Effect of Heat Treatment on Strain Rate Sensitivity in Tensile Properties of 245 kg/mm² Grade and 210 kg/mm² Grade Maraging Steels*

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Synopsis

Tensile properties and their strain rate sensitivity have been investigated on 245 kg/mm² grade and 210 kg/mm² grade maraging steels, aged in the temperature range from 400° to 575°C, as a function of aging time. A crosshead travel speed in tensile test was varied from 0.005 to 1.0 mm/min. The change in electrical resistivity at liquid nitrogen temperature has also been measured.

Embrittlement caused by decreasing a crosshead speed is observed in the 245 kg/mm² grade maraging steel aged for relatively short periods in the temperature range from 400° to 500°C. The embrittlement is also observed in the 210 kg/mm² grade maraging steel aged for relatively short periods at temperatures of 400° and 425°C. Prolonged aging leads to the decrease of the embrittlement. The critical aging time beyond which the elongation becomes insensitive to the crosshead speed is determined. The embrittled behavior at a decreased strain rate is interpreted by assuming that hydrogen diffused to the regions having intense stress concentrations leads to the initiation of cracking. It is considered that relatively intense stress concentrations tend to be produced in plastic deformation of the maraging steels containing coherent precipitates. When partially coherent precipitates are increased by prolonged aging or by raising an aging temperature, the decrease in the embrittlement at a low strain rate results from the decrease in producing intense stress concentrations in plastic deformation.

I. Introduction

The development of 210 kg/mm² grade and 245 kg/mm² grade maraging steels has provided a class of alloys possessing high strengths and toughness. A typical composition of the 210 kg/mm² grade maraging steel is 18%Ni, 9%Co, 5%Mo, 0.7%Ti, and that of the 245 kg/mm² grade maraging steel is 17.5%Ni, 12.5%Co, 3.75%Mo, 1.7%Ti.

Precipitation-hardening of the maraging steels is commonly carried out at about 500°C for periods of about 5 hr. It has been found that the tensile properties are strain rate sensitive in the maraging steels aged at temperatures less than about 460°C.¹⁻⁶⁾ As a strain rate decreases, ductility and strength decrease. It has also been found that the strain rate sensitivity is more evident in the 245 kg/mm² grade maraging steel than in the 210 kg/mm² grade maraging steel.¹⁻⁶⁾ However, the effect of heat treatment on the strain rate sensitivity has not yet been investigated thoroughly.

In order to suppress delayed brittle failure at a low stress, the strain rate sensitivity must be eliminated by

selecting aging temperature and time. The present paper is concerned with the strain rate sensitivity in the tensile properties of the maraging steels subjected to isothermal agaisngs in the range from 400° to 575°C. The mechanism of the embrittled behavior at a low strain rate will also be considered.

II. Experimental Procedure

The chemical compositions of commercial 210 kg/mm² grade and 245 kg/mm² grade maraging steels used for the present investigation are given in Table 1. Plates of 2 mm in thickness were solution-treated for 1 hr at 950°C, and were cold rolled to 0.5 mm in thickness. Tension specimens shown in Fig. 1 were punched from the plates. The final solution treatment for the 245 kg/mm² grade maraging steel was 30 min annealing at 815°C, and that for the 210 kg/mm² grade maraging steel was 30 min at 825°C. Austenitic grain size numbers after the final solution treatments were ranged from ASTM No. 10.5 to No. 11.5. Subsequently, the specimens were aged isothermally in the temperature range from 400° to 575°C. Crosshead travel speeds adopted in the present investigation were 0.005 (strain rate, $\dot{\epsilon} \doteq 0.00045 \text{ min}^{-1}$), 0.01 ($\dot{\epsilon} \doteq 0.00091$), 0.1 ($\dot{\epsilon} \doteq 0.0091$) and 1 mm/min ($\dot{\epsilon} \doteq 0.091$). Tensile strengths and elongations were determined from load-elongation curves recorded.

Measurements of the change in electrical resistivity were carried out with wires of 0.6 mm in diameter at liquid nitrogen temperature in order to investigate the precipitation behavior. Electrical resistivity was determined from a potential difference along a constant length of a wire charged with a stationary current of 0.1 A.

Aging for periods of 1 000 min and less was carried out in a salt bath. For aging over 1 000 min, speci-

Fig. 1. Tensile test specimen

Table 1. Chemical compositions of the maraging steels investigated (wt%)

No.	Ni	Co	Mo	Ti	Al	C	Si	Mn	P	S
245-E	17.50	12.80	4.00	1.70	0.08	0.004	0.05	0.03	0.005	0.005
210-G	18.08	9.52	5.29	0.90	0.07	0.004	0.03	0.04	0.005	0.006

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mens aged for 1 000 min in the salt bath were transferred into an electric furnace filled with argon, and aging period was extended.

III. Results and Discussion

1. Tensile Properties of the Aged 245 kg/mm² Grade Maraging Steel

Tensile properties obtained at a crosshead speed of 1 mm/min are shown in Fig. 2 as a function of aging time. Each measurement in Fig. 2 shows an average taken from triplicate tensile tests. In case of aging at 400°C for the periods from 100 to 10 000 min, the fracture takes place at small elongation, and then the strength appears not to be true but incidental because of premature brittle failure. It is also evident that the elongation after the isothermal aging in the temperature range from 425° to 500°C is relatively small in the early stage of aging. In those isothermal agings, increase in elongation with aging time is found before the strength reaches its maximum. For example, the elongation of a specimen aged at 450°C increases in the range from 100 to 500 min, and it is found to decrease with the further increase in strength. The aging period, during which the elongation increases with aging time, decreases as the aging temperature is raised. The elongation *vs.* aging time of a specimen aged at 575°C is shown in Fig. 2 as a typical example of the specimens aged in the range from 525° to 575°C. The initial drop in elongation is not found even after aging for 1 min.

The effect of a crosshead speed on elongation is shown in Figs. 3 to 7. As seen in Fig. 3, it is evident that the specimens aged at 425°C are embrittled by decreasing a crosshead speed. The embrittlement at a low strain rate tends to be relieved by prolonged aging. However, the specimens aged for 20 000 min at 425°C are still embrittled in the tensile test at a crosshead speed of 0.005 mm/min. As seen in Figs. 4 to 6, the specimens aged for relatively short periods in the temperature range from 450° to 500°C are

also embrittled by decreasing a crosshead speed. As the aging period at these temperatures is extended, the strain rate sensitivity in elongation is decreased. Figure 7 shows the tensile properties after aging at

Fig. 3. Effect of a crosshead travel speed on the tensile properties of the 245-E maraging steel aged at 425°C for various times

Fig. 4. Effect of a crosshead travel speed on the tensile properties of the 245-E maraging steel aged at 450°C for various times

σ_B : Tensile strength
 ϵ_t : Total elongation

Fig. 2. Tensile properties of the aged 245-E maraging steel, extended at a crosshead travel speed of 1 mm/min

Fig. 5. Effect of a crosshead travel speed on the tensile properties of the 245-E maraging steel aged at 475°C for various times

Fig. 6. Effect of a crosshead travel speed on the tensile properties of the 245-E maraging steel aged at 500°C for various times

Fig. 7. Effect of a crosshead travel speed on the tensile properties of the 245-E maraging steel aged at 525°C for various times

525°C. Regarding the specimens aged for the periods ranged from 50 to 500 min, the elongations are insensitive to the crosshead speeds adopted.

As seen in Fig. 2, the elongations are relatively small in the specimens aged for relatively short periods in the temperature range from 400° to 500°C. This fact may be interpreted on the basis of the strain rate sensitivity in elongation, as seen in Figs. 3 to 6. A critical aging time beyond which the elongation becomes insensitive to the crosshead speed is within the range 60 to 70 min for aging at 500°C, 350 to 500 min for aging at 475°C, 2 000 to 2 500 min for aging at 450°C, and more than 20 000 min for aging at 425°C. From the relation between the logarithm of the critical aging time and the reciprocal of the absolute aging temperature, an activation energy is calculated to be about 80 kcal/mol. This value of the activation energy suggests that the strain rate sensitivity in elongation declines gradually through the microstructural change with the movements of the substitutional atoms.

Figure 8 shows the changes in electrical resistivity measured at liquid nitrogen temperature and log (time) derivative of an isothermal aging at 475°C as a function of aging time. From Fig. 8 (B), it is considered that a two stage precipitation takes place in the isothermal aging at 475°C. Accordingly, such a two stage precipitation will also be revealed by pro-

Fig. 8. Resistivity curves of the 245-E maraging steel, (A), and a curve of log (time) derivative of an isothermal aging at 475°C, (B)

longing the aging periods at the temperatures less than 475°C. However, it is not necessarily clear in the present data given in Fig. 8 (A) whether the two stage precipitation takes place in the isothermal agings at the temperatures above 475°C or not.

The existence of two types of precipitates has been revealed in Fe-Ni-Co-Mo quaternary maraging steels.⁷⁻¹¹⁾ In our previous papers,^{10,11)} a precipitate formed in the first precipitation stage was referred to phase-L, and another formed in the second precipitation stage was referred to phase-H. For example, the two types of precipitates in a 18Ni-10Co-5Mo quaternary maraging steel are found in isothermal agings at 450°C and below.¹⁰⁾ On the other hand, the phase-H is precipitated directly from the solution in isothermal agings at 475°C and over.¹⁰⁾

As stated, the two stage precipitation takes place in aging of the 245 kg/mm² grade maraging steel. However, the effect of adding Ti has not yet been investigated thoroughly on the precipitates formed in the quaternary maraging steels. Accordingly, it is not clear whether the two stage precipitation in the 245 kg/mm² grade maraging steel corresponds to that in the quaternary maraging steels or not. Detailed discussion of the precipitates formed in the Fe-Ni-Co-Mo-Ti maraging steels including the 245 kg/mm² grade and the 210 kg/mm² grade maraging steels remains as a future problem. No evident change in precipitation is observed on the resistivity curves in the neighborhood of the critical aging times where the elongations become insensitive to the crosshead speeds.

It is well known that in most precipitation-hardening alloys the initial precipitate produced at low aging temperatures and short periods of aging is coherent with the matrix and small in size.¹²⁾ Further aging leads to the formation of partially coherent precipitates of much larger particle size and finally to non-coherent precipitates of an intermetallic compound.¹²⁾ Further, deformation of precipitates and slip line observations in deformed alloys containing precipitates have been extensively investigated.¹²⁾ Al-

though the investigations have been mainly focused to precipitation-hardening aluminum and copper alloys, the following results have been obtained:¹²⁾ (1) Dislocations are able to pass through small coherent precipitates, and partially coherent precipitates are sheared after relatively large strains. Non-coherent precipitates are difficult to be sheared. (2) Slip in an alloy containing small coherent precipitates tends to be confined to a narrow region. Therefore, slip bands are distributed unevenly. (3) When partially coherent precipitates are increased by prolonged aging at a certain temperature, slip lines become finer and less distinct in plastic deformation.

From the results stated above, we conclude that slip in the maraging steel containing coherent precipitates is confined to a narrow region and tends to pass through the matrix grains. Accordingly, it is considered that the plastic deformation of the maraging steel containing coherent precipitates tends to produce relatively intense stress concentrations in regions where slip bands cross the grain boundaries or pass through a tension specimen. It is also considered that crossing of the slip bands produce intense stress concentrations. When the partially coherent precipitates are increased gradually by prolonged aging or by raising an aging temperature, slip bands observed distinctly and those passing through the matrix grains are decreased gradually in plastic deformation. Thus, producing intense stress concentrations during the plastic deformation of the maraging steel is considered to be also decreased gradually.

On the other hand, it is well known that hydrogen contained in a high strength steel causes a considerable loss of ductility at a low tensile strain rate.¹³⁾ The embrittlement in the maraging steel extended at a low strain rate may be reasonably interpreted on the assumption that hydrogen diffused to the regions having intense stress concentrations leads to the initiation of cracking. Perhaps, hydrogen contained in the maraging steel and that absorbed during the present experiments are very small. However, it has been known that very small contents of hydrogen are possible to cause the hydrogen embrittlement in a high strength steel.¹³⁾ Thus, the maraging steel containing small coherent precipitates shows conspicuously the embrittlement at a low tensile strain rate, as its plastic deformation tends to produce relatively intense stress concentrations. When the partially coherent precipitates are increased by prolonged aging at a certain temperature or by raising an aging temperature, the decrease in the embrittlement at a low tensile strain rate results from the decrease in producing intense stress concentrations in plastic deformation.*

2. Tensile Properties of the Aged 210 kg/mm² Grade Maraging Steel

Tensile properties obtained at a crosshead speed of 1 mm/min, and changes in electrical resistivity measured at liquid nitrogen temperature are shown in Figs. 9 and 10 as a function of aging time, respectively. The two stage precipitation is found in the resistivity curves of the specimens aged in the temperature range from 450° to 500°C. As an aging temperature is raised to 525°C and above, it is not clear whether the two stage precipitation takes place or not. From comparison of Fig. 9 with Fig. 10, the tensile strength reaches maximum near the end of the resistivity decrease in the first precipitation stage, and decreases gradually with the progress of the second precipitation. In the temperature range from 525° to 575°C, the tensile strength reaches maximum at

Fig. 9. Tensile properties of the aged 210-G maraging steel, extended at a crosshead travel speed of 1 mm/min

Fig. 10. Resistivity curves of the 210-G maraging steel, (A), and a curve of log (time) derivative of an isothermal aging at 475°C, (B)

* The embrittlement at a low tensile strain rate is essentially caused by the combined effect of intense stress concentrations produced in plastic deformation and hydrogen contained. In the present paper, the relation between the formation of stress concentrations and the microstructures after aging has been considered in detail. However, it is considered that the austenitic grain size of the tension specimens has also an important effect on the formation of stress concentrations. As metallographic factors except the microstructures after aging were not varied in the present investigation, other factors affecting the formation of stress concentrations remain as a future problem.

about 80% of the total resistivity decrease.

The effect of a crosshead speed on elongation was investigated on the specimens aged in the temperature range from 400° to 450°C. The results obtained are shown in Figs. 11 and 12. Each mark in Figs. 11 and 12 is an average taken from seven tensile test results. In case of aging at 450°C, the tensile test at a crosshead speed of 0.005 mm/min was made on the specimens aged for periods more than 50 min, and resultant tensile properties did not differ from those at a crosshead speed of 1 mm/min. As seen in Fig. 11, the elongation after aging at 400°C for 2 500 min or less is evidently less in case of a crosshead speed of 0.005 mm/min than in that of 1 mm/min. Intergranular fracture along the prior austenite grain boundaries was observed in the fractured surfaces of the specimens, aged at 400°C for 2 500 min or less, and extended at a crosshead speed of 0.005 mm/min. However, the elongation after aging at 400°C for 5 000 min or 10 000 min is insensitive to the crosshead

speed.

In case of aging at 425°C, the elongations of the specimens aged for 100~500 min are relatively small at a crosshead speed of 0.005 mm/min, while the elongations of the specimens aged for 1 000 min and more are insensitive to the crosshead speed as seen in Fig. 12. Critical aging time required for the elongation to become insensitive to the crosshead speed is considered to be 2 500~5 000 min for aging at 400°C, and 500~1 000 min for aging at 425°C. The critical aging time is within the first stage of the resistivity decrease. As discussed in the former section, relatively intense stress concentrations tend to be produced during plastic deformation of the maraging steel containing small coherent precipitates, and lead to a considerable loss of elongation in the tensile tests at a crosshead speed of 0.005 mm/min.

With regard to aging at a given temperature, the critical aging time is longer in the 245 kg/mm² grade maraging steel than in the 210 kg/mm² grade maraging steel. This fact is considered to be attributable to the difference in the size and density of the coherent precipitates contained in the maraging steels after aging for a given period. On the other hand, by comparing the resistivity curves in Fig. 8 with those in Fig. 10, the progress in precipitation is considered to be more rapid in the 210 kg/mm² grade maraging steel than in the 245 kg/mm² grade maraging steel. For example, the decrease in the resistivity of the 210 kg/mm² grade maraging steel ceases after relatively short aging periods in the isothermal aging in the temperature range from 450° to 500°C. Accordingly, it is suggested that the microstructural change with aging time or with raise in an aging temperature is more rapid in the 210 kg/mm² grade maraging steel than in the 245 kg/mm² grade maraging steel. This fact also implies that the aging condition leading to an embrittled behavior at a decreased strain rate is much more limited in the 210 kg/mm² grade maraging steel than in the 245 kg/mm² grade maraging steel.

IV. Summary

(1) The elongations of the 245 kg/mm² grade maraging steel aged for relatively short periods at the temperatures of 500°C and less are very strain rate sensitive. The strain rate sensitivity in elongation is also observed in the 210 kg/mm² grade maraging steel aged for relatively short periods at the temperatures of 400° and 425°C. From the viewpoints based on the precipitation hardening and on the decrease in resistivity, it is concluded that the strain rate sensitivity in elongation is observed in the aging stage of the formation of the coherent precipitates. Prolonged aging leads to the decrease of the strain rate sensitivity in elongation.

(2) The critical aging time beyond which the elongation becomes insensitive to the crosshead speeds ranged from 0.005 to 1 mm/min is determined. The critical aging time is decreased, as an aging temperature is raised. The aging condition leading to the embrittled behavior at a decreased strain rate is much

Fig. 11. Effect of a crosshead travel speed on the tensile properties of the 210-G maraging steel aged at 400°C for various times

Fig. 12. Effect of a crosshead travel speed on the tensile properties of the 210-G maraging steel aged at 425°C for various times

more limited in the 210 kg/mm² grade maraging steel than in the 245 kg/mm² grade maraging steel. From the resistivity curves measured, the microstructural change with aging time or with raise in an aging temperature is considered to be more rapid in the 210 kg/mm² grade maraging steel. The difference of aging kinetics between the both grade maraging steels should be considered as a factor for the interpretation of the aging condition leading to the embrittled behavior at a decreased strain rate.

(3) The embrittled behavior at a decreased strain rate is interpreted by assuming that hydrogen diffused to the regions having intense stress concentrations leads to the initiation of cracking. The maraging steels containing the coherent precipitates tend to produce relatively intense stress concentrations in deformation. When the partially coherent precipitates are increased by prolonged aging or by raising an aging temperature, the decrease in the embrittlement at a low tensile strain rate results from the decrease in producing intense stress concentrations in plastic deformation.

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