittlement susceptibility of tempered martensitic steel

Yuji Sakiyama^{1*}, Tomohiko Omura¹, Kazuki Sugita², Masataka Mizuno², Hideki Araki², Yasuharu Shirai²

¹ Nippon Steel & Sumitomo Metal Corporation, Steel Research Laboratories, Amagasaki, 660-0891 Japan

² Center for Atomic and Molecular Technologies, Graduate School of Engineering, Osaka University, Suita, 565-0871 Japan

Abstract: The effect of strain rate on hydrogen embrittlement susceptibility of high strength tempered martensitic steel was investigated by tensile tests under cathodic hydrogen charge. Fracture elongation was decreased with a decrease in strain rate and increase in diffusible hydrogen concentration. The mechanism of hydrogen embrittlement susceptibility was investigated based on vacancy-type lattice defects formation by positron lifetime spectroscopy. Dislocation density and vacancy density decreased with a decrease in strain rate. This means that the both parameters decreased with a decrease in fracture elongation, and do not reflect hydrogen embrittlement susceptibility. On the other hand, vacancy clustering was promoted with a decrease in strain rate. Therefore, it is assumed that strain rate dependence of hydrogen embrittlement susceptibility is determined by vacancy clustering process.

1. INTRODUCTION

It is known that hydrogen embrittlement is promoted with a decrease in strain rate in tensile tests under hydrogen charge. It is considered that the strain rate dependence of hydrogen embrittlement susceptibility is controlled by hydrogen diffusion into stress concentration area or the interaction between dislocation motion and hydrogen diffusion. However, the mechanism has not been clarified yet.

In the present study, the effect of strain rates on hydrogen embrittlement susceptibility of high strength tempered martensitic steel was investigated using tensile tests under hydrogen charge. For discussing the mechanism, the lattice defects formation in the fractured specimens was investigated by positron lifetime spectroscopy.

2. EXPERIMENTAL PROCEDURE

2.1. Materials

Low-alloy steel, JIS (Japanese Industries Specification)-SCM435, with chemical composition in Table 1 was used in the present study. The steel was melted in a vacuum induction furnace and cast into an ingot. The ingot was hot rolled into 10 mm thick plates. The plates were kept at 1153 K for one hour, quenched by water, and then tempered at 683 K for one hour. The obtained sample steel comprised tempered martensitic structure, and its tensile strength was 1411MPa.

Table 1. Chemical composition of steel used (mass%).

C	Si	Mn	P	S	Al	Cr	Mo	N
0.33	0.19	0.76	0.011	0.005	0.03	1.05	0.20	0.003

2.2. Tensile tests under cathodic hydrogen charge

Tensile test specimens with 25mm gauge length and 4mm diameter were machined from the sample steel. The specimen surfaces were mechanically polished by water-proof abrasion papers with grit number of up to 600. The specimens were strained at different strain rates between $5 \times 10^{-7} \text{ s}^{-1}$ and 10^{-2} s^{-1} until fracture either in air or under cathodic hydrogen charge at room temperature. The cathodic hydrogen charge was conducted under current control with a Pt electrode in any of the following three conditions: (A) at $i_c=1 \text{ A/m}^2$ in 3% NaCl, (B) at $i_c=10 \text{ A/m}^2$ in 3% NaCl, and (C) at

* Corresponding author. E-mail: sakiyama.76d.yuuji@jp.nssmc.com, telephone: +81 06 6489 5716.

$i_c=10 \text{ A/m}^2$ in 3% NaCl + 3 g/L NH_4SCN , where i_c is cathodic current density. In general, the charging condition is more severe in the order of the conditions A, B, and C. Before the straining, precharge was conducted for 24 hours at room temperature to saturate hydrogen into the specimen. Cathodic hydrogen charge was performed by covering the tested area of tensile specimen with a cell filled with the solution. Fracture elongation was obtained based on the change in the total length of specimen before and after the test. Hydrogen embrittlement susceptibility was evaluated by relative fracture elongation, which is a ratio of fracture elongation under hydrogen charge to that in air. The fracture surface of the specimen was also observed by a scanning electron microscope (SEM).

Diffusible hydrogen concentration in the specimens was measured by means of thermal desorption analysis (TDA), with gas chromatography as the mass spectrometer. Hydrogen was charged to thin plates with thickness of 1.0mm in the above three conditions for 24 hours. The heating rate was 100 K/h. Diffusible hydrogen concentration, defined as summation of the first peak in the TDA spectrum below 573 K, was 0.3 wt. ppm for the condition A, 0.6 wt. ppm for the condition B, and 1.7 wt. ppm for the condition C.

2.3. Positron lifetime spectroscopy

Effect of hydrogen on lattice defects formation due to plastic deformation was investigated by positron lifetime spectroscopy. The parallel part of fractured specimen was used avoiding the local deformation area. The measurements were performed by using a digital oscilloscope system with photomultiplier tubes mounted with BaF_2 scintillators, and a time resolution (FWHM) was 175 ps. Na-22 positron source with 0.7 MBq was used, and acquisition time to acquire 3 million counts in the positron lifetime spectrum was about nine hours. The measured positron lifetime spectra were analysed by the programs, RESOLUTION[1] and POSITRONFIT Extended[2]. The amounts of various kinds of lattice defects (dislocation density, vacancy density and positron life time of vacancy cluster) were estimated by multi component analysis. The maximum value of lifetime was fixed to the value of 500 ps, which is corresponded to the lifetime for Fe metal surface.

3. RESULTS

3.1. Strain rate dependence of hydrogen embrittlement susceptibility

Fig. 1 shows the relationship between relative fracture elongation and strain rate obtained by the tensile tests. In the condition A, no hydrogen embrittlement appeared at strain rates higher than 10^{-3} s^{-1} . The relative fracture elongation was decreased with a decrease in strain rates below 10^{-3} s^{-1} , and hit the bottom at 10^{-5} s^{-1} . In the condition B and C, hydrogen embrittlement appeared at all tested strain rates between 10^{-6} and 10^{-2} s^{-1} . Especially, in the condition C, hydrogen embrittlement was severe, and relative fracture elongations were already minimum at 10^{-3} s^{-1} . Thus, threshold strain rate below which hydrogen embrittlement appeared increased with an increase in diffusible hydrogen concentration. The fracture surfaces at relative fracture elongations higher than 0.8 were dimple and similar to those in air. The fracture surfaces at relative fracture elongations between 0.4 and 0.8 were quasi-cleavage. The fracture surfaces at relative fracture elongations lower than 0.4 were intergranular. Fig. 2 shows the representative fracture surfaces of the ductile, quasi-cleavage and intergranular fractures.

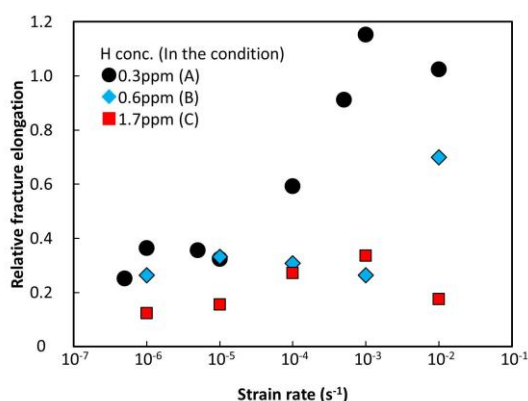


Fig. 1. Relationship between relative fracture elongation and strain rate by tensile tests under cathodic hydrogen charge.

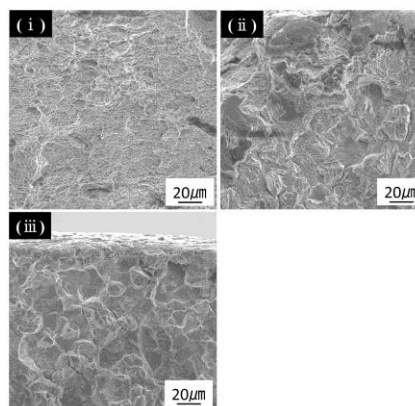


Fig. 2. The fracture surfaces of the (i) ductile, (ii) quasi cleavage and (iii) intergranular fractures.

3.2. Strain rate dependence of lattice defects formation under cathodic hydrogen charge

Fig. 3 shows the relationship between average positron lifetime and strain rate. Average positron lifetime in the condition A decreased with a decrease in strain rate, and that at strain rate 10^{-2} s^{-1} increased with an increase in diffusible hydrogen concentration. Fig. 4 shows the relationship between dislocation density and strain rate. Dislocation density slightly decreased with a decrease in strain rate and an increase in diffusible hydrogen concentration. Fig. 5 shows the relationship between vacancy density and strain rate. Vacancy density is monovacancy equivalent density of vacancy-type lattice defects (*i.e.*, monovacancies and vacancy-clusters). Vacancy density decreased with a decrease in strain rate and an increase in diffusible hydrogens concentration. Fig. 6 shows the relationship between positron lifetime of vacancy-cluster and strain rate. Positron lifetime of vacancy-cluster in the condition A at strain rate 10^{-2} s^{-1} was 430 ps. That in the condition A at strain rate 10^{-4} s^{-1} was saturated to 500 ps. That in the condition B at strain rate 10^{-2} s^{-1} was 460 ps, and that in the condition C was saturated to 500 ps. Therefore, positron lifetime of vacancy-cluster increased with a decrease in strain rate and an increase in diffusible hydrogen concentration.

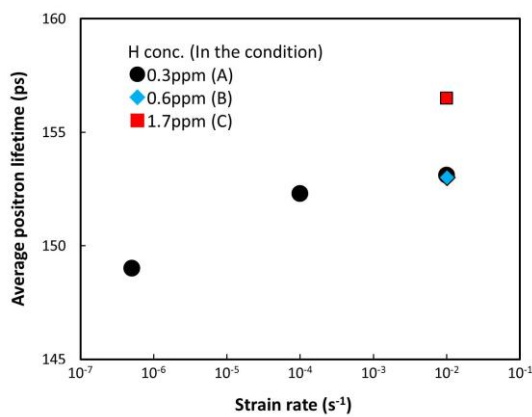


Fig. 3. Relationship between average positron lifetime and strain rate.

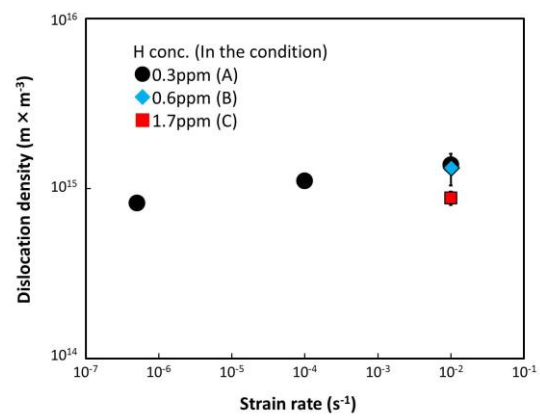


Fig. 4. Relationship between dislocation density and strain rate.

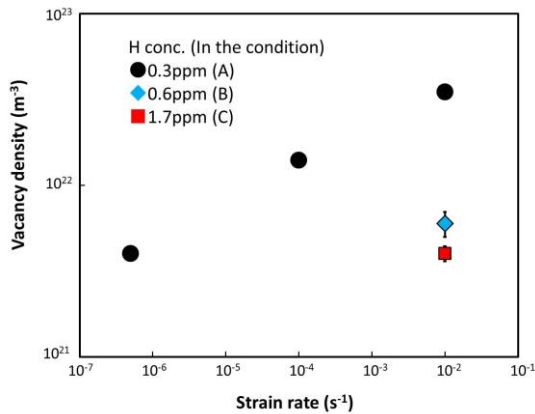


Fig. 5. Relationship between vacancy density and strain rate.

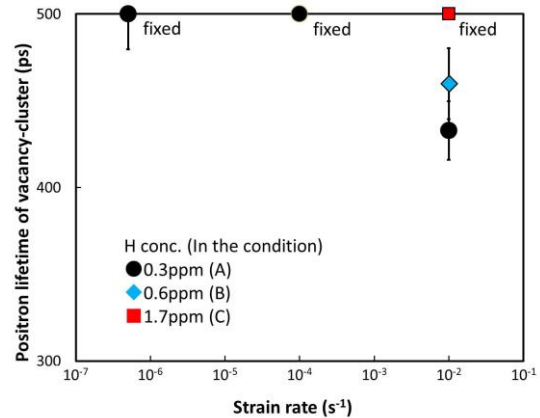


Fig. 6. Relationship between positron lifetime of vacancy-cluster and strain rate.

4. DISCUSSION

Dislocation density and vacancy density decreased with a decrease in strain rate and an increase in diffusible hydrogen concentration as shown in Figs. 4 and 5. On the other hand, positron lifetime of vacancy-cluster increased with a decrease in strain rate and an increase in hydrogen concentration as shown in Fig. 6. Vacancy density and positron lifetime of vacancy-cluster were plotted against fracture elongation as shown in Figs. 7 and 8. Decrease in fracture elongation means that hydrogen embrittlement susceptibility increased. Vacancy density decreased with a decrease in fracture elongation. This indicates that vacancy density uniquely depended on fracture elongation, and did not

reflect hydrogen embrittlement susceptibility. Dislocation density showed the similar tendency to vacancy density. On the other hand, positron lifetime of vacancy-cluster increased with a decrease in fracture elongation. This indicates that positron lifetime of vacancy-cluster is increased with a decrease in strain rate and an increase in diffusible hydrogen concentration. Therefore, it was considered that vacancy clustering is important in the mechanism of hydrogen embrittlement. Sugita *et al.* has reported the similar tendency on martensitic stainless steel[3][4].

In the present study, it was clarified that only the vacancy clustering was promoted with a decrease in strain rate. This indicates that vacancy clustering process requires time for diffusion and cohesion of vacancies. Therefore, it is assumed that time for vacancy clustering determines the strain rate dependence of hydrogen embrittlement susceptibility. Furthermore, it is expected that an increase in diffusible hydrogen concentration accelerate vacancy clustering process.

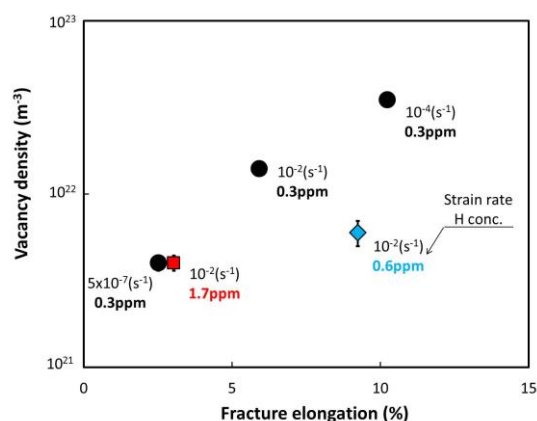


Fig. 7. Relationship between vacancy density and strain.

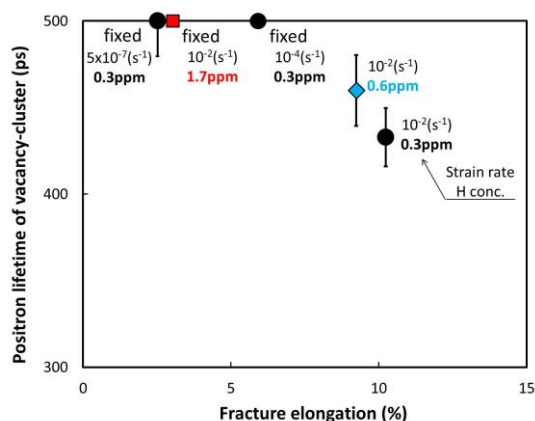


Fig. 8. Relationship between positron lifetime of vacancy-cluster and strain.

5. CONCLUSION

The strain rate dependence of hydrogen embrittlement susceptibility of tempered martensitic steel was investigated by tensile tests under cathodic hydrogen charge, and the formation of various kinds of lattice defects was investigated by positron lifetime spectroscopy. The following results were obtained.

1) The relative fracture elongation decreased with a decrease in strain rate. The threshold strain rate which hydrogen embrittlement appeared increased with an increase in diffusible hydrogen concentration.

2) Dislocation density and vacancy density apparently decreased with a decrease in strain rate and an increase diffusible hydrogen concentration. However, it is considered that both parameters were uniquely dependent on the degree of plastic strain.

3) Positron lifetime of vacancy-cluster increased with a decrease in strain rate and an increase in diffusible hydrogen concentration. This indicates vacancy clustering was promoted with a decrease in fracture elongation. Therefore, it is assumed that the strain rate dependence of hydrogen embrittlement susceptibility is determined by vacancy clustering process.

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