

## Effect of Aging Condition on Fatigue Strength of Maraging Steel in Long Life Region

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**Abstract** A novel aging treatment was proposed to improve the fatigue strength of maraging steel by taking the effects of aging condition and humidity into account. Rotating bending fatigue tests were carried out for a 350 grade of 18% Ni maraging steel in the long life region up to  $10^8$  cycles in the relative humidity of 25% and 85%. Aging conditions under investigation included a conventional aging or the so called single aging at 753K, and a two-step aging or double aging by ageing at lower temperature of 473K succeeded to the single aging. Through the double aging, the susceptibility of fatigue strength to humidity was significantly improved without any deleterious effects. The main reasons for the decrease in fatigue strength in high humidity are due to the promotion of crack initiation and the acceleration of small crack propagation. The improvement of fatigue property in high humidity by the double aging was explained from the roles of the additional precipitation of supersaturated Mo atoms during the second lower temperature aging.

**Keywords** Fatigue, Maraging steel, Aging, Humidity, Rotating bending

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### 1. Introduction

Maraging steel has the highest tensile strength and high ductility among steels used in practice [1]. However, the fatigue strength of maraging steel is much lower than expected from its high static strength [2]. The reason for the lower fatigue strength is due to the high sensitivity of maraging steel to both notch [3] and humidity [4]. Since maraging steel is mainly hardened by the precipitation of intermetallic compounds such as  $\text{Ni}_3(\text{Ti}, \text{Mo})$  and  $\text{Fe}_2\text{Mo}$  in martensite structure, the strength of the steel depends largely on the aging conditions. Many studies have been carried out on the precipitations in the steel. For example, it is well known that the in-coherent particle of  $\text{Ni}_3(\text{Ti}, \text{Mo})$ , which is one of the primary strengthening particles, precipitates at high aging temperature beyond 723K, while the particles of  $\text{Fe}_2\text{Mo}$ ,  $\omega$  phase and Mo cluster precipitate at low aging temperature below 723K [5]. Precipitation at high temperature is usually referred to as high temperature phase whilst that at low temperature as low temperature one. Considering the above-stated relation between aging temperature and precipitation, it is expected that additional precipitation and hardening may happen during a two-step aging or the conventional high temperature aging followed by a low temperature ageing, because the concentration of Mo exceeds its solubility at low temperature. However, aging treatment of maraging steel is usually conducted under a constant high temperature only, and the study of the mechanical properties, especially the fatigue strength, of the two-step aged steel at high and low temperatures has not been reported.

In the present study, the effect of aging condition on the fatigue strength of 18%Ni maraging steel of grade 350 in long life region was investigated. A novel aging treatment was proposed for improving fatigue properties of the maraging steel. Fatigue tests up to  $10^8$  cycles were performed in relative humidity of 25% and 85% under rotating bending. Aging conditions examined were the conventional aging at 753K or the so called single aging, and a two-step aging or double aging at 473K subsequent to the conventional aging at 753K.

## 2. Experimental procedure

The material used was a commercial 350-grade maraging steel. The chemical composition was shown in Table 1. The steel was solution treated for 5.4ks at 1123K in vacuum followed by air cooling and age hardened in salt bath. The mean grain size of prior austenite was about 20  $\mu\text{m}$ .

Figure 1 shows aging curves, in which hardness is increased by the two-step aging as stated in introduction. The reason for the increase in hardness by the two-step aging will be discussed later. Aging conditions investigated were a single peak-aging at the conventional aging temperature of 753K, which is denoted as SA-P, and a two-step or double aging by including an under-aging treatment at 473K after the conventional aging SA-P, which is denoted as DA-U, hereinafter. The two-step aging showed a definite hardening. These conditions were indicated by open marks in Fig.1 and were specified in Table 2. Further study on the variation of mechanical properties with different combination of two-step aging conditions can be found in the related presentation. [6]

Figure 2 shows the shape and dimensions of plain specimen. Specimens were machined after the solution treatment, and then age hardened as specified in Fig. 1. Prior to fatigue testing, specimens were sanded by emery paper followed by electro-polishing to remove work affected layer and secure surface state for easier observation. Fatigue tests were carried out using a Ono-type rotating bending fatigue machine with a capacity of 15 N·m, operating at about 50Hz in relative humidity (RH) of 25% and 85%, respectively. The humidity was moderated in a range of  $\text{RH} \pm 5\%$ .

Table 1. Chemical composition (mass%)

Grade	C	Si	Mn	Ni	Mo	Co	Ti	Al	Fe
350G	0.001	0.01	0.01	17.89	4.27	12.36	1.3	0.08	Bal.

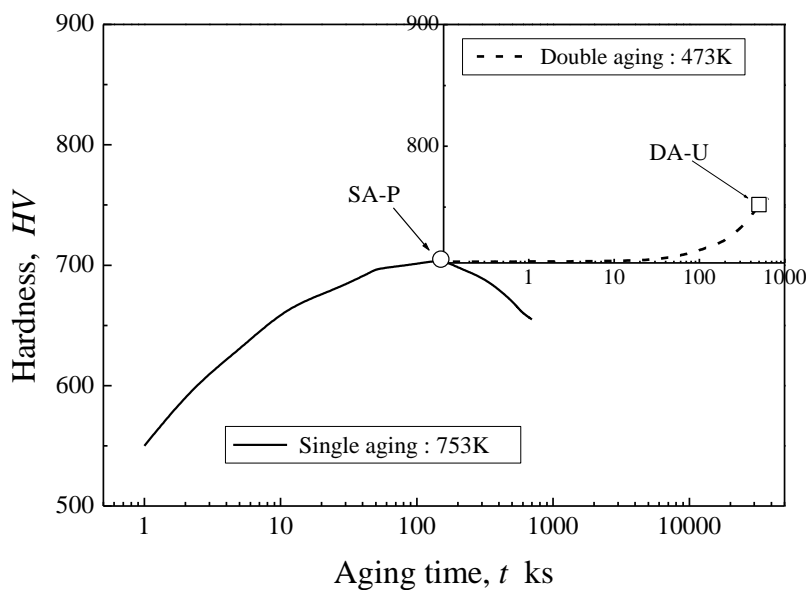


Figure 1. Aging curves of maraging steel

Table 2. Mechanical properties

Material	Aging condition	Vickers hardness $HV$	0.2% proof stress $\sigma_{0.2}$ (MPa)	Tensile strength $\sigma_B$ (MPa)	Reduction of area $\phi$ (%)
SA-P	753K-150ks	705	2300	2370	54
DA-U	753K-150ks, 473K-500ks	750	2315	2378	55

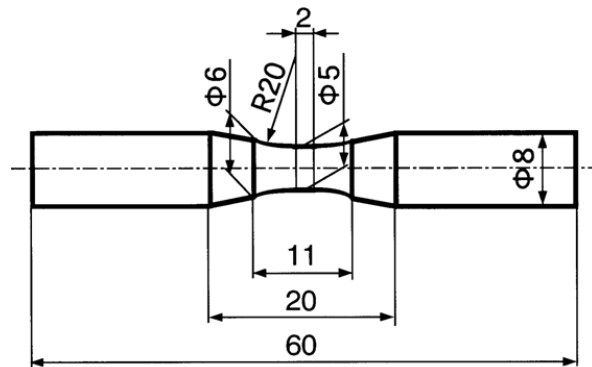


Figure 2. Shape and dimensions of plain specimen

The atmospheric temperature was not controlled and in the range of  $295 \pm 3K$ . Surface cumulative damage observation and crack length measurement were carried out by plastic replication technique. Crack length,  $\ell$ , was defined as a surficial length along the circumferential direction vertical to stress axis. Microstructure and fracture morphology were examined using both transmission electron microscope (TEM) and scanning electron microscope (SEM).

### 3. Results and discussion

Figure 3 shows  $S-N$  curves of the steels. Fracture occurred from specimen surfaces, irrespective of the age treatment and the humidity. Moreover, in case of low humidity, none of fatigue cracks were observed for specimens non-fractured after fatigued for  $10^8$  cycles, suggesting that fatigue limit defined as fatigue strength at  $10^8$  cycles was mainly controlled by the resistance to crack initiation. Fatigue limits in both steels are nearly the same in low humidity, meaning that the resistance to crack initiation was improved somewhat by the double aging, because the fatigue strength of a steel usually decreases with strengthening due to increasing notch sensitivity [7]. On the other hand, fatigue strength decreased in high humidity, and the extent of fatigue strength decrease was larger in SA-P steel than in DA-U steel.

To clarify the reason for the difference in high humidity susceptibility of fatigue limit between the SA-P and DA-U steels, successive observation of surface failure due to stress repetitions was conducted.

Figs. 4 and 5 show crack growth curves and relation between crack growth rate and stress intensity factor range in 25% and 85%, respectively. It can be seen that the initiation and early propagation of cracks short than a few grain sizes are definitely accelerated by high humidity and this trend is more remarked at lower stress levels, because the lower the stress level, the longer the crack initiation life and the stronger the influence of humidity. In other words, it is the acceleration of crack initiation and its early growth that caused the fatigue strength decrease in high humidity.

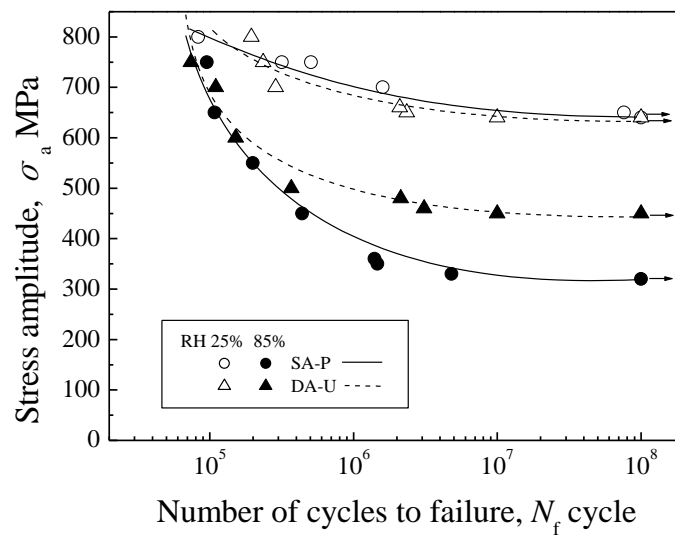


Figure. 3 S-N curves

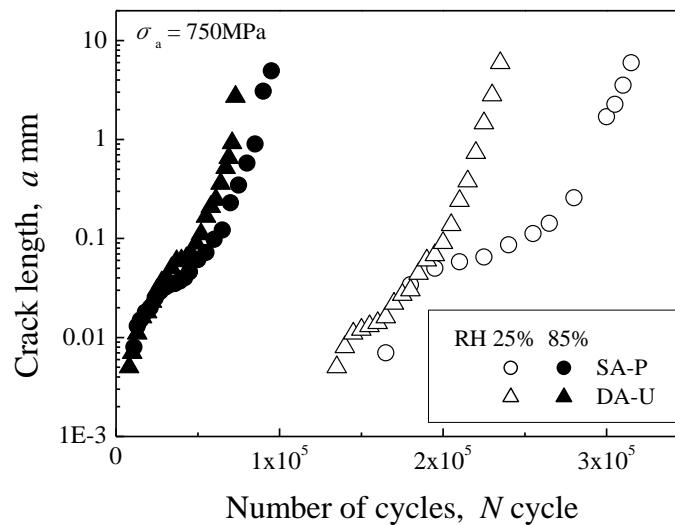


Figure 4. Crack growth curves

In case of SA-P steel subjected to fatigue in long life region in RH 85%, corrosion debris (Fig. 6a) and multi cracks initiated from a corroded pit (Fig. 6b) are confirmed on specimen surface, implying that crack initiation was promoted by anodic dissolution.

Figure 7 shows SEM morphology of SA-P steel fractured in both low and high humidity. In case of low humidity, slip steps appear on a facet in the vicinity of crack initiation site, as shown in Fig. 7a-1)), meaning that slip deformation or stage I cracking dominate crack nucleation [8]. On the other hand, in high humidity, crack initiation is characterized by many facets, especially river pattern-like facets (Fig. 7b-1), suggesting that hydrogen embrittlement assisted fracture occurred.

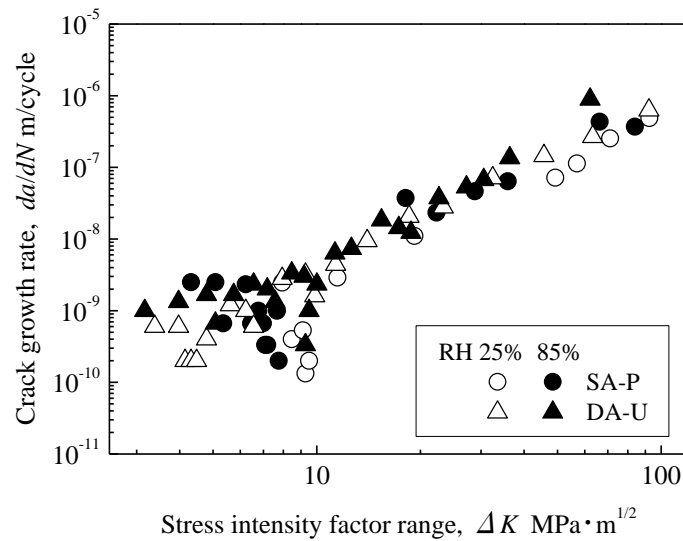
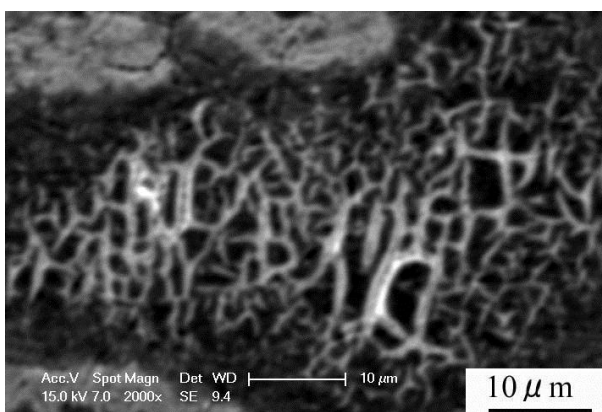
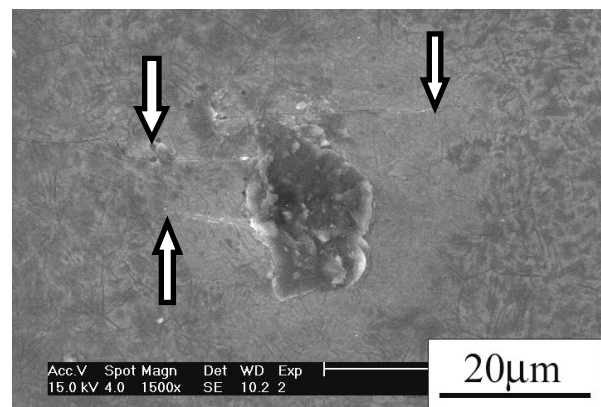


Figure 5. Crack growth rate vs. stress intensity factor range

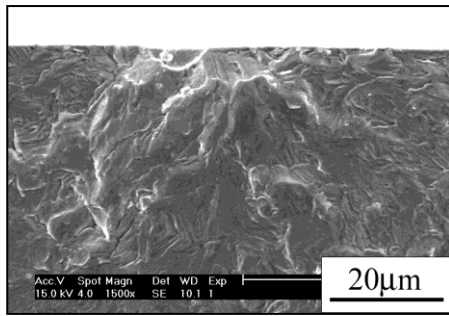


(a) Corroded area

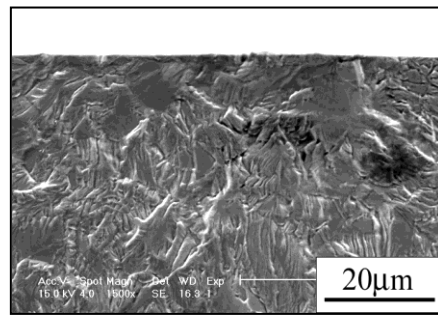


(b) Cracks initiated at corrosion pit

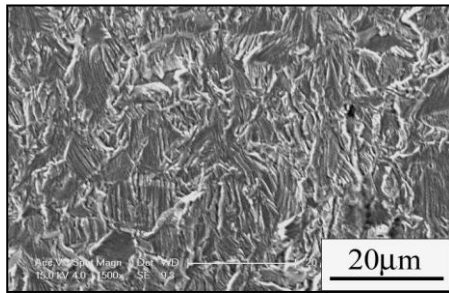
Figure 6. Surface state of SA-P specimens subjected to fatigue in long life region in RH 85% (Arrows indicate crack tips)



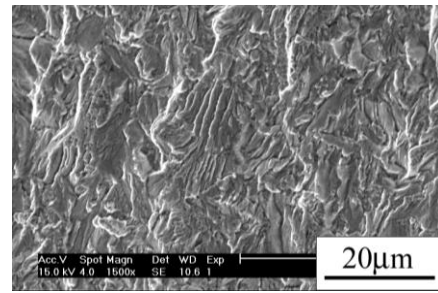
(a-1) Crack initiation site



(b-1) Crack initiation site



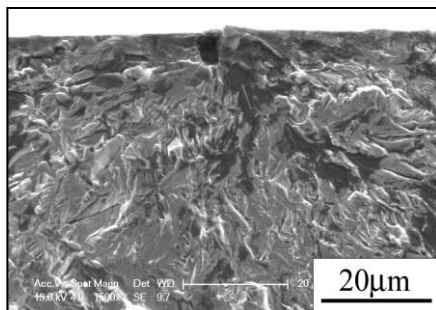
(a-2) Inner area (~200µm from surface)



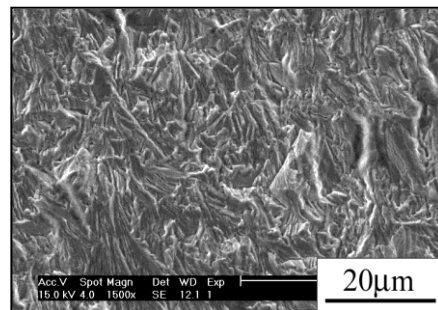
(b-2) Inner area (~200µm from surface)

Figure 7. Fracture surfaces of SA-P steel tested in (a) RH 25% and (b) RH 85%

Similar fatigue morphology can be found in DA-U steel tested in high humidity, as shown in Fig. 8. It is hard to distinguish any difference in fracture mechanism between the SA-P and the DA-U steels failed in high humidity, depending on the analysis of the results shown in Figs. 7 and 8. Therefore, it may be induced that the propagation of small cracks was accelerated by embrittlement due to hydrogen generated in cathode reaction. In addition, most of fracture surfaces in the region beyond crack initiation are occupied by martensite lath cracking regardless of humidity and aging condition, which may explain that little difference in growth rate of larger cracks is recognized between different aging conditions and humidity, as seen in Fig. 5.



(a) Crack initiation site



(b) Inner area (about 200µm from surface)

Figure 8. Fracture surfaces in DA-U steel at RH 85%

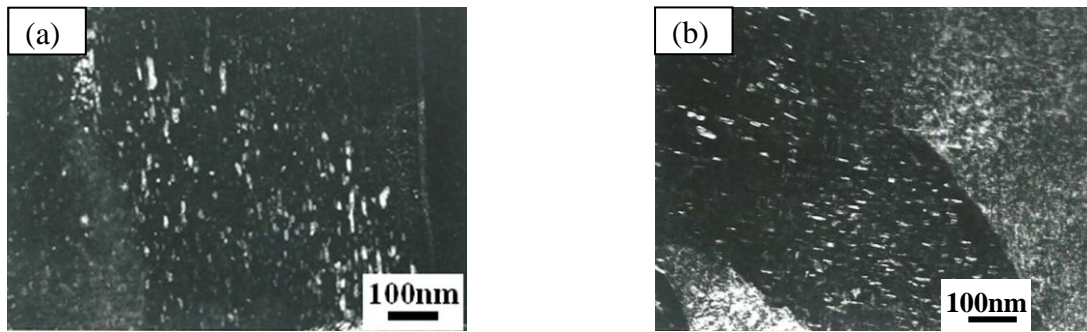


Figure 9. TEM micrographs showing microstructure of (a) SA-P and (b) DA-U

Figure 9 shows microstructures of SA-P and DA-U steels observed by TEM. Rod-shaped particles of ~10nm or less in length and spherical ones are observed in SA-P steel, which were identified as  $\text{Ni}_3(\text{Ti}, \text{Mo})$  and  $\text{FeMo}$  respectively by their diffraction patterns of TEM and the X-ray diffraction of electrolyte-extracted particles. In DA-U steel, it is difficult to confirm different particles by means of their microstructural features. Tewari et al. reported that fine particles of nano-sized  $\omega$ -phase precipitated at low aging temperature in DA-U steel [5]. However, any other precipitates were difficult to distinguish, though it can be predicted from the phase diagram of Fe-Mo alloy that Mo atoms are supersaturated during the second aging at 473 K. Since a high density of dislocations are initially introduced by martensitic transformation, the supersaturated Mo atoms may migrate by the pipe diffusion along those dislocations so as to form the moderation of concentration or the clusters around the dislocations, which impede the motion of dislocations and cause the hardening at 473 K. DA-U steel is in such a way strengthened by the second aging at 473k.

On the other hand, in high humidity, hydrogen atoms generated in accompany with anodic reaction may diffuse into the matrix and concentrate at grain boundaries, inducing hydrogen embrittlement [9, 10]. It is concerned that the clusters of supersaturated Mo atoms around dislocations and the irregularity of concentration in the second aging may trap some hydrogen so as to suppress hydrogen embrittlement in high humidity.

#### 4. Conclusions

The effect of aging condition on the fatigue strength of 18% Ni maraging steel of grade 350 in long life region in high humidity was investigated under rotating bending in relative humidity of 25% and 85%. Fatigue strength of both single aging and double aging hardened steels was markedly decreased in high humidity environment. However, the decrease of fatigue strength was suppressed by the double aging. Although most of fracture surface was characterized with lath boundary cracking regardless of the humidity and aging condition, a few brittle facets of prior austenite comparable to a grain size were observed at the origin of fracture in high humidity. The main reason for the decrease of fatigue strength in high humidity was the acceleration of both crack initiation and its early growth due to hydrogen embrittlement. The decrease in humidity susceptibility by the

double aging was explained by the precipitation of supersaturated Mo atoms as well as the fine particles of nano-sized  $\omega$ -phase diffused in the matrix during the second aging of lower temperature.

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