Preparation and Properties of Oriented Silicon Steel Insulation Coating with Repair Function

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Abstract. In order to improve the comprehensive performance of oriented silicon steel insulation coatings and repair surface defects, an insulating coating for oriented silicon steels with repair function was prepared. The repair function was investigated by a simulation experiment. Adhesion and magnetic characteristics were measured. Corrosion resistance was tested by neutral salt spray test. The structure of the insulating coating was characterized by infrared spectroscopy. Results exhibit that the coating has good performance and long service life. The surface is smooth, bright and even, the adhesion is A and the coating is able to embellish the defects of crystal exposing on the matrix layer. There is no corrosion occurred after 24h salt spray test; the magnetic induction intensity $B_{500}$ is 1.81T, which meets the technical requirements of domestic steel mill products.

1. Introduction

Oriented silicon steel is a kind of soft magnetic functional material, which is mainly used as the core of motors and transformers. It’s one of the main functional materials supporting the development of the electromechanical industry and has a unique sandwich structure. The sandwich structure consists of a substrate, a magnesium silicate ($\text{MgSiO}_3$) interlayer[1], and an insulating coating. The $\text{MgSiO}_3$ interlayer is an ideal base for insulation coatings, can prevent the adhesion among the steel strips during the recrystallization annealing[2]. The insulating coating has good insulation, adhesion, corrosion resistance and high temperature resistance, can reduce the eddy current loss of the core, and makes the oriented silicon steel meet the manufacturing requirements of the iron core[3]. Both the quality of the $\text{MgSiO}_3$ interlayer and the insulating coating will affect the appearance quality of the silicon steel strip. The inferior $\text{MgSiO}_3$ interlayer makes the colour of the steel strip nonuniform, and cause exposure points of substrate and affect the quality of silicon steel products [4], while kinds of insulating coating have different influences [5]. Organic coating is consisted of organic resins with well insulation, film-forming, shock resistance and interlayer resistance, but poor hardness and weldability, deform or creep after heated, and release pollution gas. It’s gradually eliminated from the market[6]. Semi-inorganic coating is the combination of organic resins, phosphate or chromate and other inorganic salt[7]. With the organic resin part, the performance of coating is greatly reduced after annealing. Inorganic coating has the advantages of good adhesion and welding performance, and high temperature resistance. However, the comprehensive properties of inorganic insulating coating are poor, the coating is too hard that the punching and shearing properties are poor. In addition,
commercially available inorganic coating doesn’t have the repair function required by manufacturers.

To improve the comprehensive performance and repair the surface, large-particle silica sol and aluminum dihydrogen phosphate were selected as film-forming agents, and small-particle silica sol, titanium dioxide, chromic anhydride and montmorillonite was also added[8]. Through infrared analysis technology, adhesion performance test, neutral salt spray test and magnetic performance test, the structure, thermal stability, adhesion, corrosion resistance, and magnetic characteristics of the coating were characterized. In addition, a simulation experiment to test the repair function was desired.

2. Experiments

2.1. Sample Preparation

The oriented silicon steel sheet is cut into a size of 100 mm × 30 mm with a cutting machine. Clean the small silicon steel sheet with distilled water, ethanol cleaning, 5vol% sulfuric acid and distilled water in sequence and quickly dry it to remove stain, oxide scale or dust on the surface to make the coating solution adhere better to the surface of the silicon steel sheet[7].

Firstly, Mix 2wt% montmorillonite and 40.4wt% deionized water. Secondly, add 15wt% aluminum dihydrogen phosphate, 20wt% large particle silica sol, 20wt% small particle silica sol, 2.5wt% chromic anhydride and 0.1wt% titanium dioxide in sequence, make the solution uniform by stirring. The samples need heat treatments of 475°C curing temperature for 5s and 800°C sintering temperature for 40s. The thickness of the coating film was controlled at 3μm ± 0.5.

2.2. Property Characterization

According to GB/T 2522 – 2007 test method for insulation resistance and adhesion of coating on surface of electrical steel sheet (belt), the adhesion performance of coating after heat treatment was tested.

According to GB/T10125-2012 salt spray test for artificial atmosphere corrosion test, continuous neutral salt spray test was conducted. NaCl solution with mass concentration of 50±5g/L was used to accelerate corrosion by continuous spray[9]. During the test, the salt spray can settle freely on the experimental sample, and the side of the test sample should be 15~30° from the vertical direction, in the same plane as the spraying direction of the salt spray, and the amount of salt spray output should be kept at 1~2mL/80cm² per hour. A 100-cell transparent plastic sheet with a cell area of 1 mm² is covered in the board, if the corrosion area in the cell exceeds more than 50%, then corrosion area is recorded as 1. Otherwise, it will not increase.

The samples of 30mm×30mm were cut from the silicon steel sheet after heat treatment, and the magnetic characteristics of oriented silicon steel were tested by the MPG-100D soft magnetic material tester of Brockhaus measurement.

VERTEX70 Fourier transform infrared spectrometer from German company BRUKER was used for infrared spectrum detection. First of all, the coating solution(1g) is weighed and dried at 100°C to constant weight, and then grind it into powder. The powder and spectrally pure potassium bromide are mixed at a ratio of 1:10, grind and press it into tablets, and then the tablets were tested by infrared spectroscopy to analyse the chemical bond changes in the oriented silicon steel coating.

A certain amount of coating is dried to constant weight at 100°C, and then grind it into powder, using STA449F3 thermal analyser for thermal analysis of insulating coating, that is, in the air atmosphere, within the range of 30 – 900°C for isokinetic temperature rise test.

3. Results and Discussions

3.1. Adhesion Test and Coating Appearance

According to the above method, in the adhesion test, the adhesion of the coating was tested at bending diameters of 10, 20, and 30 mm, respectively. The coating did not fall off, and the test result was A. As shown in figure 1a, there are scratches on the surface of the substrate. After the transparent insulating coating is applied, the scratches are covered, and the surface is even and flat. The colour
difference test is $\Delta E = 0.12$, and the colour is greenish and blackish. This shows that the self-made insulating coating has surface modification properties.

![Figure 1](image1.png)

**Figure 1.** Surface morphologies of the substrate and the home—made coating. (a) Substrate, (b) Home—made coating

### 3.2. Neutral Salt Spray Test

Neutral salt spray test was conducted on the substrate (figure 2a) and the sample plate (figure 2b) of self-made silicon steel coating. In the neutral salt spray test, one hour later, obvious rust spots appeared on the substrate (figure 2c), while no corrosion occurred on the sample plate (figure 2d). The substrate (figure 2e) was completely corroded after eight hours, while the sample plate (figure 2f) had no rust spots. After twenty-four hours, the substrate (figure 2g) had been completely corroded, while the sample plate (figure 2h) only had a small amount of rust spots, and the corrosion area fraction was less than 1%, which fully met the industrial requirements. Hence, the self-made silicon steel coating can greatly improve the corrosion resistance of silicon steel.

![Figure 2](image2.png)

**Figure 2.** Photos showing the appearance of different samples after neutral salt spray test

(a) Substrate, 0h. (b) Sample plate, 0h. (c) Substrate, 1h. (d) Sample plate, 1h. (e) Substrate, 8h. (f) Sample plate, 8h. (g) Substrate, 24h. (h) Sample plate, 24h
3.3. Magnetic Properties

The magnetic properties of the self-made coating with thickness of 2.5μm were tested, including the iron loss P1.7/50 when the magnetic density was 1.7T and the frequency was 50Hz, and the magnetic induction intensity B_{800} for the 5000A/m alternating magnetic field. The results showed that P1.7/50 was 2.50W/kg, and B_{800} was 1.81T.

3.4. Infrared Spectrum Analysis

The infrared spectrum was measured to obtain the infrared spectra of the coating, as shown in Figure.3. The absorption peaks near 3437cm⁻¹ and 1634cm⁻¹ are stretching and bending absorption peaks of water [10], with a large peak area, which may be interlayer water in montmorillonite. At 1105cm⁻¹, there are anti-symmetric stretching vibration absorption peaks of Si-O-Si, while at 470cm⁻¹ and 800cm⁻¹, there are symmetric stretching vibration and bending vibration peaks of Si-O bond. At 953cm⁻¹, there is the absorption peak of silicon hydroxyl group(Si-OH), which has a small area, indicating that silicon hydroxyl group(Si-OH) is almost completely condensed into Si-O-Si bond in the sintering process, like figure.3.

![Infrared spectra of the coating cured at 450°C](image)

**Figure 3.** Infrared spectra of the coating cured at 450°C

4. Conclusion

The insulating coating of oriented silicon steel was prepared by using large particle silica sol, aluminum dihydrogen phosphate, small particle silica sol, montmorillonite, chromic anhydride, titanium dioxide and deionized water as the raw materials. The formula used was silica sol, 20.0wt%, aluminum dihydrogen phosphate, 15.0wt%, small particle silica sol, 20.0wt%, chromic anhydride, 2.5wt%, montmorillonite, 2.0wt%, titanium dioxide, 0.1wt% and deionized water, 40.1wt%. After the coating is cured, the coating surface is even and smooth, and the scratch on the silicon steel sheet disappears, that is, the coating has good repair function. When the silicon steel sheet with the coating was tested in the neutral salt spray for 24 hours, the silicon steel sheet was hardly corroded. And the coating has good insulation and magnetic properties.

5. Acknowledgement

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6. References

[1] Wang C, Wang K, Xiao X B, Ding H, Wang B S, Mao C W, Zhang W L and Jin G N 2018 J. Journal of Materials Engineering 46 51-57

[2] Ma L Y, Chen W L, Li N, Wang Y S and Zhang D B 2011 J. Inorganic Chemicals Industry 43
8-11

[3] Zhang Z G and Li Z K, 2013 *J. Corrosion Science and Protection Technology* **25** 425-428

[4] Peng Z H 2014 *J. Guan Dong Science & Technology* **23** 160-168

[5] Wu X, Zhang W K, Chen X and Wu X M 2017 *J. Hot Working Technology* **46** 182-184

[6] Hu Z Q, Zhang W K, Guang H B and Gu X Y 2012 *J. Electrical Engineering Materials* 33-36

[7] Meng X N, Wu L, Zhuo Z H, Yue C, Ding J Q, Chen Y Q and Yi D L 2015 *J. Electroplating & Finishing* **34** 1275-1281

[8] Peng Z H 2014 *J. Guan Dong Science & Technology* **23** 160-168

[9] Liu X S, Du W W, Wang Y and Tao J F 2017 *J. Materials Protection* **50** 61-64

[10] Wang H, Wei C P, Peng C J, Li C Y and Cheng G 2012 *J. Bulletin of the Chinese Ceramic Society* **31** 411-415