Recrystallization and magnetic properties of purified 3% silicon steels

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Recrystallization and magnetic properties of purified 3% silicon steels

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The effect of impurities on the tertiary recrystallization and magnetic properties of the thin (10 to 100 μm) silicon steels was investigated. For purification, the conventional grain oriented silicon steels, which were used as starting materials with a thickness of 0.30 mm were pre-annealed at 1200 °C in a vacuum of 1×10^{-3} Pa before cold rolling. In sheets without pre-annealing, the tertiary recrystallization was observed after annealing for at least 3 h over 1200 °C in a vacuum. On the other hand, in pre-annealed sheets, the tertiary recrystallization was completed within 10 min at an annealing temperature of 1150 °C in a vacuum. Through recrystallization, B_8 increased to 1.95 T. Even in sheets annealed at 1050 °C, the B_8 increased with increasing the annealing time, and became 1.95 T after anneals of 2 h. The pre-annealed sheets were also annealed in hydrogen atmosphere. In this case, at annealing temperatures over 1100 °C, the tertiary recrystallization was completed in less than 1 h. Using the purified sheets, silicon steels with B_8 over 1.9 T can be obtained by short time and low temperature annealing in either a vacuum or a hydrogen atmosphere.

I. INTRODUCTION

Tertiary recrystallization occurred after cold rolling and annealing conventional grain oriented silicon steels.[1] After the recrystallization grains with sharp (110)[001] texture occupied the whole surface of the silicon steel sheet. Through such treatment, we can obtain grain oriented silicon steel sheets having large magnetic induction and very low core loss.[2–4]

However, tertiary recrystallization needs long annealing times of several hours above 1150 °C in a vacuum. It is difficult to apply this process to a commercial production. It is therefore very important to determine which factors dominate the tertiary grain growth. The conventional grain oriented silicon steels are well known to contain a large amount of impurities for obtaining a sharp (110)[001] grain texture. These impurities are thought to have an influence on the surface energy[5] controlling grain growth.

In this paper, we report the tertiary recrystallization and the magnetic properties of the purified silicon steel.

II. EXPERIMENT

The conventional grain oriented silicon steels which were used as starting materials with thickness of 0.30 mm were pre-annealed at 1200 °C in a vacuum of 1×10^{-3} Pa, for more than 10 h. With this treatment, the amounts of Sn and Cu were decreased to less than 10 ppm, but the magnetic properties were not changed. After the pre-annealing the samples were cold rolled to less than 100 μm. The annealing for recrystallization was carried out using an infrared image furnace in a vacuum of 1.3×10^{-3} Pa or in a hydrogen atmosphere. 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 with a dc B–H loop tracer.

III. RESULTS

Figure 1 shows the dependence of the magnetic induction at 800 A/m (B_8) and the coercive force (H_c) on annealing time of the sample cold rolled to 60 μm without pre-annealing. Through the tertiary recrystallization, the B_8 increased and the H_c decreased. In this experiment, complete tertiary recrystallization occurred after annealing of 3 h at 1200 °C. When the annealing temperature was decreased to 1150 °C, tertiary recrystallization was not completed although the sheets were annealed for more than 10 h.

Figure 2 shows the relationship between the annealing temperature and the B_8 of the sample with and without pre-annealing. The annealing was carried out in a vacuum. In the sample without pre-annealing, B_8 decreased with increasing the annealing temperature and no tertiary recrystallization was observed. In the samples with pre-annealing, the B_8 increased rapidly at annealing temperatures over 1000 °C, and reached 1.95 T at 1100 °C after completion of the tertiary grain growth.

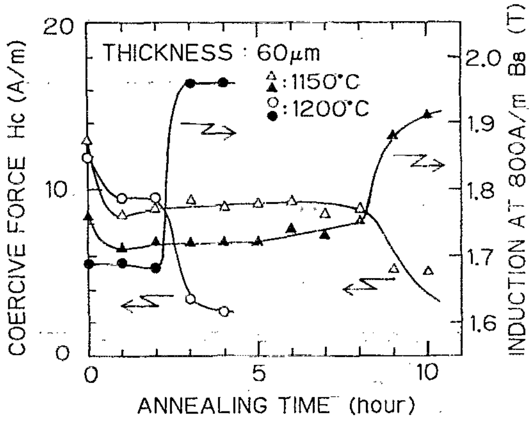

FIG. 1. Annealing time dependence of B_8 and H_c.

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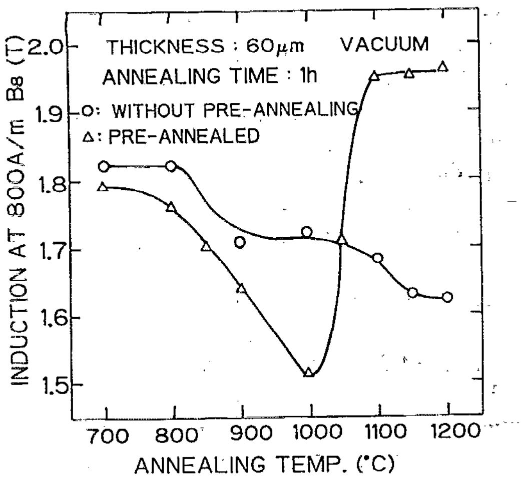

FIG. 2. Induction B_8 over annealing temperature for samples with and without pre-annealing.

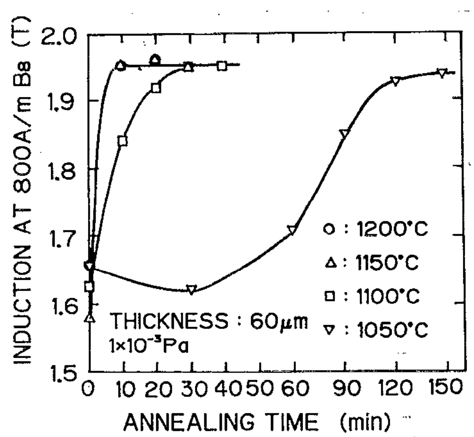

FIG. 4. The B_8 over annealing time.

The development of grain growth can be described by the grain size. Figure 3 shows the grain size of the pre-annealed sample. At the annealing temperature of 700 °C, the sample had only primary recrystallized grains with the size of about 30 μm. At 800 °C, the secondary grains appeared and began to grow. At 1050 °C, the tertiary recrystallized grains having (110)[001] texture appeared and the tertiary recrystallization was completed at 1100 °C. The size of the tertiary recrystallized grain was 1–3 mm. From this figure one can declare that the increasing B_8 shown in Fig. 2 occurs through tertiary recrystallization. In the pre-annealed sample tertiary recrystallization was begun and completed at lower temperature and shorter time as in the samples without pre-annealing.

The influence of annealing time in vacuum upon B_8 in pre-annealed samples is shown in Fig. 4. The tertiary recrystallization was completed within 10 min at annealing temperature over 1150 °C. At 1100 °C the samples needed 30 min to be completely tertiary recrystallized. Even at 1050 °C the B_8 reached around at 1.95 T when annealed over 120 min. This means that thin grain oriented silicon steels can be produced at lower temperature and shorter annealing time by using the samples having less impurities.

Figure 5 shows how the completion of tertiary recrystallization depends on the pre-annealing time. The recrystallization behavior was observed by annealing in a hydrogen atmosphere. The longer the pre-annealing time was, the larger the area of the tertiary recrystallized grains became. Therefore it is clear that the tertiary recrystallization occurs even by annealing in a hydrogen atmosphere if the sample contains few impurities.

The relationship between the area of tertiary recrystallized grains and the content of Cu and Sn in the samples is shown in Fig. 6. The annealing temperature was 1100 °C and the annealing time was 1 h. With this annealing con-

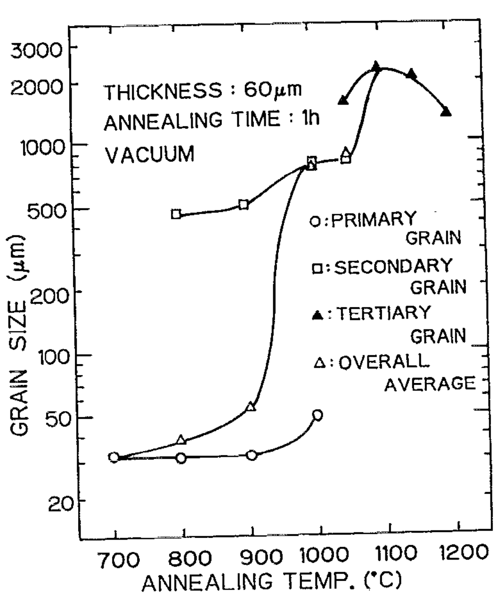

FIG. 3. Grain size over annealing temperature.

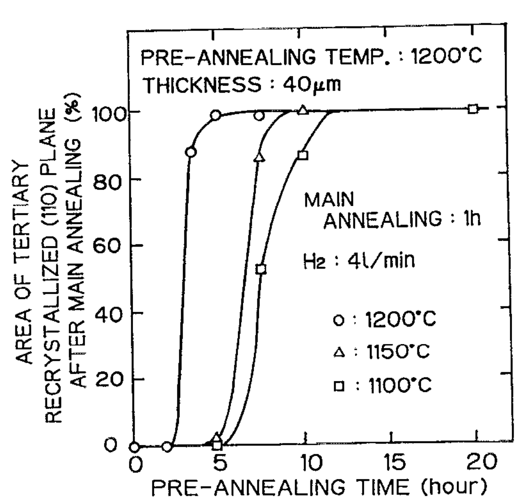

FIG. 5. Tertiary recrystallized area over pre-annealing time.

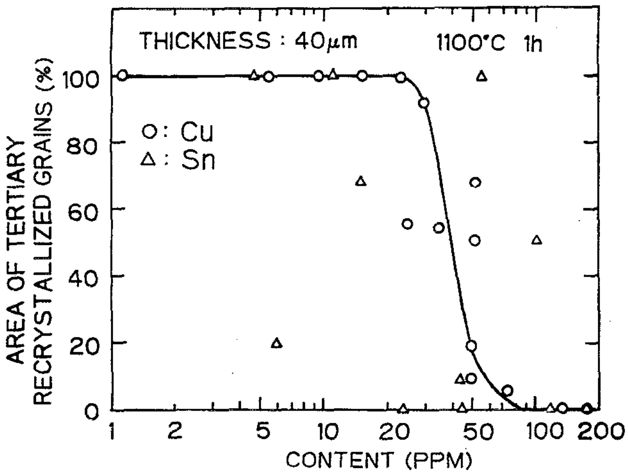

FIG. 6. Tertiary recrystallized area over impurity content.

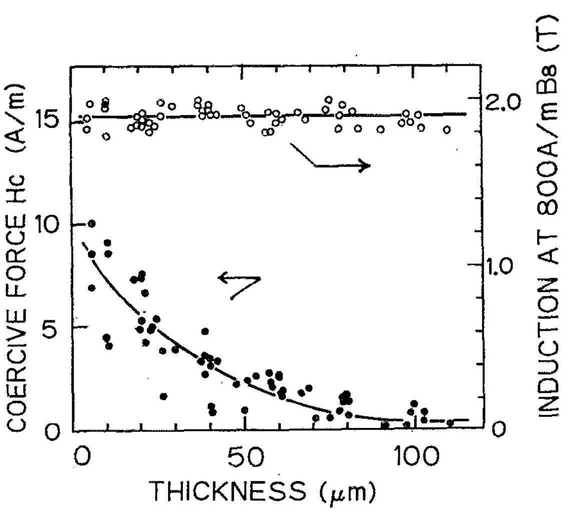

FIG. 7. B_8 and H_c over sample thickness.

dition, all of the samples with a Cu content of less than 20 ppm recrystallized to complete tertiary grains. In the samples with more than 100 ppm of Cu, no growth of the tertiary grains was observed with this anneal. The Sn content had no significant influence on the area of tertiary recrystallization. Our experiments show that Cu is the dominant element for inhibiting the tertiary grain growth.

Figure 7 shows the dependence of the B_8 and the H_c on the thickness of the tertiary recrystallized samples. Tertiary recrystallization occurred in samples from 5 to 120 μm thickness. All of the samples had high induction $B_8 > 1.9$ T. The values of H_c were very low, 1 or 2 A/m in samples with thickness of 100 μm. It increased with decreasing thickness of the samples. The reason of the increase of the H_c is considered to be the surface roughness formed by the cold rolling and annealing.

IV. CONCLUSION

By using purified grain oriented silicon steels as starting materials, thin grain oriented silicon steels can be produced by tertiary recrystallization in a vacuum at lower temperature and shorter annealing times than in samples containing higher impurities. Annealing in hydrogen atmospheres also caused tertiary recrystallization. The obtained samples have excellent soft magnetic properties. The B_8 is over 1.9 T and the H_c is 1 to 5 A/m depending on the thickness of the samples.

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