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Effect of Cyclic Heat-Treatments on the Martensitic Transformation in Iron-Nickel Binary Alloys*

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Synopsis

The effect of the cyclic heat-treatments on the martensitic transformation was examined with iron-nickel binary alloys containing 24.50 to 30.70 atomic per cent nickel from the observations of dilatation curves, microstructures and mechanical properties. Ms temperatures lowered with the increase in the number of cyclic heat-treatments so that the amount of martensite transformed also decreased. The optical and the electron microstructures of martensite after cyclic heat-treatments consisted of finer martensite plates with high dislocation density. The hardness and the tensile strength of austenite were remarkably increased by cyclic heat–treatments at an early state, but in the case of martensite they were not remarkably increased. It was suggested that these results would be explained by mechanisms based on the hardening of austenite and the partial decomposition of martensite due to the cyclic heat-treatments.

I. Introduction

In recent years, many investigations(1),(2) have been made of the high strength iron-nickel base steels strengthened by martensitic transformation and precipitation hardening. It has been known that the austenite produced by the reverse martensitic transformation in iron-nickel binary alloys is significantly altered in microstructure and strength characteristics, compared with the virgin austenite. Several works(3)~(7) have been reported about these phenomena, but no systematic investigation was carried out concerning various nickel contents.

The object of the present work is to clarify the effect of cyclic (martensite$\rightleftarrows$austenite) heat-treatments on the martensitic transformation behaviour, microstructure and mechanical properties of iron-nickel alloys containing various amounts of nickel.

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* The **1318th** report of the Research Institute for Iron, Steel and Other Metals.

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(2) D.T. Peters and C.R. Cupp, Trans. AIME, **236** (1966), 1420.
(3) G. Wassermann, Archiv. Eisenhütt., **6** (1933), 347.
(4) B. Edmondson and T. Ko, Acta Met., **2** (1954), 235.
(5) G. Krauss and M. Cohen, Trans. AIME, **224** (1962), 1212.
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II. Experimental procedure

The iron-nickel alloys were melted in vacuum. After casting into a chill mould and then hot-rolling to 10 mm diameter, the specimen for dilatometric measurement was machined to 5 mm diameter and 70 mm length, that for hardness measurement was of 10 mm diameter and 6 mm length and that for tensile test was of 5 mm diameter and 30 mm gauge length. Thin plates cold-rolled to 0.15 mm thickness were used for examinations of X-ray diffraction and of transmission electron microstructure. These specimens were annealed at 1250°C to eliminate strain and preferred orientation after recrystallization. This annealing was utilized also for the decarbonization of the specimens. Accordingly, the specimens were annealed at 1250°C for 6 hr in dry hydrogen. The compositions of iron-nickel alloys of the five kinds under investigation are listed in Table 1. The specimens were solution-treated at 1100°C for 30 min in vacuum and then water-quenched.

Table 1. Chemical composition (wt%)

Specimen	C	Si	Mn	P	S	Ni
Fe-24.50 at% Ni	0.001	0.007	0.001	0.001	0.001	25.43
Fe-27.57 at% Ni*	trace	0.04	0.16	trace	0.005	28.57
Fe-28.77 at% Ni	0.002	trace	trace	0.001	0.003	29.80
Fe-29.96 at% Ni	0.004	0.005	0.001	0.001	0.001	31.00
Fe-30.75 at% Ni	0.002	trace	trace	0.001	0.001	31.77

* air-melted

On cyclic heat-treatment, austenitizing temperatures were selected to be just above the Af points, at which the martensite-to-austenite transformation finished (Fe–24.50 at % Ni: 650°C, -27.57 at % Ni: 620°C, -28.77 at % Ni: 600°C, -29.96 at % Ni: 540°C and -30.70 at % Ni: 530°C) and cooling temperature was selected to be −196°C.

In the present work, the number of cyclic heat-treatments was defined, for convenience, as follows: The 0-cycled martensitic transformation is that of virgin austenite to martensite by cooling to −196°C and then the transformation of martensite to austenite on heating is the 1-cycled reverse martensitic one; the transformation by the second cooling to −196°C is the 1-cycled martensitic one. Thus, the subsequent numbers of cyclic heat-treatments in the martensite/austenite transformation are 2,3,4,.... 10 and 20.

The dilatation curves were obtained with a differential dilatometer and a total dilatometer above and below room temperature, respectively.

The amount of martensite transformed was determined from lattice constants of austenite and martensite, and from the amount of dilatation accompanying martensitic transformation, that is, the volume change accompanying martensitic transformation corresponds to the difference between volumes occupied by an atom in austenite and in martensite lattice. Then the calculated change in length corresponding to a complete martensitic transformation is one-third of the volume change. Accordingly, the amount of martensite transformed can be estimated by comparing the calculated values with ones obtained from the dilatation curves.

III. Results and discussions

1. Ms temperature and the amount of martensite

Fig. 1 shows the relationship between the number of cyclic heat-treatments and Ms temperatures determined from dilatation curves. In low nickel alloys, Ms temperatures do not remarkably change by cyclic heat-treatments, but in high nickel alloys, they decrease with the increase in the number of cyclic heat-treatments

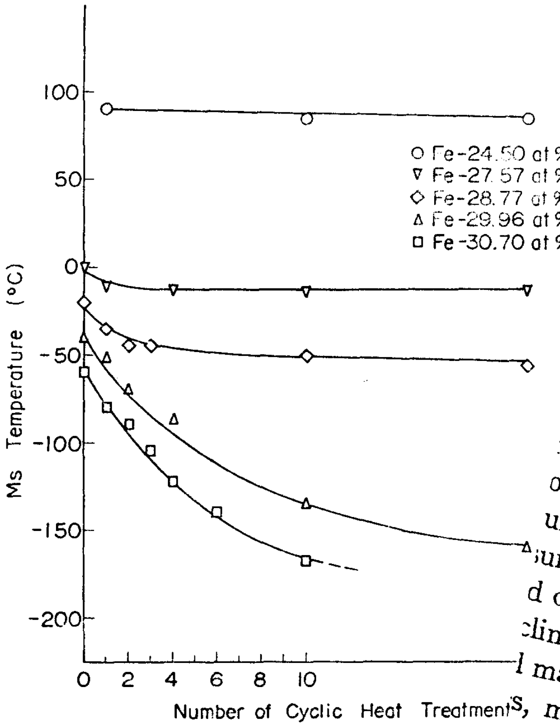

Fig. 1. Effect of the number of cyclic heat-treatme re.

(these results will be discussed later). This effect h as a result of the stabilization of austenite by numerous investi Krauss et al.⁽⁸⁾ observed the absence of stabilization by martensitic specimens from room temperature and concluded tion occurs during slow heating because of a diffusion-controll to low-nickel bcc ferrite and high-nickel fcc austenite in the ge where the

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(8) G. Krauss and M. Cohen, Trans. AIME, 227 (196

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latter two phases comprise the equilibrium structure. Since the heating rate in this study is 3°C/min because of measuring dilatation of specimens, decomposition of martensite predicted by them will occur. Yet, Ms temperature of the up-quenched specimen by using a lead-bath decreased by cyclic heat-treatments, though it did not so remarkably as in Fig. 1 (−60 to −80°C after 5 cycles in an iron–30.70 at % nickel alloy). This result shows that the decrease of Ms temperature in the up-quenched specimen may be explained by either decomposition of martensite due to insufficiently rapid heating or the hardening of austenite due to cyclic heat-treatments or by both effects.

The amount of martensite transformed decreases with the increase in the number of cyclic heat-treatments as shown in Fig. 2, especially in higher nickel alloy.

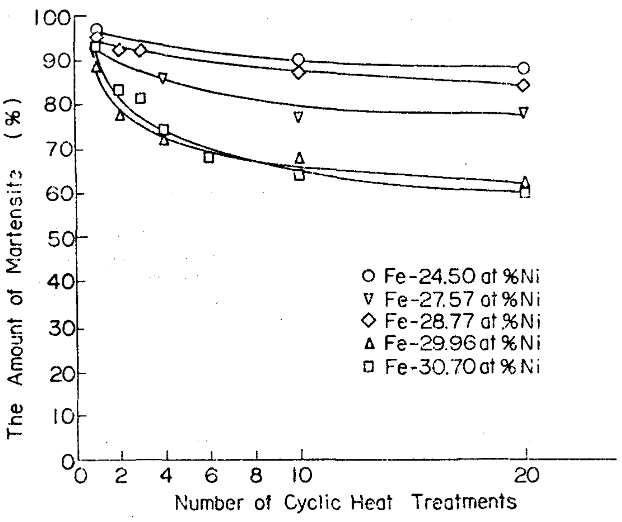
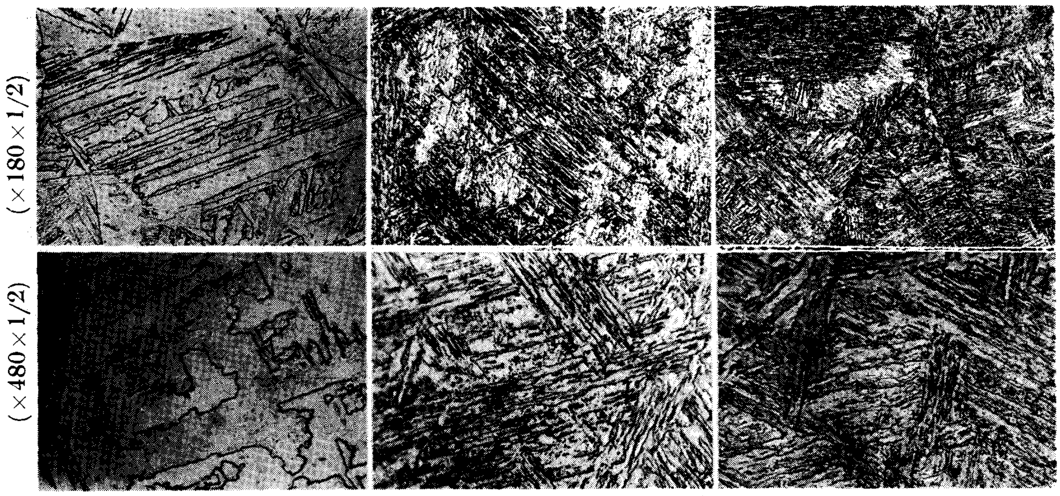


Fig. 2. Effect of the number of cyclic heat-treatments on the amount of martensite.

2. Microstructures

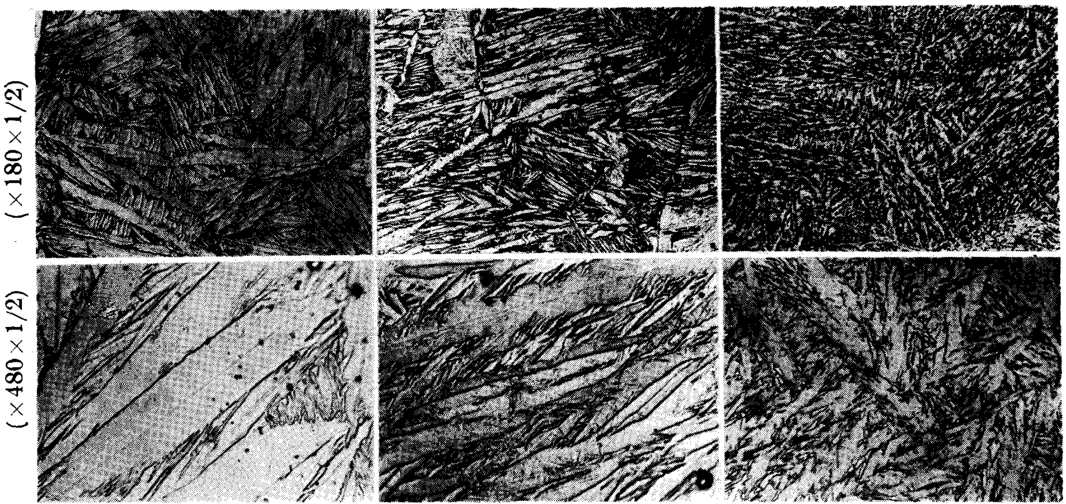
Photo. 1 shows the effect of cyclic heat-treatments on the martensite microstructures in iron–24.50 at % nickel alloy. (a) shows the first-generated (O-cycled) martensite by cooling to −196°C after solution-treatment. (b) shows one-cycled martensite, which becomes very fine. However, martensite microstructures do not become finer by a number of cycles as shown in (c) of 20 times of cyclic heat-treatments.

Photo. 2 shows the effect of cyclic heat-treatments on the martensite microstructures in iron–30.70 at % nickel alloy. (a) shows the first-generated martensite microstructure by cooling to −196°C, in which large martensite plates are observed. However, the one-cycled microstructure shows finer martensite plates, being irregular at austenite/martensite interface as shown in (b). This trend is increasingly observed in the martensite microstructures produced after a number of cycles as shown in (c) corresponding to that of 10 times.



(a) (b) (c)

Photo. 1. Optical micrographs of martensite in the iron-24.50 at % nickel alloy. The number of cyclic heat-treatments; (a): 0, (b): 1 and (c): 20.



(a) (b) (c)

Photo. 2. Optical micrographs of martensite in the iron–30.70 at % nickel alloy. The number of cyclic heat-treatments; (a): 0, (b): 1 and (c): 10.

Photo. 3 shows transmission electron micrographs of martensite in iron–30.70 at % nickel alloy. In (a) fine twins are observed in large martensite plates, which are first-generated by cooling to –196°C. As shown in one-cycled (b) and five-cycled (c), martensite becomes fine plate with high dislocation density. On the contrary, fine twins decrease with the increase in the number of cyclic heat-treatments, which will result in requiring high energy for producing twin in martensite which inherits strain from austenite.

Photo. 4 shows transmission electron micrographs of austenite in iron–30.70 at % nickel alloy. (a) shows the austenite structure water-quenched after solution-treatment. Dislocation density is very low and nearly straight dislocations are observed. In (b) of three times of cyclic heat-treatments, the dislocation density is very high and highly tangled dislocations are observed, being similar to the

(a) (b) (c)

Photo. 3. Transmission electron micrographs of martensite in the iron–30.70 at % nickel alloy. The number of cyclic heat-treatments; (a): 0, (b): 1 and (c): 5.

(a) (b) (c)

Photo. 4. Transmission electron micrographs of austenite in the iron–30.70 at % nickel alloy. The number of cyclic heat-treatments; (a): 0, (b): 3 and (c): 10

structures shown by other investigators(9). This trend is more significantly observed with the increase in the number of cyclic heat-treatments as shown in (c) of 10 times. It seems that these results interpret the martensite plates becoming fine by cyclic heat-treatments. It has been generally accepted that martensite is nucleated at some imperfections such as dislocations in austenite(10)-(12). As shown in Photos. 4(b) and (c), the dislocation density increases by cyclic heat-treatments and so the nucleation of martensite will also increase. Accordingly, martensite plates become fine.

Furthermore, it is noticeable that cell structures similar to the work-hardened structure in metals and alloys of high stacking fault energy are observed as shown in Photo. 4(c). This suggests that transformation strains are stored in the alloy during cyclic heat-treatments below recrystallization temperature.

It has been reported that iron–27.57 at % nickel alloy subjected to cyclic heat-treatments recrystallized at about 650°C(13). Austenitizing temperature of each alloy in the present case is between 530 and 620°C, except for iron–24.50 at %

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(9) G. Krauss, Acta Met., **11** (1963), 499.
(10) F.C. Frank, Acta Met., **1** (1953), 15.
(11) H. Suzuki, Sci. Rep. RITU, **A6** (1954), 30.
(12) H. Knapp and U. Dehlinger, Acta Met., **4** (1956), 289.
(13) M. Izumiyama, Sci. Rep. RITU, **A14** (1962), 11.

nickel alloy. It was ascertained by hardness and microstructure examination that these temperatures are below recrystallization temperatures. For example, after 20 times of cyclic heat-treatments, when the alloys are heated to respective temperatures between 600 and 1000°C then water-quenched, the hardness of austenite decreases remarkably between 600 and 700°C as shown in Fig. 3. Therefore, it will be seen that the alloys recrystallize between 600 and 700°C.

Photo. 5 shows microstructural changes of martensite in iron-29.96 at % nickel alloys which are heated at 600, 700 and 1000°C after 20 times of cyclic heat-treatments and then cooled to −196°C. Martensite plates become remarkably large between 600 and 700°C, because the strain stored in austenite is released by heating to such a temperature range.

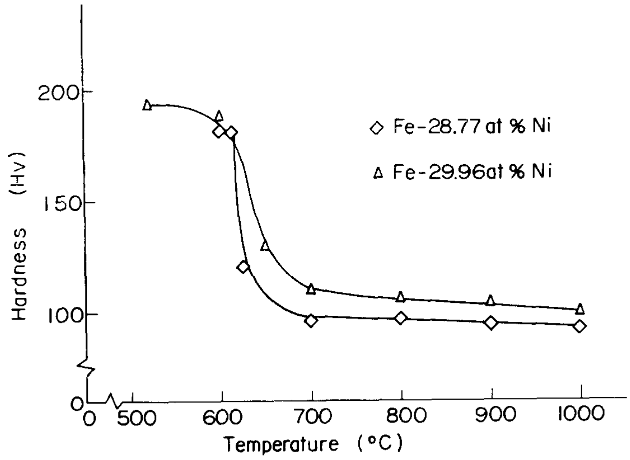

Fig. 3. Effect of austenitizing temperature on hardness of austenite after 20 times of cyclic heat-treatments.

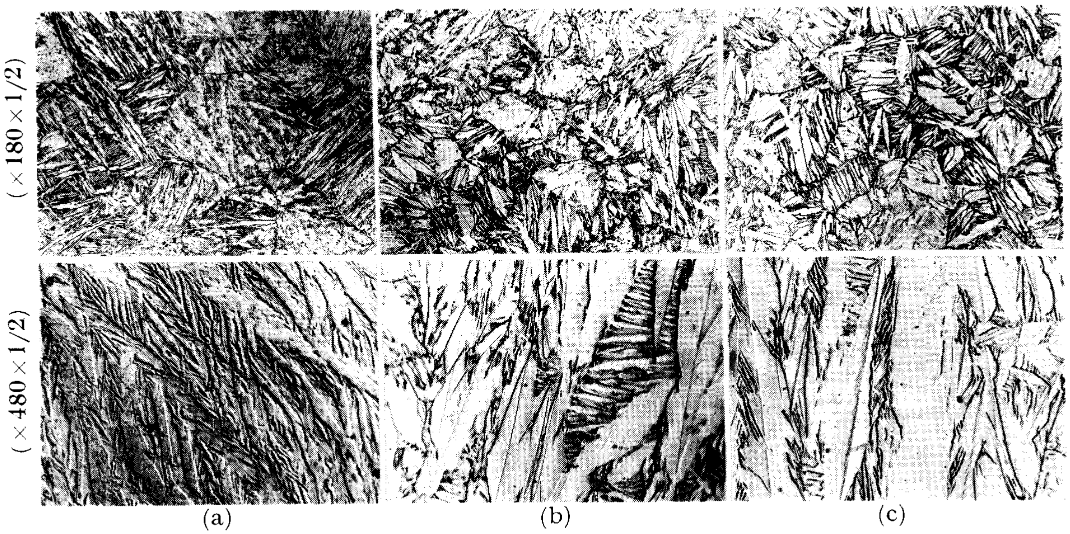

Photo. 5. Optical micrographs of martensite in the iron–29.96 at % nickel alloy. (a): 20 times of cyclic heat-treatments→600°C→−196°C, (b): 20 times→700°C→−196°C and (c) 20 times→1000°C→−196°C

In the transformation to martensite of austenite hardened by cyclic heat-treatments below such a strain-releasing temperature, it is considered that more driving force than that of austenite without hardening will be required, corresponding to the extent of hardening. Consequently, it seems that austenite must be further supercooled. This effect should be taken into consideration as another cause of Ms temperature depression shown in Fig. 1. In this study, the lattice parameter of the austenite did not change during cyclic heat-treatments and X-ray diffraction did not reveal the presence of bcc phase. Therefore, even if bcc phase is produced by the decomposition of martensite during heating, the amount of this phase is slight. Accordingly, the decrease in Ms temperatures shown in Fig. 1 will come from both the decomposition of martensite and the stabilization of austenite.

To clarify the extent of each effect, the relation between Ms temperature changes by cyclic heat-treatments and heating rates should be investigated in detail.

3. Mechanical properties

Fig. 4 shows the effect of cyclic heat-treatments on the hardness of austenite. The hardness increases by 1.7 to 1.8 times through 2 to 3 cycles of heat-treatments and saturates beyond these cycles. On the contrary, the hardness of martensite does not change significantly by cyclic heat-treatments as shown in Fig. 5.

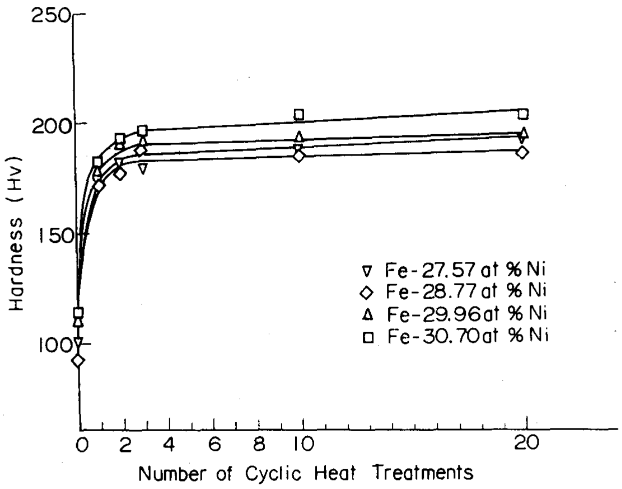

Fig. 4. Effect of the number of cyclic heat-treatments on hardness of austenite.

The relation between the tensile strength and the number of cyclic heat-treatments is shown in Fig. 6. The tensile strength of austenite increases about 1.5 times by cyclic heat-treatments, but that of martensite does not increase so remarkably as that of austenite.

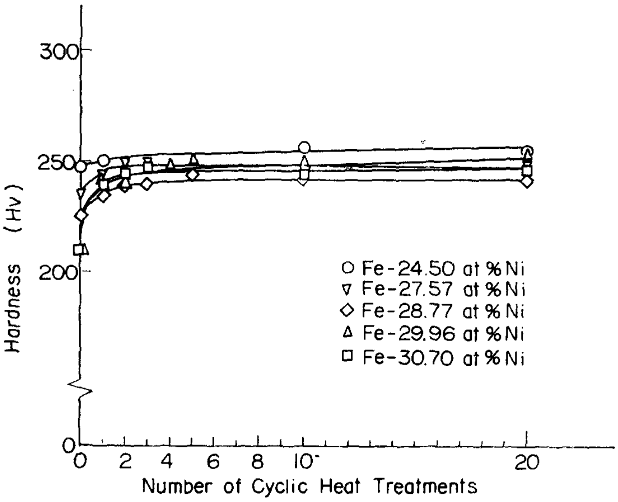

Fig. 5. Effect of the number of cyclic heat-treatments on hardness of martensite.

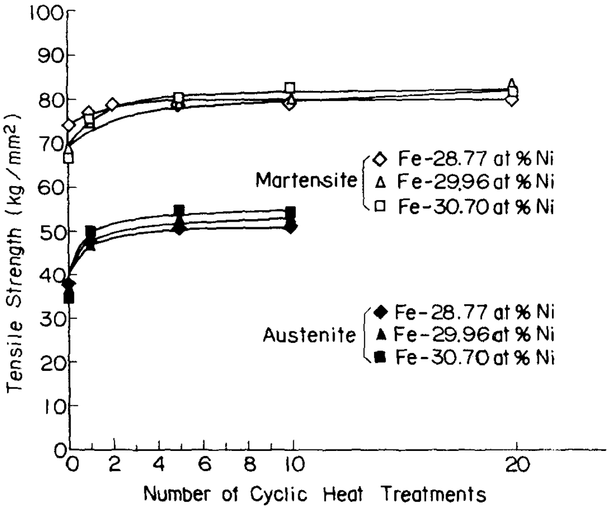

Fig. 6. Effect of the number of cyclic heat-treatments on tensile strength of martensite and austenite.

Both elongation and reduction of area in iron–30.70 at % nickel alloy do not decrease significantly by cyclic heat-treatments, but those in iron–28.77 at % nickel alloy lower remarkably as shown in Fig. 7. Cracks induced by cyclic heat-treatments are observable along the grain boundaries in the latter alloy as seen in Photo. 6. It is considered that this grain boundary crack causes the decrease in the elongation and in the reduction of area in this alloy.

As shown in Fig. 2, the amount of martensite transformed from austenite after a number of cycles is larger in the alloy with less contents of nickel. Such observation can be understood on account of the fact that the larger the amount of transformation with less retained amount of the austenite is, the more is the volume change in the heat-treatment.

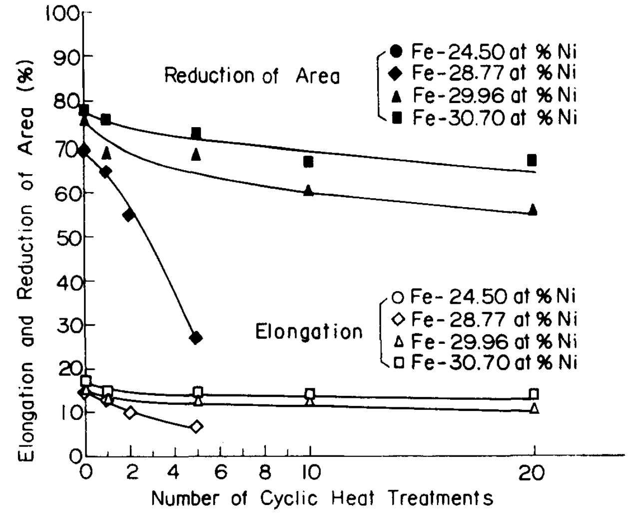

Fig. 7. Effect of the number of cyclic heat-treatments on elongation and reduction of area of martensite.

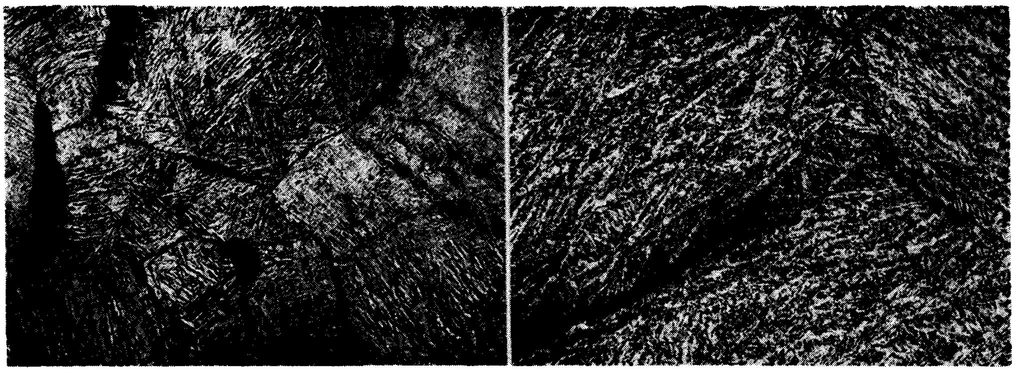
(a) (×120×3/4) (b) (×120×3/4)

Photo. 6. Grain boundary cracks generated by 20 times of cyclic heat-treatments in the iron-24.50 at% nickel alloy (a) and in the iron-28.77 at% nickel alloy (b).

Summary

Results obtained in the present investigation may be summarized as follows:

(1) Ms temperature in iron-nickel alloys lowers with the increase in the number of cyclic heat-treatments, especially in the higher nickel alloys. This phenomenon cannot be explained only by the decomposition of martensite during heating, but probably the stabilization of austenite due to the cyclic heat-treatments will play an important role.

(2) The amount of retained austenite is increased by cyclic heat-treatments in iron-high nickel alloys.

(3) Martensite plate becomes remarkably fine by cyclic heat-treatments.

(4) Both austenite and martensite subjected to cyclic heat-treatments contain many tangled dislocations.

(5) Hardnesses of austenite and of martensite reach the respective satura-

tion values. The hardness of austenite subjected to cyclic heat-treatments increases by 1.7 to 1.8 times than that of virgin austenite and that of martensite about 1.2 times at the most.

(6) Tensile strength of austenite increases by about 1.5 times through 5 cycles of heat-treatments, but that of martensite about 1.2 times.

(7) Grain boundary crack occurs in iron-low nickel alloys by cyclic heat-treatments and, consequently, the elongation and the reduction of area both decrease.