Structure at Near-Surface of TiN-Coated Grain Oriented Silicon Steel Sheet Characterized by Ultra-Low Iron Loss[†]

Yukio Inokuti

Research Laboratories, Kawasaki Steel Corp., 1, Kawasaki-cho, Chuo-ku, Chiba 260, Japan

The mechanism responsible for ultra-low iron loss in TiN-coated silicon steel sheet was clarified by electron microscope observation of the magnetic domain and the structure at the interface of the TiN film and single crystal of silicon steel, and by X-ray pole figure measurement. Marked domain refinement due to thin TiN film coating was accomplished, satisfying the coherency relationship of $(1\bar{1}1)_{\text{TiN}} // (011)_{\text{Si-steel}}$, $[110]_{\text{TiN}} // [100]_{\text{Si-steel}}$ between the TiN film and single crystal matrix, by applying a strong surface tension to the $[100]_{\text{Si-steel}}$ direction (elastic strain along only one axis). Fine transverse fringes were observed at the interface using a transmission electron microscope, and the elliptical shape of the $\{200\}_{\text{Si-steel}}$ pole peaks was detected by measuring the dual textures of X-ray pole figures.

(Received September 11, 1996)

Keywords: grain oriented silicon steel, TiN film, domain refinement, coherency, single crystal, atomic arrangement, dual phase, elliptical shape, strong tensile stress, ultra-low iron loss

I. Introduction

Grain oriented silicon steel sheet is a representative functional product manufactured using the highest level techniques in Japan's steel industry, and its technology plays a leading role in iron and steel production worldwide. Grain oriented silicon steel sheet, in which secondary recrystallized grains are highly aligned in the $(011)[100]$, or Goss orientation, are mainly used as core material for transformers and other electrical machinery and equipment. Figure 1 shows a schematic diagram of a grain oriented silicon steel sheet⁽¹⁾. The direction of secondary recrystallized grains with highly oriented products deviates no more than 3 to 4 degrees from the rolling direction (RD). The surface of the steel sheet is formed by a forsterite film, with an insulation coating with a low thermal expansion coefficient applied to the upper surface.

The grain oriented silicon steel sheet must provide a high magnetic flux density and a low iron loss; in addition to these superior magnetic properties, its magnetostriction characteristics and lamination factor must also be good. The conventional manufacturing process involves a number of complicated steps, which has earned silicon steel products the dubious reputation of an "artistic product of iron". However, energy conservation has made it important to produce the grain oriented silicon steel sheet with lower core loss.

Table 1 shows a summary⁽²⁾ of the factors, together with the improvement methods that influence the iron loss in grain oriented silicon steel sheet. At present, the

iron loss has been reduced to the lower limits possible with conventional manufacturing processes. Nevertheless, efforts to achieve further improvement in the iron loss of grain oriented silicon steel sheet are needed.

In a recent innovation⁽²⁾⁽³⁾, ultra-low iron loss has been obtained by applying ceramic coatings such as TiN, CrN or TiC to polished silicon steel sheets by physical vapor deposition (PVD) or chemical vapor deposition (CVD). With this technique, the ultra-low iron loss is generally attributable to the improvement in hysteresis loss (Wh) obtained by using a polished steel surface that is clean and free from impurities and to the reduced eddy current loss (We) arising from the strong straining of the ceramic film, but a fundamental clarification of the mechanism is important. In particular, the key point is to directly observe the magnetic domain and the structure at the near-

Fig. 1 Schematic diagram showing (a) Goss orientation and (b) grain oriented silicon steel sheet. (α in (b) represents deviation angle from the $[001]$ axis of secondary recrystallized grains.)

[†] This paper was originally published in Japanese in J. Japan Inst. Metals, **60** (1996), 781; Boundary, **10** (1994), 40; Boundary, **12** (1996), 26.

Table 1 Factors in and improvement of iron loss in grain oriented silicon steel sheet.

Iron loss	Factor	Improvement	
		Method	State
1) Hysteresis loss	a) Orientation	Sharpness of secondary grains	Ideal (011)[100]
	b) Impurities	Purification	Low level
	c) Surface and interface	Smooth	Mirror surface
2) Eddy current loss	A) Thickness	Decrease of sheet thickness	Free from sharpness of secondary grains
	B) Resistivity	Increase of Si content	Free from deterioration in steel production
	C) Domain walls	Grain size	Small grains
		Tension	Surface tension by coating
		Artificial grain boundary	Domain refining by Laser, Plasma, etc.

surface of the TiN-coated single crystal of silicon steel with the (011)[100] orientation, and to measure directly the dual textures between the TiN film and single crystal matrix. For this purpose, it is preferable to use an ideal (011)[100] single crystal of silicon steel.

This paper presents typical experimental results of (1) electron microscopic observation of the magnetic domain structure after making distinct chemically polished and TiN-coated areas inside a single giant single crystal with the ideal (011)[100] orientation, (2) transmission electron microscope (TEM) observation of the structure and analysis at the interface between the TiN film and the single crystal of silicon steel, a sample which was taken by mechanical processing from the defined area and thinned by using the focused ion beam (FIB) technique, and (3) X-ray pole figure measurement of the dual textures of the TiN film and single crystal matrix of the silicon steel.

II. Experimental Procedure

The starting materials were grain oriented silicon steel sheets of 0.23 mm in thickness, the chemical compositions of the raw material being in mass% C: 0.043, Si: 3.31, Mn: 0.072, Se: 0.019, Sb: 0.023, and Mo: 0.012. This silicon steel product was cut down to a sample size of $0.23 \times 30 \times 300 \text{ mm}^3$, and strain-relief annealing was carried out for 7.2 ks at 1073 K. (The specimen size was determined by the frame size of the single sheet tester (SST) usually applied in magnetic measurements.) Since the magnetic flux density of the silicon steel products

used in this experiment was closely related to the degree of alignment of the (011)[100] orientation, an initial measurement of the magnetic flux density was made to assess the suitability of samples for this experiment. Samples with high magnetic flux densities of above 1.93 Tesla⁽⁴⁾ were selected.

The insulation coating of the silicon steel samples so far obtained was removed by dipping in an NaOH solution, and the forsterite film was then removed by dipping in a 10% HCl solution at 353 K. The orientation of samples with large secondary recrystallized grains of about 25–30 mm ϕ was measured using a fully automatic apparatus⁽⁵⁾ which is capable of measuring the orientation of single crystals. A further selection was next made to obtain samples with an ideal orientation of the (011) crystallographic plane relative to the [100] axis, in which α , β , and γ ⁽⁶⁾ were each less than 1 degree. The surface of these silicon steel samples was chemically polished by dipping in a 3% HF + 97% H₂O₂ solution. The method of selecting the single crystal was the same as that used when observation of the magnetic domain in TiN-coated silicon steel sheet was attempted⁽⁴⁾.

The TiN ceramic coating was applied to the polished silicon steel samples by an ion plating apparatus of the hollow cathode discharge (HCD) type, as shown in Fig. 2(a). This HCD method has the merit of enabling deposition at a high ionization rate of 43% with a plasma beam of low voltage (40 V) and high current (500 A). Figure 2(b) shows the sample holder used in the HCD apparatus, which made it possible to apply the tension of 0.5–20 MPa (normal tension: 3.5 MPa) to the specimen while rotating the specimen holder in one direction to simultaneously deposit a uniform coating on both surfaces. This specimen holder also prevented deformation of the thin silicon steel samples during coating. To check in detail the domain-refining of the TiN-coated silicon steel sheet, distinct areas were made by chemical polishing after partial wrapping with stainless foil (60 μm thick) and TiN-coating inside a single crystal (giant secondary Goss grain) of (011)[100] orientation, as shown schematically in Fig. 2(c).

Ion plating by the HCD method was performed at 20 kW power, 0.093 Pa vacuum, -100 V bias, 1.5 kW focusing power, 673 K substrate temperature, 150 SCCM reactive N₂ gas, 0.1 $\mu\text{m}/\text{min}$ deposition rate, and 0.7 μm thickness. The magnetic domains⁽⁷⁾ after application of the TiN ceramic coating were observed under an accelerating voltage of 200 kV and beam current of 25 μA using an electron microscope (H-700). This sample has been set in the central position of the specimen holder of Fig. 1(b), and other four samples have been used to check the magnetic properties after TiN coating.

The sample for TEM observation of the TiN-coated silicon steel sample after the observation of the magnetic domain was taken initially by mechanical processing from an area of marked domain refinement of the TiN-coated silicon steel sheet, and thinned by using the FIB technique shown in the schematic diagram in Fig. 3. (Note that the sample was taken by mechanical proc-

Fig. 2 Schematic diagram of (a) hollow cathode discharge (HCD) ion plating apparatus, (b) tensile holder used in TiN coating, and (c) distinct areas of chemical polishing by stainless foil wrapping and TiN coating inside the (011)[100] single crystal of a silicon steel sheet.

Fig. 3 Schematic diagram of sample thinning for TEM observation, using FIB technique. (Note that the crystallographic orientation of the (011)[100] single crystal of silicon steel seen at top left is consistent with the direction of the white line in the circle in Fig. 4(c).)

essing (Nikoukikai Corp. NSG-52-ACE) parallel to the direction of the $[011]_{\text{Si-steel}}$ denoted by the white line in the circle in Fig. 4(c).) An FIB apparatus (Seiko Electronic Corp., SMI-8300) was used for thinning, by irradiating the sample with a Ga ion beam under an accelerating voltage of 35 kV. The thin sample for TEM was observed under an accelerating voltage of 400 kV and beam current of $8 \mu\text{A}$ under an electron microscope (JEM-4000FX).

The dual textures⁽⁸⁾ of the TiN film and (011)[100] single crystal matrix of the silicon steel after application of the TiN ceramic coating were also measured under an accelerating voltage of 60 kV and beam current of 250 mA

using a solid state detector (SSD) auto pole figure apparatus (Rigaku Corp. RU-300). From the crystallographic planes and lattice spacings of TiN (NaCl structure) and $\alpha\text{-Fe}$ (BCC structure), as can be seen in Table 2, it was possible to simultaneously measure the $\{111\}_{\text{TiN}}$ and $\{100\}_{\text{Si-steel}}$ or $\{110\}_{\text{TiN}}$ and $\{110\}_{\text{Si-steel}}$ pole figures of the TiN films and the single crystal matrix of silicon steel, respectively, with the same sample. Furthermore, to check directly the difference of textures between the (011)[100] single crystal itself (chemically polished area) and the TiN-coated area, an ideal giant (011)[100] single crystal of about 60 mm ϕ was selected and polished, and then TiN-coated by making distinct areas of chemical polishing and TiN coating, as shown with the dotted line in Fig. 2(c).

III. Experimental Results

1. Observation of magnetic domain

The left and right hand areas of Fig. 4(a) show respectively the surface appearance of a chemically polished (silver) and TiN-coated (gold) single crystal of the silicon steel sheet which was TiN-coated by the HCD method in Fig. 1(a). Optimum tensioning of the silicon steel sheet was applied (see Fig. 1(b)) after making distinct chemically polished and TiN-coated areas inside the (011)[100] single crystal, as shown with dotted line in Fig. 2(c).

Figure 4(b) shows the domain structure of the chemically polished and TiN-coated areas lying inside the (011)[100] single crystal in Fig. 4(a). Photographs of this domain structure have been taken successively by shifting the sample, using a 200 kV electron microscope with a magnification of five times. The domain structure of the chemically polished area in Fig. 4(b) shows an original 180° main domain with large black and white wall spac-

Fig. 4 (a) Surface appearance of chemically polished (silver) and TiN-coated (gold) silicon steel sheet, (b) montage of domain structure of chemically polished and TiN-coated (011)[100] single crystal of silicon steel, and (c) enlarged domain structure of chemically polished and TiN-coated (011)[100] single crystal. (The sample was taken by mechanical processing parallel to the direction of the $[011]_{\text{Si-steel}}$ denoted by the white line in the circle.)

Table 2 JCPDS of TiN and α -Fe and marks of poles in pole figures of TiN and α -Fe.

TiN (38-1420)*				α -Fe (06-0696)*			
$d(\text{\AA})$	Int.	hkl	Mark**	$d(\text{\AA})$	Int.	hkl	Mark**
0.24492	72	111	▲	0.20268	100	110	○
0.21207	100	200	■	0.14332	20	200	□
0.14997	45	220	●	0.11702	30	211	
0.12789	19	311		0.10134	10	220	

* JCPDS

** Notation of poles in pole figures

ings elongated in the rolling direction, while that of the TiN-coated area shows marked domain refinement due to the strong tensile stress existing in the TiN film. At the borderline between the chemically polished area and the TiN-coated area, as can be seen in Fig. 4(c), the domain

changes drastically, showing four to six narrow wall spacings in the latter for each wall spacing in the former. Lens-like refined domains also form from the large black and white 180° main domains at the beginning of the TiN coating area. On the chemically polished side of this line, longitudinally striped domains form distinctively within regions about $\pm 45^\circ$ to the rolling direction due to local strain arising from the strong surface tension in the TiN film.

2. TEM observation

Figure 5(a) shows a TEM micrograph taken at the near-surface of the single crystal of silicon steel observed after the sample was thinned by the FIB technique. Figure 5(a) shows an intermixed layer of TiN and silicon steel about 10 nm in width with fine transverse fringes, and Fig. 5(b) shows diffraction patterns of the (a) TiN film and (b) single crystal of silicon steel taken from the positions indicated by ☆ and *, respectively, in Fig. 5(a).

TiN has a NaCl structure having a lattice constant of 0.424 nm, in which Ti and N atoms are arranged alternately. From these diffraction patterns, the crystallographic plane of the TiN film is $(110)_{\text{TiN}}$, and the direction

Fig. 5 (a) TEM observation at near-surface of TiN-coated silicon steel sheet and (b) diffraction patterns of (a) TiN film and (b) $(100)[011]$ single crystal of silicon steel.

of the TiN film is $[1\bar{1}1]_{\text{TiN}}$, although the diffraction pattern of the TiN films shows weak ring patterns. The diffraction patterns among the $(110)_{\text{TiN}}$ planes include one of $[2\bar{2}0]_{\text{TiN}}$ deviated 35° from $[1\bar{1}1]_{\text{TiN}}$ in the clockwise direction and one of $[002]_{\text{TiN}}$ deviated 55° from $[1\bar{1}1]_{\text{TiN}}$ in the counterclockwise direction. In contrast, the crystallographic plane of the single crystal of silicon steel is $(100)_{\text{Si-steel}}$, and the direction of the single crystal is $[011]_{\text{Si-steel}}$.

From the diffraction patterns of the TiN film and single crystal of silicon steel, it is considered that there is an orientation relation between the TiN film and single crystal of silicon steel, as shown by

$$(110)_{\text{TiN}} // (100)_{\text{Si-steel}}, \quad [1\bar{1}1]_{\text{TiN}} // [011]_{\text{Si-steel}} \quad (1)$$

3. Measurement of dual textures

Figure 6 shows a typical example of a $\{111\}_{\text{TiN}}$ pole figure of the TiN film ($0.7 \mu\text{m}$ thick) deposited on the $(011)[100]$ single crystal of silicon steel. The crystallographic orientation of the dominant texture of the TiN film is $(1\bar{1}1)[110]$, with a strong $\{111\}_{\text{TiN}}$ pole peak (maximum peak intensity, 11) at the center position and six strong $\{111\}_{\text{TiN}}$ pole peaks (intensity, 9–11) at intervals of 60° from a circular position 70.5° left of center in the $\{111\}_{\text{TiN}}$ pole. It should be noticed that there are seven $\{111\}_{\text{TiN}}$ pole peaks in the $\{111\}_{\text{TiN}}$ pole figure with strong intensities of 9–11, and these intensities are similar to

Fig. 6 $\{111\}_{\text{TiN}}$ pole figure of TiN-coated $(011)[100]$ single crystal of silicon steel. The numbers of the $\{111\}_{\text{TiN}}$ pole peaks represent peak intensities from 1.5 to 11.

those⁽⁸⁾ of TiN $1.0 \mu\text{m}$ in thickness.

Figure 7 shows a typical example of (a) $\{111\}_{\text{TiN}}$ pole figure of the TiN film and (b) $\{100\}_{\text{Si-steel}}$ pole figure of the $(011)[100]$ single crystal of silicon steel that were measured simultaneously using the SSD auto pole figure ap-

Fig. 7 (a) $\{111\}_{\text{TiN}}$ pole figure of TiN film and (b) $\{100\}_{\text{Si-steel}}$ pole figure of $(011)[100]$ single crystal of silicon steel. The schematic diagram at left shows a cross-sectional sheet having the dual textures of the TiN film and single crystal and the method used in measuring the X-ray pole figure.

paratus. The left-hand diagram in this figure shows schematically the dual structure of the TiN film and single crystal and the method of measuring the X-ray pole figure. From Fig. 7, the crystallographic orientation of the dominant texture of the TiN film in (a) $\{111\}_{\text{TiN}}$ pole figure is $(1\bar{1}1)[110]$, whereas that of the $(011)[100]$ single crystal of the silicon steel sample in (b) $\{100\}_{\text{Si-steel}}$ pole figure is $(011)[100]$. Two $\{200\}$ pole peaks of the $(011)[100]$ single crystal sample showed an elliptical shape. Also, it should be noted in Fig. 7(b) that six $\{220\}$ pole peaks of the TiN film, the crystallographic orientation of which was $(1\bar{1}1)[110]$, were detected simultaneously.

These textural analyses indicated the following orientation relationship between the TiN film and single crystal of silicon steel:

$$(1\bar{1}1)_{\text{TiN}} // (011)_{\text{Si-steel}}, \quad [110]_{\text{TiN}} // [100]_{\text{Si-steel}} \quad (2)$$

IV. Discussion

1. Atomic arrangements of TiN film and single crystal of silicon steel

Figure 8 shows a schematic diagram of the orientation relationship of the TiN film and single crystal of silicon steel obtained in the direction of TEM observation and that obtained in the SSD auto pole figure measurement. From Fig. 8, it is evident that the TEM result of the orientation relationship in (1) is consistent with the texture relationship in (2). Figure 9(a) and (b) show a schematic diagram of the atomic arrangements of the TiN film and single crystal of silicon steel and a three-dimensional

Fig. 8 Schematic diagram showing the crystallographic orientations of the TiN film and $(011)[100]$ single crystal of silicon steel, the orientations of which were determined by both TEM observation and X-ray pole figures. (ND: Normal Direction, RD: Rolling Direction and TD: Transverse Direction)

Fig. 9 (a) Schematic diagram of atomic arrangements of TiN film and $(100)[011]$ single crystal of silicon steel and (b) three-dimensional schematic diagram.

schematic diagram, respectively. The misfit from coherency between the $[110]_{\text{TiN}}$ and $[100]_{\text{Si-steel}}$ directions shows a good value of 4.5%. Moreover, Fig. 10(a) and (b) show the arrangements of the stacked Ti atoms with the HCP structure and those of alternately stacked Ti and N atoms of $(111)_{\text{TiN}}$ with the NaCl structure, respectively. Figure 10(c) shows the atomic positions of layers A, B and C when the Ti and N atoms were stacked alternately in a closely packed state. It should be noticed from Figs. 9(a) and 10(b) that the six stack layers of the alternating atoms of Ti and N in the TiN film in this experiment are A(Ti), B(N), C(Ti), A'(N), B'(Ti), C'(N). The six stacking layers of the TiN film are different from three stacked layers A, B, C,—in which the same metallic atoms are stacked.

On the other hand, from the TEM observation of the intermixed layer of TiN and the single crystal (about 10 nm in width) in Fig. 5(a), it is worth of noting that the size of the fine fringes is about 2 nm, which is estimated to be 5 atomic layers of Fe-Fe atomic spacings in the $[011]_{\text{Si-steel}}$ direction in the $(100)[011]$ single crystal of silicon steel. From the experimental fact⁽⁹⁾ that no fine fringes could be observed between a TiN film and Si-wafer, it is considered that the fringes in this experiment were created by the strong surface tension (elastic strain)

Fig. 10 Comparison of atomic layers of (a) α -Ti (HCP structure) and (b) TiN (NaCl structure); (c) shows the positions of Ti and N atom packing in the A, B, and C layers.

imparted to the near-surface of the single crystal of silicon steel by TiN ceramic coating, which made it possible to obtain ultra-low iron loss in the TiN-coated silicon

steel sheet by inducing radical domain refinement, as seen in Fig. 4(c). It should also be emphasized that the ultra-low iron loss was obtained because of the good coherency between the TiN film and single crystal, in which the difference of the thermal expansion coefficients of the ceramic TiN and silicon steel sheet after cooling from 973 K (plasma coating temperature) to room temperature strongly tensions the near-surface of the TiN-coated silicon steel sheet to the $[100]_{\text{Si-steel}}$ direction (strong elastic surface tension along only one axis). In this case, good adhesion between the thin TiN ceramic film and single crystal matrix of the silicon steel sheet is absolutely essential.

2. Formation of elliptical $\{200\}$ pole peaks at near-surface of single crystal of TiN-coated silicon steel

The schematic diagram of Fig. 11 shows the peak shape of two $\{200\}_{\text{Si-steel}}$ poles of a (011)[100] single crystal of silicon steel of a chemically polished area (A) and TiN-coated areas (B). Also, the peak shape of seven $\{111\}_{\text{TiN}}$ poles (C) of the TiN films deposited on (011)[100] single crystal of silicon steel is shown in Fig. 11. In the $\{100\}_{\text{Si-steel}}$ pole figure of the chemically polished area, the peak shape of two $\{200\}_{\text{Si-steel}}$ poles, which manifest a dominant texture of (011)[100], is round $[a_1/a_2(\text{Av.}):1.01]$. In contrast, in the $\{100\}_{\text{Si-steel}}$ in the TiN-coated areas (B), the peak shape of the two $\{200\}_{\text{Si-steel}}$ poles is elliptical

Fig. 11 Schematic diagram of (A) $\{100\}_{\text{Si-steel}}$ pole figure in chemically polished area, and (B) $\{100\}_{\text{Si-steel}}$ pole figure and (C) $\{111\}_{\text{TiN}}$ pole figure in TiN-coated area inside a (011)[100] single crystal of silicon steel. The $\{100\}_{\text{Si-steel}}$ pole figures in (A) and (B) show two $\{200\}_{\text{Si-steel}}$ pole peaks, and the $\{111\}_{\text{TiN}}$ pole figure in (C) shows a $\{111\}_{\text{TiN}}$ pole peak at the central position. Also, a_1/a_2 , b_1/b_2 and C_1/C_2 in the pole figures represent the peak width ratio of RD/TD (Rolling Direction/Transverse Direction) detected at the pole peak intensity of 1.5. Av. indicates their average values, and N represents the number of the pole peak in the sample used in the X-ray pole figure measurement.

Table 3 Values of tensile stress applied to grain oriented silicon steel sheet by TiN coating.

① Magnetic measurement due to applying tensile stress	② Measurement of compressive stress sensitive of magnetostriction
8–10 MPa	8 MPa

$[b_1/b_2(Av.):1.46]$; also see Fig. 7.]. However, in the $\{111\}_{\text{TiN}}$ pole figures (C), the peak shape of the central $\{111\}_{\text{TiN}}$ pole, the peak of which manifests the dominant texture of $(1\bar{1}1)[110]$ orientation, is round $[c_1/c_2(Av.):0.99]$.

It is particularly important to point out that the formation of an elliptical shape is due to the strong tensile stress (elastic strain) of the single crystal imparted to the $[100]_{\text{Si-steel}}$ direction of the rolling direction (RD) by the TiN coating, which produces ultra-low iron loss by inducing radical domain refinement, as seen in Fig. 4(b) and (c). In addition to the ultra-low iron loss (drastic improvement in iron loss of about 40%⁽²⁾⁽³⁾), the high magnetic flux density ($\Delta B_8(T)=0.014-0.016$ Tesla⁽¹⁰⁾) was also increased by the strong tension in the vicinity of the single crystal surface with a silicon steel matrix about 300 times thicker than the thin TiN film of 0.7 μm . It should be emphasized that the formation mechanism of the elliptical shape of $\{200\}_{\text{Si-steel}}$ poles as observed by X-ray pole figure measurement is consistent with that of the fine fringes observed at the interface between the TiN film and single crystal by TEM. With respect to the formation area of the elliptical shape of $\{200\}_{\text{Si-steel}}$ pole peaks, detailed discussion will be given elsewhere.

On the other hand, the strong tensile stress at near-surface of TiN-coated silicon steel sheet was investigated by the magnetic measurement in order to evaluate the improvement of iron loss due to applying tensile stress⁽¹⁰⁾ and by the measurement of the compressive stress sensitivity of magnetostriction⁽¹¹⁾. The measured values of

the tensile stress are listed in Table 3. It should be noted that the strong surface tension of 8–10 MPa was imparted to the $[100]_{\text{Si-steel}}$ direction of the silicon steel sheet by the TiN ceramic coating. In addition, it is worthy of note that this TiN-coated silicon steel sheet also shows good lamination performance and low noise, the production of which has become important from an environmental point of view.

Finally, it may be pointed out that Fe_{16}N_2 nitride with a giant magnetic moment, which Kin and Takahashi⁽¹²⁾ discovered, but which has remained unconfirmed to date, has an atomic structure strongly strained only along one axis, and this condition is similar to the strong elastic surface tension in only one direction which produces ultra-low iron loss by magnetic domain refinement in the TiN-coated silicon steel sheet. Obtaining samples with an ideal (100) single crystal of pure iron⁽¹³⁾ will be the key point for a further examination of this problem.

REFERENCES

- (1) Y. Inokuti: Bulletin Japan Inst. Metals, **27** (1988), 250.
- (2) Y. Inokuti, K. Suzuki and Y. Kobayashi: Mater. Trans., JIM, **33** (1992), 946.
- (3) Y. Inokuti, K. Suzuki and Y. Kobayashi: Mater. Trans., JIM, **34** (1993), 622.
- (4) Y. Inokuti: J. Japan Inst. Metals, **59** (1995), 347.
- (5) M. Katayama, J. Shimomura, H. Yamashita, T. Kikuchi and Y. Inako: Bulletin Japan Inst. Metals, **33** (1994), 625.
- (6) T. Nozawa, T. Yamamoto, Y. Matsuo and Y. Ohya: IEEE Trans. Mag., MAG-14 (1978), 252.
- (7) For example, B. Fukuda, K. Sato, T. Ichida, Y. Itoh and H. Shimanaka: IEEE Trans. Mag., MAG-17 (1981), 2878.
- (8) Y. Inokuti: ISIJ International, **36** (1996), 579.
- (9) M. Miyake: ISIJ International, **33** (1993), 907.
- (10) Y. Inokuti, K. Suzuki and Y. Kobayashi: J. Japan Inst. Metals, **60** (1996), 674.
- (11) Y. Inokuti, K. Suzuki and Y. Kobayashi: Mater. Trans., JIM, **36** (1995), 1081.
- (12) T. Kim and M. Takahashi: Appl. Phys. Lett., **20** (1972), 492.
- (13) Y. Inokuti, N. Nishida and N. Ohashi: Met. Trans., **6A** (1975), 773.