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IRON LOSS OF TERTIARY RECRYSTALLIZED SILICON STEEL (INVITED)

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ABSTRACT

Tertiary recrystallization was observed after cold-rolling and annealing conventional silicon steels. The magnetic induction B_8 at 800A/m of tertiary recrystallized sheets about 75μm thick is as high as 1.97T. Magnetic domain width in these sheets is wider than 1mm, hence the domain refining techniques such as mechanical scratching and chemical etching are employed in order to reduce the iron loss. The iron loss of the sheet mechanically scratched under the application of a tensile stress of $4kg/mm^2$ are as follows: W17/50=0.42W/kg and W13/50=0.19W/kg. However, this domain refining effect disappears after annealing for 30 minutes at 800°C. On the other hand, the domain refining by the chemical etching is effective for the iron loss of the sheets even if they are annealed, and the iron loss of the sheet 71μm thick under the application of a tensile stress of $4kg/mm^2$ is as follows: W17/50=0.35W/kg and W13/50=0.17W/kg. The iron loss of a chemically etched sheet with thickness of 31μm is as follows: W17/50=0.21W/kg and W13/50=0.13W/kg.

INTRODUCTION

One of the most important problems for silicon steels is the reduction of iron loss. In the past, the iron loss was reduced by increasing the perfection of the (110)[001] grain texture, so called the Goss texture[1][2]. At the present time, the misorientation angle between the rolling direction and the [001] crystal axis is less than three degrees. One important method for more reduction of the iron loss is to reduce the thickness of silicon steels. Many studies to obtain thin silicon steels with low iron loss were carried out[3][4][5]. However, it is very difficult to make thin steels having good grain texture by the conventional rolling and annealing methods. Another method to reduce the loss is magnetic domain refining by mechanical scratching or laser irradiation. However, the domain refining effect on the loss by these techniques completely disappeared after an annealing to release the stress stored during the process of manufacturing cores.

In a previous paper[6], we reported briefly about the grain texture and magnetic properties of tertiary recrystallized thin silicon steel sheets obtained by rolling and annealing conventional grain oriented silicon steels. In this paper, we report that the changes of the magnetic properties such as magnetic induction B_8 and coercive force Hc with the progress of recrystallization by annealing and also iron loss before and after applying domain refining techniques using thin silicon steel sheets thinner than 150μm obtained by cold-rolling.

EXPERIMENTAL PROCEDURE

Grain oriented silicon steels 300μm thick (Z-6H) were dipped into a mixed solution of hydrofluoric acid and sulfuric acid, and then into nitric acid to remove the surface insulator coating. Next, the steels were cold rolled into thin sheets to various thickness thinner than 150μm. The cold-rolled sheets were cut into pieces 100mm long and 5mm wide, followed by annealing in a vacuum of $1.3x10^{-3}$Pa using an infrared image furnace. In this annealing, the heating rate was kept at 3°C/sec, and the cooling rate was about 10°C/sec down to 600°C. The rolled and recrystallized textures of the sheets were observed by x-ray diffraction. The average grain diameter was measured by observing the sheet surface using an optical microscope. The static coercive force and the magnetic induction were measured by using a DC B-H loop tracer. The iron loss was measured by using a single sheet tester at 50Hz. The hysteresis loss at 50 Hz was obtained by multiplying the measured DC loss by 50. The eddy current loss at 50Hz was determined by subtracting the hysteresis loss from the iron loss. The static magnetic domain width was observed using a scanning electron microscope. In order to refine the domain width and to reduce the iron loss, a chemical etching technique as well as a mechanical scratching technique are employed in this experiment. In the mechanical scratching technique, we scratched the sheet surface using a weighted needle transverse to the rolling direction. By this treatment, the sheet gets local stress around the scratches. In the chemical etching technique, the sheet surface was coated by an acid proof resist, scratched transverse to the rolling direction to remove locally the resist with a needle, and then dipped into a nitric acid solution for several minutes. After rinsing in water, the resist was dissolved completely using acetone. After this treatment, heat proof grooves were formed on the sheet surface. Before the measurement of the loss, the chemically etched sheet was annealed to release the stored mechanical stress.

RESULTS AND DISCUSSION

Rolled texture

It is well known that the as-rolled grain texture has strong effects on the grain growth and recrystallized texture by annealing. Figure 1 shows x-ray diffraction intensities of as-rolled sheet surfaces. In the sheet 150μm thick, only the (110) diffraction is observed. When the sheet became thinner than 120um, both the (222) and the (110) diffractions are observed, and with decreasing sheet thickness the (110) diffraction decreased while the (222) diffraction increased. Successively, we observed x-ray diffraction intensities of the inside of each as-rolled sheet after etching chemically to remove the surface layer in a mixed solution of hydrofluoric and hydrogen peroxide until a half of the as-rolled thickness was removed. As a result, it is found that as the diffraction point approaches the center of the sheet thickness, the (110) diffraction becomes weaker, while the (222) diffraction becomes stronger. The higher the roll reduction rate is, the more pronounced this tendency becomes. These facts mean that the grain texture of the as-rolled sheets thinner than 120μm is changed by rolling from the (110) to the (111) near the center of the sheet thickness at the beginning; the area with the (111) texture spreads toward the sheet surfaces with increasing amounts of rolling reduction until finally the area reaches the sheet surface.

Primary and secondary recrystallization

Figure 2 shows the annealing temperature dependence of the magnetic induction B_8 at 800A/m of sheet 80μm thick. With an increase of the annealing temperature, the magnetic induction increases linearly at first because of recovery from stored

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stress and crystal defects, and then the primary recrystallization begins at about 550°C. This recrystallization is completed at 600°C, and the magnetic induction B_8 at this temperature is about 1.85T and has a tendency to decrease slightly until 1000°C. The main grain texture of the primary recrystallization is determined by an x-ray pole figure to be (110)[001].

Figure 3 shows the iron loss at 50Hz of the primary recrystallized sheet 80μm thick annealed at 700°C as a function of the magnetic induction. From this figure, it is found that this thin sheet has a high iron loss because the B_8 is not high enough; moreover, the coercive force is as large as 23A/m. For example, the iron loss W15/50 is 1.53W/kg, about twice the loss of conventional grain oriented silicon steels.

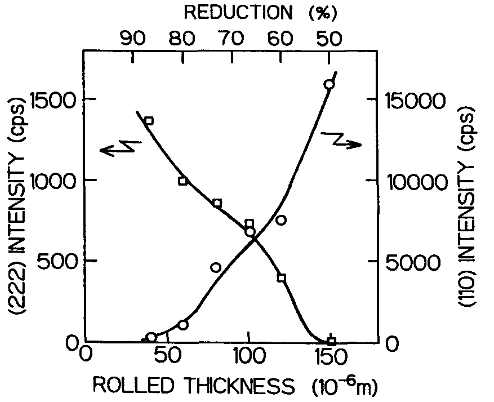


Fig.1 X-ray diffraction intensity as a function of rolled thickness: the circles are for (110) intensity and the squares are for (222) intensity.

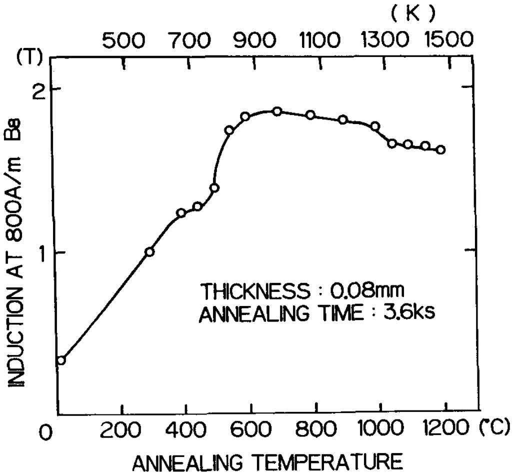


Fig.2 Magnetic induction B_8 at H=800A/m as a function of annealing temperature.

The magnetic induction B_8 dependence upon the sheet thickness is shown in Figure 4. The sheets thinner than 120μm have a constant B_8 after being annealed at 750°C; however, for the sheets thicker than 150μm, the B_8 decreased drastically, and the primary grain texture is weak (110)[001]. It seems that this fact has a close relation with the as-rolled grain texture, that is, the (111) rolled texture in the as-rolled state is necessary to obtain a high B_8 and to have (110)[001] grain texture in the primary recrystallization.

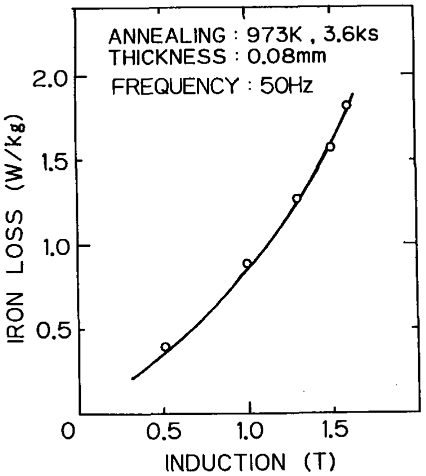


Fig.3 Iron loss of the primary recrystallized sheet as a function of the magnetic induction.

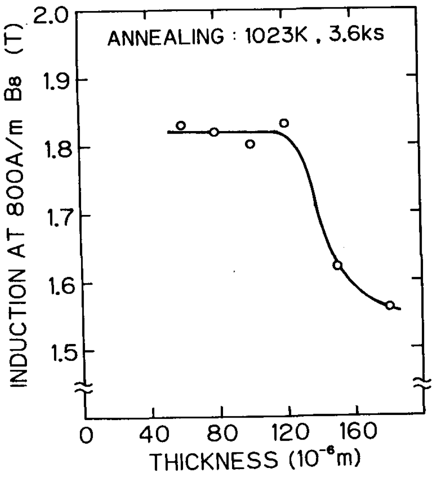


Fig.4 Magnetic induction B_8 dependence on the sheet thickness of the primary recrystallized sheets.

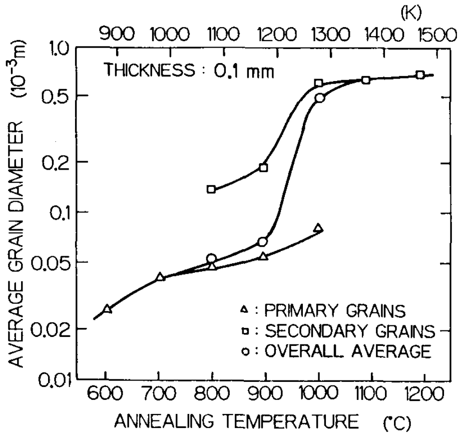


Fig.5 Average grain diameter as a function of the annealing temperature.

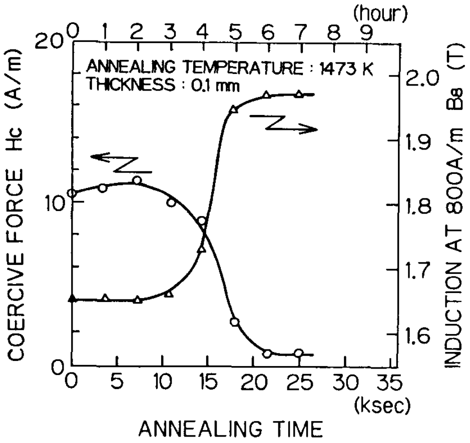


Fig.6 Magnetic induction B_8 and coercive force Hc at 800A/m as a function of the annealing time.

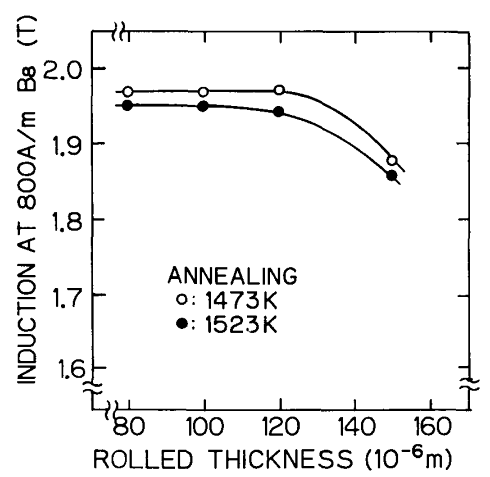


Fig.7 Magnetic induction B_8 dependence upon the rolled thickness of the tertiary recrystallized sheets.

The primary and secondary grain diameters were observed, and the results for sheets 100μm thick are shown in Figure 5. The higher the annealing temperature is, the larger the grain diameter becomes. At 800°C, secondary grains among the primary grains are found for the first time, grow rapidly with increasing annealing temperature and occupy the whole surface of the sheet about 1000°C. In this secondary recrystallized sheet, there are very few grains with the (110)[001] texture. Therfore with progress of secondary recrystallization, the B_8 becomes smaller as shown in Figure 2.

Tertiary recrystallization

In order to obtain a more excellent soft magnetic thin sheet having a higher B_8 and lower Hc than those of the primary or the secondary recrystallized sheets, we tried to realize the tertiary grain growth. In this experiment, the sheets were annealed for a long time above 1150°C. Figure 6 shows the dependences of the coercive force Hc and the B_8 upon the annealing time at 1200°C for the sheet 100μm thick. Until 3 hours, the magnetic properties remained almost unchanged, but drastic changes on both the Hc and the B_8 are observed after annealing for more than 3 hours. By this annealing, the (110)[001] grains grow and occupy the whole sheet surface. When the sheet is annealed for more than 6 hours, the B_8 and Hc reach constant values of 1.97T and 1.0A/m respectively. This value of B_8 corresponds to 97% of the saturation induction of 3wt% silicon steel.

The magnetic properties and the tertiary grain growth were observed for sheets with different thickness at various annealing temperatures. As a result, it was found that the annealing temperature must be higher than 1200°C to grow the tertiary grains. The maximum values of the B_8 for the sheets with various thickness annealed at temperatures of 1200°C and 1250°C are shown in Figure 7. From this figure, it is found that all sheets having the (110)[001] primary texture exhibit high values of B_8 after annealing. This fact is thought to be that some selected primary grains with the excellent (110)[001] grain orientation remain among the secondary grains with other orientations and grow as seeds of the tertiary recrystallization with increasing annealing temperature.

Figure 8 shows the iron loss, eddy current loss and hysteresis loss of the tertiary recrystallized sheet 75μm thick as a function of the magnetic induction. The DC magnetic properties of the sheet are as follows: magnetic induction B_8 is 1.98T, and the coercive force Hc is 2.8A/m. However, the iron loss W17/50 is 0.81W/kg, only 20% lower than that of the conventional grain oriented silicon steels. This is due to the effect of the large eddy current loss. At 1.7T, the eddy current loss comprises about 80% of the iron loss. In this sheet, the magnetic domain width observed is as wide as 1mm, and this wide width is the reason why the sheet has a large eddy current loss. In order to reduce the eddy current loss, it is necessary to apply the domain refining techniques.

One of the domain refining techniques employed in this experiment is to apply a tensile stress to the sheet. Figure 9 shows the iron and the hysteresis losses at 1.3T as a function of the external tensile stress. The eddy current loss decreased from 0.30 to 0.21W/kg by applying a tensile stress of 4kg/mm^2. In this experiment, the hysteresis loss also decreases by applying the tensile stress and consequently the iron loss changes from 0.38W/kg to 0.23W/kg.

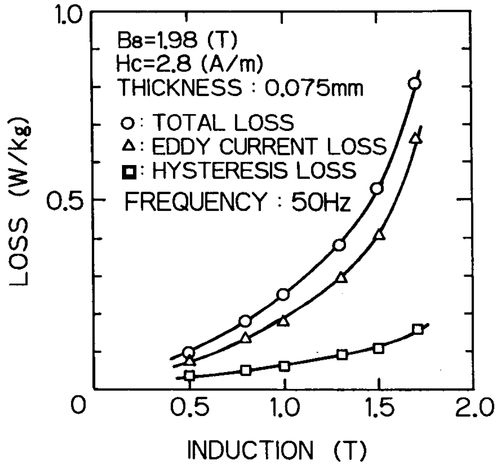


Fig.8 Iron loss of the tertiary recrystallized sheet.

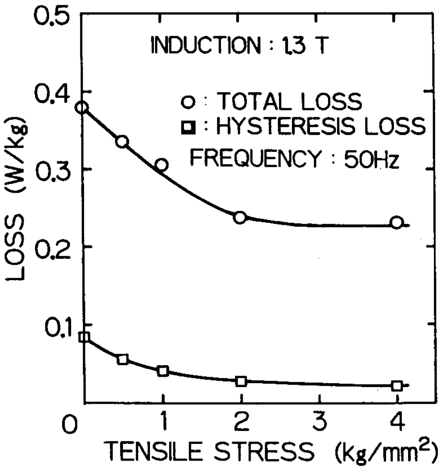


Fig.9 Iron loss dependence upon the tensile stress.

In order to reduce the eddy current loss further, the needle scratching is well known to be effective. Using the same sheets as those measured for loss shown in Figure 8 and 9, we scratched the sheet surface by a microcomputer controlled needle. Figure 10 shows the losses of this sheet under applying a tensile stress of 4kg/mm^2. In this scratching, a weight of 10g was applied on the needle to obtain a scratch width of about 15μm and a depth of about 0.1μm, and the spacing between each scratch was 1.25mm. This condition is the best for decreasing the iron loss. The losses W13/50 and W17/50 obtained in this experiment were 0.19 and 0.42W/kg, respectively. The former value is comparable with that of iron based amorphous materials, and the latter is less than half of that of the conventional grain oriented silicon steels.

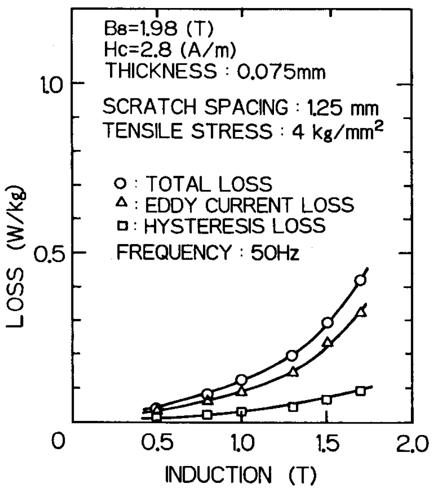


Fig.10 Iron loss of the needle scratched sheet.

Although we can get a thin silicon steel sheet with the low iron loss by the needle scratching method, this domain refining effect almost disappears after an annealing for a few minutes at temperatures around 800°C because the locally stored stress caused by the needle scratching is completely released, and as a result, the iron loss returns to the value obtained before scratching. This means that we cannot use this sheet as a toroidal core or a cut core because annealing is necessary to release the stress introduced during the process of winding and cutting.

Therefore, we tried to make heat proof grooves on the sheet surface by a chemical etching technique. In this method, the width and the depth of the grooves and the spacing between them can be changed independently, and every chemically etched sheet was annealed at 800°C for 30 minutes before measuring the magnetic properties in order to make clear only the effect of grooves. Figure 11 shows the iron loss, the hysteresis loss and the eddy current loss as a function of the spacing between the grooves. The eddy

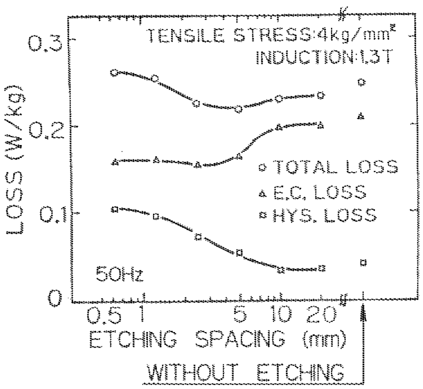
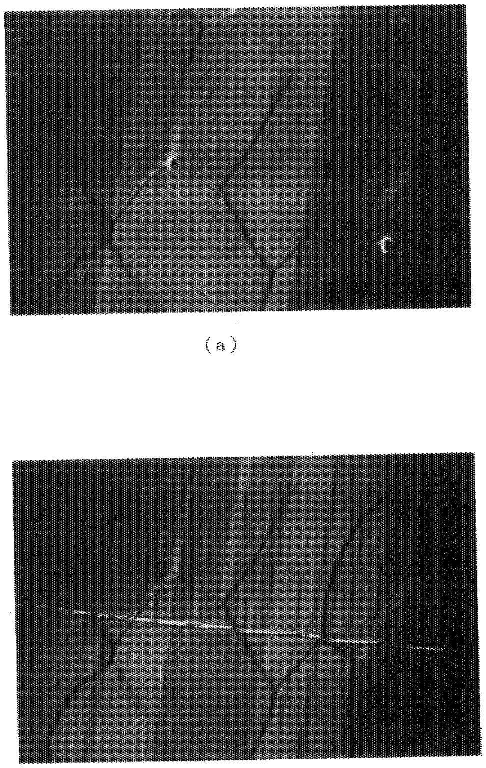


Fig.11 Iron loss dependence upon the spacing between chemically etched grooves: the sheet thickness=71um, B_8=1.94T and Hc=1.4A/m.



(a)

(b)

Photo.1 Magnetic domains before (a) and after (b) chemical etching.

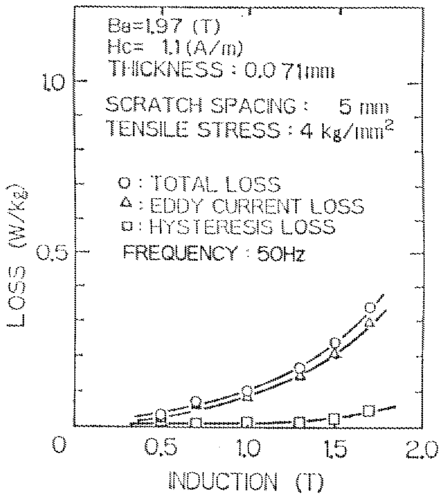


Fig.12 Iron loss of the chemically etched sheet.

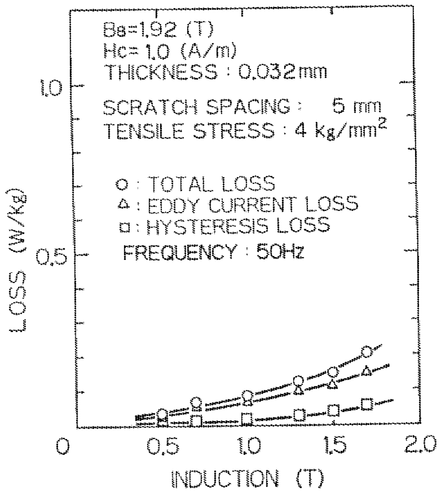


Fig.13 Iron loss of the chemically etched thin sheet.

current loss decreases while the hysteresis loss increased with decreasing spacing. From this figure, it is found that the iron loss had the minimum value at the spacing of 5mm. The increase of the hysteresis loss is due to the increase of the surface roughness caused by the grooves. Photo 1 shows the magnetic domains of the same area of the sheet surface with and without grooves. The curved lines like grain boundaries in this photo are traces of secondary recrystallized boundaries and their depth is less than 1um. From this photograph, it is found that the domains are divided into many subdomains by the chemical etching to lower the magnetostatic energy caused by magnetic free poles around the edge of the grooves[7].

Table 1 Magnetic properties of conventional grain oriented silicon steel, amorphous sheets and tertiary recrystallized sheets.

SAMPLE	GRAIN ORIENTED SILICON STEEL (WITH COATING)	Fe-BASE AMORPHOUS SHEET (FIELD ANNEALED)	TERTIARY RECRYSTALLIZED SILICON STEEL SHEET (DOMAIN REFINED) (MECHANICAL)	(CHEMICAL)	
THICKNESS(mm)	0.3	0.02~0.04	0.075	0.071	0.032
Bs (T)	2.03	1.5~1.6	2.03	2.03	2.03
W13/50 (W/kg)	0.6	0.15~0.30	0.19	0.17	0.13
W17/50 (W/kg)	1.02	--	0.42	0.35	0.21

Figure 12 shows the iron loss, eddy current loss and hysteresis loss of the sheet 71μm thick having chemically etched grooves with 40μm of width and 2.5μm of depth under an external tensile stress of $4kg/mm^2$. The iron loss W13/50 becomes an extremely low value of 0.17W/kg, almost the same as that of the iron based amorphous materials. Further, the loss 0.35W/kg at W17/50 is about 30% of that of conventional grain oriented silicon steels. These values of iron loss are completely reproducible.

Figure 13 shows the iron loss, eddy current loss and hysteresis loss of the sheet 32μm thick with the same grooves as those of the above 71μm thick and with the application of the tensile stress of $4kg/mm^2$. The iron losses W17/50 and W13/50 are surprisingly low values at 0.21 and 0.13W/kg, respectively.

Table 1 shows the magnetic properties of conventional grain oriented silicon steels, iron based amorphous sheets and tertiary recrystallized and domain refined silicon steel sheets developed in this study. In this table, the sheet with chemically etched grooves on its surface have the same iron loss even after it is annealed at 800°C for 30 minutes. Moreover, this sheet has far lower iron loss W13/50 and higher saturation magnetization than those of the amorphous materials having the same thickness, and the loss W17/50 is about one fifth of that of the conventional silicon steels. From these results, the tertiary recrystallized silicon steel in this study is expected to become one of the most excellent soft magnetic materials with low iron loss as well as high magnetic induction.

CONCLUSION

Conventional grain oriented silicon steels were cold rolled and annealed in a vacuum. The as-rolled and annealed grain textures of these sheets were observed, and the iron losses of the tertiary recrystallized and domain refined silicon steels were also measured. The results are summarized as follows.

(1) By cold-rolling the grain oriented silicon steel, the grain texture changes from the (110) to the (111) with an increase in the amount of cold reduction, and after primary recrystallization, the induction B_8 becomes high for sheets having the (111) as-rolled texture.

(2) By annealing above 1200°C for more than 6 hours, the tertiary recrystallization is completed, and the sheet 75μm thick having the (111) rolled texture has a high magnetic induction B_8 of 1.97T.

(3) When the surface of the tertiary recrystallized sheet 75μm thick is scratched by a needle, and the iron loss under application of the tensile stress of $4kg/mm^2$ is measured, the loss becomes as follows: W17/50=0.42W/kg and W13/50=0.19W/kg.

(4) By chemical etching, heat proof grooves are formed on the sheet surface. Under the application of the tensile stress of $4kg/mm^2$, the iron loss of the sheet 71μm thick is as follows: W17/50=0.35W/kg and W13/50=0.17W/kg.

(5) For the sheet 32μm thick with the chemically etched grooves, the iron loss under application of a tensile stress of $4kg/mm^2$, is as follows: W17/50=0.21W/kg and W13/50=0.13W/kg.

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