Preparation of high-gradient ferromagnetic matrices for magnetic filters (separators) with fractal surface structure by magnetoelectrolysis

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A method for steel etching under a magnetic field to obtain functional surfaces which are used as high-gradient ferromagnetic matrices of separators was describe.

Описан способ управления процессом травления стали под действием магнитного поля для получения функциональных поверхностей, которые применяются в качестве высокоградиентных ферромагнитных насадок сепараторов.

Magnetic separators are used in different branches, including waste water purification, biology and medicine, if magnetic properties are given to traditional sorbents and biosorbents being used to separate the target objects from the working media. The nano- and micromagnetic particles (for example, nanomagnetite Fe_3O_4) are used for the magnetic labeling; to minimize the amount of magnetic material used for magnetic labeling of the sorbents. Magnetic fields (MF) are created in the separator operating volume using high-gradient ferromagnetic matrices (HGFM). Today, different methods of HGFM preparation are known, including hydrostatic extrusion, nickel vapor deposition onto a stainless wire, controlled corrosion, etc. [1]. The hydrostatic extrusion and the vapor deposition provide preparation of HGFM having a high separation efficiency of the target objects, but are very costly. The controlled corrosion is environmentally hazardous and does not provide a high HGFM capacity due to unsufficient MF homogeneity.

The method of magnetically operated corrosion is proposed in this work. It is less environmentally hazardous, provides the obtaining of HGFM with a ramified dendritic surface structure, and thus enables to get a high matrix capacity. The ecological compatibility is provided by the possibility to select the accelerated etching rate mode using MF application [2]. This problem is solved by the study of the magnetic field influence on electrochemical reactions near the metal-electrolyte interface and is one of magnetoelectrolysis research branches. As a rule, a change of mass transfer in the electrolyte under the magnetic field influence causes a structure change of the metal surface and rate of electrochemical processes (etching, dissolution, deposition).

In [3, 4], the mechanism of magnetic field influence on the etching process in electrolytes is explained. The steel etching process in weak nitric acid solution has a self-oscillating character [2] which is connected with temporary periodic changes of the sample surface structure [5]. A rather weak permanent magnetic field (about 300 Oe) influences considerably the self-oscillating etching process [4].

In this work, the mode of magnetically controlled etching is considered for the obtaining of HGFM with optimum charac-

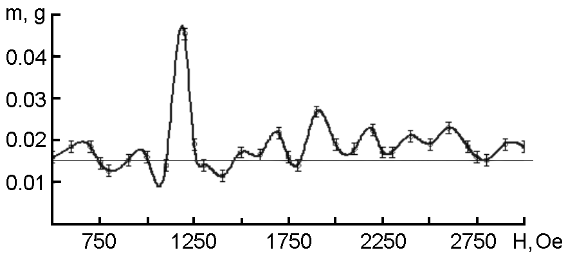

Fig. 1. The mass loss dependence on a magnetic field strength (etching duration is equal to 10 minutes).

teristics. Physical parameters affecting the formation of functional surface during magnetically controlled corrosion are studied, including influence of an external magnetic field, electrolyte concentration, etching duration, temperature, and the system geometry.

The quantitative characteristics of self-oscillating steel etching process were studied in this work depending on the external magnetic field strength. Corrosion of steel surface under a magnetic field and intensification possibility of steel etching process were studied in a weak nitric acid solution under a permanent magnetic field in the 0–3000 Oe range. The studies were carried out using the setup described in [2]. The magnetic field dependence of the etched steel mass was obtained for samples of steel (Ukrainian standard 14959-7, 65 G grade) shaped as $25\times20\times0.18$ mm^3 plates. The chemical composition of the steel was as follows: Si 0.17–0.37 %, Cu 0.20 %, Mn 0.90–1.20 %, Ni 0.25 %, P 0.035 %, Cr 0.25 %, S 0.035 %. Prior to the experiments, the sample was pre-treated by surface polishing by rotating felt disk (2000 rpm) and polishing paste. The plate was coated with nitro lacquer using a disk-shaped mask of 10 mm diameter on both sides to form the etching region. The plates were weighed on analytical balance at 10^{-4} g accuracy. The mass loss was averaged for 5 samples, the deviation from the average value did not exceed 5 %. The etching was conducted in weak nitric acid solution of 3.5 % concentration and 15 ml volume under a MF. This concentration was chosen because the etching rate under MF influence at higher concentrations did not provide the functional coating that satisfies the required HGFM criteria. The experiments were carried out at 22±2C°. The temperature control has shown that the temperature change by 1°C gives approximately 1 % of mass loss change. The permanent homogeneous magnetic field from 0 up to 3000 Oe was applied perpendicular to the sample surface and parallel to gravity force. Research was conducted on the device which includes the electromagnet, visualization system, and computer [2].

It has been determined in experiment that dependence of mass loss on the magnetic field strength has oscillating character (Fig. 1). The horizontal line corresponds to the mass loss value at $H = 0$ in Fig. 2a, the horizontal line corresponds to 0.015 g.

The etching surface microstructure was studied using a scanning electron microscope SEM-103 "Selmi". Fig. 2 shows a typical image of the corroded microstruc-

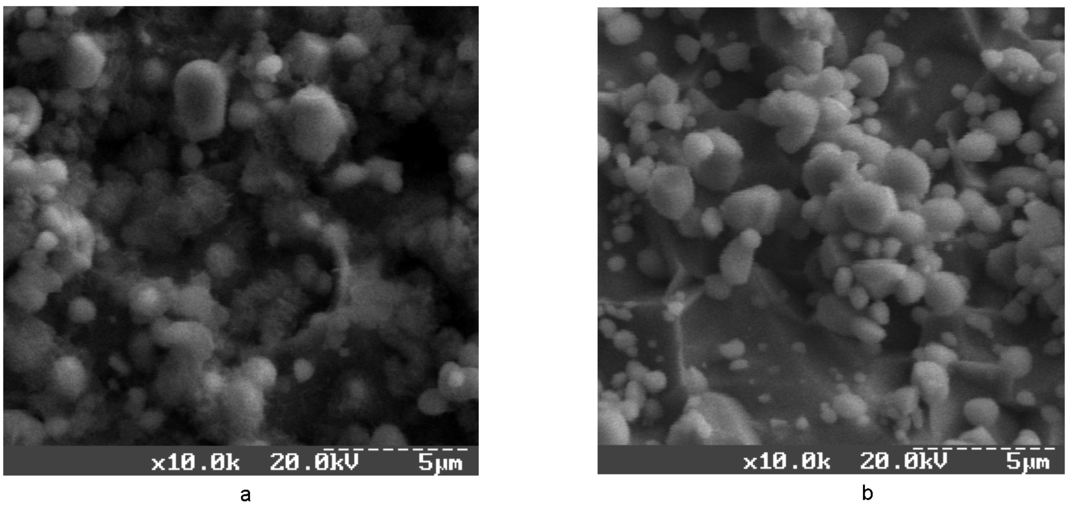

Fig. 2. Image of the corroded microstructure surface that corresponds to minimum (a) and maximum (b) of the mass loss dependence on a magnetic field strength. Etching duration is equal to 10 minutes: a) magnetic field is 2500 Oe, b) magnetic field is 1100 Oe.

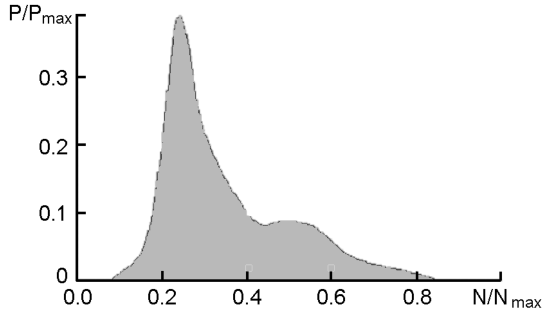

Fig. 3. Typical distribution of grey color of corroded surface. P is the number of pixels, P_{max} is total number of pixels, N is the number of grey color, N_{max} is the total number of grey colours.

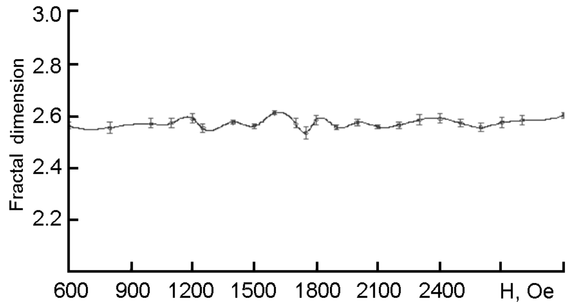

Fig. 4. Dependence of fractal dimension on the magnetic field strength.

ture surface after etching in a nitric acid solution which corresponds to the maximum (Fig. 2a) and minimum (Fig. 2b) in the mass loss dependence on a magnetic field.

The distribution of grey color in the corroded surface images shows two maxima, thus suggesting that the corroded surface has a two-component composition (Fig. 3). The bright grains correspond to the passivating film, the dark grey regions, to the steel surface. This statement is our hypothesis, it is to note that a further experimental study of the surface is necessary for a more detailed explanation.

The digital image processing was also used to study the etched surface. Gwyddion is a modular software for data analysis. It is used to analyze the images obtained by scanning electron and scanning probe microscopes (AFM, MFM, STM). One of the main advantages of that program is connection of the modules for the analysis of two-dimensional data that can be easily expanded by means of additional programs.

The result of fractal analysis using this software applying method of variation is represented in Fig. 4. The dependence was obtained by the etched surface image analysis at certain values of magnetic field strength. It is represented in Fig. 4. Each value of the magnetic field corresponds to eight surface images scanned at different magnifications. The images with 10 000, 5000 magnification factor have been chosen. The mean square error is 0.02.

As is known, the surfaces of solids are rough at a microscopic scale. This surface roughness can be described using fractal dimensionality. No natural length scale exists for fractal objects. They have the same look at various magnifications. The fractal dimensionality of solid objects influences significantly the physical phenomena that occur therein, in particular, the etching process in a magnetic field. Basing on that consideration, it can be stated that the fractal dimension affects the resistance (impedance) of the electrode and therefore the magnetic field affects the electrode resistance, so it can be concluded that the magnetic field affects the material etching process through the influence on fractal dimension.

Note that the sustained phase element makes a significant contribution to classical electrolyte-electrode systems [6], its presence is connected with the fractal surface geometry. The Cantor set forms the basis of the classical model of the surface, it is believed that the number of blocks $N = 2$ and the scale factor $r = 1/\xi$, $\xi > 2$. Taking this into consideration, the surface looks like a self-similar branching. Impedance of the system should satisfy the condition $Z(\omega \sim (i\omega)^{-\eta}$, where $\eta = 1 - \log_{\xi}2 = 3 - D$. D is fractal dimension, η is a quantity depending on the average number of branches.

Thus, it is shown in Fig. 4 that fractal dimension is maximum for the field value 1650 Oe because you can get the HGFM matrices of a magnetic filter with the most branched surface structure in this mode, and therefore with a maximum capacity.

These HGFM were examined to study the operating efficiency of separation. The effectiveness of HGFM was determined basing on the difference of the solution optical density prior to and after separation, which indicates a concentration change of target objects that are removed from the solution (magnetically controlled biosorbent is *S.cerevisiae* yeast used to purify the solution from copper ions). The liquid with magnetically controlled biosorbent was fed through a magnetic separator at 0,01 ml/s rate. The sampling of the purified liquid was carried out after 10, 30, 60, 150 minutes.

Table. The time dependence of the liquid separation efficiency at different concentration of yeast

Yeast concentration, cells/ml	Separation efficiency, %			
	10 min	30 min	60 min	150 min
$4 \cdot 10^6$	63	77	92.5	100
$6 \cdot 10^6$	44.4	63	77	92.5
$8 \cdot 10^6$	60	66.6	66.6	66.6

The time dependence of the liquid separation efficiency is presented in Table at different concentration of yeast. This HGFM is characterized by a high separation efficiency, thus, it is highly efficient. Tens, sometimes even hundreds of HGFM layers are used in industrial separators that allow to purify large volumes of liquids.

To conclude, the characteristics of HGFM functional surface have been found to depend on the formation parameters at magnetically controlled corrosion. The character of metal etching has quasi-periodic dependence on external magnetic field strength and the corroded surface consists of two components. The surface structure produced by etching depends on external magnetic field strength. Thus, application of an external magnetic field is a way to control the self-oscillating process of steel sample etching as well as the corroded surfaces structure at corresponding system parameters. Besides, fractal dimension and self-organized passive film contribute considerably to the resistance of the functional surface. Thus, the control of fractal dimension and relative component composition by means of the magnetic field allows to choose the optimal magnetic field influence mode for fabrication of magnetic filter HGFM for magnetically controlled biosorption.

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Отримання високоградієнтних феромагнітних насадок для магнітних фільтрів (сепараторів) з фрактальною структурою поверхні методом магнітоелектролізу

С.В.Горобець, О.Ю.Горобець, О.М.Било, М.М.Потьомкін

Розглянуто спосіб керування процесом травлення сталі під дією магнітного поля для отримання функціональних поверхонь, що застосовуються як високоградієнтні феромагнітні насадки сепараторів.