Production of Primary Recrystallized Si-Fe Foil with Orientation near (110)[001] and Magnetic Properties

N. Abe, M. Iwasaki, K. Kosuge, Y. Ushigami, and T. Nozawa

The magnetic properties and features of magnetic domain structures of recently developed grain-oriented 15 to 50 μm thick Si-Fe foil with very high permeability were investigated. The reported induction values of these materials were 1.70 to 1.95 T at 800 A/m. These high values produced low core losses. The losses of toroidal cores made of 50-μm thick material was $W_{15/400}$ = 8.0 W/kg and $W_{18/400}$ = 12.5 W/kg. Despite the presence of a very small primary recrystallized grain size, they consisted of simple magnetic domains with 180° domain walls; ac magnetization was achieved by simple 180° domain wall displacement. The cut core, made of 20-μm thick material, was $W_{10/10k}$ = 330 W/kg and $W_{15/10k}$ = 580 W/kg, which compares to those of amorphous cut cores.

Keywords
core loss, cut core, foil, magnetic materials, Si-Fe foil

1. Introduction

LINE frequency loss has been improved primarily by improving crystal orientation and decreasing anomalous eddy current loss by increasing the number of effective 180° domain walls. On the other hand, these thick materials are not suitable for higher frequency magnetic applications because of higher eddy current density. Thin (110)[001] grain-oriented Si-Fe foil is required for high-frequency electrical apparatus. Particularly low core losses and high permeabilities at higher design flux densities and higher excitation frequencies greater than 400 Hz are necessary.

In 1949, Littmann studied noninhibitor processes for developing Si-Fe foil.[1] During his studies, when a material initially oriented in the (110)[001] fashion (permeability of 1740 at 800 A/m), having a satisfactory large grain size (grain diameter of from 0.05 to 10 mm), was subsequently cold rolled and recrystallized, permeabilities at 800 A/m were 1600 to 1700 at thicknesses of 25 to 125 μm, and core losses were high particularly at high excitation levels.

The purpose of this article is to present the newly developed Si-Fe foil with low core loss at high design inductions for high-frequency applications,[2] as well as their magnetic properties and characteristics of domain wall behavior. Additionally, an attempt has been made to manufacture the cut core and compare the core loss with that of amorphous cut cores.

N. Abe, M. Iwasaki, and **K. Kosuge,** Hirohata R&D Lab., Nippon Steel Corp., Himeji, Hyogo, Japan; and **Y. Ushigami** and **T. Nozawa,** Steel Research Labs., Nippon Steel Corp., Futtu, Chiba, Japan.

2. Experimental

2.1 *Production of Very Highly (110)[001] Grain-Oriented Si-Fe*

Many studies of cold rolling and primary recrystallization[2,3] indicated that, if starting materials have a sharp (110)[001] orientation, it is possible to produce sharp (110)[001] oriented Si-Fe foil. The grain boundaries of the starting material affect the cold rolled recrystallization texture. Grains with {110}⟨001⟩ type orientation nucleate at starting material grains and grow. On the other hand, grains with (111)[011] type orientation occur in the vicinity of old grain boundaries of the starting material.

Figure 1 shows the etch pits of Si-Fe foil. The (100) grains appear in grains and the (111) grains appear near the grain boundaries of the starting materials. According to these experimental facts, selection of starting materials with both ideal (110)[001] oriented grains and less grain boundary area is significant.

This unique starting material, in the form of a 5-ton coil, was developed by temperature-gradient secondary recrystallization annealing using a special furnace.[4] Figure 2 shows a portion of the macrostructure of this strip, and Fig. 3 shows the dc hys-

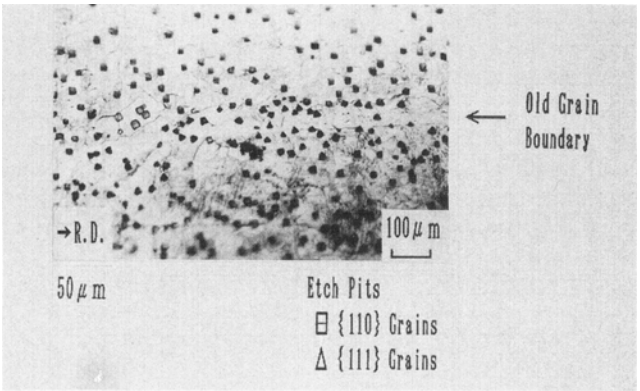

Fig. 1 Etch pits in Si-Fe foil.

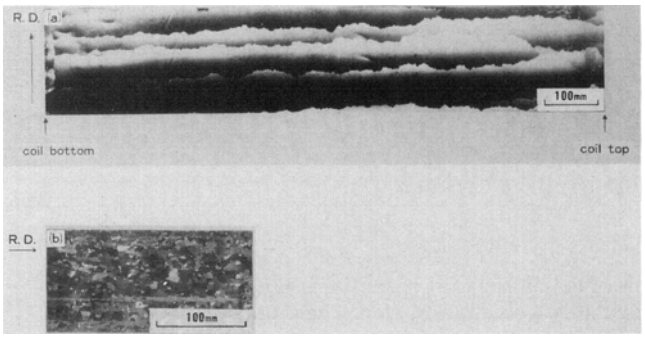

Fig. 2 Macrostructure of grain-oriented 3Si-Fe strip. (a) Starting material of large grain diameter (40 × 1000 mm) and high B_8 (2.00 T). (b) Starting material of small grain diameter (3 × 5 mm) and low B_8 (1.85 T).

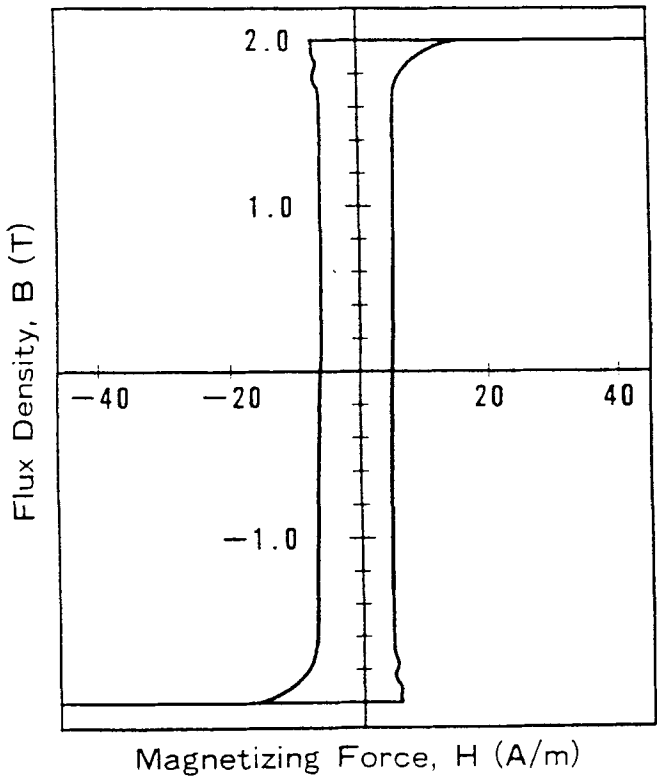

Fig. 3 dc hysteresis loop of the starting material with large grain diameter (40 × 1000 mm) and high B_8 (2.00 T).

teresis loop of this material. The maximum permeability is about 250,000. The perfect squareness ratio, Br/Bs = 1.0, and reentrant shape hysteresis loop correspond to the ideal (110)[001] orientation and a very simple domain structure of 180° domain walls.

2.2 *Production of Si-Fe Foil with Orientation near (110)[001]*

The starting materials (B_8 = 1.98 T, grain size = 40 × 1000 mm, thickness = 150 to 200 μm) were cold rolled using a

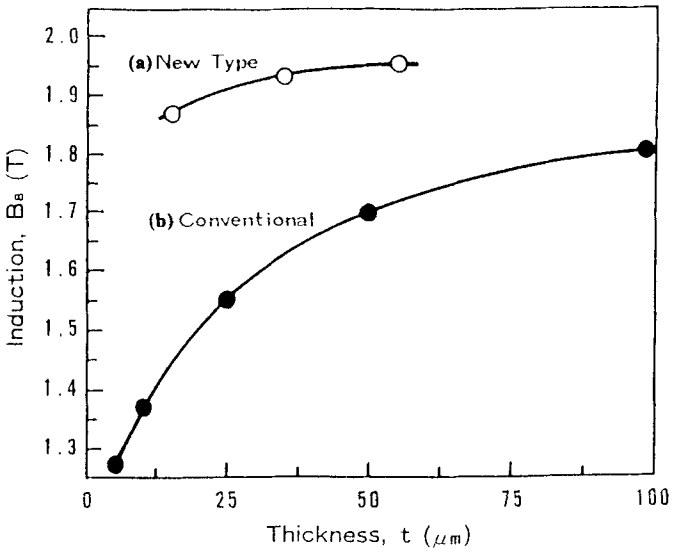

Fig. 4 Relationship between induction B_8 of primary recrystallized 3Si-Fe foil and sheet thickness. (a) Starting material of large grain diameter (40 × 1000 mm) and high B_8 (2.00 T). (b) Starting material of small grain diameter (3 × 5 mm) and low B_8 (1.85 T).

sendzimir mill with 18 back-up rolls and a 10.5-mm diameter work roll to a final thickness of 15 to 50 μm. Specimens were then annealed at 800 to 1000 °C for 30 to 600 s in a hydrogen atmosphere, followed by annealing in a nitrogen atmosphere to form insulating films.

Tape-wound toroids of Si-Fe foil were stress relief annealed. Some of these toroids were laser irradiated after stress relief annealing.

The domain patterns were observed using high-voltage scanning electron microscopy (SEM). Dynamic domain wall behavior was observed using a newly developed SEM line-sampling stroboscopic method.[5]

3. Results and Discussion

Figure 4 shows the relationship between induction, B_8, of primary recrystallized foil and foil thickness. The B_8 values of the foil strongly depend on two variables—the B_8 value and grain size of the starting material.

Figure 5 shows grain structures for foils and crystal orientations of each grain determined by the electron channeling pattern method. This figure shows that the crystal orientations of primary recrystallized foil made from highly grain-oriented large grain starting material are near (110)[001].

Figure 6 shows the relationship between core loss at f = 400 Hz and the maximum exciting flux densities. The core losses are $W_{15/400}$ = 9 W/kg and $W_{18/400}$ = 22 W/kg. After laser irradiation, the losses decrease to 8 W/kg and 14 W/kg, respectively. These values are about 40% lower than those of conventional materials. These very low losses at higher inductions are a distinct feature of highly oriented (110)[001] Si-Fe foil. The decrease in core loss caused by use of laser irradiation

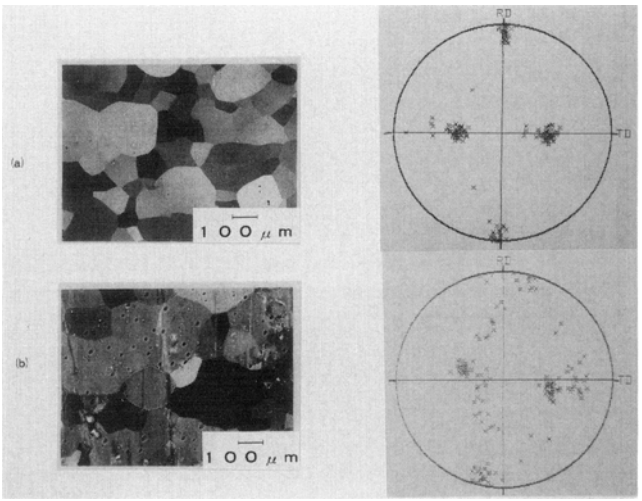

Fig. 5 Grain structures for Si-Fe foil and crystal orientations of each grain determined by the electron channeling pattern method. (a) Grain structures. (b) Crystal orientations of each grain.

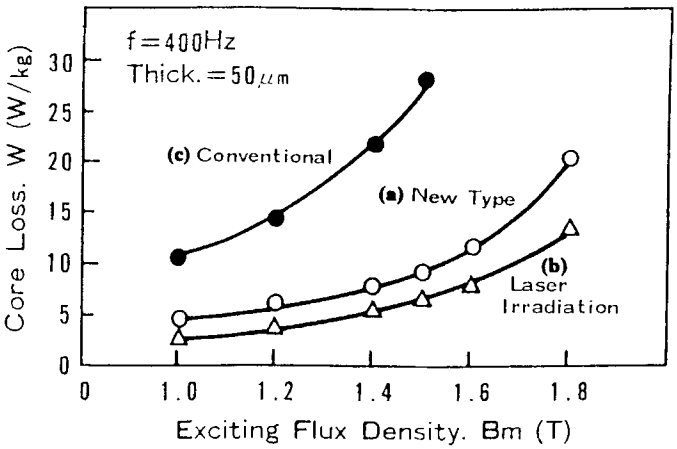

Fig. 6 Relationship between core loss of Si-Fe foil toroidal core at $f = 400$ Hz and maximum exciting flux density. (a) Starting material of large grain diameter (40×1000 mm) and high B_8 (2.00 T). B_8 of Si-Fe foil is 1.95 T. (b) After laser irradiation of (a). (c) Starting material of small grain diameter (3×5 mm) and low B_8 (1.85 T). B_8 of Si-Fe foil is 1.60 T.

is due to an increase in the number of effective domain walls (see Fig. 9).

Figure 7 shows the hysteresis loop at 400 Hz magnetization. The loop shape change (resembling a snake-like pattern) corresponds to an increase in the number of domain walls that contribute to reverse magnetization (see Fig. 9).

Figure 8 illustrates the dependence of decreasing core loss on B_8 as a function of isotropic tensile stress (coating) and local stress (laser irradiation) in material of various thicknesses. This figure shows that the losses at 400 Hz do not depend on thickness, but primarily on B_8 value. The decrease in loss in higher B_8 materials is due primarily to the domain wall spacing refining effect (see Fig. 9 and 10).

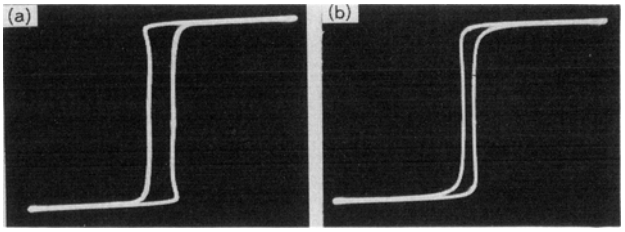

Fig. 7 Hysteresis loop at $f = 400$ Hz magnetization of Si-Fe foil. (a) Before laser irradiation. (b) After laser irradiation.

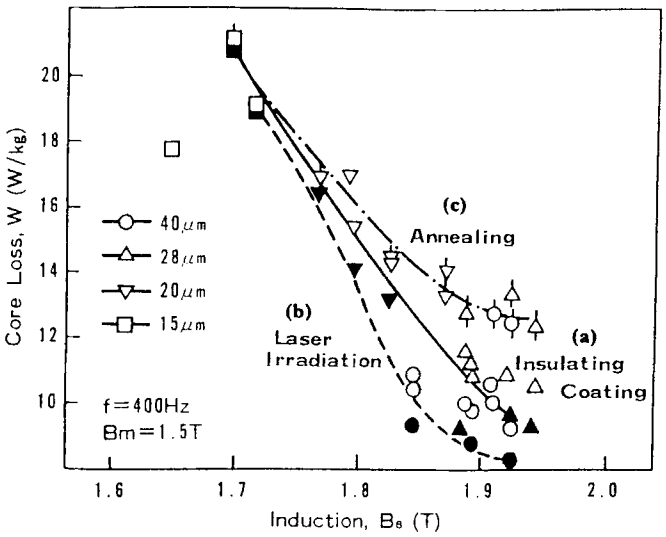

Fig. 8 Induction B_8 dependence of loss decreasing effect due to isotropic tensile stress (coating) and local stress (laser irradiation). (a) Before laser irradiation with coating. (b) After laser irradiation with coating. (c) Before laser irradiation with no coating.

On the other hand, the ineffectiveness in lower B_8 material can be attributed to a local stress and a magnetostatic defect induced at grain boundaries based on the large variation of crystal orientation from grain to grain.

Figures 9 and 10 illustrate the domain patterns of Si-Fe foil. Despite the small grains (see Fig. 4), they consist of large slab-like domains. Using laser irradiation decreases the main domain wall spacings to about one tenth. The striped patterns near the laser spots are supplemental domains with transverse magnetization. There are many fine spots in the main domains. They are supplemental domains caused by demagnetizing fields near grain boundaries and/or tilting of the [001] component out of the sheet plane. In poorly grain-oriented material, the magnetic domain images generally are not clear. Narrow, snaky domains, like the growth rings of a tree, are observed. The main domain wall spacings of Si-Fe foil are refined, largely due to isotropic tensile stress due to surface coating (see Fig. 10).

Figure 11 shows the dynamic domain patterns corresponding to each phase during ac magnetization. These patterns illustrate that the magnetization of highly grain-oriented Si-Fe foil is achieved mainly by 180° domain wall displacement. On the

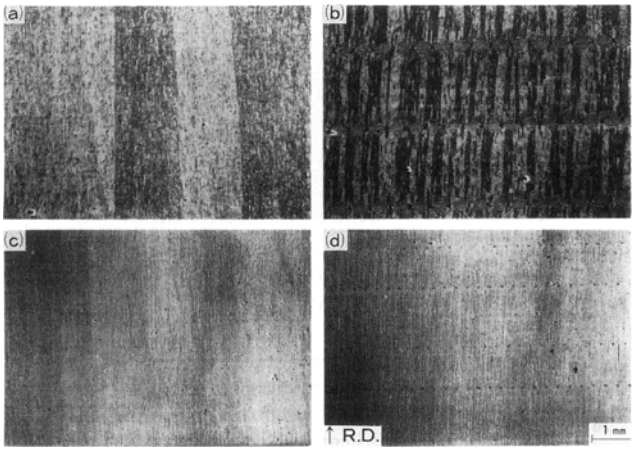

Fig. 9 Demagnetized domain patterns of Si-Fe foil. (a) Starting material of large grain diameter (40 × 1000 mm) and high B_8 (2.00 T). B_8 of Si-Fe foil is 1.95 T. (b) After laser irradiation of (a). (c) Starting material of small grain diameter (3 × 5 mm) and low B_8 (1.85 T). B_8 of Si-Fe foil is 1.60 T. (d) After laser irradiation of (c).

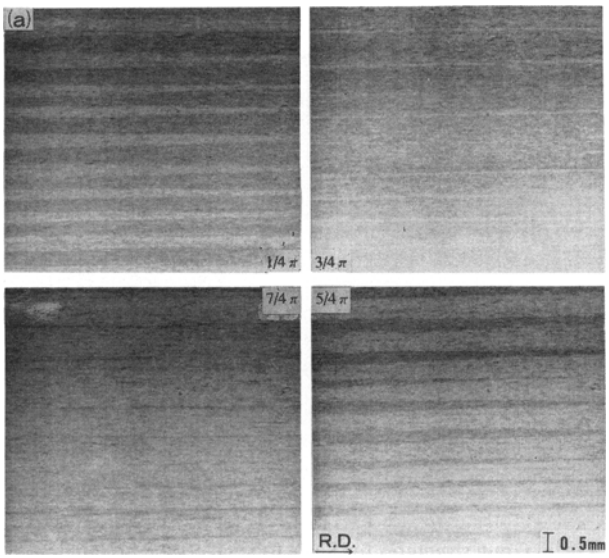

Fig. 10 Demagnetized domain patterns of Si-Fe foil.(a) Starting material of large grain diameter (40 × 1000 mm) and high B_8 (2.00 T). B_8 of Si-Fe foil is 1.95 T. (b) After coating of (a). (c) Starting material of small grain diameter (3 × 5 mm) and low B_8 (1.85 T). B_8 of Si-Fe foil is 1.60 T. (d) After coating of (c).

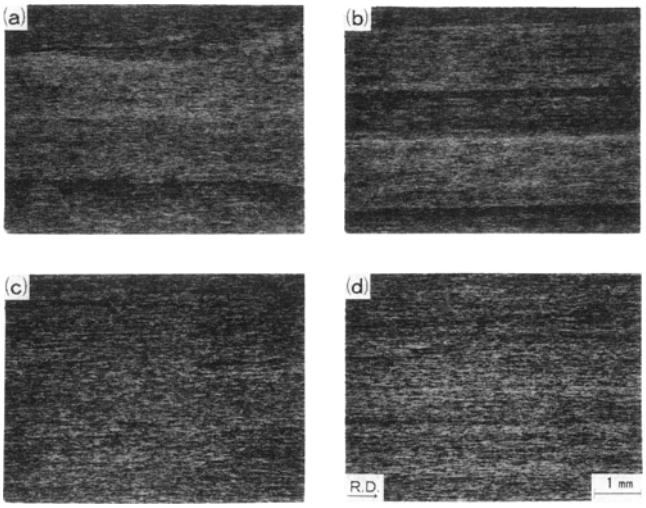

Fig. 11(a) Dynamic domain patterns corresponding to each phase during ac magnetization in higher B_8 material before laser irradiation with coating. Starting material of large grain diameter (40 × 1000 mm) and high B_8 (2.00 T). B_8 of Si-Fe foil is 1.95 T.

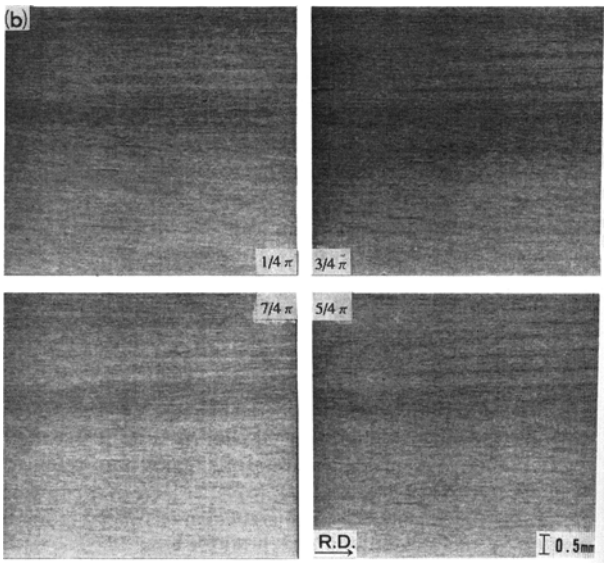

Fig. 11(b) Dynamic domain patterns corresponding to each phase during ac magnetization in higher B_8 material before laser irradiation with coating. Starting material of small grain diameter (3 × 5 mm) and low B_8 (1.85 T). B_8 of Si-Fe foil is 1.60 T.

other hand, the wall behavior in lower B_8 material is not uniform. These figures suggest that domain wall velocities change from place to place. Furthermore, low magnetic domain image contrast implies that the magnetization is achieved by low reproducible main wall behavior and/or rearrangement of supplementary domains during dc magnetization.

Figure 12 shows the relationship between core loss per frequency and the frequency at various thicknesses. The linear core loss increase up to about 5 kHz suggests that magnetization is achieved by simple main domain wall displacement similar to dynamic domain wall behavior at 50 Hz magnetization. The rapid increase in core loss at more than 5 kHz reflects the changes in the magnetization processes. At 10,000 Hz, the low core loss does not only depend on B_8 values, but also on sheet thickness, as can be seen by comparing to Si-Fe foils with thickness of 40 and 28 μm, which have the same B_8 values. The lowest core loss at 10,000 Hz is 480 W/kg using laser-irradiated Si-Fe foil with a thickness of 28 μm. Therefore, to achieve low core loss, high B_8 values and many domain walls are needed.

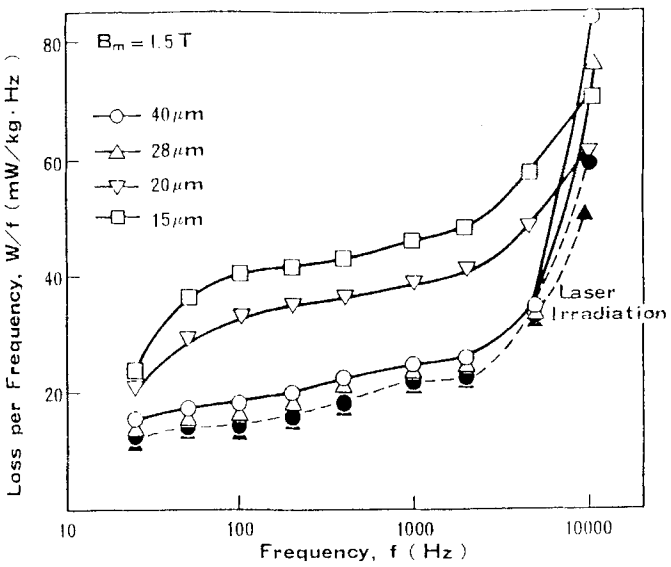

Fig. 12 Relationship between core loss per frequency and frequency.

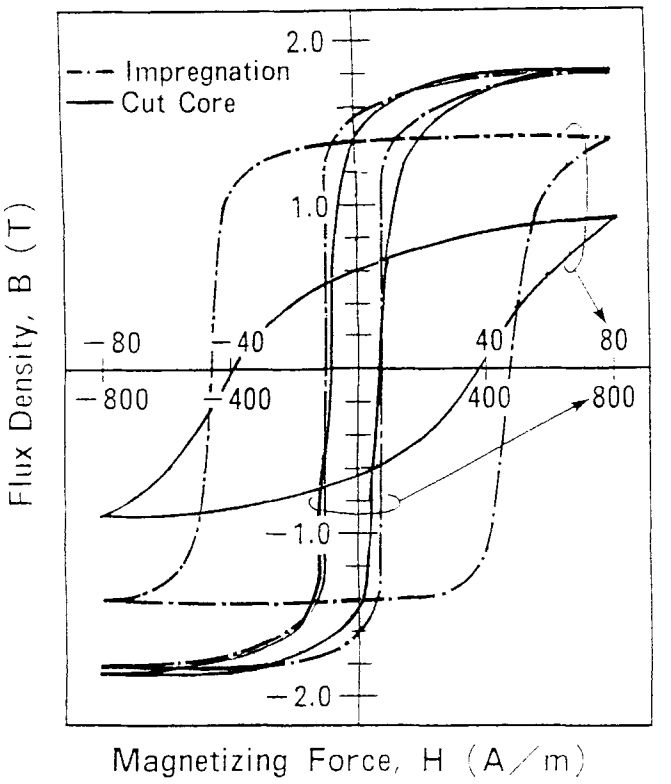

Fig. 13 dc hysteresis loop of the impregnation core (non-cut core) and the cut core.

Figure 13 shows the dc hysteresis loop of the impregnation core and cut core. In spite of impregnation, the shape of the dc hysteresis loop does not change. However, after cutting, the shape became inclined, and coercive forces decreased slightly, most likely because stress introduced by impregnation was relieved by cutting.

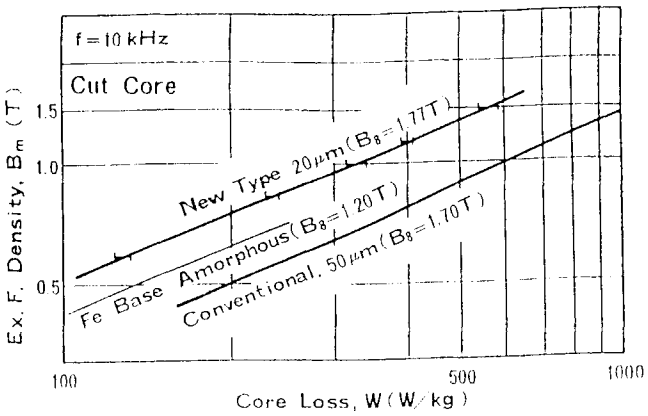

Fig. 14 Relationship between core loss of Si-Fe cut core at $f = 10$ kHz and maximum exciting flux density.

Figure 14 shows the relationship between core loss at 10 kHz and exciting flux density. The new type of cut core can be used at densities above 1.0 T, and the core loss compares with those of Fe-Si-B amorphous cut cores. The core loss at 1.0 T was 330 W/kg and at 1.5 T it was 580 W/kg.

4. Conclusions

Grain-oriented Si-Fe foil with orientation near (110)[001] has been developed recently. By refining of main domain wall spacing due to isotropic tensile stress and/or periodically induced local stresses, core losses decreased significantly. Core losses of these materials at middle frequencies were low, particularly at higher inductions. Although very small in grain size, they are composed of simple domains, and ac magnetization up to about 5 kHz is achieved by simple 180° main domain wall displacement.

The core losses of cut cores compare with those of Fe-Si-B amorphous cut cores at 10 kHz. The present results were 330 W/kg at 1.0 T and 580 W/kg at 1.5 T. These materials are useful for transformers and/or inductors for middle frequencies and higher design induction applications.

Acknowledgment

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References

1. M.F. Littmann, US patent No. 2,473,156
2. N. Abe, Y. Ushigami, M. Iwasaki, and T. Nozawa, 14th Ann. Conf. on Magnetics in Japan, The Magnetics Society of Japan, Matsuyama C., Japan, 1990, p 402
3. T. Taoka, *Tetsu Hagane*, Vol 54 (No. 2), 1968, p 22
4. T. Nozawa, T. Nakayama, Y. Ushigami, and T. Yamamoto, *J. Magn. Magn. Mater.*, Vol 58, 1986, p 67-77
5. H. Mogi, M. Yabumoto, Y. Matsuo, and K. Koyanagi, to be published, 1993