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Effects of titanium on magnetic properties of semi-processed non-oriented electrical steel sheets

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The effects of titanium inclusions on magnetic properties of non-oriented electrical steel sheets were investigated. The magnetic induction and core loss of test specimens deteriorated as the titanium content increased. Electron microscopic study revealed that the deterioration was classified into two types: one was caused in the steels containing < 0.016 wt % titanium by the ''pinning effects'' of titanium carbonitrides on the recrystallization of cold-rolled sheets, and the other occurred in the steels containing > 0.016 wt % titanium by numerous (Fe, Ti)P precipitates in both grains and grain boundaries.

1. Introduction

Electrical steel sheets are consumed in millions of tons annually as the core material of small motors and ballasts [1]. Due to environmental problems, decreasing total carbon dioxide emission must be an urgent process. Thus, much attention has been paid to improving the magnetic properties, which leads to a decrease in power supply consumption. As improved magnetic properties are realized through the control of chemistry, grain size and texture, several new products with low core loss and high induction have been developed by the precise control of chemistry and mill processing [2–4]. This improvement has been achieved with the advances in steelmaking technology, which has its roots in the relationship between structure and magnetic properties. Advances in steelmaking technology now allow production of ultra-low carbon lean-alloyed steels, which have realized high induction or relative permeability and low core loss on semi-processed non-oriented electrical steel sheets.

Ultra-low carbon steels are expected to have improved magnetic properties with respect to typical low carbon (0.02–0.06 wt % carbon) steels having similar alloy composition. Lowering carbon contents leads to improved magnetic properties [5]. It is well known that the carbon in steel deteriorates magnetic properties on ageing. Honma *et al.* [6] showed this deterioration in 3.2 wt % silicon steel and concluded that the steel containing 0.0038 wt % carbon suffered from ageing, while the steel containing 0.0020 wt % did not. To solve the ageing problem, Allan and James [7] or Senuma [8] proposed the addition of titanium in order to precipitate the interstitial solid solute carbon as carbonitrides. However, these precipitates deteriorate the magnetic properties. Shimoyama *et al.* [9] investigated TiN and ZrC in 3 wt % silicon steel and inferred that these inclusions inhibited the grain growth on the recrystallization of cold-rolled sheet. Like titanium, vanadium also forms carbonitrides in silicon steels. Nakayama and Takahashi [10] studied the effects of vanadium on magnetic properties of semi-processed non-oriented electrical steel sheets and concluded that the size of vanadium carbonitride affected the grain growth on recrystallization and the magnetic properties deteriorated at 0.016 wt % vanadium content.

In this study, we investigated the effect of lean alloying, and especially the effect of titanium on magnetic properties of semi-processed electrical steel sheets.

2. Experimental details

Melts of various titanium content steels were prepared in a 50 kg vacuum induction furnace. Table I lists the chemical composition of the steels. The proportions of titanium are 0.001, 0.002, 0.003, 0.004, 0.006, 0.016, 0.042, 0.078, and 0.110 wt %, denoted as T-1, T-2, T-3, T-4, T-6, T-16, T-42, T-78 and T-110, respectively. The contents of other elements are 0.30 wt % silicon, 0.30 wt % manganese, 0.28 wt % aluminium, 0.100 wt % phosphorus, 0.004 wt % sulfur, < 0.001 wt % carbon and 0.001 wt % nitrogen. The steel ingots were machined to 45 mm thickness and reheated to 1453 K, then hot rolled to 4.5 mm thick sheets at a finishing temperature of 1103 K. After being air cooled to 943 K, the hot-rolled sheets were kept in a furnace at 943 K for 5 h, and then furnace cooled. These sheets were uniformly ground to 2.3 mm in thickness to remove any scale, and cold rolled to 0.5 mm thickness by a four-high-pilot cold-rolling mill with a total reduction of 78%. Continuous annealing was

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TABLE I Chemical composition of steels (wt %)

Steels	C	Si	Mn	P	S	Al[a]	Ti	N	O
T-1	< 0.0010	0.30	0.30	0.100	0.0041	0.28	0.001	0.0014	0.0034
T-2	< 0.0010	0.30	0.30	0.099	0.0041	0.28	0.002	0.0015	0.0019
T-3	< 0.0010	0.31	0.30	0.098	0.0045	0.27	0.003	0.0013	0.0010
T-4	< 0.0010	0.31	0.30	0.098	0.0046	0.28	0.004	0.0013	0.0035
T-6	< 0.0010	0.31	0.30	0.104	0.0045	0.27	0.006	0.0014	0.0022
T-16	< 0.0010	0.31	0.30	0.100	0.0044	0.27	0.016	0.0013	0.0011
T-42	< 0.0010	0.29	0.30	0.100	0.0047	0.28	0.042	0.0012	0.0032
T-78	< 0.0010	0.29	0.30	0.098	0.0045	0.28	0.079	0.0011	0.0013
T-110	< 0.0010	0.29	0.29	0.099	0.0047	0.27	0.110	0.0011	0.0012

[a] Acid soluble.

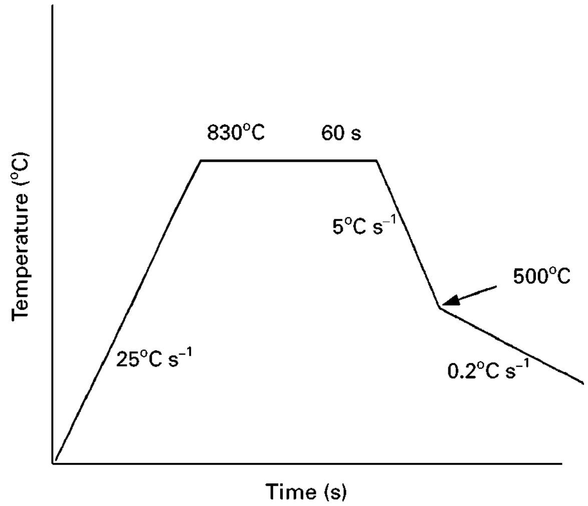

Figure 1 Heat diagram of continuous annealing.

performed with a Shinku-Riko ULVAC CCT-QB controller following the thermal profile as shown in Fig. 1. Continuous-annealed sheets were cut to 30 mm × 100 mm in dimension, either longitudinally or transversely to the rolling direction for 10 cm miniature Epstein frame specimens. These specimens were annealed at 1023 K for 2 h in nitrogen gas for stress relieving and then furnace cooled. Magnetic properties including core loss and induction were measured by 10 cm miniature Epstein frame of Yokogawa Electric Works Magnetic Measuring System. Texture examinations were performed using, a X-ray diffractometer (Material Analysis and Characterization Inc., MXP-3), with a Mo target operated at 46 kV and 16 mA. An inclusion study was done by the extraction replica method using a transmission electron microscope (TEM) with an EDAX facility for chemical analysis (Hitachi HU-700H operating at 100 kV).

3. Results and discussion

3.1. Grain size and microstructure

Fig. 2 shows the microstructures of continuous annealed sheets. The grain size decreases with increasing titanium content. Although the steel contains < 0.016 wt % titanium, the shape of the grains was equiaxed, while in the titanium content range > 0.016 wt %, the grains were deformed. Vanderschueren [11] studied the mechanism of recrystallization of IF steel and pointed out that the deformed ferrite grains were very difficult to recrystallize. Moreover, Park *et al.* [12]

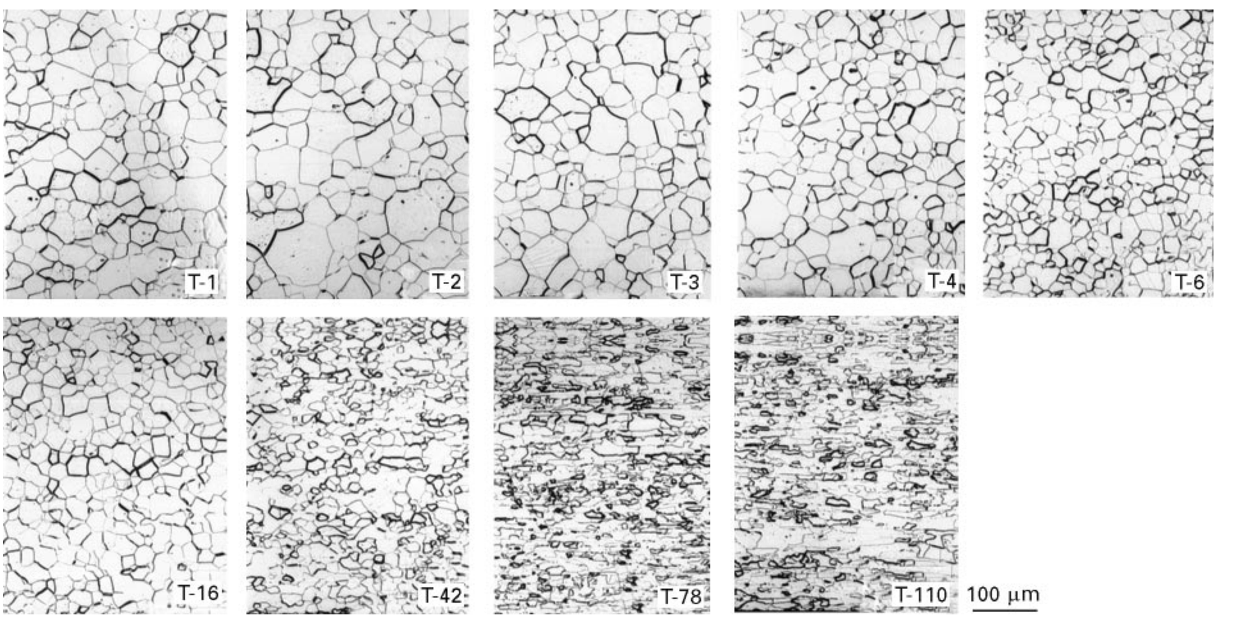

Figure 2 Microstructures of titanium containing cold-rolled and recrystallized steels after stress relief annealing.

investigated phosphorus in titanium-stabilized IF steel and summarized that phosphorus retarded the recrystallization of cold-rolled steel. Furthermore, Brun *et al.* [13] and Jeong and Chung [14] investigated this retardation and concluded that the precipitation of phosphides as (Fe, Ti)P was inevitable in steel containing much titanium and phosphorus unless the coiling temperature was < 773 K during hot rolling.

Fig. 3 shows the TEM replica of the various titanium containing steel sheets after continuous annealing. No titanium inclusions but large Al_2O_3 ones were observed in steel T-1. In steel T-6, titanium carbonitride pinning down the grain boundary was observed along it. Therefore the effect of titanium carbonitride on the retardation of grain growth is more pronounced than that of compound inclusions in steel T-1. However, in steels with more titanium content (> 0.016 wt %) numerous iron–titanium phosphide (Fe, Ti)P precipitates were observed both in grains and along grain boundaries (T-110). In our case, as the titanium content in steels containing 0.1 wt % phosphorus increases, the precipitating behaviour occurs at a point between 0.006 and 0.016 wt % titanium, and the major precipitates change from titanium carbonitride to iron–titanium phosphides.

3.2. Magnetic properties

Fig. 4 shows the relationship between core loss at 1.5 T and 50 Hz (W15/50) and titanium contents after stress relief annealing. It is well known that grain size

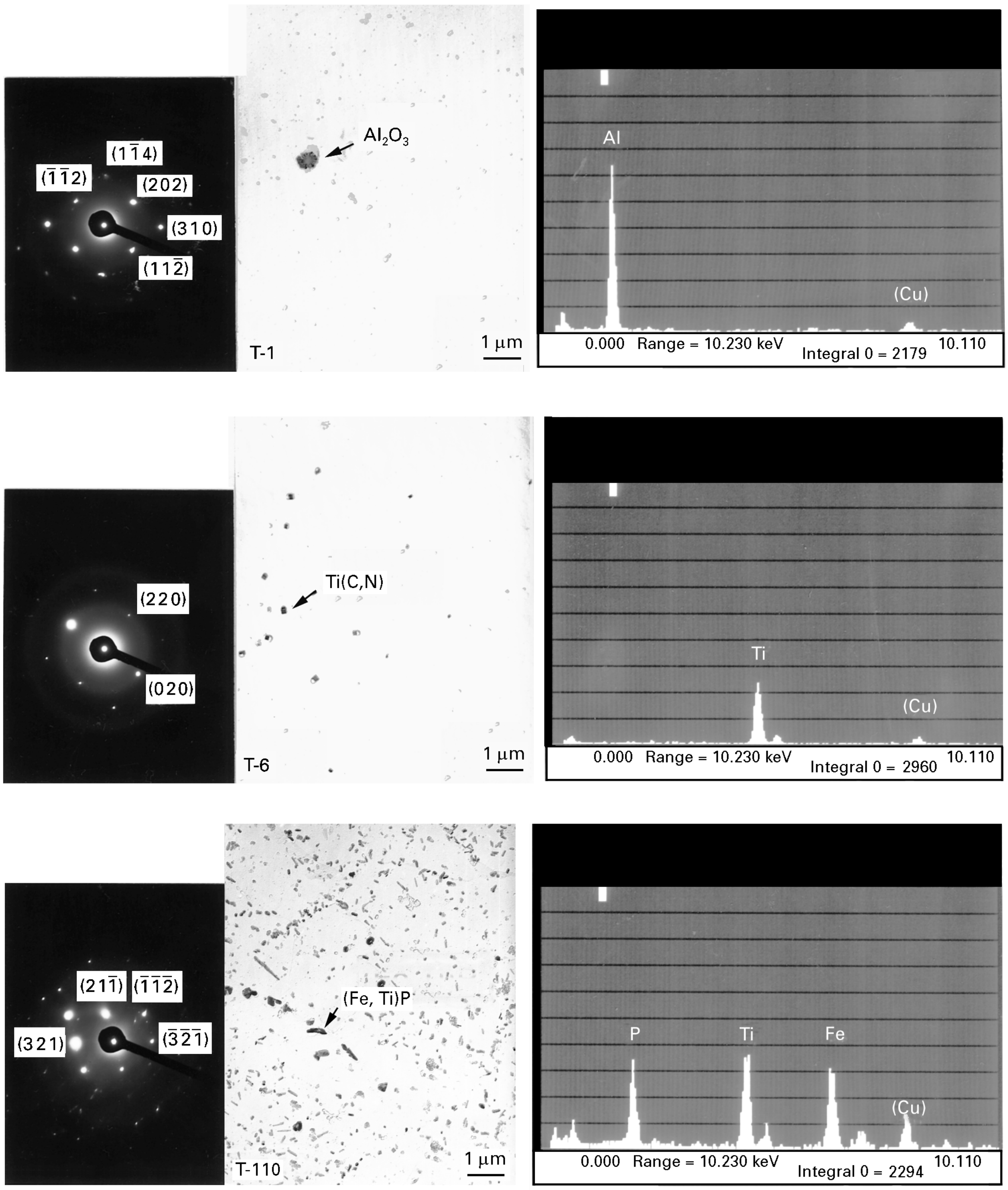

Figure 3 Inclusions observed by transmission electron microscope (TEM) after stress relief annealing.

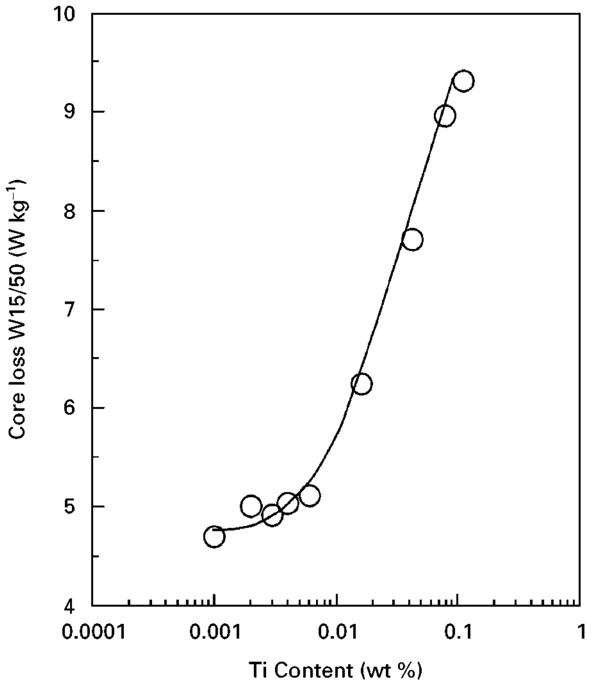

Figure 4 Relationship between core loss (W15/50) and titanium content after stress relief annealing.

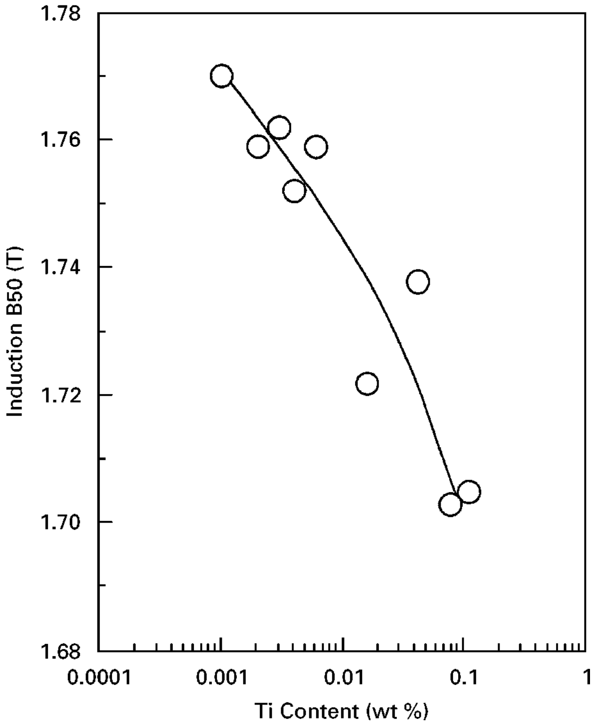

Figure 5 Relationship between induction (B50) and titanium content after stress relief annealing.

affects magnetic properties [15–17]. Matsumura and Fukuda [18] reviewed non-oriented electrical steel sheet and mentioned that the grain size affected the core loss and the lowest core loss was achieved at a size of about 150 μm. For grain sizes smaller than 150 μm, core loss increased with decreasing grain size. In our case, the grain size was less than 40 μm, so that the core loss increased with increasing titanium content (Fig. 4) as grain size decreased (Fig. 2). Further more, Matsumura *et al.* [19] pointed out that core loss increases with increasing numbers of inclusions. In our inclusion studies, steels containing < 0.016 wt % titanium have fewer inclusions such as titanium carbonitrides, while steels containing > 0.016 wt % titanium have numerous inclusions such as (Fe, Ti)P. This is the reason why the core loss increases dramatically in steel containing > 0.016 wt % titanium.

By contrast with the core loss, magnetic induction at 5000 A m^{-1} (B50) decreased with increasing titanium content < 0.016 wt % and dropped dramatically for-titanium contents > 0.016 wt % (Fig. 5). Magnetic induction or permeability is affected by the grain size and texture of hot bands. Fig. 6 shows microstructures of hot bands. Steels < 0.016 wt % titanium content had equiaxed grains, while steels containing > 0.016 wt % titanium partially recrystallized. Therefore, these unrecrystallized hot bands lead to the low magnetic induction at 5000 A m^{-1}. Moreover Figs 7 and 8 show that the inverse pole intensity as a function of titanium content $\{222\}$, which is not

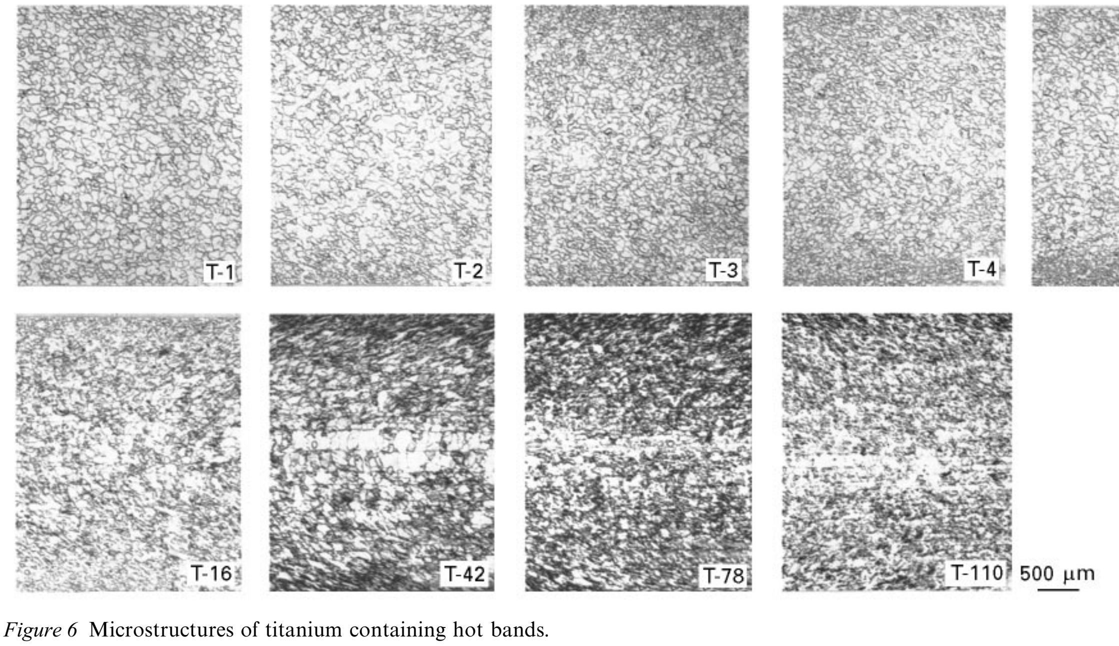

Figure 6 Microstructures of titanium containing hot bands.

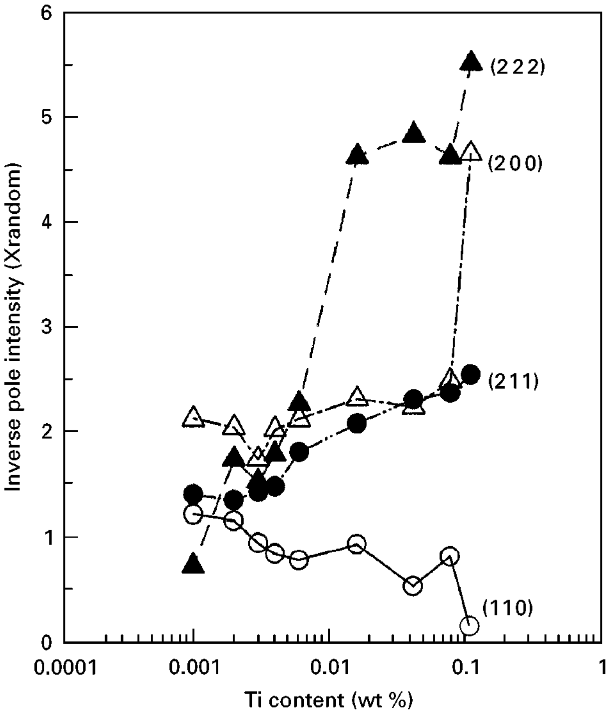

Figure 7 Relationship between inverse pole intensity and titanium content in hot bands.

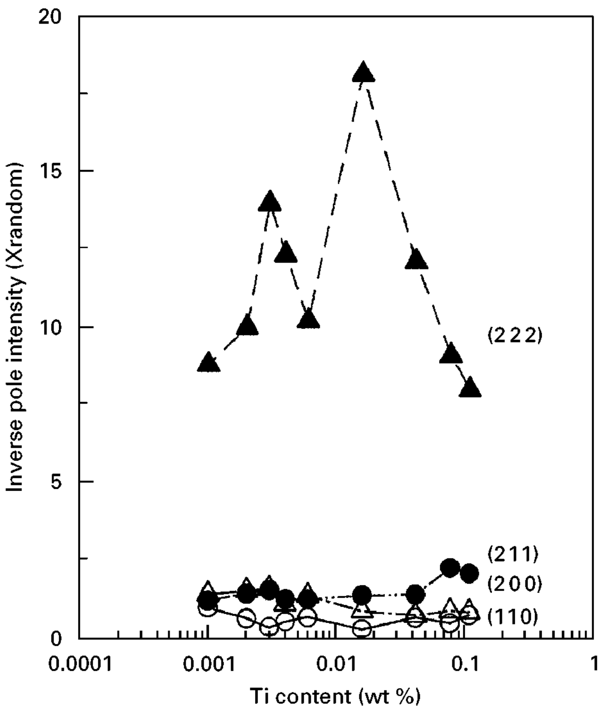

Figure 8 Relationship between inverse pole intensity and titanium content in cold-rolled and recrystallized sheets after stress relief annealing.

easy to magnetize, developed both in hot bands (Fig. 7) and cold-rolled and recrystallized sheet after stress relieving (Fig. 8). Although $\{222\}$ in hot bands developed with increasing titanium content, $\{222\}$ in cold-rolled and recrystallized sheet did not exhibit the same trend. This may be an effect of the two different types of precipitates in the titanium range > 0.016 wt % and < 0.016 wt %. $\{200\}$ in cold-rolled and recrystallized sheet, which is easy to magnetize, decreased with increasing titanium content. This is an another reason why the magnetic induction was decreasing with increasing titanium content.

4. Conclusions

The effect of titanium in semi-processed silicon steels is as follows.

1. The grain size decreases with increasing titanium content. Studies revealed that titanium carbonitrides were major precipitates in steels containing < 0.016 wt % titanium along the grain boundary acting as "pinning" inclusions. In steels containing > 0.016 wt % titanium numerous (Fe, Ti)P precipitates both in grains and along grain boundaries, retarded grain growth.
2. Magnetic properties were affected by the titanium content. As titanium content increases, core loss at 1.5 T and 50 Hz (W15/50) increased and induction decreased, due to grain size decreases both in hot bands and cold-rolled and recrystallized sheet caused by inclusions, and magnetically favorable textures decreased in cold-rolled and recrystallized sheet after stress relief annealing.

References

1. F. E. WERNER, in "Energy Efficient Electrical Steels" edited by A. R. Marder and E. D. Stephenson (TMS-AIME, Warrendale, PA, 1981) p. 1.
2. E. D. STEPHENSON and A. R. MARDER, *IEEE Trans. Mag.* **MAG-22** (1986) 101.
3. A. R. MARDER, *Metall. Trans. A* **17A** (1986) 1277.
4. J. M. SHAPIRO, in "Energy Efficient Electrical Steels", edited by A. R. Marder and E. D. Stephenson (TMS-AIME, Warrendale, PA, 1981) p. 33.
5. P. K. RASTOGI and G. LYUDKOVSKY, *IEEE Trans. Mag.* **Mag-20** (1984) 1539.
6. K. HONMA, T. NOZAWA, H. KOBAYASHI, Y. SHINOMIYA, I. TACHINO and K. MIYOSHI, *IEEE Trans. Mag.* **MAG-21** (1984) 1903.
7. C. ALAN and P. H. R. JAMES, British Patent 7931139 (September, 1979).
8. T. SENUMA, Japanese Patent Kokai 5-186855 (July, 1993).
9. Y. SHIMOYAMA, K. MIYOSHI, M. TANINO and T. WADA, *IEEE Trans. Mag.* **MAG-19** (1990) 2013.
10. T. NAKAYAMA and M. TAKAHASHI, *J. Mater. Sci.* **30** (1995) 5979.
11. J. VANDERSCHUEREN, in "Forum Book of The International Forum for Physical Metallurgy" (ISIJ, Tokyo, 1994) pp. 145–8.
12. Y. B. PARK, H. J. KANG and S. K. CHANG, *ibid.* pp. 245–8.
13. C. BRUN, P. PATOU and P. PARNIERE, in "Metallurgy of Continuous Annealed Sheet Steel", edited by B. L. Bramfitt and P. L. Mangonon (TMS-AIME, Warrendale, PA, 1982) p. 173.
14. W. C. JEONG and J.-H. CHUNG, *Tetsu-to-Hagane* **75** (1989) 74.
15. T. D. YENSEN, *Trans. Amer.* **43** (1924) 145.
16. T. D. YENSEN and N. A. ZIEGLER, *Trans. ASM* **23** (1935) 556.
17. *Idem, ibid.* **24** (1936) 337.
18. K. MATSUMURA and B. FUKUDA, *IEEE Trans. Mag.* **MAG-20** (1984) 1553.
19. K. MATSUMURA, B. FUKUDA, K. KINOSHITA, T. IMAI, R. KOBATA and S. MIYAZAKI, *Kawasaki Seitetsu Gihou* **15** (1983) 208.

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