Recrystallization of Reversed Austenite and Subsequent Martensitic Transformation in 18%Ni Maraging Steel*

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Synopsis

Recrystallization behaviors of reversed austenite from either martensitic (nondeformed) or deformed martensitic (50% rolled) 18%Ni maraging steels, and martensite structures formed from either unrecrystallized or recrystallized austenite were studied by the microstructural observations. The main results obtained are as follows.

(1) Right after the completion of $\alpha' \rightarrow \gamma$ reverse transformation, the reversed austenite grain boundaries exactly correspond to the prior austenite grain boundaries of the original specimen before heating in both nondeformed and deformed specimens, and thus no change in the austenite grain size is observed. The reversed austenite is recrystallized to a finer grain size by further heating.

(2) The recrystallization of the reversed austenite in deformed specimen starts and finishes at lower temperatures in comparison with nondeformed specimen. In the case of nondeformed specimens, the recrystallized grains form preferentially along the reversed austenite grain boundaries. On the other hand, in the deformed specimens, the recrystallized grains form finely and evenly in the reversed austenite grain. Owing to such a difference in preferential nucleation site of recrystallized grain, a deformation of martensite prior to heating enhances the refinement of reversed austenite grain.

(3) In the case that martensites are formed from the unrecrystallized austenite right after the completion of $\alpha' \rightarrow \gamma$ transformation by heating and quenching, martensite structure such as packets and blocks after the $\alpha' \rightarrow \gamma \rightarrow \alpha'$ transformation almost exactly corresponds to that of the original specimen before the heating. Such a microstructural reproducibility was also observed in the deformed specimens. However, the width of martensite laths formed from the unrecrystallized austenite is smaller in comparison with that formed from the recrystallized austenite.

I. Introduction

Since a grain size refinement is effective for improvement of both strength and ductility, several thermo-mechanical treatments such as cold rolling before solution treatment,¹⁾ hot rolling during cooling from the solution temperature²⁻⁴⁾ and $\alpha' \rightarrow \gamma$ cyclic transformation^{5,6)} have been attempted in the maraging steels. It is known that the mechanism of refinement of austenite grain in maraging steels is different from that of carbon steels; i.e., the grain size of reversed austenite† right after the $\alpha' \rightarrow \gamma$ reverse transformation is identical with the prior austenite grain

size of the specimen before heating,⁷⁻⁹⁾ and the reversed austenite is recrystallized to a finer grain size with further heating to higher temperatures.^{5,8-11)} However, the recrystallization behavior of the reversed austenite has been little studied.

The purpose of this investigation is to make clear the effects of holding temperature, holding time and prior deformation on the recrystallization of reversed austenite in 18%Ni maraging steel, and also to study the martensite structures formed from either unrecrystallized or recrystallized austenites.

II. Experimental Procedures

Chemical compositions of 18%Ni maraging steel (210 kg/mm² grade) used are shown in Table 1. As-received plate materials of about 5 mm thick were cold rolled to sheets of about 0.5~3 mm in thickness. These were solution treated in vacuum at 1300°C for 1 h and water quenched. An average prior austenite grain size of 390 μ m was obtained by this solution treatment. Part of solution treated specimens were rolled by 50% at room temperature. Solution treated (nondeformed) and 50% rolled specimens were heated at selected temperatures between 725° and 1300°C at a constant heating rate of 100°C/min, and held for various periods of time, and then cooled to room temperature at cooling rate of 100°C/min. Microstructures of these specimens were observed with optical and transmission electron microscopes. Specimens for optical micrographs were etched with the solution of FeCl₃ 10 g, HCl 30 cc and H₂O 120 cc for revealing the martensite structure. In order to observe the prior austenite grain boundaries, some of the specimens were aged at 500°C for 20 min and electrolytically etched with CrO₃ 10 g+H₂O 100 cc. Prior austenite grain sizes were determined by the linear analysis on the optical micrographs. Transformation behavior of the specimens during heating or holding at a constant temperature was examined by the electrical resistivity change.

Table 1. Chemical compositions of 18%Ni maraging steel investigated. (wt%)

Ni	Co	Mo	Ti	Mn	Cr	S	P	Si	C	Fe
18.30	8.95	5.00	0.70	<0.01	<0.01	0.008	0.002	<0.001	0.015	bal.

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† In this paper, the austenite formed from the martensite by heating is referred to as a reversed austenite, regardless of a mechanism of the reverse transformation (e.g., whether a shear transformation or a diffusional transformation).

III. Results and Discussion

1. Recrystallization Behavior of Reversed Austenite in Martensitic Specimens

Figure 1 shows an example of resistivity *vs.* temperature curve for the solution treated (nondeformed) specimen. Specimens were heated at 950°C and then immediately cooled to room temperature at a rate of 100°C/min. The average A_s and A_f temperatures determined by resistivity curves in eight samples were 684°C and 786°C, respectively.

Photograph 1 shows the optical micrographs of the specimens which were heated at various temperatures above A_f and immediately cooled to room temperature. The solution treated specimen (Photo. 1 (a)) shows a typical lath martensite structure which is characterized by the well-developed blocks in a prior austenite grain.¹²⁾ Photograph 1 (b) is the microstructure of the specimen heated at 800°C (just above A_f) and immediately cooled, showing the same austenite grain size as that of original specimen before heating (Photo. 1 (a)). Hosomi *et al.*⁸⁾ have observed that the location of the reversed austenite grain boundaries in 18%Ni maraging steel exactly corresponds to the prior austenite grain boundaries of the original specimen and hence there is no change in austenite

Fig. 1. Resistivity change of 18%Ni maraging steel; solution treated specimen.

grain size by the $\alpha' \rightarrow \gamma$ reverse transformation. These phenomena were also observed in the present study as will be shown later. When the specimen is heated at 900°C, small austenite grains are locally observed (Photo. 1 (c)). These small grains are formed by the recrystallization of reversed austenite. It is clear that the recrystallized grains form preferentially along the reversed austenite grain boundaries. When heated at 950°C, as shown in Photo. 1 (d), the recrystallization of reversed austenite is completed and austenite grains become fine. Hereafter, the reversed austenite having the same grain size as that of original specimen such as Photo. 1 (b) is referred to as the unrecrystallized austenite.

The reverse transformation proceeds during holding the specimen at the temperatures between A_s and A_f which were determined by continuous heating at a rate of 100°C/min. Figure 2 shows the progress of reverse transformation during holding the specimen at 725°C. Figure 2 (a) shows the resistivity change of the specimen heated at 725°C and immediately cooled to room temperature. During cooling of the specimen, the slight change in the slope of the resistivity-temperature curve is observed at M_s temperature, indicating that the reversed austenite is partially formed during heating at 725°C. When the specimen is held at 725°C for 30 min, the resistivity increases during the holding and the slope of resistivity-temperature curve on cooling changes widely at M_s temperature (Fig. 2 (b)). As is seen in Fig. 2 (c), when the specimen is again heated at a higher temperature after holding at 725°C for 30 min, the resistivity increases linearly with temperature and shows no evidence of progress of further reverse transformation. It is clear from these resistivity measurements that the $\alpha' \rightarrow \gamma$ reverse transformation completes by heating at 725°C for 30 min.

Photograph 2 shows the microstructure change with the holding time at 725°C. The specimen immediately cooled from 725°C without any holding time (Photo. 2 (a)) consists of both the tempered martensite and the freshly transformed martensite

(a) Before heating (as solution treated: 1300°C 1 h → W.Q.)
(b) $T=800^\circ\text{C}$ (c) $T=900^\circ\text{C}$
(d) $T=950^\circ\text{C}$

Photo. 1.

Optical micrographs of 18%Ni maraging steel held at various temperatures (T) and immediately quenched (heating and cooling rates: 100°C/min); solution treated specimen. Etchant: FeCl_3 10 g + HCl 30 cc + H_2O 120 cc.

formed from the reversed austenite. The lightly etched regions in Photo. 2 (a) may correspond to the freshly transformed martensite. As is evident from Fig. 2 (b), the reverse transformation is completed by holding at 725°C for 30 min, and the reversed austenite is unrecrystallized (Photo. 2 (b)). When the specimen is held for longer time at 725°C, the reversed austenite is partially recrystallized by holding for

Fig. 2. Resistivity change of 18%Ni maraging steel; solution treated specimen.

50 h (Photo. 2 (c)) and completely recrystallized by holding for 110 h (Photo. 2 (d)).

Photograph 3 shows the optical micrographs of the specimens held at various temperatures for 1 h. These micrographs indicate that the reversed austenite is unrecrystallized at 725°C (Photo. 3 (a)), partially recrystallized at 750°C (Photo. 3 (b)) and 775°C (Photo. 3 (c)), and completely recrystallized at 800°C (Photo. 3 (d)).

Based on the microstructural observations such as Photos. 1 to 3, the recrystallization behavior of reversed austenite is summarized as shown in Fig. 3 as a function of holding temperature and time. The reversed austenite right after the completion of $\alpha' \rightarrow \gamma$ transformation is unrecrystallized irrespective of holding temperature (*i.e.*, the grain size of reversed austenite is identical with the prior austenite grain size of the original specimens). With an increase in holding time or in holding temperature, the reversed austenite changes from the unrecrystallized to the recrystallized austenites. Because of the recrystallization of the reversed austenite, the austenite grain size once becomes fine, and then increases with an increase in holding time or holding temperature due to the grain growth.

In the case of Fe-30~34%Ni alloys in which $\alpha' \rightarrow \gamma$ reverse transformation takes place at fairly low temperatures by the mechanism of shear transformation, grain boundaries of the reversed austenite are known to correspond to that of original specimen¹³⁾ and the recrystallization of reversed austenite occurs by further heating because of a high density of lattice defects caused by shear mechanism of martensitic reverse transformation.^{14,15)} The present observation in 18%Ni maraging steel is similar to that in Fe-high Ni alloys. But the reverse transformation in maraging steel might take place partly by the diffusional process in addition to the shear mechanism because of high A_s and A_f temperatures.

(a) $X=0$ min (b) $X=30$ min
(c) $X=50$ h (d) $X=110$ h

Photo. 2.

Optical micrographs of 18%Ni maraging steel; solution treated and held at 725°C for various period (X) (heating and cooling rates: 100°C/min). Etchant: FeCl_3 10 g + HCl 30 cc + H_2O 120 cc.

(a) $T=725^{\circ}\text{C}$ (b) $T=750^{\circ}\text{C}$
(c) $T=775^{\circ}\text{C}$ (d) $T=800^{\circ}\text{C}$

Photo. 3.
Optical micrographs of 18%Ni maraging steel; solution treated and held at various temperatures (T) for 1 h (heating and cooling rates: $100^{\circ}\text{C}/\text{min}$). Etchant: FeCl_3 10 g + HCl 30 cc + H_2O 120 cc.

Fig. 3. Structure change of reversed austenite with holding (austenitizing) temperature and holding time in 18%Ni maraging steel (heating rate: $100^{\circ}\text{C}/\text{min}$; solution treated specimen).

2. Recrystallization Behavior of Reversed Austenite in Prior-deformed (50% Rolled) Martensitic Specimens

The effect of prior deformation of martensite on the reverse transformation and the recrystallization of reversed austenite was examined. The specimens solution treated at 1300°C for 1 h were cold rolled by 50% at room temperature and heated at several temperatures above A_f .

Figure 4 shows the resistivity-temperature curve of 50% rolled specimen. This curve is similar to that of nondeformed specimen, indicating that the prior deformation of 50% does not have any significant effect on the reverse transformation. The A_s and A_f temperatures of 50% rolled specimen were 695° and 790°C , respectively. These temperatures are slightly higher than those of nondeformed specimens.

Photograph 4 shows the optical micrographs of specimens which were deformed by 50% at room temperature and heated at several temperatures above A_f and then immediately cooled to room temperature. Photograph 4 (a) shows the martensite structure deformed by 50% at room temperature. Prior austenite grain boundaries and martensite block boundaries are heavily distorted. Photograph 4 (b) shows the

Fig. 4. Resistivity change of 18%Ni maraging steel; 50% rolled specimen.

microstructure of a specimen heated at 800°C (just above A_f temperature) after 50% deformation. The reversed austenite grain size and morphology of martensite seem to be the same as that of the original deformed specimen. It is of interest that not only prior austenite boundaries but also the martensite block boundaries are distorted even after a cyclic transformation. Furthermore, fine recrystallized grains are observed in the prior austenite grain, as can be seen clearly in Photo. 4 (c) which is the higher magnification micrograph of Photo. 4 (b), indicating that the recrystallization of reversed austenite formed from the deformed martensite occurs right after the completion of reverse transformation. Thus, the recrystallization temperature of reversed austenite is lowered by the prior deformation of martensite. It is also to be noted that the nucleation site of recrystallized grain of reversed austenite is different between deformed and nondeformed specimens. Whereas the recrystallized grains form preferentially along the reversed austenite grain boundaries (corresponding to the prior austenite grain boundaries before heating) for the nondeformed specimens, they form evenly in the reversed austenite grain for deformed specimens.

The recrystallization of reversed austenite progresses with an increase in holding temperature as shown in Photo. 4 (d) and is completed at 850°C

Photo. 4. Optical micrographs of 18%Ni maraging steel; deformed of 50% by rolling and held at various temperatures (T) and immediately quenched (heating and cooling rates: $100^{\circ}\text{C}/\text{min}$). Etchant: FeCl_3 10 g + HCl 30 cc + H_2O 120 cc.

(Photo. 4 (e)). The recrystallization of reversed austenite is completed at lower temperature in deformed specimens than in nondeformed as is evident from the comparison of Photos. 4 (e) and 1 (c). Koppenaal and Gold¹⁵⁾ reported that the deformation of martensite prior to reverse transformation increases the driving force for recrystallization of reversed austenite in Fe-31%Ni-0.03%C alloy.

3. Change in Reversed Austenite Grain Size with Austenitizing (Holding) Temperature in Nondeformed and Deformed Specimens

In nondeformed (solution treated) and deformed (50% rolled) specimens, the change in reversed austenite grain size with holding temperature was observed. Photograph 5 shows an example of the effect of prior deformation on the reversed austenite grain size. These micrographs were taken from the specimen held at 900°C for 1 h in nondeformed (Photo. 5 (a)) and deformed (Photo. 5 (b)) specimens. It appears that the reversed austenite grain size of deformed specimen is smaller than that of nondeformed specimen.

Figure 5 shows the change in the reversed austenite grain size with holding temperature in nondeformed and 50% rolled specimens. The reversed austenite grain size increases with an increase in holding temperature higher than A_f in both specimens. As can be seen, prior deformation of martensite is effective for the refinement of reversed austenite grain size, although this effect gradually lessens with increasing holding temperature. Such a difference in reversed austenite grain size between nondeformed and deformed specimens is due to the change in the preferential nucleation site of recrystallized grain of the reversed austenite as mentioned in Section III. 2. The reversed austenite formed at 725°C for 1 h in non-

Photo. 5. Optical micrographs of 18%Ni maraging steel held at 900°C for 1 h; (a) nondeformed (solution treated) specimen, (b) 50% rolled specimen. Electrolytically etched with CrO_3 10 g + H_2O 100 cc.

deformed specimen is unrecrystallized and thus the austenite grain size is the same as that of the original specimen as shown in Fig. 5.

4. Morphological Characteristics of Martensites Formed from the Unrecrystallized Reversed Austenite

Photographs 6 (a) and (b) are the optical micrographs taken from the identical area of the specimen before and after the cyclic $\alpha' \rightarrow \gamma$ (unrecrystallized) $\rightarrow \alpha'$ transformation. Specimen was heated at 725°C

for 1 h to produce the reversed austenite (unrecrystallized) and then water quenched. It is to be noted the reversed austenite grain boundaries after the cyclic transformation (Photo. 6 (b)) exactly corresponds to that of original specimen before the cyclic transformation (Photo. 6 (a)). Furthermore, the martensite structure does not change by the cyclic transformation. In Photo. 6, martensite packet boundaries are observed. Thus, it was found that the microstructure (such as reversed austenite grain boundary, martensite packet boundary and maybe block boundary) is reproducible, provided that the reversed austenite is unrecrystallized. This microstructural reproducibility was also observed in the prior deformed specimens.

Photograph 7 shows the transmission electron micrographs of lath martensites in the specimens which solution treated (Photo. 7 (a)), held at 725°C for 1 h (Photo. 7 (b)) and at 820°C for 1 h (Photo. 7 (c)). Lath martensites in Photos. 7 (a) and (c) were formed from the recrystallized austenite and

those in Photo. 7 (b) were formed from the unrecrystallized austenite. Lath martensites in all the specimens contain a high density of dislocations. The width of martensite laths formed from the unrecrystallized austenite (Photo. 7 (b)) is fairly small in comparison with that formed from the recrystallized austenite. Photographs 6, 7 (a) and 7 (b) indicate that, as a characteristic of lath martensite structure formed from the unrecrystallized austenite, the microstructure on the scale of optical micrographs (*i.e.*, reversed austenite boundary, martensite packet and block boundaries) is identical with that of the original state, but the lath size is different from that of the original specimen.

Fig. 5. Change in the reversed austenite grain size with holding temperature (holding time: 1 h) in non-deformed (solution treated) and 50% rolled 18%Ni maraging steel.

(a) Solution treated (1 300°C 1 h → W.Q.)
(b) (a) + 725°C 1 h → W.Q.

Photo. 6. Optical micrographs taken from the identical area of the specimen in 18%Ni maraging steel before and after $\alpha' \rightarrow \gamma \rightarrow \alpha'$ cyclic transformation. Electrolytically etched with CrO_3 10 g + H_2O 100 cc.

(a) Solution treated (1 300°C 1 h → W.Q.) (b) (a) + 725°C 1 h → W.Q. (c) (a) + 820°C 1 h → W.Q.

Photo. 7. Transmission electron micrographs of lath martensites of 18%Ni maraging steel; solution treated and then held at various temperatures.

$\bar{D}_A$ = prior austenite grain size

- (a), (c), (d): martensites formed from the recrystallized reversed austenite.
 (b): martensites formed from the unrecrystallized reversed austenite.

Fig. 6. Lath width distributions for 18%Ni maraging steel held at various temperatures for 1 h; solution treated specimen.

Figure 6 shows the lath width distribution in the specimens held at various temperatures. About 300 laths in each specimen were measured on the micrographs such as Photo. 7. The distribution of lath width in the specimen quenched from the recrystallized austenite state is almost the same irrespective of holding temperature as shown in Figs. 6 (a), (c) and (d). The most frequently observed width is about $0.20 \sim 0.25 \mu\text{m}$. This indicates that the size of martensite laths is not affected by the prior austenite grain size. On the other hand, in the case of lath martensite formed from the unrecrystallized austenite, the lath width distribution becomes narrow and shifts to the smaller size. The most frequently observed lath width is between 0.10 to $0.15 \mu\text{m}$.

IV. Summary

The recrystallization behavior of the reversed austenite in solution treated (nondeformed) and deformed (50% rolled) 18%Ni maraging steel and the martensite structure formed from the unrecrystallized and recrystallized austenite were studied mainly by the microstructural observations. The main results obtained are as follows.

(1) Right after the completion of $\alpha' \rightarrow \gamma$ transformation, the grain boundaries of reversed austenite exactly correspond to the prior austenite grain boundaries of the original specimen before heating in both

nondeformed and deformed specimens, indicating that no change in the austenite grain size is observed. The reversed austenite is recrystallized to a smaller grain size by further heating.

(2) The recrystallization of the reversed austenite in deformed specimen starts and finishes at lower temperatures in comparison with the nondeformed specimen. The recrystallized grains form preferentially along the reversed austenite grain boundaries for the nondeformed specimens. On the other hand, in the deformed specimens, the recrystallized grains form finely and evenly in the reversed austenite grain. Owing to such a difference in preferential nucleation site of recrystallized grains, a deformation of martensite prior to heating enhances the refinement of reversed austenite grain.

(3) Martensite structure such as packets and blocks after the $\alpha' \rightarrow \gamma \rightarrow \alpha'$ transformation almost exactly corresponds to that of the original specimen before heating, provided that the reversed austenite is unrecrystallized. This microstructural reproducibility was also observed in the deformed specimens. Irrespective of such a microstructural reproducibility on a scale of optical microstructure, the lath size of martensites formed from the unrecrystallized austenite becomes smaller than that of the original specimen. The most frequently observed lath width changes from about $0.20 \sim 0.25 \mu\text{m}$ (martensites formed from the recrystallized austenite) to about $0.10 \sim 0.15 \mu\text{m}$ (martensites formed from the unrecrystallized austenite).

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