

## Using EIS to Assess the Protection Afforded by Phosphate Layers to PM Produced Nd-Fe-B Magnets

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### Using EIS to assess the protection afforded by phosphate layers to PM produced Nd-Fe-B Magnets

Emerson A. Martins<sup>1</sup>, Hercílio G. De Melo<sup>2</sup>, Isolda Costa<sup>1</sup>

<sup>1</sup> IPEN - Instituto de Pesquisas Energéticas e Nucleares

<sup>2</sup> Chemical Engineering Department of the Polytechnic

School of the São Paulo University. P.O. Box 61548,

CEP: 05424-970 – São Paulo – SP – Brazil.

Permanent sintered Nd-Fe-B magnets present excellent magnetic properties finding many applications in different fields such as dentistry, where they are employed to fix dental prosthesis and to correct dental malocclusion [1,2]. However, these materials are highly susceptible to corrosion due to their complex microstructure and inherent porosity [3]. For applications in corrosive environments they are usually surface treated and coated or encapsulated.

In this work, the effect of phosphate pre-treatments towards the corrosion protection of powder metallurgy (PM) produced Nd-Fe-B magnets was investigated in a phosphate buffered solution (PBS) using EIS. The study was complemented by SEM-EDS surface observations to identify the protection mechanism afforded by the pre-treatment. The pre-treatment procedure was accomplished by immersion of polished samples in two different phosphating baths for varying periods.

Figure 1 presents SEM-EDS image of a polished magnet surface. The gray phase (matrix) constitutes the Fe-rich magnetic phase and the two other phases indicated, the light gray one and the white one, are Nd-rich phases. These latter are anodic in relation to the matrix [4]. Galvanic coupling between the Nd-rich and Fe-rich phases is partially responsible for the low corrosion resistance (CR) of this material. Porosities associated to the fabrication process also decrease the CR of the magnet investigated.

The OCP of the magnet stabilized quickly upon immersion in the test solution (less than 30 minutes), and there was almost no evolution of the EIS response up to 4 hours of immersion in the test electrolyte. Figure 2 depicts one EIS diagram obtained after 1h of immersion in the PBS solution. It is composed of two depressed capacitive loops. The high frequency (HF) one makes an angle of 45° with the real axis, while the one at low frequency (LF) of 22.5°. This kind of response is typical of porous electrodes with infinite length pores [5]. The HF loop is ascribed to the Rct in parallel with the double layer capacity (reaction takes place in the pore walls, whose length is greater than the depth of signal penetration) and the LF one is attributed to a diffusion controlled process within the pores and in series with the Rct. Cathodic polarization curves acquired after 4h of immersion in the PBS solution confirmed that oxygen diffusion controls the corrosion process.

Samples treated with a tricationic phosphate bath during 30 min and 1h prior to their immersion in the PBS test solution were also tested. EIS experiments after 30 min of immersion in the test solution showed an increase of the impedance, however increasing the pre-treatment time significantly decreased the impedance response. The shape of the EIS diagram obtained after the 1h pre-treatment was similar to the one of the polished sample, indicating that this treatment was not efficient in blocking the porosities present in the sample microstructure.

Figure 3 presents the results of the EIS experiments, after 30 min in the PBS solution, for samples treated during different times in a 10 g.L<sup>-1</sup> NaH<sub>2</sub>PO<sub>4</sub> solution (pH = 3.8). An increase of the impedance with pre-treatment time was observed. Evaluation of the Bode phase angle diagrams showed the presence of a HF time constant that can be ascribed to a thin phosphate layer on the sample surface

(confirmed by EDS analysis and visual observation through interference colours). The angle between the HF capacitive loop and the real axis shows that the electrode behaves like a porous electrode. However, the presence of the phosphate layer provokes an increase of the loop diameter and also displaces the LF loop to lower frequencies. This indicates that the phosphate layer is also formed on the pore walls hindering the corrosion reaction.

SEM observation of the magnet surface after immersion in the phosphate bath shows only little corrosive attack in the anodic Nd-rich phase. This indicates that a thin phosphate film is formed on these particles protecting them from corrosion. This hypothesis is supported by the low solubility of neodymium phosphates.

### References

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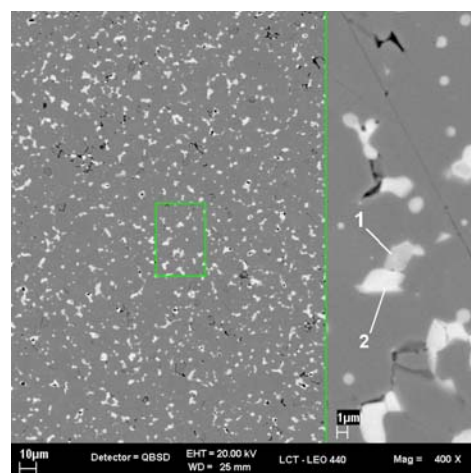


Figure 1 – SEM micrograph of the magnet surface. 1 and 2 Nd-rich phase. Matrix - magnetic (Fe-rich) phase.

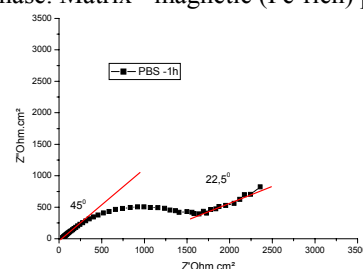


Figure 2 – EIS diagram for a polished magnet sample after 1 hour immersion in the PBS solution.

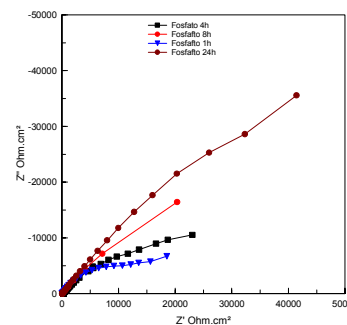


Figure 3 – EIS diagrams of samples treated for various treatment times in NaH<sub>2</sub>PO<sub>4</sub> (10 g.L<sup>-1</sup>) solution. Results obtained for 30 min of immersion in the PBS solution.