Comparative Studies on *Aspergillus niger* Biocorrosion of Alnico and NdFeB Magnetic Materials

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Bio-corrosion of both, magnetized and unmagnetized Alnico and NdFeB magnetic materials exposed in saline gel type Czapek Dox (comparatively sterile and inoculated with Aspergillus niger, was investigated by XRF, XRF, gravimetric, optical and SEM techniques. It was concluded that both Alnico and NdFeB are thermodynamic instable – in sterile media one achieved an appreciable corrosion rate, respectively in unmagnetized state it was approx. 2.5·10^{-6} g/h·cm^2 for Alnico and approx. 5.28 ·10^{-6} g/h·cm^2 for NdFeB, and in magnetized state it was around 2 times higher. Also, after the development of Aspergillus niger the corrosion rate was significantly higher comparatively to the exposure in sterile media (5-6 times higher).

Keywords: magnets, Alnico, NdFeB, biocorrosion, Aspergillus niger

Magnetic materials like AlNiCo (containing on iron, aluminum, nickel, cobalt and copper), and NdFeB (containing neodymium, iron, boron) are widely used in electrical machines and automations.

Even thought the magnetic materials based on NdFeB exhibit the best magnetic properties [1], due to their Nd-rare earth content, they are expensive and their corrosion resistance is weaker in comparison with AlNiCo magnets [2-7]. After their magnetization, it was noticed a change in their electrochemical behavior and also an increase of the corrosion rate [3,8,9].

Metallic materials being in contact with organic products (oils, lubricants, etc. – with carbon easy to be metabolized by microbial cultures) are susceptible to the microbiologic corrosion [10]. Several studies and analysis have been revealed the accelerated degradation by corrosion of metallic materials due to filamentous fungi. It was reported an accelerated corrosion due to *Aspergillus niger* of the metals such as: carbon steel [11-14], the austenitic steel [15-17], copper [18-20], aluminum [21, 22] and also of the complex structures such as underground power cables [23-25].

Aspergillus niger is a filamentous fungus having a wide geographic distribution [26, 27], which is due to outstanding tolerance to extreme environmental conditions: it can develop in a wide range of temperatures (10-50^{o}C), *p*H (2-11), salinity (up to 34 %) [28]. It is resistant to the herbicide products, pesticides, including toxic heavy metal salts, which are absorbed in the medium [29]. Along with other microorganisms, *Aspergillus niger* plays an important role in the biodegradation of the pollutants, respectively it has a role in the bioremediation of soil and/or surface water [30] - reducer and bio- absorber of the compounds of hexavalent chromium [31], biodegradation of mineral oil and some petroleum products [32] etc. Recent studies [33, 34] have been revealed the accelerated growth of *Aspergillus niger* when exposing the culture medium to electric (or magnetic) fields of 50Hz, up to 15V/cm and consequently the maturation time it is reduced by approx. 30%, the spore production increasing by approx. 60%. It was also found that at the applied electric field above approx. 30V/cm *Aspergillus niger* growth is inhibited, and when applying more than 50V/cm on the culture medium, the growth is completely inhibited. Several studies [11, 12, 35] have been noticed that aggressive corrosion process of *Aspergillus niger* is due to the citric acid resulting from the metabolism processes [36, 37].

Given the arguments presented above, the aim of our study is to assess comparatively the biocorrosion of magnetic materials, Alnico and NdFeB type (both in a magnetized and unmagnetized state) in contact with gels salt formed from the solution of mineral salts gelled with Agar-Agar inoculated with *Aspergillus niger* spores.

Experimental part

Alnico and NdFeB magnetic materials were investigated, both compositional and structural, using X-ray diffractometry (XRD - D8Advance Bruker diffractometer), scanning electron microscopy (SEM- InspectF FEI microscope) and X-ray fluorescence spectrometry (XRF - S8Tiger Bruker instrument).

Samples of Alnico and NdFeB magnetic materials (exposed surface of approx. 40cm^2), both in magnetized and unmagnetized state [3], were immersed in 60g of saline gel type Czapek-Dox. Aiming the evaluation of the bio-corrosion of these samples, it was performed specific microbiologic determinations, gravimetric evaluations (weight loss determinations using a digital analytical balance type N92, LAB A&D Ltd.) and by XRF it was determined the concentration of the dissolved metals in the biomass.

Bio-corrosion determinations were made both in a buffered mineral solution type Czapek - Dox, prepared from MERCK p.a. reactives, by dissolving in 1000 mL of distilled water of: 2g $NaNO_3$; 0.7g KH_2PO_4; 0.3g K_2HPO_4; 0.5g KCl; 0.5g $MgSO_4 \cdot 7H_2O$; 0.01g $FeSO_4$ and gelled by adding 30g of Agar-Agar (a difficult assimilable carbon source) and in the mineralized gel with added sucrose 30g / L (source of food - easily digestible carbon microorganisms). In order to emphasize the contribution of filamentous fungus *Aspergillus niger* mold to the corrosion of materials investigated, measurements were made both in the gelled medium, with (B) and without sucrose (A) as sterile medium, and inoculated medium with the inoculum of approx. 10^6 spores / mL of *Aspergillus niger* (ATCC 16404).

Control samples (sterile gel) and inoculated samples were incubated at 30 ± 2 °C with relative humidity of 90 ± 5 %, in the dark. Samples were analyzed periodically at 24,

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Table 1

CHEMICAL COMPOSITION OF THE ALNICO SAMPLE

Element	Fe	Co	Ni	Al	Cu	Si
Content [wt %]	50.85	23.79	12.46	8.81	3.91 %	0.17

48, 72 and 120 h, macroscopic and microscopic (stereomicroscopy).

Result and discussions

The morphology of the Alnico magnetic material sample investigated through scanning electron microscopy – SEM (fig.1), revealed that the material is homogenoeus in distribution of the major crystalline phases of FeCo and AlNi (identified by XRD – fig. 2).

Table 1 shows the chemical composition in weight percentage of the investigated Alnico sample, as measured by XRF. It was found, by analysis, that in addition to the usual constituents of the investigated magnets type AlNiCo, it contains also 0.17 % Si as an impurity (fig. 1).

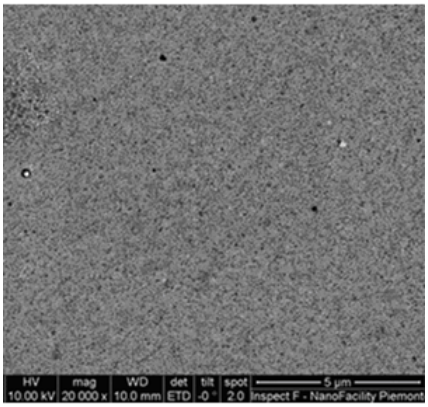

Fig. 1. SEM morphology of the Alnico investigated sample

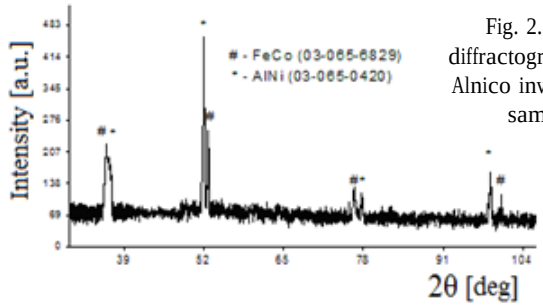

Fig. 2. X-ray diffractogram of the Alnico investigated sample

The SEM morphology of the NdFeB sample shown in figure 3 reveals a uniform distribution of the crystalline phases of $Nd_2Fe_{14}B$ and α Fe (evidenced by XRD – fig.4).

Table 2 shows the chemical composition of the investigated NdFeB sample, as measured by XRF. XRF method can not determine the content of the elements having lower atomic weight, less than approx. 24 (Boron have the atomic weight 10.81). The impurities revealed in the investigated sample NdFeB were: Si, Al and Cr, and by difference the Boron content was approx. 0.35%.

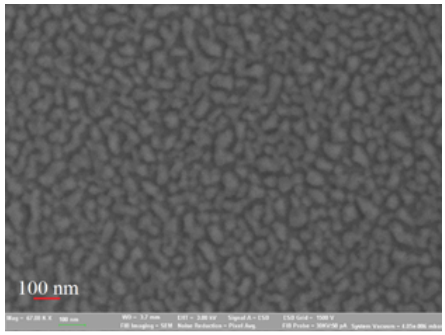

Fig. 3. SEM morphology of the NdFeB investigated sample

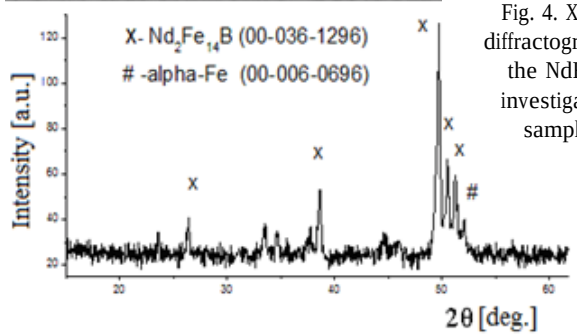

Fig. 4. X-ray diffractogram of the NdFeB investigated sample

The gravimetric evaluation results as weight loss after exposing in Czapek - Dox sterile gels, respectively inoculated gels of the magnetic materials samples - both magnetized and unmagnetized state, are summarized in table 3.

Analyzing the values presented in table 3, it has been revealed that in *Aspergillus niger* inoculated gels, with the specified test conditions, the weight loss (and therefore corrosion rates) are more than double higher than in sterile gels, which can be explained by increasing the aggressiveness of the environment resulting from the formation of organic acids (particularly citric acid [11, 12, 35]), in the metabolism of the mold. This explanation is supported also by the fact that gels with sucrose *B*, medium having easily digestible carbon (sucrose), the metabolism processes are more intense and therefore the weight losses are 1.25 ÷ 1.5 times higher than the sucrose -free gels.

The results of the XRF analysis on metal content of the investigated Czapek - Dox gels (*A* - without sucrose, *B* – with sucrose), on the reference gel prior to the immersion of the samples of magnetic material and after the exposure of the samples for 120 h at 30 ± 2 ° C, are summarized in table 4.

Analyzing the data presented in table 4 it is shown that, for the Alnico, both in magnetized and unmagnetized state, the corrosion occurs in sterile gels only by dissolving iron

Table 2

CHEMICAL COMPOSITION OF THE NdFeB SAMPLE

Element	Fe	Nd	Si	Al	Cr	B
Content [wt %]	72.24	27.12	0.13	0.1	0.06	**Δ% 0.35**

Tabel 3

GRAVIMETRIC EVALUATION RESULTS – WEIGHT LOSS OF THE MAGNETIC MATERIALS SAMPLES EXPOSED TO THE BIOLOGIC MEDIA

Sample		**Δm – Weight loss (after 120 hours at 30 ±2°C) [g]**			
		Sterile gel		***Aspergillus niger* inoculated gel**	
		Without sucrose „A"	With sucrose „B"	Without sucrose „A"	With sucrose „B"
Alnico	unmagnetized	0.0127	0.0132	0.0645	0.0813
	magnetized	0.0238	0.0262	0.1294	0.1610
NdFeB	unmagnetized	0.0257	0.0317	0.0706	0.0877
	magnetized	0.0539	0.0579	0.1349	0.2086

Sample		Elemental content [wt%] x10^{-4}											
		P	S	Cl	K	Fe	Al	Ni	Co	Cu	Si	Nd	Cr
Reference Czapek Dox „A" gel		5.1	2.9	2.8	4.9	5.0	–	–	–	–	–	–	–
Reference Czapek Dox „B" gel		4.9	2.7	2.6	4.7	4.9	–	–	–	–	–	–	–
AlNiCo in sterile „A"	unmagnetized	5.1	2.9	2.8	4.9	170	35	0.01	0.05	0.02	–	–	–
	magnetized	4.9	2.7	2.6	4.7	332	69	0.02	0.09	0.04	–	–	–
AlNiCo in sterile „B"	unmagnetized	5.1	2.9	2.8	4.9	185	39	0.01	0.11	0.03	–	–	–
	magnetized	4.9	2.7	2.6	4.7	366	74	0.02	0.32	0.09	–	–	–
AlNiCo in „A" with *A. niger*	unmagnetized	5.1	2.9	2.8	4.9	430	87	320	203	39.7	0.01	–	–
	magnetized	4.9	2.7	2.6	4.7	832	167	670	411	81.2	0.02	–	–
AlNiCo in „B" with *A. niger*	unmagnetized	5.1	2.9	2.8	4.9	515	112	432	251	49.6	0.01	–	–
	magnetized	4.9	2.7	2.6	4.7	1023	205	870	498	94.5	0.02	–	–
NdFeB in sterile „A"	unmagnetized	5.1	2.9	2.8	4.9	335	0.01	–	–	–	–	92	–
	magnetized	4.9	2.7	2.6	4.7	669	0.03	–	–	–	–	225	–
NdFeB in sterile „B"	unmagnetized	5.1	2.9	2.8	4.9	396	0.01	–	–	–	–	131	–
	magnetized	4.9	2.7	2.6	4.7	725	0.03	–	–	–	–	239	–
NdFeB in „A" with *A. niger*	unmagnetized	5.1	2.9	2.8	4.9	875	0.02	–	–	–	0.01	295	0.01
	magnetized	4.9	2.7	2.6	4.7	1682	0.05	–	–	–	0.02	561	0.02
NdFeB in „B" with *A. niger*	unmagnetized	5.1	2.9	2.8	4.9	1072	0.03	–	–	–	0.01	381	0.02
	magnetized	4.9	2.7	2.6	4.7	2598	0.09	–	–	–	0.03	867	0.03

Table 4
INVESTIGATED MEDIA ELEMENTAL CONTENT-SYNTETIC RESULTS OF XRF MEASUREMENTS

Table 5
THE DISOLVED METAL CONTENT IN THE INVESTIGATED GELS, XRF MEASUREMENTS

Sample		Disolved elements weight [g]·10^{-4}								Total [g] *weight*	v_{corr}·10^{-6} [g/h·cm^2]
		Fe	Al	Ni	Co	Cu	Si	Nd	Cr		
AlNiCo in sterile „A"	unmagnetized	99.00	21.00	0.01	0.03	0.01	–	–	–	0.012005	2.50
	magnetized	196.26	41.40	0.01	0.05	0.02	–	–	–	0.023775	4.95
AlNiCo in sterile „B"	unmagnetized	108.00	23.40	0.01	0.07	0.02	–	–	–	0.013149	2.73
	magnetized	216.66	44.40	0.01	0.19	0.05	–	–	–	0.026132	5.44
AlNiCo in „A" with *A. niger*	unmagnetized	255.00	52.20	192.00	121.80	23.82	0.01	–	–	0.064483	13.4
	magnetized	496.26	100.20	402.00	246.60	48.72	0.01	–	–	0.129379	26.9
AlNiCo in „B" with *A. niger*	unmagnetized	306.00	67.20	259.20	150.60	29.76	0.01	–	–	0.081277	16.9
	magnetized	610.86	123.00	522.00	298.80	56.70	0.01	–	–	0.161137	33.6
NdFeB in sterile „A"	unmagnetized	198.00	0.01	–	–	–	–	55.20	–	0.025321	5.28
	magnetized	398.46	0.02	–	–	–	–	135.00	–	0.053348	11.1
NdFeB in sterile „B"	unmagnetized	234.60	0.01	–	–	–	–	78.60	–	0.031321	6.52
	magnetized	432.06	0.02	–	–	–	–	143.40	–	0.057548	12.0
NdFeB in „A" with *A. niger*	unmagnetized	522.00	0.01	–	–	–	0.01	177.00	0.01	0.069902	14.6
	magnetized	1006.26	0.03	–	–	–	0.01	336.60	0.01	0.134291	28.0
NdFeB in „B" with *A. niger*	unmagnetized	640.20	0.02	–	–	–	0.01	228.60	0.01	0.086884	18.1
	magnetized	1555.86	0.05	–	–	–	0.02	520.20	0.02	0.207615	43.3

and aluminum, and in inoculated medium- due to the action of *Aspergillus niger* – it dissolved the main elements of the material (table 1 - Fe, Al, Co, Ni, Cu).

This finding can be explained by the ability of the *Aspergillus niger* to extract from the culture media and to retain the heavy metals in biomass [15-19, 27, 29, 31, 39, 40], to produce changes in the Langmuir - Blodgett layers, due to the presence of microbial culture [41, 42], which leads to the acceleration of the general corrosion process (1), with respect to the anodic reaction.

$$Me \rightarrow Me^{z+} + ze^{-} \quad (1)$$

where:

Me - dissolved metal;

z - valence of dissolved metal;

Me^{z+} - the formed metallic ion with z valence;

ze^{-} - number of released electrons.

By X-ray fluorescence spectrometry (table 4) was evidenced the dissolution of the elements Fe and Nd (in the case of NdFe) in both sterile and *Aspergillus niger* inoculated media. Given the low atomic weight (10.81) of the Boron, this was not revealed by this technique. In the inoculated medium, under the action of *Aspergillus niger*, corrosion is particularly intense and as a result in the formed biomass there are present even aluminum, silicon and chromium which dissolves simultaneously with major constituents (Nd, Fe and B).

Given that each magnetic material sample was exposed to the same amount of gel (60 g), from the data of table 4

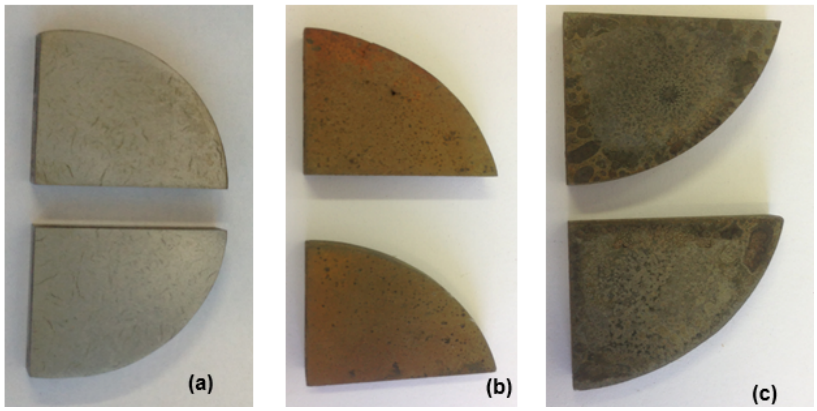

Fig. 5. The NdFeB unmagnetized sample before (a) and after exposure to Czapek-Dox gel - b) sterile environment and c) medium inoculated with Aspergillus niger

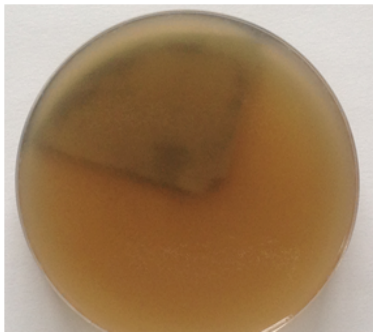

Fig. 6. The Alnico unmagnetized sample exposed to the Czapek-Dox sterile medium without sucrose - 120 h at 30 ± 2 °C

we can calculate the amount of dissolved metal; data are summarized in table 5.

A comparative analysis of the values presented in tables 3 and 5 notes that for samples of Alnico, gravimetric results show deviations up to ± 0.5 % compared to the values calculated from the results of XRF measurements. For NdFeB samples systematic deviations are positive, respectively the gravimetric results are systematically higher by 1.0 ÷ 1.5 % compared to the values calculated from the results of XRF measurements, which can be explained by the fact that the XRF technique is unable to determine the amount of boron dissolved. It also notes that in all environments the investigated magnetized samples show a corrosion rate of approx. twice higher than those unmagnetized.

In figure 5 it is presented the unmagnetized NdFeB sample, optical images before and after 120 h exposure at 30 ± 2 °C in saline gel type Czapek-Dox (*A* without sucrose), both in sterile condition and inoculated with spores of the filamentous *Aspergillus niger* mold.

The analysis of the images shown in figure 5 evidenced that the magnetic material NdFeB exposed in Czapek - Dox sterile saline gel was covered with corrosion products oxide (b), suggesting that the overall corrosion process (1) is dominated by (2):

$$_{2x}Fe + _{2(y+z)}H_2O \rightarrow {}_2Fe_xO_y(OH)_z + {}_{2y}H_2 \quad (2)$$

Also, when are exposed in the medium inoculated with *Aspergillus niger*, after 120 h of mold growth, were evidenced sample surface indentations, deep traces of corrosion - the presence of oxidized corrosion products being insignificant. These findings suggest that the biocorossion of NdFeB, according to the investigation data, is carried out in at least two main stages - a first stage of a chemical oxidation of the material forming the oxy - hydroxide complexes (2), followed by the second stage, in which the oxide corrosion products, under the action of the metabolism products of the *Aspergillus niger*, (firstly citric acid [11, 12, 35]) are dissolved (3) and form metal ions diffusing into the culture medium from which are extracted by hyphae and are retained in biomass (conidiophores and conidia) of the mold.

$$_2Fe_xO_y(OH)_z + {}_{2(2y+z)}R-COOH \rightarrow$$

$$\rightarrow {}_{2x}Fe^{2+} + {}_{2(2y+z)}R-COO^- + {}_{2(y+z)}H_2O \quad (3)$$

Representative images on observations of conducted microbiological monitoring are shown in figures 6-11.

Analyzing figure 6 and figure 8 it is evidenced that on the sterile environments there is no growth of microorganisms, but after an exposure time of 120 h, there is a color change from pale yellow to rusty brown of the gel, indicating a Czapek - Dox gel contamination with iron corrosion products.

A comparative analysis of the images in figure 7 and figure 9 shows that in the inoculated culture medium, the mould growth is being more intense and with a faster maturation (darker aspect) on the surface of the magnetized samples, according to those from [43]- the magnetic field stimulates the metabolism and thus the growth and maturation of filamentous mold *Aspergillus niger* (detailed in fig. 10 and fig. 11). With this finding,

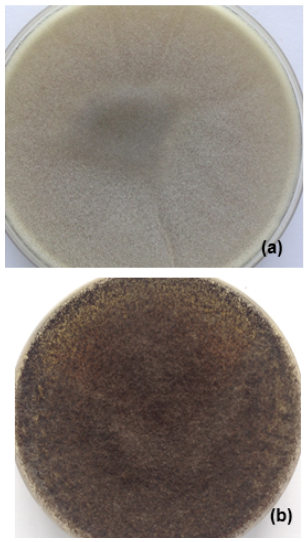

Fig. 7. The AlNiCo unmagnetized (a) and magnetized (b) sample exposed to the Czapek-Dox medium without sucrose inoculated with 10^6 spores/mL of *Aspergillus niger*, after 120 h incubation at 30± 2 °C

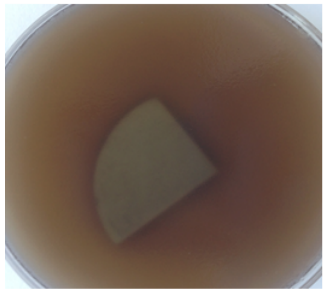

Fig. 8. The NdFeB unmagnetized sample exposed to the Czapek-Dox sterile medium without sucrose - 120 h at 30 ± 2°C

namely the increasing of the speed of formation of metabolism products, it can explain the corrosion rate of approx. twice higher than the unmagnetized samples - as shown in table 3 and table 5.

Conclusions

After processing the experimental data obtained through the techniques: XRD, XRF, gravimetry, optical microscopy and SEM, concerning the corrosion of magnetic material type Alnico and NdFeB (magnetized and unmagnetized

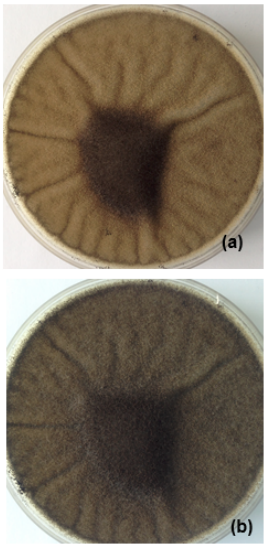

Fig. 9. The NdFeB unmagnetized (a) and magnetized (b) sample exposed to the Czapek-Dox medium without sucrose inoculated with approx. 10^6 spores/mL of *Aspergillus niger*, after 120 h incubation at 30± 2 °C

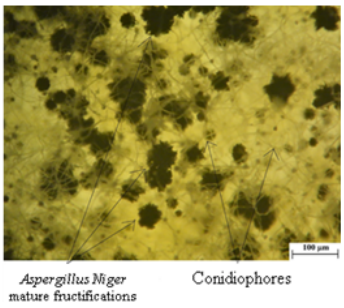

Fig. 10. Detail on the AlNiCo unmagnetized sample exposed to the Czapek-Dox medium without sucrose inoculated with approx. 10^6 spores/mL of *Aspergillus niger*, after 120 h incubation at 30± 2 °C

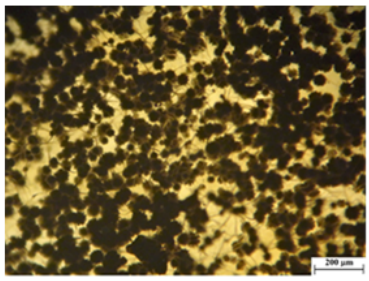

Fig. 11. Detail on the NdFeB magnetized sample exposed to the Czapek-Dox medium without sucrose inoculated with approx. 10^6 spores/mL of *Aspergillus niger*, after 120 h incubation at 30± 2 °C

states) exposed to the saline gel type Czapek Dox (in sterile condition, compared with the gel inoculated with spores of *Aspergillus niger*), were concluded the followings:

- the samples of Alnico and NdFeB magnetic materials showed an homogeneus structure, the specific main crysalline phases of FeCo and AlNi, respectively those of $Nd_2Fe_{14}B$ and α Fe are evently distributed;
- both, Alnico and NdFeB are thermodynamic instable – in sterile media, an appreciable corrosion rate it was achieved, respectively in unmagnetized state it was approx. $2.5 \cdot 10^{-6}$ g/h·cm^2 for Alnico and approx. $5.28 \cdot 10^{-6}$ g/h·cm^2 for NdFeB, and in magnetized state it was around 2 times higher (approx. 4.95 x10^{-6} g/h·cm^2, respectively approx. 11.1 x10^{-6} g/h·cm^2);
- due to the metabolic processes, after 120 h of the mould growth, the overall rate of corrosion, in the filamentous fungi *Aspergillus niger* inoculated Czapek - Dox media is significantly higher (5-6 times) than in sterile medium.

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