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Improvement of protective coating on Nd-Fe-B magnet by pulse nickel plating

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Sintered Nd-Fe-B magnets were coated by pulse nickel plating at different plating conditions. Optimal pulse plating condition was established (average current density = 1 A/dm², peak current density = 6 A/dm² with $T_{\text{on}}:T_{\text{off}} = 1:2$). In order to make a comparison, magnets with similar nickel coating thickness plated by dc were also prepared. The corrosion resistance of the coated magnets was evaluated by (i) Normal Salt Spray Test (5% NaCl, 35 °C) and (ii) potentiodynamic polarization measurement (3.5% NaCl solution). It was found that the corrosion resistance of the pulse nickel plated magnet was significantly improved as compared with that of the conventional dc plated ones, with negligible deterioration in magnetism. The microstructure of the coating was examined by optical microscopy and scanning electron microscopy. It was found that the porosity was much lower, and the grains much finer in the pulse-plated layer as compared with the dc plated ones.

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I. INTRODUCTION

The neodymium-iron-boron (Nd-Fe-B) permanent magnet, being one of the most popular magnets nowadays, derives its attractiveness and popularity from its well known excellent magnetic properties, including high remanence, high coercivity, and large energy product.¹ However, the applications of the Nd-Fe-B magnets are greatly hindered by their poor corrosion resistance in humid environments, especially when the magnets are of the sintered type.² The corrosion attack arises from the preferential oxidation of the Nd-rich phase in the grain boundary region.³ Such an intergranular mode of corrosion results in irreversible loss in flux and coercivity,⁴ contamination, and even total disintegration. The employment of coatings on the Nd-Fe-B magnets has been proven to be one feasible way to enhance the corrosion resistance without leading to significant deterioration of the magnetic properties. Among the various types of coatings applied to the Nd-Fe-B magnets, metallic coatings of Ni, Ni/Cr, Al, Zn, Cu, etc., have achieved different degrees of corrosion protection.⁵⁻⁹ Such metallic coatings were applied to Nd-Fe-B by dc electroplating, electroless plating, or immersion coating. On the other hand pulse plating, a technique which is well known for producing coatings which are more uniform, less porous, more wear-resistant and more corrosion-resistant (although with higher equipment cost, about twice that of the dc ones), has not been used on the Nd-Fe-B magnets. Thus it is the purpose of the present study to investigate the possibility of pulse plating the Nd-Fe-B magnets with Ni, to establish the optimal plating conditions, and to assess the coating properties.

II. EXPERIMENTAL PROCEDURE

Commercial Nd-Fe-B magnets composed of 15% Nd, 77% Fe, and 8% B were used in the present study. They

were in disk form with diameter and thickness of 15.7 and 2.2 mm, respectively. The samples were fabricated by conventional sintering and supplied by a local manufacturer. The magnetic properties were given as $B_r = 11.4$ kG, $H_{ci} = 12.4$ kOe, and $(BH)_{\text{max}} = 34$ MGOe.

The samples were cleaned by electrodegreasing in an alkaline solution, and then immersed in 10% hydrochloric acid at room temperature for 30 s for activation. The samples were pretreated with Ni striking at a current density of 2 A/dm² for 1 min, followed by dc plating of semi-bright Ni at 50 °C for 5 min using 1 A/dm². The samples were subsequently pulse plated with Ni using unipolar pulse in a bath having the composition and conditions listed in Table I. Four selected combinations of on and off times ($T_{\text{on}}:T_{\text{off}} = 1:1, 1:2, 1:4, 1:10$) were prepared. The mean thickness of the Ni coating film was in the range of 12–15 μm . For comparison, dc plated Ni coatings of similar thickness were also prepared using similar current densities.

The corrosion resistance of Ni coatings was investigated by (i) normal salt spray (NSS) test and (ii) electrochemical measurement. The NSS test (35 °C, 5% NaCl) was performed for 136 h using a standard chamber in accordance with ASTM G85 practice.

TABLE I. Composition and conditions for pulse nickel plating.

Nickel sulphate (NiSO ₄ ·6H ₂ O)	300 g/l
Nickel chloride (NiCl ₂ ·6H ₂ O)	60 g/l
Boric acid (H ₃ BO ₃)	40 g/l
Wetting agent	0.5 ml/l
Brightener	3 ml/l
Anode	Nickel
Average current density	1 A/dm ²
Peak current density	6 A/dm ²
Temperature	50 °C
Agitation	Moderate to vigorous

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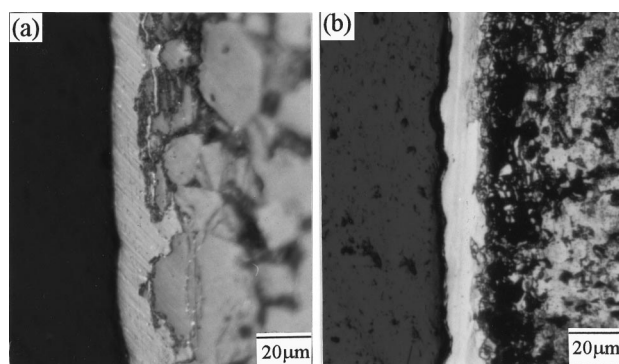


FIG. 1. Cross section of pulse Ni-plated samples (a) with pretreatment, (b) without pretreatment.

The time of initial formation of brown rust on the samples was recorded. Samples were removed at regular intervals from the chamber, carefully cleaned with de-ionized water, and dried thoroughly with warm air and weighed. The mass gain per unit surface area exposed due to the formation of corrosion product was also recorded and plotted against the exposure time.

In the electrochemical measurement of the samples, an EG&G PARC 273 potentiostat was used to obtain the potentiodynamic polarization scans. Two parallel graphite rods were used as the counter electrode and the saturated calomel electrode (SCE) was used as the reference electrode. Potentiodynamic polarization scans were carried out according to ASTM G5 practice, using deaerated 3.5% NaCl solution as the electrolyte at a scan rate of 0.6 V/h, scanning from -1 to $+1.2$ V. The corrosion potential E_{corr} and the corrosion current density i_{corr} were determined from the polarization curves using Tafel extrapolation.

Magnetic measurements were performed after 136 h of exposure in the salt spray test. The samples were pulse magnetized in a field of 35 kOe and demagnetized in a vibrating sample magnetometer (VSM) to obtain the demagnetization curves. The remanence B_r and the coercivity H_{ci} were determined.

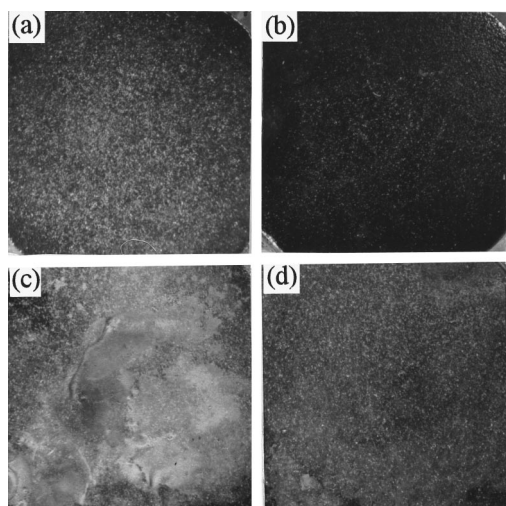


FIG. 2. Surfaces of samples before and after 16 h of exposure in salt spray test: dc-plated (a)(c); pulse-plated (b)(d). (3.5X).

TABLE II. Time of initial formation of brown rust in NSS test for different settings.

Plating technique	T_{on} (μs)	T_{off} (μs)	Time of initial formation of brown rust (h)
Pulse-plated	200	200	15
	200	400	16
	200	800	8
	200	2000	10
dc-plated	8

III. RESULTS AND DISCUSSION

Pulse Ni coating having a thickness of $12\text{--}15\ \mu\text{m}$ was obtained and the cross section is shown in Fig. 1(a). The coating was smooth and good adhesion was observed between the substrate and the coating. Pretreatment using dc plated semi-bright Ni was found to have greatly improved the adhesion of the coating to the substrate. The pretreatment prevented direct attack of the acidic bath on the magnet surface and hence prevented disintegration of the grains at the interlayer. Figure 1(b) revealed voids and pores appearing between the substrate and the coating without pretreatment. To achieve a high quality coating at a reasonable deposition rate, proper selection of current density is of primary importance. In the present study the optimal average current density was $1\ \text{A}/\text{dm}^2$ at $50\ ^\circ\text{C}$, with a peak current density of $6\ \text{A}/\text{dm}^2$. An on time of $200\ \mu\text{s}$ was employed since it is the most common selection for ferrous material with a reasonable deposition rate.

The corrosion resistance of four different pulse-plated Ni coatings, together with dc-plated Ni coating and the bare sample was assessed in the salt spray environment. The surfaces of dc-plated and pulse-plated samples before and after 16 h of salt spray test are shown in Fig. 2. It could be seen that the dc coating was seriously damaged [Fig. 2(c)] while for the pulse coating, the corrosion product just began to form [Fig. 2(d)]. The time of initial formation of brown rust was recorded and listed in Table II. It was found that the initial time for rust formation in pulse-plated samples was retarded by a factor of 2 as compared with that for the dc

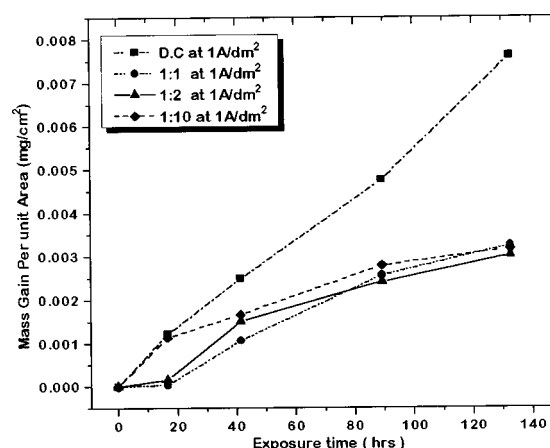


FIG. 3. Mass gain per unit sample area exposed at different exposure times in NSS test.

TABLE III. E_{corr} and i_{corr} from potentiodynamic polarization measurement of different plated samples.

Samples	E_{corr} (mV)	i_{corr} ($\mu\text{A}/\text{cm}^2$)
Pulse-plated Ni ($T_{\text{on}}:T_{\text{off}}=1:2$)	-440	0.12
dc plated Ni	-530	0.98
Uncoated Nd-Fe-B	-940.7	115.7

plated samples, extending up to 16 h for coatings with an on-off time ratio of 1:2. For bare samples, corrosion products were visible after 2 h of exposure.

The mass gain due to the formation of rust at different times of exposure is shown in Fig. 3. It was found that the mass increased at a faster rate after initial formation of rust, indicating breakdown of coating and exposure of the substrate to the salt mist environment.

Potentiodynamic polarization test of the pulse Ni-plated, dc Ni-plated and the bare samples in 3.5% NaCl deaerated solution at room temperature was performed and the corresponding corrosion potential E_{corr} and the corrosion current density i_{corr} were listed in Table III. The i_{corr} of the bare Nd-Fe-B was much greater than those of the coated ones, in fact greater by a few orders of magnitude. The i_{corr} of the pulse-plated samples was lower than that of the dc-plated samples by about an order of magnitude. The reduction in corrosion rate is probably due to the removal of discontinuities and pores which are usually present in dc plating. The more noble value of E_{corr} for pulse Ni coating indicated that the coating was more stable as compared with that of the conventional dc plating. Thus both the salt spray test and electrochemical test showed that the pulse-plated samples were more corrosion resistant than the dc plated ones. The improvement in corrosion resistance may be attributed to the structure of the coating in pulse plating, which is characterized by finer grains and lower porosity, as shown in Fig. 4(a)

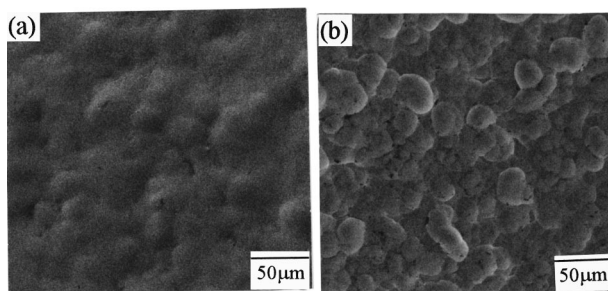


FIG. 4. Scanning electron microscope (SEM) micrographs of surfaces of pulse-plated (a) and dc-plated (b) samples.

TABLE IV. Magnetic properties before and after salt spray test.

	Before NSS test		After NSS test		Loss in B_r (%)
	B_r (kG)	H_{cl} (kOe)	B_r (kG)	H_{cl} (kOe)	
Pulse-plated Ni	11.3	12.4	11.2	12.3	0.88%
dc-plated Ni	11.3	12.3	11.1	12.3	1.77%
Uncoated	11.6	12.3	10.9	12.2	6.51%

versus the case of dc plating, Fig. 4(b).¹⁰

After 136 h of exposure in the salt spray test, the magnetic properties of the coated samples remained almost unchanged, whereas those of the bare samples decreased by about 6.5% (Table IV).

IV. CONCLUSIONS

Ni coating of thickness 12–15 μm has been successfully applied to the magnets by pulse plating. The set of conditions used was: average current density=1 A/dm², peak value=6 A/dm², temperature=50°C, on-off times=200 μs :400 μs . The coating obtained was fine-grained and low in porosity, and adhered well to the substrate. Corrosion data from both the salt spray test and electrochemical test indicated that for similar coating thickness, the corrosion resistance of the pulse Ni-plated samples was at least doubled as compared with the dc-plated ones. Magnetic measurements revealed that the remanence and the coercivity of the magnets were unaffected by the Ni coating.

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