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EFFECT OF THE TEST REGIMES ON THE CORROSION RESISTANCE OF THE Zn-1Fe-1Mg ALLOY

Elmira Damirovna Abdrakhmanova [1a], Elvira Dinifovna Khafizova [1], Milena Vladislavovna Polenok [1], Rinat Kadykhanovich Islamgaliev [1], Hakan Yilmazer [2, 3]

[1] Ufa University of Science and Technology, 32 Zaki Validi st., Ufa 450076 Russia
[2] Yildiz Technical University, Esenler 34220, Istanbul, Turkey
[3] Health Biotechnology Joint Research and Application Center of Excellence, Esenler 34220, Istanbul, Turkey
[a] elmira.abdr2019@mail.ru

ABSTRACT

Bioresorbable zinc alloys are ever more often regarded as promising materials for medical implants and vascular stents since they have a lower corrosion rate in a physiological environment in comparison with magnesium alloys. At the same time, products made of zinc alloys must have a controlled corrosion rate to provide the required time for the recovery of the body. It is known that in determining the corrosion rate, an important role is played by the choice of the test method and its parameters (corrosive environment, environment temperature, the sample's exposure time in the corrosive environment). In the present study, the gravimetric method was used, based on a precise measurement of the substance mass prior to and after testing. The surface of the samples subjected to corrosion tests was investigated using scanning electron microscopy and energy-dispersive analysis. The aim of the present study is to reveal the effect of the samples' exposure time in a corrosive environment, as well as the frequency of surface cleaning, on the corrosion resistance of the Zn-1Fe-1Mg biodegradable zinc alloy. It is shown that under the same test conditions but different frequencies of surface cleaning, the corrosion rates may differ by a factor of 3.8.

KEYWORDS

Corrosion rate; gravimetric method; zinc alloy.

ВЛИЯНИЕ РЕЖИМОВ ИСПЫТАНИЙ НА КОРРОЗИОННУЮ СТОЙКОСТЬ СПЛАВА Zn-1Fe-1Mg

Эльмира Дамировна Абдрахманова [1a], Эльвира Динифовна Хафизова [1], Милена Владиславовна Поленок [1], Ринат Кадыханович Исламгалиев [1], Хакан Йылмазер [2, 3]

[1] Уфимский университет науки и технологий, 450076, Россия, Уфа, ул. Заки Валиди, 32
[2] Технический университет Йылдыз, 34220 Турция, Стамбул, Эсенлер
[3] Центр совместных научных исследований и применений в области биотехнологий для здравоохранения, 34220 Турция, Стамбул, Эсенлер
[a] elmira.abdr2019@mail.ru

АННОТАЦИЯ

Биорезорбируемые цинковые сплавы все чаще рассматриваются как перспективные материалы для медицинских имплантатов и сосудистых стентов, поскольку они обладают меньшей скоростью коррозии в физиологической среде по сравнению с магниевыми сплавами. Вместе с тем изделия, сделанные из цинковых сплавов, должны обладать контролируемой скоростью коррозии для обеспечения нужного временного интервала для восстановления организма. Известно, что при определении скорости коррозии важную роль играет выбор метода испытаний и его параметров (коррозионная среда, температура окружающей среды, время выдержки образца в коррозионной среде). В настоящей работе был использован гравиметрический метод, основанный на точном измерении массы вещества до и после испытаний. Поверхность образцов, подвергнутых коррозионным испытаниям, исследовали с применением методов сканирующей электронной микроскопии и энергодисперсионного анализа. Целью настоящей работы является определение влияния времени выдержки образцов в коррозионной среде, а также периодичности очистки поверхности на коррозионную стойкость биодеградируемого цинкового сплава Zn-1Fe-1Mg. Показано, что при одинаковых условиях испытаний, но при разной периодичности очистки поверхности скорость коррозии может отличаться в 3,8 раза.

КЛЮЧЕВЫЕ СЛОВА

Скорость коррозии; гравиметрический метод; цинковый сплав.

Introduction

Zinc alloys are known to be subject to corrosion due to chemical or electrochemical interaction with a physiological environment. At the same time, the fast interaction of zinc alloys with a physiological environment can be used for medical purposes to create biodegradable medical implants. In this case, the implant material must have certain strength and functional properties, be harmless for the body and degrade with a controllable rate in order to provide the required time for the recovery of the body. The material must retain its strength and functional characteristics in the presence of a corrosive environment since the human body is saturated with various ions, in the first place, Cl^-, which makes the environment rather aggressive. When studying bioresorbable metallic materials, the following test methods are used: the gravimetric (weight) method [1–7], the hydrogen evolution method [4–7] and the potentiodynamic method [2, 8–10]. The gravimetric method is one of the simplest methods and is based on a precise measurement of the substance mass prior to and after testing. The choice of the corrosive environment, the environment temperature, the sample's exposure time in the corrosive environment play a significant role in the test results.

Corrosion processes start from the surface of metallic products, and in some cases, they combine with the action of different mechanical factors (tensile or alternating stresses, friction, impact, etc.).

The corrosion properties of biodegradable materials are tested under the conditions similar to those of the human body, such as the temperature, composition of the corrosive environment and the pH level. In such studies, when selecting the corrosive environment, often applied are Hanks' solution (in one liter of distilled water: 8 g NaCl, 0.4 g KCl, 0.12 g $Na_2HPO_4 \cdot 12H_2O$, 0.06 g KH_2PO_4, 0.2 g $MgSO_4 \cdot 7H_2O$, 0.35 g $NaHCO_3$ and 0.14 g $CaCl_2$; the pH value of the solution is 7.4) [4, 9, 10, 11] and Ringer's solution (8.60 g NaCl, 0.30 g KCl and 0.33 g $CaCl_2$ per one liter of water) [1, 3, 5, 12], and less often applied is a simulated body fluid – SBF (a solution with an ion concentration close to the ion concentration in the human body plasma) [13–15]. The ion concentration in the solutions is presented in Table 1.

Table 1. Ion concentrations (mmol) in corrosive environments

Таблица 1. Концентрации ионов (мМоль) в коррозионных средах

	Na	K	Mg	Ca	Cl	HCO.3	HPO.4	SO.4
Body plasma / Плазма крови	142	5	1.5	2.5	103.0	27.0	1.0	0.5
SBF	142	5	1.5	2.5	148.8	4.2	1.0	0
Ringer's solution / Раствор Рингера	147	4	–	2.25	155.6	–	–	–
Hanks' solution / Раствор Хэнкса	146	5	2.4	18.9	160	4.1	6.2	0.5

The exposure time in a corrosive environment in finding the corrosion rate of bioresorbable metallic materials by the gravimetric method influences the test results. For example, there are intermittent tests [1, 3, 16–18], where samples, owing to frequent mass measurements, enable evaluating the sample's corrosion rate and establishing a certain dependence. Many studies also practice a long-term, for 7 and more days, exposure in a corrosive environment where the pH is maintained at 7.2–7.8 by means of replacing the corrosive environment [2, 4–7, 19, 20].

Therefore, the aim of this study is to reveal the differences in intermittent and long-term exposures of samples in a corrosive environment when finding the corrosion rate of bioresorbable materials by the gravimetric method through the example of the Zn-1Fe-1Mg zinc alloy.

1. Materials and Methods

The material of the study is the Zn-1Fe-1Mg zinc alloy that was smelted in a chamber furnace in a graphite crucible 20 mm in diameter with a closed lid at a temperature of 580 °C. Further, the samples were subjected to homogenizing annealing at a temperature of 300 °C for 12 hours. Chemical composition was found using a Thermo Scientific ARL Optim'X X-ray fluorescent spectrometer.

Using an ARTA 120 CNC electrical discharge machine, discs with a diameter of 20 mm and thickness of 1.8 mm were cut. The preparation of samples for structural studies included mechanical grinding and polishing on diamond suspensions with abrasive particle sizes of 1 and 0.5 μm.

Corrosion tests were performed on three samples having the same shape, sizes and surface preparation. Using a drilling machine, holes Ø1 mm were drilled in the upper part of the samples, serving for the hanging of the samples in a corrosive environment.

Corrosion tests were conducted by the gravimetric method, specifically, using intermittent and long-term exposure in a corrosive environment for 60 days.

The intermittent test method consisted in sample exposure in a corrosive environment – Ringer's solution (composition: 8.6 g/l NaCl, 0.3 g/l KCl, 0.25 g/l $CaCl_2$, pH 7). The tests were conducted at a constant temperature of 38±1 °C. The samples were weighed and photographed before and after cleaning from corrosion products every two days. The cleaning from corrosion products was performed in accordance with ASTM G1-03-E in a solution of chromium(VI) oxide (200 g of the reagent per 1000 ml of distilled water), then in distilled water with the use of a KAISI-105 ultrasonic bath. After the cleaning, the samples were dried and weighed on an EJ-123 electronic scale with measurement accuracy up to 0.01 mg. After the cleaning from corrosion products and weighing, the samples were again subjected to the action of the corrosive environment. The surface of the samples was studied by scanning electron microscopy and energy-dispersive analysis every 14 days.

In the long-term exposure, the corrosion tests included exposure in Ringer's solution for 10, 20, 30, 40, 50, and 60 days. There were two samples per each exposure. The tests were conducted at a constant temperature of 38±1 °C.

During the tests, the pH was maintained at 7–7.8 by means of measuring the pH and replacing the corrosive environment without the extraction of the samples. After the specified time ran out, the samples were extracted from the corrosive environment, dried and photographed with corrosion deposits on the sample surface, and weighed. Further, the material was cleaned in a solution of chromium(VI) oxide, then in distilled water using a KAISI-105 ultrasonic bath. After that, the samples were weighed and photographed once again.

The corrosion rate (*CR*), mm/year, was calculated in accordance with ASTM G3–63592 by the formula:

$$CR = \frac{87.6\ (M_0 - M_1)}{St\rho},$$

where *CR* is the corrosion rate, mm/year; *S* is the sample surface area, cm^2; M_0 is the initial mass, mg; M_1 is the mass after immersion, mg; *t* is the exposure time, h; ρ is the metal density, g/cm^2.

Mass loss in % was calculated by the formula:

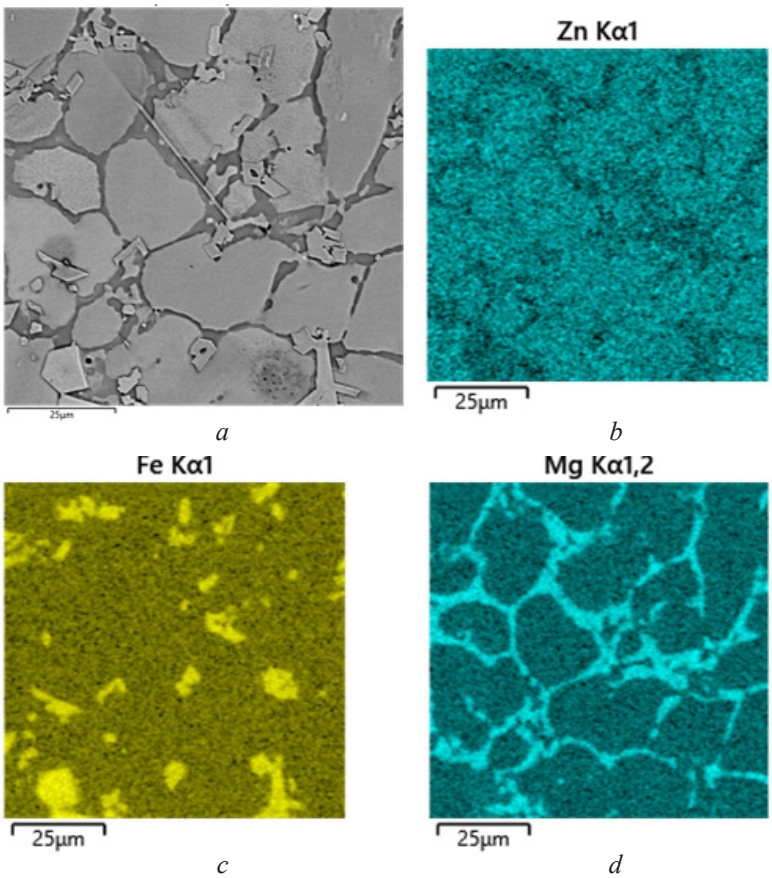
$$ML = \frac{(M_o - M_1)}{M_o} \cdot 100\%,$$

where *ML* is the mass loss, %.

Afterwards, the surface of the corroded samples was studied using a JSM-6490LV scanning electron microscope.

2. Results and Discussion

Fig. 1 shows the structure of the initial cast material after a homogenizing annealing. Presumably, the phases Zn, Mg_2Zn_{11} and $FeZn_{13}$ are present in the alloy, which is in agreement with the phase diagram of Zn-Mg [21] and Zn-Fe [22]. According to enegy-dispersive X-ray spectroscopy (EDS), the Mg_2Zn_{11} phase is located at the boundaries of the Zn phase (Fig. 1, *d*). Calculations show that the average grain diameter of the Zn phase is 60 μm, while the $FeZn_{13}$ phase size varies from 1.5 to 30 μm (Fig. 1, *a*, *b*).



a *b* *c* *d*

Fig. 1. Structure of the Zn alloy in the initial state (*a*), and the EDS analysis of the alloy (*b, c, d*)

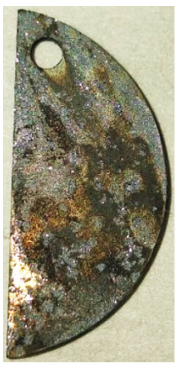
Рис. 1. Структура цинкового сплава в исходном состоянии (*a*) и ЭДС анализ сплава (*b, c, d*)

Table 2 presents the results of photographing after the intermittent and long-term exposures of the Zn alloy in Ringer's solution. In the intermittent test method, the sample surface was visually little different from that at the moment of immersion; it did not become covered with degradation products, and therefore it was not photographed prior to cleaning.

The corrosion damage of the Zn alloy is conditioned by its non-uniformity in terms of its chemical and phase composition. Table 2 shows the effect of Ringer's solution on the structure of the initial alloy during a period of 60 days. Here, we observe an increase in the corroded surface area with increasing duration of stay in the solution, as well as the penetration of corrosion into the depth of the material, which correlates with the mass loss of the samples presented in Table 3. In the intermittent method, the mass loss of the samples is much smaller than that after long-term exposure (Table 3). In the former case, the corrosion rate is 0.044 mm/year on average, and in the latter case, for long-term exposure for 10 days we observe the maximum corrosion rate of 0.315 mm/year which decreases two-fold after an exposure for 60 days. Thus, as a result of the formation of a passivating film, the rate of metal dissolution in the case of long-term exposure decreases, but still remains higher than that in the case of the intermittent method.

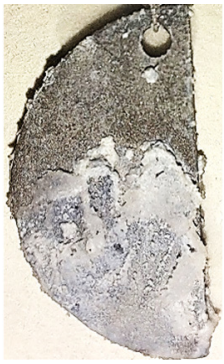
Table 2. View of Zn-1Fe-1Mg samples after testing in Ringer's solution

Таблица 2. Вид образцов Zn-1Fe-1Mg после испытаний в растворе Рингера

	Intermittent exposure / Прерывистая выдержка	Long-term exposure / Длительная выдержка	
	After cleaning / После очистки	Before cleaning / До очистки	After cleaning / После очистки
10 days / 10 дней			
20 days / 20 дней			

The end of table 2

Окончание таблицы 2

	After cleaning / После очистки	Before cleaning / До очистки	After cleaning / После очистки
30 days / 30 дней			
40 days / 40 дней			
50 days / 50 дней			
60 days / 60 дней			

During the stay of the samples in Ringer's solution there was no gas emission, which is in agreement with [23, 24]. During the stay of the samples in Ringer's solution the pH gradually increased with time, which indicates the absorption of hydrogen ions in the solution, therefore during long-term exposure the solution was often renewed during the experiment, whereas in the intermittent study it was replaced every 48 hours. During intermittent exposure, on the sample surface there formed a film (Fig. 2) where there were predominantly corrosion deposits containing Na, Zn, O.

Table 3. Mass loss of the samples during the corrosion tests, %

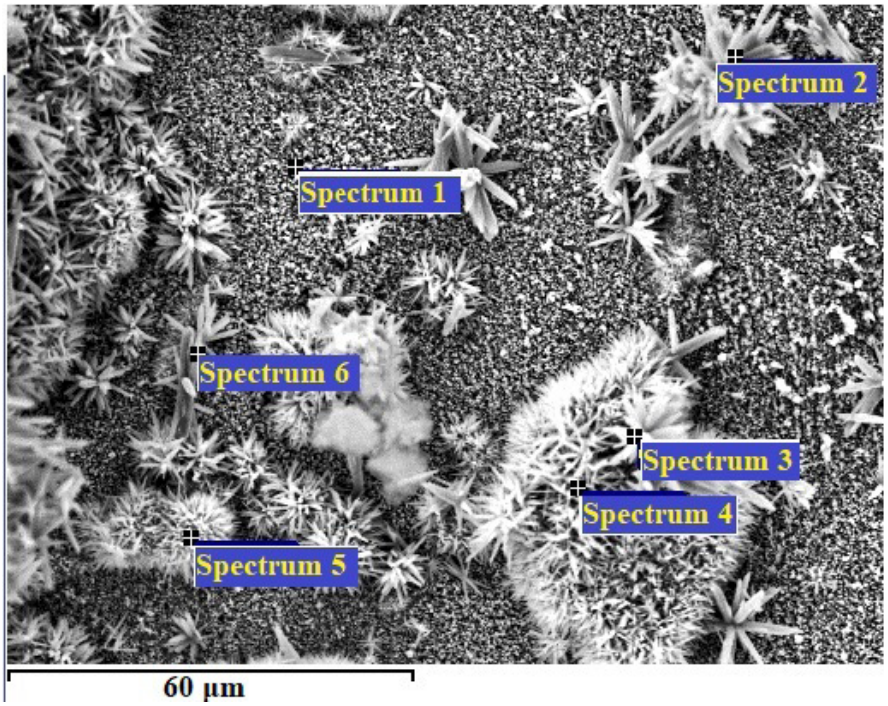
Таблица 3. Потеря массы образцов во время коррозионных испытаний, %

	10 days / 10 суток	20 days / 20 суток	30 days / 30 суток	40 days / 40 суток	50 days / 50 суток	60 days / 60 суток
Intermittent exposure / Прерывистая выдержка	0.31%	0.41%	0.71%	0.89%	1.06%	1.48%
Long-term exposure / Длительная выдержка	2.27%	2.05%	3.97%	5.43%	4.35%	7.30%

Table 4. Corrosion rate, mm/year

Таблица 4. Скорость коррозии, мм/год

	10 days / 10 суток	20 days / 20 суток	30 days / 30 суток	40 days / 40 суток	50 days / 50 суток	60 days / 60 суток
Intermittent exposure / Прерывистая выдержка	0.056	0.03	0.045	0.042	0.040	0.047
Long-term exposure / Длительная выдержка	0.315	0.247	0.258	0.198	0.182	0.179

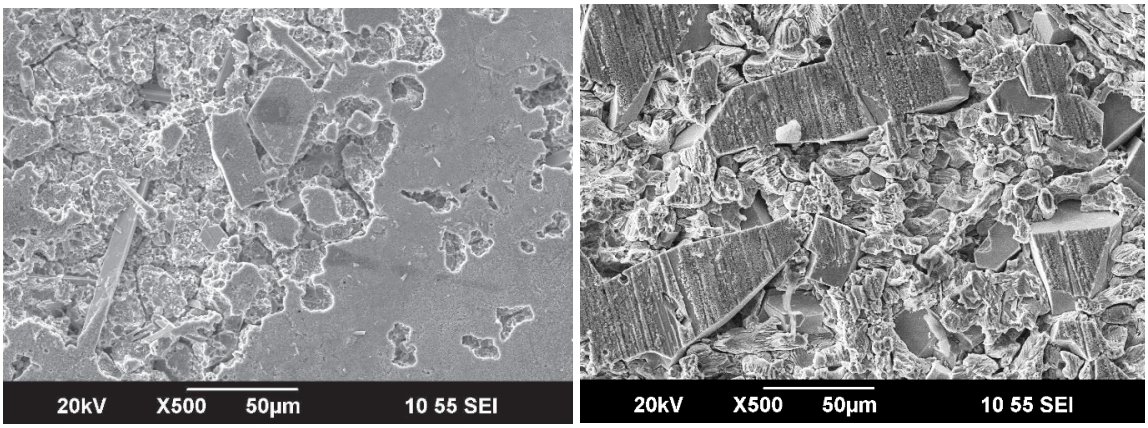


Spectrum	O	Na	Mg	K	Ca	Fe	Zn
Spectrum 1	17.51	18.83	0.00	0.12	0.00	0.00	63.55
Spectrum 2	14.69	15.67	0.00	0.21	0.00	0.00	69.42
Spectrum 3	8.19	5.86	0.00	0.00	0.00	0.00	85.95
Spectrum 4	8.88	6.09	0.00	0.00	0.00	0.29	84.74
Spectrum 5	8.62	8.54	0.00	0.00	0.17	0.00	82.67
Spectrum 6	13.12	10.00	0.00	0.00	0.00	0.39	76.49

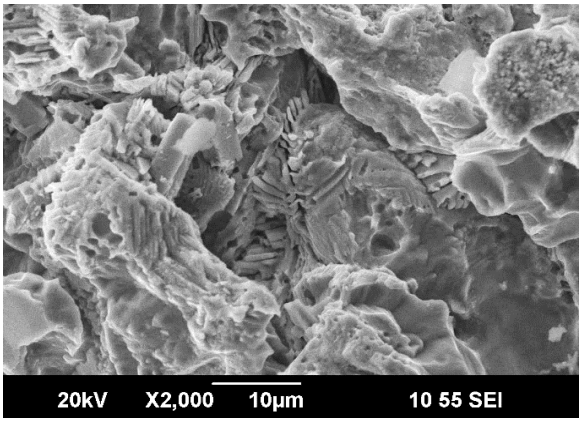
Fig. 2. Image of the sample surface obtained by a scanning electron microscope (SEM) after exposure for 20 days without cleaning

Рис. 2. Изображение поверхности образцов на растровом электронном микроскопе (РЭМ) после выдержки в течение 20 дней без очистки

Fig. 3 displays the images obtained by a scanning electron microscope. During the corrosion tests by the intermittent method for a period of 20 days, the dissolution of the whole surface adjacent to the $FeZn_{13}$ particles was observed. There can be seen in Fig. 3, *c* in large magnifications plates of the eutectic phase where the Mg_2Zn_{11} plates turned out be dissolved as a result of corrosion. On day 60 of the corrosion tests, the degradation of the $FeZn_{13}$ particles that had previously dissolved the alloy's other phases while remaining fully intact (Fig. 4), became noticeable.



a *b*



c

Fig. 3. Image of the sample surface obtained by a SEM after exposure for 20 days after cleaning:
a – intermittent method; b, c – long-term exposure method

Рис. 3. Изображение поверхности образцов на РЭМ после выдержки в течение 20 дней после очистки:
a – прерывистый метод; b, c – метод с длительной выдержкой

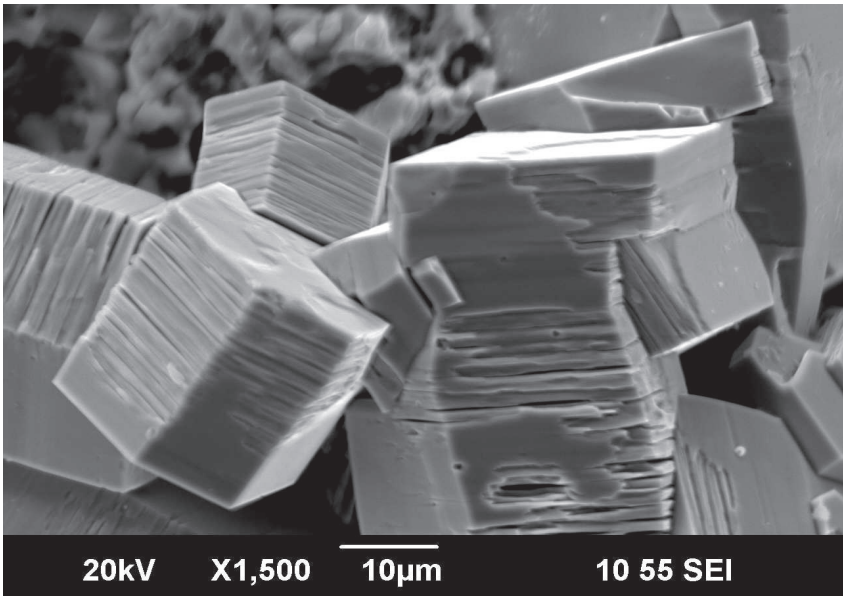


Fig. 4. Image of the sample surface after a long-term exposure for 60 days

Рис. 4. Изображение поверхности образцов после длительной выдержки в течение 60 дней

The corrosion of the Zn alloy may be conditioned by the presence of particles (Mg_2Zn_{11} and $FeZn_{13}$) which have a more electronegative potential in comparison to pure Zn. The corrosion damage of the Zn alloy depends on the activity of a metal that the particles have in their composition, i.e., on their ability to part with electrons at the outer electron shell. The degree of activity of a metal is characterized by the respective electrochemical series of metals. Among the Zn alloy's elements Zn (−0.76), Fe (−0.440), Mg (−2.363), the most active is Mg. Corrosion occurs in places where Mg is concentrated since it has a more electronegative potential and therefore acts as an anodic center and dissolves faster [25, 26]. The $FeZn_{13}$ particles are cathodic and accelerate the dissolution of the Zn matrix.

Conclusions

The studies have shown a 3.8-fold difference in the corrosion resistance after intermittent and long-term exposures of the Zn alloy in Ringer's solution as a result of the testing by the gravimetric method.

The formation of a passivating film during long-tern exposure decelerates the metal dissolution almost by a factor of 2. However, the corrosion rate values are higer than those for the intermittent method.

The intermittent method enables evaluating the corrosion rate for any time durtion. However, this method does not take into account the formation of a protective film and the change in corrosion rate due to surface passivation.

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