Assessment of the Corrosion Behavior of Nd-Fe-B Magnets used in Dentistry

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Abstract. Permanent magnets based on intermetallic compounds are employed in dentistry to fix dental prosthesis. However, these materials are very sensitive to corrosion. In this study the corrosion behavior of a sintered commercial Nd-Fe-B magnet has been investigated at neutral pH in a phosphate buffered solution (PBS). With this aim demagnetized specimens were immersed in the test solution and their corrosion behavior were monitored at increasing test times by means of electrochemical impedance spectroscopy (EIS), potentiodynamic polarization curves and surface observation by scanning electron microscopy (SEM). Experimental results indicated that the corrosive attack was verified during test periods of up 4 hours. Impedance results have indicated the occurrence of diffusion controlled phenomenon, likely linked to the presence of pores in the magnet microstructure.

Introduction

Permanent magnets based on intermetallic compounds of rare earth (RE) elements and transition metals exhibit excellent magnetic properties and are applied in numerous fields such as computer hard disc drives, electronic motors, lockers, electronic scales and biomaterials. Among them Nd-Fe-B magnets have the strongest permanent magnetic field, with energy products (BH) as high as 48 MGOe [1,2]. However, it is also well known that one of the major problems for extensive application of sintered Nd-Fe-B magnets is their poor corrosion resistance, which is mainly ascribed to the presence of a Nd-rich phase [3,4]. This phase is less noble than the other constituents of the magnet, and is susceptible to preferential corrosive attack when it is exposed to a number of electrolytes.

It is widely known that the corrosion resistance of Nd-Fe-B magnets depends on its chemical composition as well as the processing technique [5]. Powder metallurgy produced magnets are especially susceptible to corrosive attack as pores constitute preferential pathways for electrolyte penetration.

The aim of this study is to investigate the corrosion behavior of Nd-Fe-B magnets. The study was performed in phosphate borate solution (PBS) using electrochemical techniques, namely, anodic and cathodic potentiodynamic polarization curves and electrochemical impedance spectroscopy (EIS). In order to assess the influence of the microstructure in the corrosion response of the samples scanning electrochemical microscopy (SEM) observations associated with energy dispersive X-ray analysis (EDS) were performed prior and after the immersion of the magnets in the test solution.

Material and Methods

The magnet was prepared by powder metallurgy, and its chemical composition is given in Table 1. The chemical elements Dy, Al, Nb, Co, Si and Cu are added to increase and improve the magnetic and corrosion properties on the other hand Na, Ca and S are probably impurities.

Fabl	le 1	. (Chemical	composition	(wt %)) of th	ne No	d-Fe-B	used	in th	ne present	study	I
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Fe	Nd	В	Dy	Al	Co	Si	Cu	Nb	Na	Ca	S
50.59	28.31	1.0	2.09	3.73	1.28	1.39	0.18	0.66	0.41	0.15	0.16

For electrochemical evaluation disc specimens of 12 mm diameter and 4 mm thickness were embedded with cold resin setter, after all the electrical contacts had been adequately established, leaving only one face exposed. The specimens were then ground up to 600 grit emery paper and cleaned with distilled water and alcohol prior to the corrosion tests. All the corrosion studies were carried out at room temperature (25 ± 2) °C in a PBS solution, whose composition is shown in Table 2.

Table 2. Composition of the PBS solution.

NaCl g.L ⁻¹	$Na_2HPO_4 g.L^{-1}$	KH ₂ PO ₄ g.L ⁻¹
8.77	1.42	2.72

All the electrochemical tests were performed using a classical three electrodes cell with Ag/AgCl and a platinum grid as reference and counter electrode, respectively. For the EIS experiments a Solartron[®] 1260 frequency response analyser coupled to a Solartron[®] 1287 electrochemical interface was used. The measurements were performed at the open circuit potential (OCP) in the 10 kHz to 10 mHz frequency range. The acquisition rate was 10 points per decade and the ac signal amplitude 15 mV. OCP was recorded prior and after the completion of each experiment in order to verify that stationarity requirements were obeyed.

Anodic and cathodic potentiodynamic polarization curves were obtained at a 1.0 mV.sec⁻¹ scan rate using the same electrochemical interface employed in the EIS experiments. The curves were initiated after OCP stabilization and were obtained in separate measurements.

The equipment used to perform the SEM/EDS analyses was a Philips XL-30 scanning microscope equipped with an EDS spectrometer. The acceleration voltage used to perform EDS analyses was 20 keV, giving a penetration depth of approximately 1µm. Semi-quantitative elemental analyses were performed using internal standards of the equipment.

Results

Figure 1 presents the SEM micrographs prior (a) and (b) after immersion during 4 hours in the test electrolyte. In Figure 1(a) semi-quantitative EDS measurements have shown that regions labeled as (1) and (2) are Nd-rich while (3) is the iron rich magnetic phase. Secondary electrons image of a sample after 4 hours immersion in the PBS solution (Figure 1(b)) show extensive corrosion product deposition on the magnet surface, on the other hand the backscattered image show extensive intergranular corrosion as well as preferential dissolution around Nd-rich particles. Moreover, closer observation of the corroded surface has shown the existence of pitting corrosion at specific places. The secondary electrons image reveals differences in contrast inside some grains showing that some of the phases present variations in composition [6].



Figure 1 SEM micrographs of (a) polished Nd-Fe-B magnet; (b) Nd-Fe-B magnet after 4 hours of immersion in the PBS solution: backscattered and secondary electrons image of the same region.

Figure 2 presents anodic (a) and cathodic (b) polarization curves after 1 and 4 hours of immersion in the test electrolyte. Except for a small increase in the OCP obtained after longer immersion period, likely due to some test irreproducibility, there is no change in the corrosion response of the samples for different immersion times. Anodic curves present a typical active

behavior, whereas in the cathodic branches two diffusion controlled processes are clearly visible. The first one, from the OCP until nearly -0.8 V, can be undoubtedly ascribed to the diffusion process normally associated to the oxygen reduction reaction. On the other hand, from around -0.9 V until -1.2 V, the diffusion of cathodic reactants through the pores of the material can explain the behavior. As a great quantity of corrosion products was present at the electrode surface after 4 hours of immersion in the test electrolyte (Figure 1(b)) it is possible to conclude that they are not protective and do not hinder the corrosion reactions.



Figure 2 Anodic (a) and cathodic (b) polarization curves for Nd-Fe-B magnets in PBS solution.

Figure 3 presents impedance diagrams obtained after two different immersion times in the test solution: 1 and 4 hours. As in the polarization curves no relevant change is noticeable between the two experiments. Two different relaxation phenomena can be easily distinguished in the Bode phase angle diagrams. The one in the higher frequency range is very depressed with angles close to 45 degrees. The observation of the Nyquist diagrams confirms this behavior, as a 45 degree angle can be easily distinguished. This kind of impedance response is typical of diffusion controlled processes. The material used in the present investigation was fabricated by powder metallurgy. These materials are characterized by the existence of porosities. So it is possible that this relaxation phenomenon would be related to the diffusion of the test electrolyte through these microstructure features. This could explain why there is neither diminution of the corrosion susceptibility of the

magnet nor change of the corrosion mechanism with immersion time, even though a great quantity of corrosion products were formed during the test. It is likely that the penetration of the electrolyte through the pores would provoke the dissolution of the magnet at the sub-surface layers undermining the corrosion resistance of the material.

The second relaxation phenomenon visible in the impedance diagrams would be associated with the charge transfer reactions at the magnet surface. In this case the dispersion in the experimental points of the diagrams could be associated with localized corrosion processes. Indeed, as already stated, closer SEM observation of the magnet surface has shown the existence of pitting corrosion at different sites.



Figure 3 Nyquist (a) and Bode (b) diagrams for Nd-Fe-B magnets after 1 and 4 hours immersion in PBS solution.

Conclusions

The results of the present work allow us to conclude that:

- The complex microstructure of the magnets leads to extreme corrosion susceptibility.
- Corrosion is more severe in the Nd-rich phase.

- Diffusion of the electrolyte through the pores of the powder metallurgy produced magnets seems to play a key role in the low corrosion resistance of the material.

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