

Multi-layered electroless Ni–P coatings on powder-sintered Nd–Fe–B permanent magnet

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Abstract

This paper has shown a successful protective coating scheme for powder-sintered Nd–Fe–B permanent magnet using multi-layered electroless nickel (EN) deposition. A low-phosphorus nickel layer is plated with an alkaline EN solution first, followed by a high-phosphorus nickel layer plated with an acidic solution. An additional topcoat by medium-phosphorus nickel on the high-phosphorus coating is also explored. It is shown that the high-phosphorus nickel layer coated in acidic solution provides the best corrosion protection because of its dense amorphous structure. The medium phosphorus topcoat is also dense and is able to provide reasonable corrosion resistance. The low-phosphorus layer itself does not have enough corrosion resistance; its main role is to provide an intermediate coating on the powder-sintered magnet. X-ray diffraction measurement shows that the low-phosphorus coating consists of nano-crystallites, and the high- and the medium-phosphorus coatings are dominated by amorphous structure. Microscopic observation and scratch test on these composite coatings demonstrate good adhesion between the magnet and the coatings. Remanence and coercivity of the plated magnet decrease with the applied coatings, but measured values are still very attractive for practical applications among known hard magnets.

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1. Introduction

Nd–Fe–B permanent magnet was developed in the 1980s [1] with excellent magnetic properties such as high remanence, high coercivity, and large maximum energy product. The major drawback that has prevented its wide spread application is its low corrosion resistance in humid and chloride environment. To overcome this problem, a lot of research work on the protective coating on Nd–Fe–B magnet has been carried out [2–8]. The coating material can be generally categorized into two groups: polymers, including epoxy resin [2,3] and bismaleimide (BMI) resin [4], and metals [2,5–8]. Generally epoxy coating is easy to apply and less costly. However, level of corrosion protection provided by polymeric resin is relatively low compared with metallic coatings. This is because polymers are, in

general, prone to moisture attack and mechanical scratch, which will weaken the adhesion between the coating and the base material. Among polymeric coatings, BMI seemed to be able to provide the best protection [4] but the material itself is brittle and difficult to prepare. Its adhesion has to be improved by an extra layer of undercoat [4], so the advantage of simplicity and low cost of polymer resin coating is, to a certain degree, lost. Generally polymeric coatings are considered more suitable for applications in less stringent conditions such as low temperature and low humidity environment.

Metallic protection is mainly achieved by two types of techniques: vapour deposition and electrochemical plating [5]. Post-deposition heat treatment may be necessary after coating is carried out to improve the adhesion between the coating and the magnet. The advantage of metallic coatings is that they are capable of providing better protection in harsher working environment. On the other hand, the disadvantage is the higher cost due to materials,

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processes, and equipment involved. Despite the cost concern, so far the majority research work of magnet protection is still centred on metallic coatings because of the excellent protection they can provide. Coated metals that have been studied include Ni, Cr, Al, Zn, Cu, and various composite coatings such as Ti/Al, Ni/Al, Ni/Cr, and Al/Fe [2,5–10]. Successful protection has been achieved in spite of some disagreement on the superiority among some reports [2,5]. Among these metallic coatings, electrolytic Ni coating is one of the most promising candidates because of its good performance and relatively low processing cost.

This work explores the possibility of using electroless Ni–P (EN) plating for the protection of powder-sintered Nd–Fe–B permanent magnet. Electroless plating is a self-catalytic process, and, because of this uniqueness, the coatings are very uniform regardless the shape of coated parts. Electroless Ni–P plating requires less equipment set-up cost, and is most suitable for components of complex shape. Its excellent resistance to corrosion and wear has been proven by numerous other engineering applications. There has been little investigation on EN application on Nd–Fe–B magnet. Part of the reason is EN plating, especially the ones with medium- and high-phosphorus content for corrosion protection, is usually achieved in acidic solutions of pH around 4.8 [11]. However Nd–Fe–B reacts quickly with Cl^- and SO_4^{2-} in acidic solutions. Unless a layer of coating can be quickly applied, strong reaction will prevent coating from being applied despite that Fe is a catalyst for Ni reduction. The problem is extremely serious with powder-sintered magnet; the chemical reaction precedes the Ni plating reaction, resulting in complete disintegration of the sintered magnet.

The approach adopted in the current study is to coat an intermediate layer of Ni–P in an alkaline solution first. This intermediate layer itself is not enough to provide the needed corrosion protection, since it is not dense enough and only contains a low percentage of phosphorus. The role of this layer is to provide a platable surface for the corrosion-resistant Ni–P coatings plated in the conventional acidic solutions.

After the coatings were successfully applied, their cross-sectional structure, adhesion to the magnet, corrosion resistance, and effect on magnetic properties were analyzed and discussed.

2. Experiment

Commercially available powder-sintered Nd–Fe–B permanent magnet samples were used in the experiment. They were in disk form with diameter and thickness of 8.0 and 1.5 mm, respectively. Samples were first degreased in an alkaline solution at 70–80 °C for 90 s, and then immersed in 5% HNO_3 for 30 s at room temperature for oxide removal. Finally they were placed in 5% H_2O_2 + 10% acetate solution for micro-etching the surface before the Ni–P coatings were applied.

Preliminary experiment had shown that this sintered magnet reacted strongly with acidic electroless nickel solution once being placed in the bath, causing plating out of the solution and disintegration of the sintered magnet itself. Therefore, it is necessary for an intermediate layer to be plated in an alkaline nickel solution first, followed by an acidic coating. Three types of sample were prepared for the study. Sample A was coated in the alkaline solution with a low-phosphorus Ni–P layer to 20 μm thickness. Sample B was coated to 5 μm thickness with the same low-phosphorus Ni–P in the alkaline solution, followed by 15 μm -thick high-phosphorus Ni–P in an acidic solution. Sample C began with the same coatings as sample B and was further plated by 5 μm topcoat of medium-phosphorus Ni–P in acidic solution. Table 1 gives a summary of the three types of coatings employed in this study.

The morphology of coatings was observed under a scanning electron microscope (SEM). Compositions of various layers of coating were analyzed by energy dispersive X-ray spectroscopy (EDX) installed in SEM. X-ray diffraction (XRD) was carried out to determine the degree of crystallinity of the coatings.

The adhesion between the coating and the magnet was examined by the observation on cross-section under microscope and a mechanical scratch test. The scratch test was carried out in a CSEM microscratch tester. Vertical load was progressively applied so that the depth was increased linearly with time during scratching. This test also serves the purpose of qualitatively probing the mechanical properties of coatings. For example, brittle films may fracture during a scratch test, and a weakly adhered film may delaminate from the substrate.

Corrosion resistance of the above three coatings was investigated by accelerated test in 50% HCl (by volume)

Table 1
Summary of the structure and P content of the Ni–P coatings

Samples	Intermediate layer (alkaline solution)	High-P coating (Acidic solution)	Medium-P topcoat (acidic solution)
A	20 μm	No	No
B	5 μm	15 μm	No
C	5 μm	15 μm	5 μm
P content (wt%)	2.5–3.0	11.1	6.7

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