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# Corrosion protection of Nd–Fe–B magnets by bismaleimide coating

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Samples of Nd–Fe–B were coated with bismaleimide (BMI), a resin which is well known for its desirable barrier, mechanical, and thermal properties but has not been used as a coating material. The performance of BMI-coated samples (average coating thickness  $\sim 15 \mu\text{m}$ ) was compared with commercial epoxy-coated samples (average coating thickness  $\sim 20 \mu\text{m}$ ) in a series of tests. In the copper-accelerated acetic acid–salt spray test, the epoxy-coated samples failed badly (damaged area  $>50\%$ ) within 20 h while the BMI-coated samples showed no sign of failure up to 200 h. Using the cross-cut tape test for adhesion strength (ASTM D 3359), the commercial epoxy coatings suffered 25% area detachment while for the BMI coatings, no detachment was observed. The scratch resistance of the BMI coatings, measured in terms of scratch depth in the pin scratch test, was about 1.5 that of the commercial epoxy coatings, and the Vickers hardness was higher by a factor of about 2.5. When magnetized under the same magnetizing field, there was no significant difference in the magnetization obtained among bare, epoxy-coated, and BMI-coated samples. These results suggest that BMI coatings are excellent for protection of the Nd–Fe–B magnets against corrosion and function better than the commonly used epoxy resin. © 1999 American Institute of Physics. [S0021-8979(99)45108-4]

## I. INTRODUCTION

The development of permanent magnet materials has seen three milestones in the present century, namely, the appearance of the alnico system (1930s), the ferrites (1940s), and the rare earth magnets (1960s). Neodymium–iron–boron (Nd–Fe–B), a rare earth magnet developed in the early 1980s, is especially important for two reasons. In the first place, Nd–Fe–B possesses excellent magnetic properties which include high remanence, high coercivity, and large maximum energy product,<sup>1</sup> and second, it is free of cobalt and hence cheaper. However, the replacement of Co by Fe greatly reduces the corrosion resistance of Nd–Fe–B magnets, in fact to a level comparable to low alloy steel in salt spray environments.<sup>2</sup> Thus it is common to apply a coating on Nd–Fe–B magnets for corrosion protection. Protective metallic coatings employing Ni, Cr, Ni/Cr, Al, Zn, etc. on Nd–Fe–B magnets have been reported in the literature.<sup>3–6</sup> On the other hand, among the different types of polymer coatings, only epoxy resin has been attempted on Nd–Fe–B magnets. The time to failure of various epoxy-coated Nd–Fe–B magnets in the ASTM normal salt spray (NSS) test (35 °C, 5% NaCl, pH 6.5–7.2) in different studies ranged between 100 and 200 h,<sup>6,7</sup> which was comparable to a semibright Ni coating of 30  $\mu\text{m}$  (Ref. 3) and might thus be regarded as moderately corrosion resistant. As a further step in the development of polymer coating for Nd–Fe–B, the present study aims at investigating the feasibility of employing a relatively new resin, bismaleimide (BMI), as a coating material on Nd–Fe–B for corrosion protection. BMI resin has the generic chemical structure of an aromatic chemical

group that is attached to two maleimide groups. Compared with epoxy resin, BMI resin has greater thermal stability and lower moisture sensitivity, but is brittle and difficult to process.<sup>8</sup> The brittleness of BMI resin may be reduced and its processability may be enhanced by suitable modification while the desirable properties are retained. The BMI resin used in the present study was modified using 0,0'-diallyl bisphenol A (DBA). Modified BMI-resin is widely used in fiber/matrix composites for aerospace applications, but its employment as a coating material for corrosion protection has not been reported in the literature.

## II. EXPERIMENTAL PROCEDURE

### A. Sample preparation

Bare and epoxy-coated samples of sintered Nd–Fe–B magnet in the present study were supplied by a local manufacturer, the magnetic properties claimed by the supplier being  $B_r = 11.4 \text{ kG}$ ,  $H_{ci} = 12.4 \text{ kOe}$ , and  $(BH)_{\text{max}} = 34 \text{ MGOe}$ . The samples were in disk form with a diameter of 15.7 mm and thickness of 2.7 mm, and the epoxy coating had a thickness of  $\sim 20 \mu\text{m}$ . Before the application of BMI coating, the bare samples were ground with 220 grit SiC paper, cleaned by alkaline solution followed by alcohol and distilled water, and then dried. To enhance adhesion, a thin layer of primer of  $\sim 2 \mu\text{m}$  was coated on the cleaned samples using a spinner and allowed to air dry for at least 5 h. A BMI coating was then applied on the primer in a similar manner and the overall coating thickness was  $\sim 15 \mu\text{m}$ . The coating was dried at

60 °C for 2 h and then subjected to a sequence of heat treatment for crosslinking: 80 °C for 1 h, 120 °C for 1 h, 150 °C for 2 h, 180 °C for 2 h, and 200 °C for 4 h.

### B. Copper-accelerated acetic acid–salt spray (CASS) test (ASTM B 368-85)

The corrosion resistance of BMI-coated and epoxy-coated samples was assessed by the CASS test in accordance with ASTM B 368-85.<sup>9</sup> The salt solution consisted of 5 wt % NaCl, with 0.25 g CuCl<sub>2</sub>·2H<sub>2</sub>O added per liter of solution and with the pH adjusted to 3.1–3.3 by using glacial acetic acid. The temperature of the fog chamber was kept at 49 ± 1 °C.

### C. Cross-cut test for adhesion strength (ASTM D 3359-93)

In the cross-cut test for adhesion strength, the coated surface was scribed with a sharp tool steel cutter to produce two orthogonal sets of parallel scribed lines that penetrated the coating to expose the substrate.<sup>10</sup> The grid size generated by these sets of scribed lines was 2 mm×2 mm. An adhesive tape was pressed tightly on the scribed surface and then removed. The number of grids detached was recorded and the adhesive strength of the coating was rated by the percentage of area peeled off in the test.

### D. Electrochemical impedance spectroscopy (EIS)

EIS measurements of epoxy-coated and BMI-coated samples were performed in 3.5% NaCl solutions at a pH of 6.5 and 20 °C using an impedance analyzer (EG&G Model 6310). A pair of graphite rods was used as the counter electrode, a saturated calomel electrode (SCE) was used as the reference electrode, and the sample under test was the working electrode. Impedance measurements were made at –940 mV (the open-circuit potential of bare Nd–Fe–B) from 100 kHz to 10 mHz with an amplitude of 20 mV.

### E. Pin scratch test and hardness test

A tool steel pin with a 100° pointed end and under a constant load of 38.5 gf was moved over the coated surface at a speed ~20 mm/min. The load used was chosen so as not to cut through the coating. The width of the scribe (which was simply 2.38 times the depth) was measured under a microscope. This width was used as an inverse measure of the scratch resistance of the coating.

The hardness Hv of the coatings was measured by the Vickers diamond microhardness tester using a load of 25 gf and with a loading time of 15 s. The load used was chosen so that the depth of the indentation was less than one third of the coating thickness.

### F. Magnetic measurements

Bare, epoxy-coated and BMI-coated samples were magnetized under the same magnetizing field using a capacitor discharge magnetizer. The flux density at the center of the surface of the magnetized samples was measured by a Gaussmeter.

TABLE I. Comparison between BMI and epoxy coatings.

|   | Bare Nd–Fe–B samples | Commercial epoxy-coated samples | BMI-coated samples |
|---|----------------------|---------------------------------|--------------------|
| Coating Thickness                         | ...                  | ~20 μm                          | ~15 μm             |
| CASS (time to failure)                    | <1 h                 | <20 h                           | >200 h             |
| Adhesion strength (fractional detachment) | ...                  | 25%                             | 0%                 |
| Scratch Resistance (scribe width)         | ...                  | 13.5 μm                         | 9.3 μm             |
| Vickers hardness                          | ...                  | 11                              | 28                 |
| Hv (0.025)                                | ...                  | 11                              | 28                 |
| Flux density (at surface center)          | 1.68 kG              | 1.66 kG                         | 1.67 kG            |

## III. RESULTS AND DISCUSSIONS

The results of the experiments to compare the corrosion resistance and other properties of BMI coatings with those of commercial epoxy coatings are summarized in Table I.

In the present study the corrosion resistance of coated Nd–Fe–B samples was assessed by the CASS test. It is quoted in BS 1224<sup>11</sup> that a test duration of 16 h in the CASS test is equivalent to 96 h in the acetic acid–salt spray (ASS) test, and presumably to more than 96 h in the normal salt spray (NSS) test. The commercial epoxy-coated samples failed badly (damaged area >50%) within 20 h in the CASS test [Fig. 1(a)]. Thus the NSS equivalent duration of the commercial epoxy-coated samples was comparable to the results of other studies. On the other hand, there was no sign of corrosion for the BMI-coated samples in a test period of more than 200 h in the CASS test [Fig. 1(b)]. The excellent corrosion resistance of the BMI coating may be partly attributed to its well-known resistance to hot/wet environments

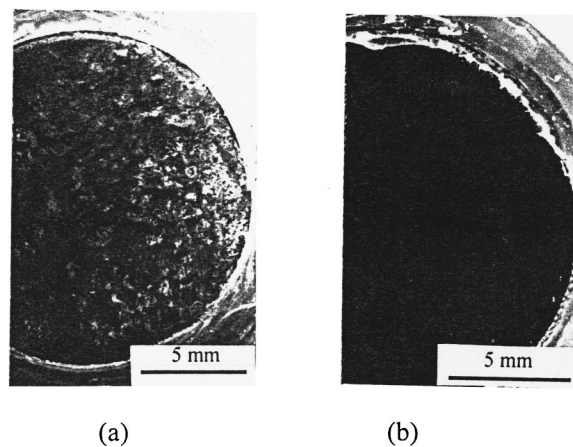


FIG. 1. Surfaces after exposure in CASS test (a) epoxy-coated sample, 20 h; (b) BMI coated sample, 200 h.

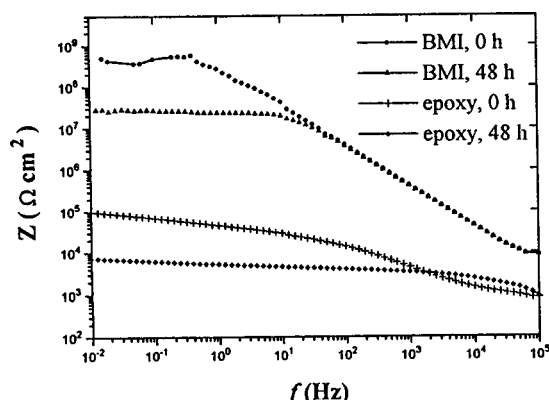


FIG. 2. Bode plots ( $\log|Z|$  vs  $\log f$ ) of epoxy-coated and BMI-coated samples in 3.5% NaCl solution.

compared with epoxy resin.<sup>12</sup> Crosslinking in a thermosetting resin enhances the barrier property of the resin while at the same time weakens its adhesion to a metal substrate.<sup>13</sup> As the barrier property of a polymer coating and its adhesion strength to a metal substrate both contribute to the corrosion protection of a metal by a polymer coating,<sup>14</sup> highly crosslinked BMI coatings might not offer optimal protection. To overcome the problem of adhesion, a primer was used as the undercoat, and strong adhesion was achieved as indicated by the absence of peeled cells in the adhesion test.

As a supplement to the CASS test for overall corrosion resistance, EIS measurements were also performed. The EIS data in 3.5% NaCl solution for both BMI-coated and epoxy-coated samples at the beginning and after 48 h of immersion are given in the Bode format ( $\log|Z|$  vs  $\log f$ ) in Fig. 2, where  $Z$  is the impedance and  $f$  is the frequency. The excellence in the protectiveness of the BMI coatings over the epoxy coatings is obvious from the curves shown. The coating resistance of the BMI coatings, as given by the low frequency limit of the impedance  $Z$ , was higher than that of the epoxy coatings by more than 3 orders of magnitude. Such a difference in coating resistance, which was a reflection of the difference in the barrier property of the coatings to water and ions, was consistent with the results of the CASS test.

The scratch resistance of the BMI coatings, as measured by the reciprocal of the scribe depth or width, was about 1.5 times that of the commercial epoxy coatings. Since the corrosion protection of a metal substrate relies heavily on the integrity of the coating and scratching is not uncommon during storage, transport or operation, scratch resistance is an important factor to consider.

It is essential that the corrosion protection measures for a magnet should not affect its magnetic performance. Bonded magnets, which are composed of magnet particles dispersed in a polymer matrix, may offer good corrosion resistance since the particles are encapsulated by polymer. However, the magnetic properties are reduced, although bonded mag-

nets possess other advantages such as easy processing. The simple magnetic measurement on bare, BMI-coated and epoxy-coated samples revealed that both types of coatings did not result in reduction of the magnetic performance. This is not unexpected as polymer materials are nonmagnetic in nature and the coatings used in the present study were very thin.

#### IV. CONCLUSIONS

BMI was used as a coating material for the first time and was successfully coated on sintered Nd-Fe-B magnet samples for corrosion protection. A thin primer was used as the undercoat to enhance the adhesive strength to the substrate. The BMI-coated samples (coating thickness  $\sim 15 \mu\text{m}$ ) could withstand more than 200 h in the copper-accelerated acetic acid-salt spray test, which was equivalent to more than 1200 h in the normal salt spray test, and was about an order higher than in the case of epoxy coating. The excellent corrosion resistance might be attributed to the barrier property of BMI and the adhesive strength of the primer/BMI system to the substrate. The BMI coatings were also more scratch resistant than the commercial epoxy coatings. Thus BMI coatings may be regarded as an alternative to, if not a replacement of, the commonly used epoxy resin in the corrosion protection of sintered Nd-Fe-B magnets.

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