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THE EFFECT OF CORROSION PROCESS ON THE SURFACE TOPOGRAPHY OF Nd–Fe–B TYPE MAGNETS BONDED WITH BIOPOLIMER

WPŁYW PROCESU KOROZYJNEGO NA TOPOGRAFIĘ POWIERZCHNI MAGNESÓW TYPU Nd–Fe–B WIĄZANYCH BIOPOLIMEREM

A b s t r a c t

The article presents the results of surface topography measurements of the Nd–Fe–B type bonded magnetic material before and after the corrosion test in aggressive environments. The roughness of composite materials is closely related to the technological process. The increase in values of surface roughness parameters weakens the corrosion resistance by development of the actual surface and increases the contact surface with corrosive media. The results are a contribution to further work on the selection of appropriate technological parameters – among others enlargement of the bio-polymer content which contributes to the homogenisation of powder composition and thus has a beneficial effect on the resistance of material to an aggressive environment in a long-term use.

Keywords: *bonded magnets, corrosion, surface roughness*

S t r e s z c z e n i e

W artykule przedstawiono wyniki badań topografii powierzchni wiązanego materiału magnetycznego Nd–Fe–B przed i po testie korozyjnym w agresywnym środowisku. Chropowatość materiałów kompozytowych tego typu jest ścisłe związaną z procesem technologicznym. Większa chropowatość powierzchni osłabia odporność korozyjną wskutek zwiększenia rzeczywistej powierzchni styku korodującego elementu z medium korozyjnym. Wyniki są przyczynkiem do dalszych prac nad doborem odpowiednich parametrów technologicznych. M.in. zwiększenie zawartości spojnika biopolimerowego przyczyniłoby się do zwiększenia homogenizacji kompozycji proszkowej, a tym samym do zwiększenia odporności materiału na działanie agresywnego środowiska przy dłuższej eksploatacji.

Słowa kluczowe: *magnesy wiązane, korozja, chropowatość powierzchni*

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1. Introduction

In the era of dynamic development of computers, electronics and automation there has been a steady growth of interest in magnetic materials. The most commonly used materials are hard magnetic materials based on rare earth (RE) and transition metals (M) [1–2]. This group includes, among others, Nd–Fe–B neodymium magnets with unique magnetic properties due to the presence of $\text{Nd}_2\text{Fe}_{14}\text{B}$ ferromagnetic phase. The sintering with liquid phase and the consolidation of high-coercive powders with polymeric binders are among the most commonly used methods of preparing these kind of materials. Sintered materials are characterized by poor resistance to corrosion due to the high content of highly active rare earth element $E_{\text{Nd}_3+/Nd}^0 = -2,43 \text{ V}$ [3]. These features restrict their use and reduce the life of devices they are part of. Bonded RE-M-B material with lower content of rare earth element obtained in the form of a high-coercive powder [4] is an alternative to sintered materials. These powders, because of the high affinity of oxygen to neodymium, oxidize both in the manufacturing and storage process [5]. The presence of oxide layers on the surface of powder particles may adversely affect the process of consolidation. Insufficiently tight, weakly adhering adhesive coating makes the final material more porous, exhibits inferior magnetic properties and corrosion resistance [6, 7]. In papers [7–9] it has been concluded that loosely connected oxide products can be removed in the process of etching, and then the cover of the surface of powder particles can be coated with a coating and/or bonding material (encapsulation or biencapsulation).

As it has been shown in the studies [7–9] bonding of magnetic powder with the epoxy resin (encapsulation) provides a suitable consistency of the material. However, considering the use of materials in medicine and prosthetics, the epoxy resin is not a suitable binder. The replacement of an epoxy resin with biopolymer does not decrease the magnetic parameters[10]. A suitable binder content can contribute to the increase in homogenization of powder composition, and thus to minimize the occurrence of voids and open pores between metal particles and to smoothen the surface. Surface roughness is recognizable visually or it is mechanically reflected as surface roughness, not due to its shape [11].

The aim of this study was to analyze the surface of a bonded magnetic material (RE-M-B powder consolidated with biopolymer) before and after exposure to aggressive corrosive media.

2. Material preparation

The bonded magnetic materials were prepared from powder $\text{Nd}_{12}\text{Fe}_{77}\text{Co}_5\text{B}_6$ (commercial MQP-B, Magnequench). The powder was produced by rapid solidification from the liquid alloy. In the process, the amorphous strip is mechanically ground, and in order to obtain a nanocrystalline structure undergoes heat treatment at the temperature of approximately 600°C. Powder particles surface etching in a 5% aqueous solution of oxalic acid were the preliminary stage of specimens preparation. To protect the etched powder surface against atmospheric agents in the later stages of sample preparation, the encapsulation processes

were used – coating with biopolymer. The composition content was 5% mass. of bio-polymer binder and 95% mass. of magnetic powder.

3. Research methodology

Potentiokinetic polarization curves were performed in 0.5 M sulphate solution (pH = 2). Electrochemical studies were carried out by means of: potential scanning 10 mV·s⁻¹ (the potential was changed from the cathode to the anode value ($E = -0.8 \div 0.8$ V vs. SCE), rotation speed of 16 rev s⁻¹ (the test samples were in the form of a rotating discs).

The surface roughness analysis was carried out by means of a contact profilometer (Taylor Hobson) and the measurement distance was 4 mm for each sample. During the measurement, the recorded profile which was plotted on the Abbott'a curve indicated a high density altitude on the unit area for sample, participation of surface vertices and distribution of their size as well as the values of stereometric parameter (Rp – maximum peak height, Rv – maximum valley depth, Rz – maximum height of the roughness profile – based on the five highest peaks and lowest valleys over the entire sampling length, Rc – average height of roughness profile elements, Rt – maximum height of the profile, Ra – arithmetic average of absolute values, Rq – Root mean square deviation of the roughness profile).

4. Experimental

Based on the analysis of potentiokinetic curve obtained in the sulphate solution acidified to pH = 2, it can be concluded that the magnetic material of chemical formula Nd₁₂Fe₇₇Co₅B₆ undergoes active digestion, and there is not any observed trend of passivation (there is no passive range) and after exceeding the corrosion potential ($E_{corr} \approx -0.68$ V), the corrosion current continuously increases with the potential increase (Fig. 1).

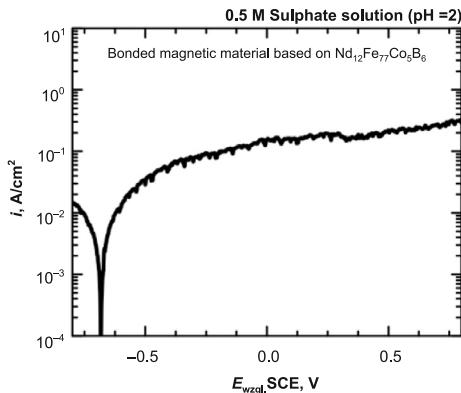


Fig. 1. The potentiokinetic polarization curves for samples obtained from Nd₁₂Fe₇₇Co₅B₆ powder encapsulated with biopolymer measured in 0.5 M sulfate solution acidified to pH = 2 (10 mV·s⁻¹, 16 r·s⁻¹, 20°C) – powder consolidated with biopolymer

The potentiokinetic studies have been complemented by roughness measurements by means of a profilographometer with a pin base and head provided with an inductive transducers. The measurements were carried out before and after the contact of sample surface with an aggressive environment. The computer system has enabled a complete analysis of the surface of magnetic material. The surface material was measured in 2D and then recorded the operating distance of the profile. Fig. 2 shows an example of the surface profile registered in an electrochemical test.

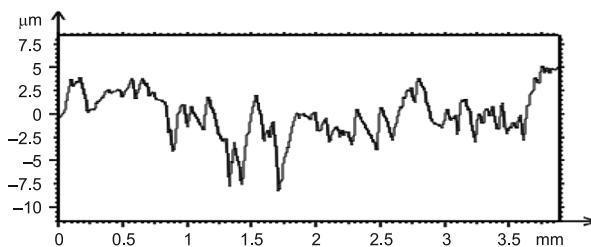


Fig. 2. Profile of operating distance registered for the bonded magnetic material – $\text{Nd}_{12}\text{Fe}_{77}\text{Co}_5\text{B}_6$ powder bonded with biopolymer

Roughness parameters from each of the isolated profile were calculated before (sample 1) and after exposition to the sulphate solution acidified to pH = 2 (sample 2) (Tab. 1), statistical analysis was used and the obtained results were compared in Table 3 and Fig. 3.

Table 1
The values of roughness parameters for $\text{Nd}_{12}\text{Fe}_{77}\text{Co}_5\text{B}_6$ bonded magnetic material – powder bonded with biopolymer

Symbol	$R_p, \mu\text{m}$	$R_v, \mu\text{m}$	$R_z, \mu\text{m}$	$R_c, \mu\text{m}$	$R_t, \mu\text{m}$	$R_a, \mu\text{m}$	$R_q, \mu\text{m}$
Sample 1	2.8 ± 0.8	3.3 ± 0.9	6.1 ± 1.6	3.3 ± 0.5	7.9 ± 2.3	1.0 ± 0.2	1.3 ± 0.3
Sample 2	7.9 ± 0.7	10.8 ± 0.6	18.6 ± 1.2	10.1 ± 0.1	22.9 ± 0.6	3.6 ± 0.4	4.4 ± 0.3

The R_a parameter values for the measured surface of magnetic materials based on $\text{Nd}_{12}\text{Fe}_{77}\text{Co}_5\text{B}_6$ are $R_a = 1.04 (\pm 0.18)$, and the values of roughness are well tolerated even by

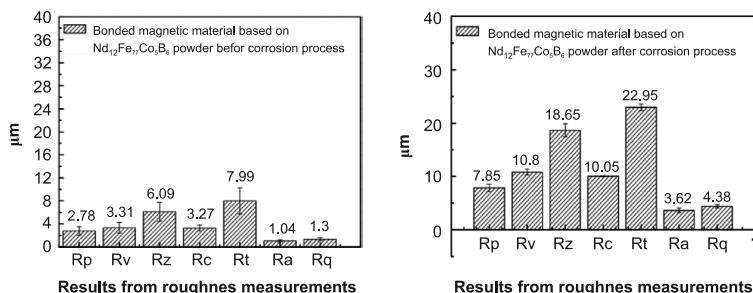


Fig. 3. Selected parameters of the surface profile for $\text{Nd}_{12}\text{Fe}_{77}\text{Co}_5\text{B}_6$ bonded magnetic material (powder bonded with biopolymer): a) before corrosion test, b) before corrosion test

cells in the body ($R_a < 4 \mu\text{m}$ [11]). Also other parameters of altitude are relatively low, which is closely correlated with favorable functional properties of the material [12].

The $\text{Nd}_{12}\text{Fe}_{77}\text{Co}_5\text{B}_6$ bonded magnetic material (powder bonded with biopolymer) was subjected to a 10-minute (dry contact) exposure to aggressive environments – sulphate acidified to $\text{pH} = 2$ solution (accelerated corrosion test). As it was concluded in work [12], height roughness parameters have the greatest impact on the intensity of corrosive wear surface and for the tested material these parameters increased after the accelerated corrosion test – revealing a significant development of the area. Particular increases were recorded for parameters R_t and R_z (overall and maximum height of roughness profile) (Tab. 3, Fig. 3b).

The increase in other parameters of height R_p (the maximum height profile), R_v (the maximum depth of the Valley) and R_c (the average height of profile elements) also points to the dominance of deeper valleys, which is associated with a progressive deterioration of surface material. Despite relatively low values of roughness parameters of the material, (sample 1) in a structure there may be present discontinuities which in aggressive environmental conditions are associated with an increased susceptibility to corrosion. However, it should be noted that even after corrosion processes, the R_a value is still less than $4 \mu\text{m}$.

The elevation distribution of the magnetic material surface and the Abbott curve (unfiltered individual parameters) were also recorded before and after the corrosion test (Fig. 4).

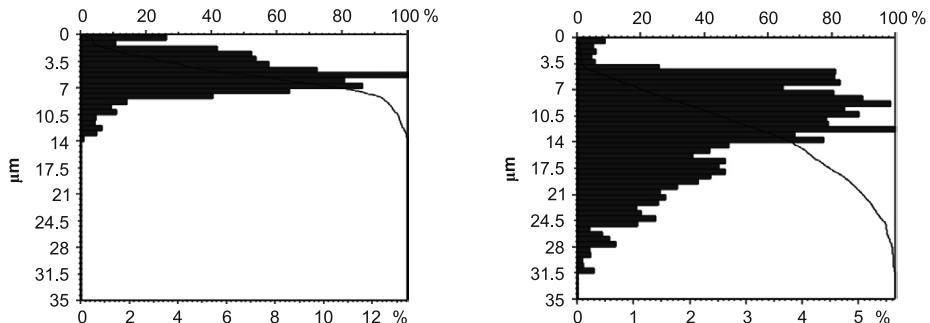


Fig. 4. Abbott curve and distribution of surface elevations for $\text{Nd}_{12}\text{Fe}_{77}\text{Co}_5\text{B}_6$ bonded magnetic material (powder bonded with biopolymer): a) before corrosion test, b) before corrosion test

The resulting elevations distribution of tested material surface after the corrosion test (Fig. 4b) testifies to surface development and progressive degradation into the material (to the depth of 30 m).

5. Conclusion

The tested composite – the $\text{Nd}_{12}\text{Fe}_{77}\text{Co}_5\text{B}_6$ bonded magnetic material (powder bonded with biopolymer) was actively digested in aggressive corrosive media (acidified to $\text{pH} = 2$ sulfate solution). As a result of this process, the surface of the material degraded as evidenced

by the increase in the height of roughness parameters. The results indicate a further direction of research – a change in process parameters (pressure, binder content, increasing the adhesion of powder particles to the binder) – which would allow for obtaining a material with favorable roughness parameters.

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