Effect of titania addition on the ceramic film of a high permeability silicon steel

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This study investigates the impact of titania (TiO_2) addition on the microstructure and surface quality of ceramic film on grain-oriented silicon steel sheet. Titania was added to magnesia slurry in the range of 0 - 8 wt%. The ceramic film without TiO_2 was discontinue, non-uniform and presented stains. Therefore, it resulted in low insulation resistivity, oxidation resistance, and surface quality. The addition of 2% TiO_2 improved the film microstructure and coating properties. As the addition of titania increases, it is observed that the coating properties are altered, and defects begin to emerge. The ceramic film with 8% TiO_2 shows bare spots (100 – 200 µm) that decreased the insulation resistivity and oxidation resistance.

Keywords: Grain-oriented; Ceramic film; Glass film; TiO_2

1. Introduction

Grain-oriented silicon steel is widely used in transformers cores. Such applications require low core loss, high permeability, and an insulation coating. To provide a proper defect-free ceramic coating, additives are commonly used. Titania (TiO_2) is known to enhance the formation of forsterite [1, 2]. However, the role of TiO_2 in the microstructure and defects of the ceramic film needs further investigation.

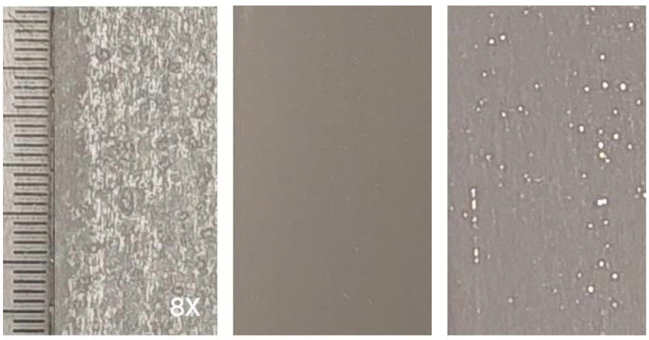
2. Experiments

The samples (305 x 120 x 0.27 mm) used in this study were made of 3% Si steel, sourced from a decarburized coil. The surface consisted of an oxidized sublayer with a thickness of 3 µm, an oxygen content of 870 ppm, and a fayalite/silica ratio of 0.16.

The samples were coated in the laboratory with a magnesia slurry composed of 17 wt% MgO, borax, and chloride. The addition of TiO_2 varied at 0%, 2%, 5%, and 8%. The applied coating weight was 7 g/m². Final annealing was carried out in an H_2-N_2 atmosphere, with a heating rate of 10°C/h above 650°C and a pH_20/pH_2 ratio of 0.06, up to 800°C. The soaking temperature was 1200°C for 15 hours in 100% H_2.

3. Results and discussion

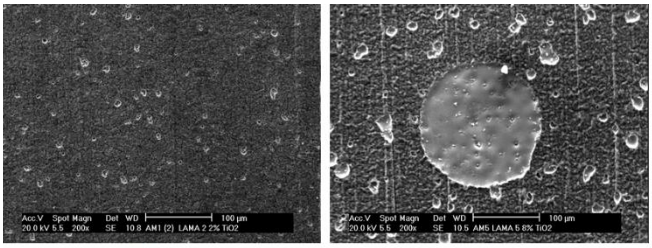
Figure 1 shows that the surface quality, both without and with 8% TiO_2, was poor. The former exhibited significant stains, while the 8% TiO_2 sample displayed numerous bare spots. The best quality was achieved with 2% TiO_2, which resulted in an uniform light grey film without any bare spots.



0% TiO_2 2% TiO_2 8% TiO_2

Figure 1: Ceramic coating without and with titania.

Figure 2 shows the microstructures of the film with 2% and 8% TiO_2 from surface. The coating with 2% TiO_2 (Fig. 2a) exhibited small discontinuities (< 10 µm), which can be observed under 200x magnification. At the same magnification, the film with 8% TiO_2 (Fig. 2b) shows a high incidence of bare spot defect. The typical defect has a circular shape with dimension from 100 to 200 µm consisting mainly of exposed base metal. It is unclear the mechanism of this defect. In the EDS analyses of the continuous film, Ti was not identified. There is evidence that Ti occupies the Si site in the forsterite lattice [3]. However, significant amounts of Ti were observed at the edges of the bare spot defect.



2% TiO_2 (a) 8% TiO_2 (b)

Figure 2: Ceramic coating with small discontinuities and with bare spots.

Table 1 summarizes the main properties of the coatings. The best results were obtained with 2% TiO_2.

Table 1. Coating properties.

TiO2 addition	Insulation [ohm.cm2]	Adhesion [mm]	Surface quality	Oxidation resistance
0%	0.987	20	Bad (stains)	Bad
2%	2.17	20	Great (no defects)	Good
8%	1.825	25	Bad (bare spots)	Bad

References

[1] Masui, H. et al., Int. CI6 B32B15/04 (1992), 5,565,272.
[2] Toda, H. et al., JP20071388201A (2005), JFE Steel Corp.
[3] Berry, A. et al., Chem. Geol. 242 (2007), 176-186.

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