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The Relationship of Microstructure and Magnetic Properties in Cold-rolled 6.5% Si-Fe Alloy

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Abstract — The effect of microstructures on the magnetic properties in the 6.5% Si-Fe alloy was studied. The alloy was subjected to two patterns of heat treatments, and the microstructures were observed by means of TEM. The alloy after an appropriate treatment has shown excellent soft magnetic properties, with the maximum permeability of 7.25×10^4 and the core loss $W_{10/50}$ of 0.39 W/kg. The corresponding microstructure was characterized by the extremely fine domains of DO_3 order phase existing in B2 order phase. However, the coarsening of the DO_3 domains was found to reduce the maximum permeability and increase the core loss of the alloy. It is suggested that the coarsening of the DO_3 domains be accompanied with the formation of magnetic contrast around the boundary of the DO_3 and B2 phases. Therefore, it is assumed that the phase boundary tends to oppose the displacement of magnetic domain walls due to the existence of the magnetic contrast.

I. INTRODUCTION

In the Fe-Si system, an alloy with 6.5 % Si has shown a high saturation magnetization and near zero magnetostriction [1]. This alloy is expected to be very useful for a number of applications. On the basis of the Fe-Si phase diagram [2], there are two kinds of order phases, DO_3 and B2, in the 6.5 % Si-Fe alloy. The magnetic properties of the alloy were reported to vary with their microstructures [3]. Since a clear understanding of microstructure is effective for improving the magnetic properties, the purpose of this work was to investigate the magnetic properties and to observe the microstructure in order to clarify its effect on the magnetic properties.

II. EXPERIMENTAL

Table I shows the chemical composition of the alloy used in this work. Specimens were prepared to a thickness of 0.35 mm by cold-rolling after hot-rolling. Rings of 25 mm x 15 mm were then cut from the plate. The rings were first annealed at 1200℃ for one hour, and then furnace cooled to room temperature for the purpose of recrystallization and grain growth. We call this heat treatment normalization treatment. Following the normalization treatment, the specimens were subjected to different heat treatments. As shown in Fig.1, two patterns of heat treatments were performed. In the pattern-A heat treatment, specimens were heated directly to the temperatures in the range from 400℃ to 900℃ and then quenched into oil after isothermal annealing. In the pattern-B, specimens were first heated to 900℃, at which A2 disorder phase forms. Subsequently, they were cooled to 500℃ at a rate of 10 ℃/min, and quenched into oil after isothermal annealing. All heat treatments were carried out under vacuum.

TABLE I
CHEMICAL COMPOSITION OF THE ALLOY (wt. %).

C	Si	Mn	P	S	Sol.Al	N	O	Fe
0.0015	6.50	0.040	0.004	0.0004	0.003	0.0014	0.0022	Bal.

Maximum permeability and core loss were measured following the heat treatments. To verify the reproducibility, at least five rings were used for each result. It was confirmed that errors in the results obtained in this work are less than 6%. The results to be represented in the following are the average values. Furthermore, the specimens after heat treatments were electro-polished for observing microstructures with TEM. It has been proved that 111 and 002 reflections are attributed to the formation of DO_3 and B2 order phases, respectively. In this study, TEM dark field images using 111 and 002 reflections were observed to study the morphologies of B2 and DO_3 phases.

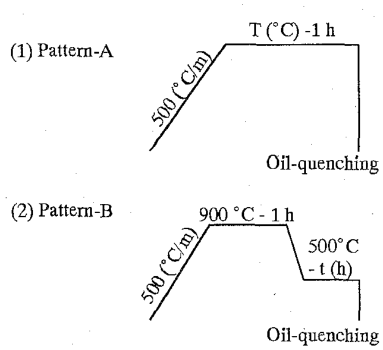

Fig.1 The two patterns of heat treatments.

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III. Results And Discussion

The magnetic properties of specimens after the normalization treatment are given in Table II. The corresponding microstructures were characterized by the formation of B2 and DO_3 order phases. The following heat treatments were performed with these specimens.

1) Pattern-A heat treatment

Fig.2 shows the dependence of maximum permeability on quenching temperature. Quenching from 500℃ results in a permeability of 6.27×10^4, which is the maximum value between 400℃ and 900℃. Fig.3 shows the dependence of core loss on quenching temperature. The variation of core loss with quenching temperature is similar to that of maximum permeability, quenching from 500℃ showing the lowest value. TEM dark field images using 111 and 002 reflections of the specimens quenched from 500℃ are shown in Photo 1. It is observed that the domain configuration of DO_3 phase is plate-shaped with the size of about 20 nm in length, existing in the matrix of B2 phase. The B2-type 1/4 <111> antiphase boundaries are also visible. The microstructures of the specimens quenched from 400℃ and 600℃ were similar to that of 500℃. It is considered that the large difference in magnetic properties is attributed to the different volume fractions of DO_3 and B2 phases changed by quenching temperatures. On the other hand, the 002 dark field image of specimens quenched from 800℃ is shown in Photo 2. The microstructure is observed to be B2 order phase, which has a granular appearance with plenty of B2-type 1/4 <111> antiphase boundaries. The similar microstructure has been found in the specimens quenched from 900℃, at which the formation of A2 disorder phase is expected. The formation of B2 order phase results from quick transformation of A2 to B2 phase during the quenching process. The results of the magnetic property and microstructure indicated that the magnetic property can be improved by the formation of DO_3 order phase, and also, is largely affected by the volume fraction of DO_3 and B2 phases. It is obtained that quenching from 500℃ in the pattern-A treatment gives the appropriate fraction for the desired magnetic properties. On the other hand, it is considered that the formation of DO_3 and B2 phases during the normalization treatment could affect the properties of the quenched specimens, the pattern-B treatment was performed in order to eliminate the effect.

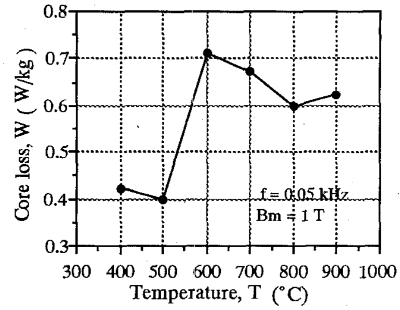


Fig.3 Dependence of core loss on quenching temperature.

2) Pattern-B heat treatment

Table III shows magnetic properties after the pattern-B heat treatment. Several results obtained from the pattern-A treatment are also listed in this table for a comparison. The mag-

TABLE II
Magnetic Properties of 6.5 % Si-Fe Alloy after Normalization Treatment.

$B_{0.8}$ (T)	μ_m ($\times10^4$)	Hc (A/m)	$W_{10/50}$ (W/kg)
1.10	3.56	6.94	0.51

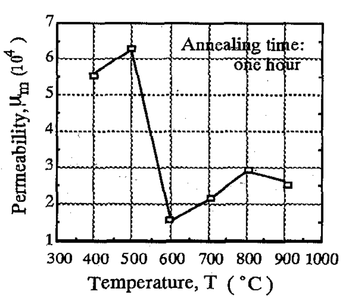


Fig.2 Dependence of permeability on annealing temperature.

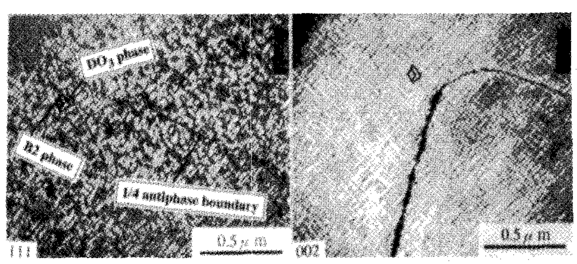


Photo 1 Microstructures of 6.5% Si-Fe alloy after pattern-A heat treatment at 500°C for one hour.

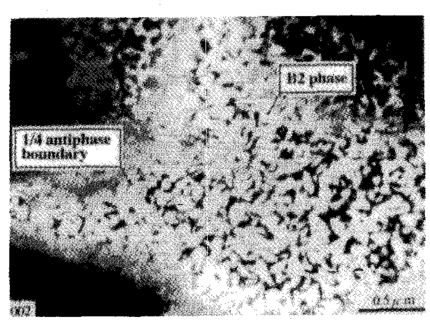


Photo 2 Microstructure of 6.5% Si-Fe alloy after pattern-A heat treatment at 800°C for one hour.

netic properties of the specimens after annealing for one hour in the pattern-B are superior to those of pattern-A and the maximum permeability reaches 7.25 x 10^4. However, annealing for two hours resulted in a drop in the magnetic properties. The magnetic properties after annealing for two hours are found to become about the same as those of pattern-A, as shown in Table III. In addition, the core losses of specimens after the two patterns of treatments and the normalization treatment are shown in Fig.4. Compared with the results of the pattern-A, it is found that the hysteresis loss decreases by a factor of 4, whereas the coercivity decreases by only 0.78 A/m in the pattern-B. Because hysteresis loss is determined by the area of BH-loop, which is affected by not only coercivity but also permeability, it indicates that the large decrease of hysteresis loss after annealing for one hour in the pattern-B is also contributed by the increase of permeability. On the other hand, it is evident that the decreases of core losses after the two patterns of heat treatments are mainly caused by the decrease of eddy current loss in the pattern-A and by the decrease of hysteresis loss in the pattern-B treatment, as shown in Fig.4. The microstructures after annealing for one and two hours in the pattern-B are shown in Photo 3. The microstructure of specimens annealed for one hour is characterized by the extremely fine domains of DO_3 phase existing in the B2 phase. The elongation of DO_3 domains seen in the pattern-A is alao observed due to the coarsening of DO_3 phase after annealing for two hours in the pattern-B. Since the DO_3 phase is formed through the spinodal decomposition of B2 phase at 500℃ [2], it is expected that the composition changes continuously across the B2 and DO_3 phases at the early stage of the decomposition reaction and begins to change discontinuously during the annealing process. It has been reported that magnetic contrast was observed around

TABLE III
MAGNETIC PROPERTIES OF 6.5 % SI-FE ALLOY AFTER PATTERN-A AND PATTERN-B HEAT TREATMENTS AT 500℃.

Properties	$B_{0.8}$ (T)	μ_m ($x10^4$)	Hc (A/m)	$W_{10/50}$ (W/kg)
pattern-A, 1 h	1.18	6.27	5.65	0.40
pattern-B, 1 h	1.20	7.25	4.87	0.39
pattern-B, 2 h	1.19	6.25	5.53	0.41

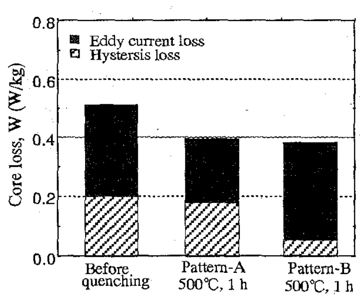
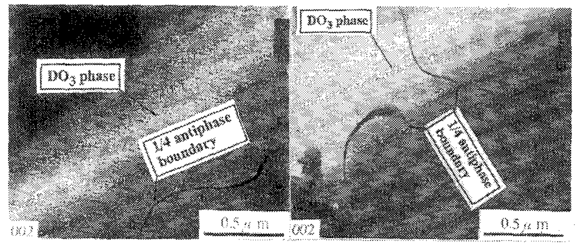


Fig.4 The effect of heat treatment condition on core losses.



one hour two hours
Photo 3 Microstructures of 6.5% Si-Fe alloy after pattern-B heat treatment at 500°C.

the boundary between B2 and DO_3 phases, and the appearance of the magnetic contrast is caused by the different magnetization of the B2 and DO_3 phases due to their different structures [4]. Therefore, magnetic contrast is expected to come to appear due to the coarsening of DO_3 domains. It is assumed that the magnetic contrast inhibits the displacement of magnetic domain walls when the specimen is magnetized, resulting in the decline of maximum permeability. The variation of other magnetic properties, such as coercivity and core loss, can be also explained by this assumption.

IV. CONCLUSIONS

Heat treatments and TEM observations were performed on the 6.5 % Si-Fe alloy. It has been found that the structure of the coexistence of B2 and DO_3 order phases could improve soft magnetic properties. The appropriate volume fraction of the two order phases for good magnetic properties was obtained by quenching from 500℃. Furthermore, the magnetic properties depend largely on the morphology of the DO_3 order phase. The maximum permeability of specimens is as high as 7.25 x 10^4 after being quenched from 500℃ in the pattern-B treatment. The corresponding microstructure is characterized by the extremely fine domains of DO_3 phase in the B2 phase. The coarsening of the DO_3 domains adversely affected the soft magnetic properties by decreasing permeability and increasing coercivity and core loss. It is assumed that magnetic contrast tends to inhibit the displacement of the magnetic domain walls, which comes to appear around the B2 and DO_3 phase boundary accompanied with the coarsening of DO_3 domains.

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