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Study of corrosion resistance of composite material samples for oil equipment

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Исследование коррозионной стойкости образцов композиционных материалов для нефтяного оборудования

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aluminum, composite materials, chromium carbide, corrosion, magnesium, oil and gas field equipment, steel, density, hardness.

The use of instrumental and structural metal matrix materials reinforced with various functional fillers is relevant to increase the corrosion resistance and operational reliability of oil and gas field equipment. A study of the corrosion resistance of a number of aluminum matrix dispersion-reinforced composites containing up to 10 % (wt.) chromium carbide and magnesium was carried out. Experimental samples of composite materials were synthesized by powder metallurgy by sintering compacted starting materials in an alundum crucible under a coal powder at a temperature of 640 °C for 1 hour. The phase composition of the resulting composites was studied on an XRD 7000 X-ray diffractometer (Shimadzu) with an attachment for X-ray spectral microanalysis. Photographs of the microstructure and distribution maps of chemical elements were obtained using a VEGA LMS scanning electron microscope. To measure the hardness of the composites, an ITV-30-AMV hardness tester was used. Corrosion tests were carried out at room temperature for 504 hours. The corrosive medium was a model electrolyte solution without forced circulation, containing 30 g/l sodium chloride and the addition of acetic acid to pH = 4.0. It has been established that the corrosion rate (P, mm/year) of the samples decreases almost twofold in proportion to the increase in the content of chromium carbide in the matrix aluminum. Additional alloying of composites with magnesium increases the corrosion rate relative to pure aluminum in proportion to the increase in magnesium content. A continuous uniform distribution of corrosion damage to the metal surface of the samples and a decrease in their hardness after corrosion resistance tests are observed in all cases. The research results indicate increased corrosion resistance of the Al-Cr_3C_2 composite, which is important for its applications as part of equipment operated in a corrosive environment.

Ключевые слова:
алюминий, композиционные материалы, карбид хрома, коррозия, магний, нефтегазопромысловое оборудование, сталь, плотность, твердость.

Для повышения коррозионной стойкости и эксплуатационной надежности нефтегазопромыслового оборудования является актуальным применение инструментальных и конструкционных металломатричных материалов, армированных различными функциональными наполнителями. Выполнено исследование коррозионной стойкости ряда алюмоматричных дисперсно армированных композитов, содержащих до 10 мас. % карбида хрома и магния. Экспериментальные образцы композиционных материалов синтезировали методом порошковой металлургии, спекая уплотненные прессованием исходные материалы в алундовом тигле под угольной засыпкой при температуре 640 °С в течение 1 ч. Фазовый состав полученных композитов изучали на рентгеновском дифрактометре XRD 7000 (Shimadzu) с приставкой для рентгеноспектрального микроанализа. Фотографии микроструктуры и карты распределения химических элементов получали на электронном сканирующем микроскопе VEGA LMS. Для измерений твердости композитов использовали твердомер ИТВ-30-АМВ. Коррозионные испытания проводили при комнатной температуре в течение 504 ч. Коррозионной средой служил модельный раствор электролита без принудительной циркуляции, содержащий 30 г/л хлорида натрия и добавку уксусной кислоты до pH = 4,0. Установлено, что скорость коррозии (П, мм/год) образцов уменьшается практически в два раза пропорционально повышению содержания карбида хрома в матричном алюминии. Дополнительное легирование композитов магнием увеличивает скорость коррозии относительно чистого алюминия пропорционально повышению содержания магния. Во всех случаях наблюдается сплошное равномерное распределение коррозионного поражения металлической поверхности образцов и снижение их твердости после испытаний на коррозионную стойкость. Результаты исследований свидетельствуют о повышенной коррозионной стойкости композита Al-Cr_3C_2, что важно для его применений в составе оборудования, эксплуатируемого в коррозионно-активной среде.

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Introduction

Oil industry is a significant element of economic development in many countries. A large number of different equipment is used for oil exploration and its subsequent refining, including those made of metal-based materials. The use of such materials for tool-making and structural purposes is associated with the problem of metal corrosion. The development of corrosion process is caused by redox reactions in which a metal, as a result of interaction with a substance from its environment, turns into an undesirable compound and loses its original functional properties. In the presence of aggressive environment impact on metal material without special protection measures the probability of equipment failure caused by corrosion can be very high [1–4]. Moreover, damage to individual components of equipment can lead to its failure as a whole, which will disrupt production and lead to serious economic losses [5, 6].

As a result of corrosion about 10 % of the total metal production in the world is lost [7], which affects manufacturing plants, transport, nuclear and military facilities [3, 8–10]. The importance of the corrosion problem for the oil and gas sector is confirmed by the large number of scientific papers posted from 2000 to 2020 on the WOS SCIE, SSCI, A & HCI and CPCI-S search platforms [9, 11]. Based on the studies conducted [7, 12] it is noted that annually the economic losses associated with corrosion damage of equipment in each country vary between 2–5 % of their GDP [3, 8, 10, 12]. In addition, up to 20–30 % of equipment downtime also occurs due to corrosion.

The IMPACT study showed [3, 12] that by implementing corrosion control techniques significant savings of 15 to 35 % of the cost of damage can be achieved, which is equivalent to a reduction in global corrosion costs.

Handling a variety of corrosive substances requires the right material of construction. The oil and gas industry has traditionally used steel throughout the hydrocarbon value chain, from well construction and drilling rigs to onshore pipelines and storage tanks. Nevertheless, steel being a universal structural has certain disadvantages, including the potential for corrosion and the significant weight of equipment madeof steel.

In the search for high-tech and low-cost alternative for steel as the main structural material many researchers have considered composite systems [2, 3, 12, 15–17]. Recently, in this direction considerable efforts have been made to develop various compositions of metal matrix composites (MMCs). Among MMCs, aluminium-based composites are considered to be the most promising structural materials due to their high corrosion resistance, wear resistance, specific modulus of elasticity and low density. Aluminium MMCs can be manufactured by many processes using different reinforcement particles such as borides, carbides, oxides, nitrides and their combinations. Due to the presence of stable reinforcing particles aluminium MMCs show excellent mechanical and wear-resistant characteristics [18, 19].

Application of composites results in lower overall structural weight, provides better corrosion resistance, reduces overall operating costs and provides greater design flexibility. Currently, composites are beginning to be used in the oil and gas industry for the production of risers, pressure vessels, tanks, drill pipes and pipeline structures, decks, etc.

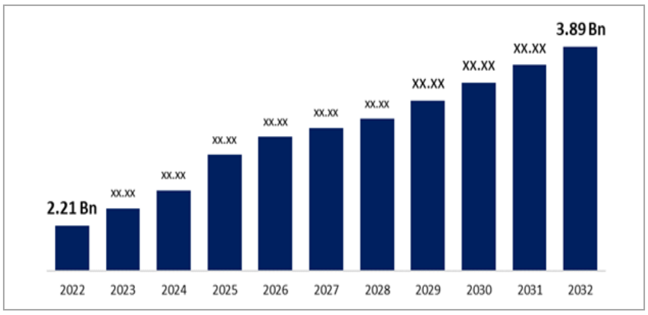

Fig. 1. Global market of composites in oil and gas industry [20]

The global Oil & Gas composites market is expected to exceed USD 3.89 billion by 2032, at a compound annual growth rate of 5.8 % during the period 2022 to 2032. [20]. Such competitive properties of composite materials as light weight, corrosion resistance, good mechanical strength can support this growth rate (Fig. 1).

Composites with metal, epoxy, phenolic and polymer matrix are already used in oil and gas industry [21–23]. Glass fibre, carbon or aramid fibre are most often used as fillers. A significant share of metal matrix materials are composites based on aluminium reinforced with graphite [24], refractories [25], carbides [26, 27] and other fillers [28–30].

Methodology of research

Aluminium powder PA-3 with purity of 99 % (GOST 6058-73) and average particle size of 250–450 mkm was used for fabrication of aluminomatrix composites.

Powders of chromium carbide (Cr_3C_2) (GOST 28377-89) were used as disperse filler with particle size of 40–100 microns and magnesium oxide (GOST 4526-75) with particle size of 10–50 microns.

Suspensions of aluminium, chromium carbide and magnesium oxide powders, taken in a given ratio, were mixed with a mechanical agitator. The obtained homogeneous powder mixture was cold pressed in a mould made of hardened steel on a uniaxial press PGR400 at a pressure of 980 MPa and holding time for 2 min. Industrial oil was used as a lubricant for the mould walls. The compacted samples were sintered under a layer of coal powder at 640 °C for 1 hr [31].

The phase composition of the obtained composite was studied on X-ray diffractometer XRD 7000 (Shimadzu) with XFlash Detector 630M (Bruker Nano GmbH). Photographs of the microstructure of the alumina matrix composite material and maps of chemical elements distribution were obtained on a TESCAN VEGA LMS electron scanning microscope. Visual study of the structure was carried out on an optical microscope Olympus BX61.

Vickers hardness measurements were carried out on an ITV-30-AM hardness tester under a load of 25 N with a delay of 15 s. According to GOST 2999-751 measurements were carried out at five points along the surface of the samples.

Corrosion tests were carried out under static conditions, without forced circulation of the corrosion medium. A model electrolyte solution containing 30 g/l NaCl and acetic acid added to pH = 4.0 was used as the test corrosion medium. The final corrosion rate values were calculated as the arithmetic mean of the results of three corresponding tests with a relative error not exceeding 5 %. The test base was 504 h, temperature – +22 °C, volume of solution in the cell with three samples – 500 ml.

Before testing, the mould samples were polished to a mirror shine, washed with ethyl alcohol and, after drying, weighed to an accuracy of ±0.0001 g. Then three samples of the same composition were placed in one cell, ensuring complete immersion in the solution and the absence of contact between the surface of the samples. To fix (suspend) the samples, a thread from synthetic material was used [32].

After the end of testing, the samples were removed from the cell, washed with warm distilled water and ethyl alcohol, dried and weighed. Additionally, it was recorded the appearance of the samples and assessed the condition of their surface.

Discussion of the results

The characteristic properties of aluminium and aluminium-based alloys are good machinability, corrosion resistance, low density, high thermal conductivity. The main disadvantage of aluminium is low hardness and strength. To increase physical and mechanical properties, the matrix is reinforced with various fillers. Sometimes reinforcement with one functional substance may not give the required properties for specific applications of alumina matrix materials. In this case, two or more different reinforcing components are used to create a hybrid composite system. Studies [33] have shown that such hybrid composite systems have better properties than conventional composites. Their advantages over conventional composites are balanced strength and stiffness, increased impact and bending strength, high and stable thermal resistance, lower cost, better fatigue resistance and improved fracture toughness, less sensitivity to cutting and ability to inhibit crack growth. Therefore, in the present work, mono- and poly-armouring of aluminium matrix has been considered.

To obtain alumina matrix composites it was used the method of powder metallurgy which makes possible to synthesise a composite material with a uniform distribution of filler particles in the volume of the metal matrix. The use of aluminium powder with high content of pure metal allowed levelling the influence of impurities on the formation of structure, properties and interfacial bonds between the reinforcing particles and the substrate.

The surface morphology of the obtained samples and the distribution of elements are shown in Fig. 2. In all cases, a smooth and relatively smooth surface is observed. According to the obtained maps, the distribution of elements over the volume of samples is quite uniform.

Density and porosity of the material are among the main parameters considered in its application. The density of samples was determined by hydrostatic weighing method. The density and porosity of the samples increased with the growth of the filler (chromium carbide) fraction. In the hybrid composites, increasing of Mg proportion from 5 to 10 % (with chromium carbide in the amount of 5 %) led to a decrease in the density of the samples and an increase in porosity (Fig. 3). At the same time, stirring and strain hardening lead to the appearance of electrochemical inhomogeneities, causing the appearance of local corrosion. As a consequence, there is some compromise between mechanical and corrosion properties in aluminium alloys.

The corrosion resistance of cast aluminium alloys has been investigated in many works, e.g. in [34–36]. From the point of view of the kinetics of corrosion processes, aluminium alloys are a short-circuited multi-electrode system [37]. The presence of dispersed phase leads to the presence of zones with different potentials on the alloy surface. In the surface zones that have reached the breakdown potential and have increased adsorption activity and electrical conductivity, active ions are adsorbed. As a result, a metal-anion complex is formed, which passes into solution and displaces oxygen. Since the solubility of many alloying elements in aluminium is rather limited, the formation of secondary phases leads to local corrosion, what is inevitable when traditional processing methods are used[31, 32].

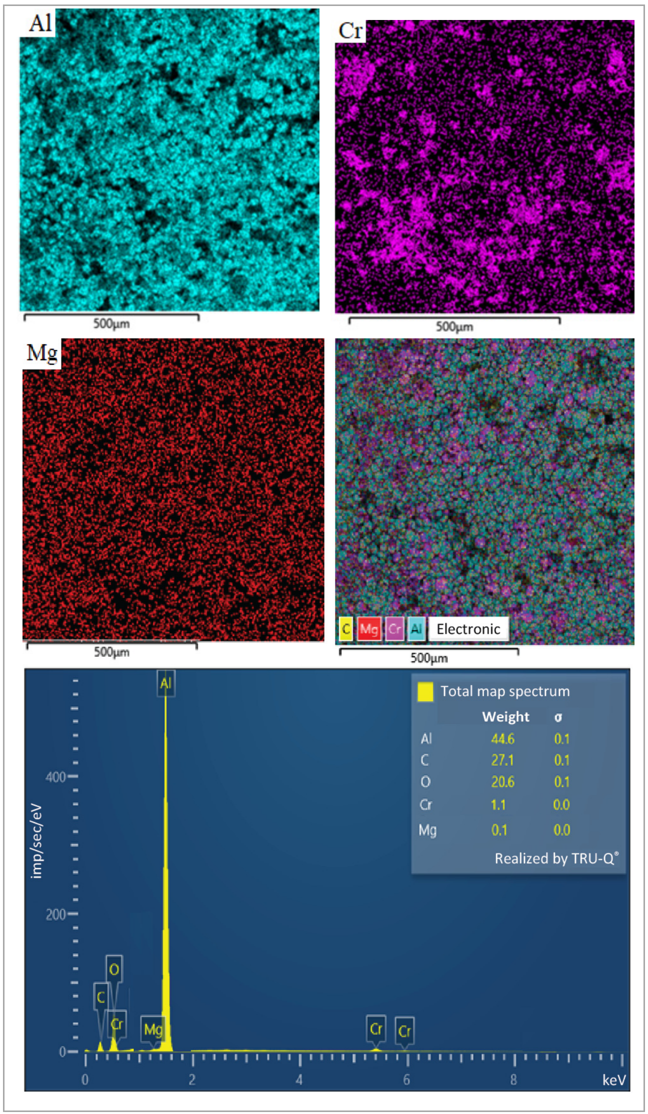

Fig. 2. Maps of distribution of surface elements of the initial sample of Al + 5 % Cr_3C_2 + 10 % Mg composition

Corrosion resistance characterises the ability of a metal or alloy to resist the corrosive effect of the environment and it can be determined by mass or rate indices. The mass corrosion rate was determined by mass loss and then converted into linear corrosion rate (GOST 9.908-85). The rate of mass loss (V_k), was determined by the ratio:

$$V_k = \frac{m_1 - m_2}{St}, \tag{1}$$

where m_1, m_2 – mass of the sample before and after corrosion, respectively, g; t – time of corrosion damage, h; S – surface area of the sample, m^2.

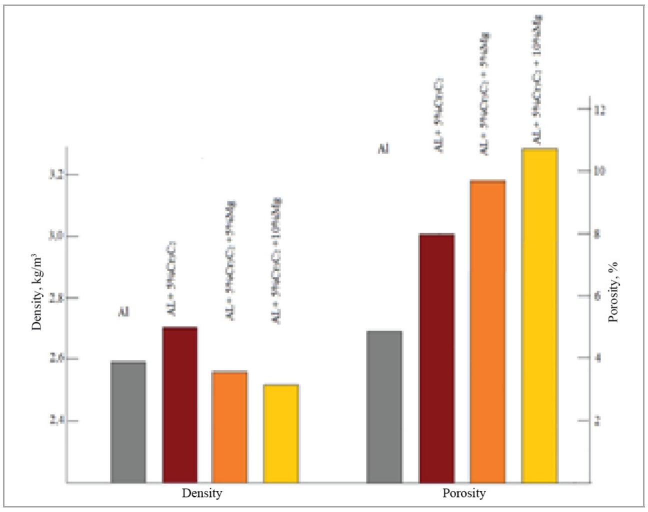

Fig. 3. Variation of density and porosity of samples depending on the composition

Mass corrosion losses were then converted to corrosion rates expressed in mm/year:

$$\Pi = 8.76\,\frac{V_k}{\rho}, \qquad (2)$$

where P – corrosion rate, mm/year; 8.76 – coefficient; V_k – corrosion rate, g/(m^2·h); ρ – metal density, g/cm^3.

As a result of the studies it was found that there is a continuous uniform corrosion for all the samples studied.

Based on the value of P the corrosion resistance of metals was assessed by a ten-point scale (GOST 9.908-85). In accordance with this scale in the conditions of the conducted experiment Al-Cr_3C_2 samples have corrosion resistance – 6 points (table).

It is well known that the passivation properties of aluminium oxide film significantly improve the corrosion resistance of aluminium alloys under normal conditions. Although the aluminium oxide film is relatively stable, aluminium oxide degrades in aggressive ion-rich environments, causing pitting corrosion. Adding other elements to pure aluminium improves its mechanical properties but reduces its corrosion resistance due to the inhomogeneity of the structure. For example, when magnesium is added to aluminium, Mg atoms diffuse to the grain boundary to form a secondary phase called β-(Al_3Mg_2) phase [38, 39]. This is a direct cause of intergranular corrosion, which results in corrosion cracking.

Corrosion pitting is prevented by the addition of small amounts of metals due to the formation of a secondary phase which creates a potential difference with the aluminium matrix. However, corrosion resistance tends to decrease when the secondary phase content exceeds a certain amount. This can be seen when comparing the corrosion rates of Al + 4 % Cr_3C_2, Al + 5 % Cr_3C_2 + 5 % Mg and Al + 5 % Cr_3C_2 + 10 % Mg samples. Increasing the magnesium content from 5 to 10 % raised the corrosion rate from 0.2189 to 0.4157 and further to 0.6831 g/(m^2·h), lowering the corrosion resistance of the material (Fig. 4). Therefore, in order to obtain highly corrosion-resistant aluminium alloys, it is important to select the optimum content of additional metals.

In the process of corrosion, the mechanical properties of materials also change. After corrosion resistance tests, the hardness of all samples decreased (Fig. 5).

The corrosion problems which may occur in alumina matrix composites can be categorised as follows:

– galvanic coupling between reinforcing phase and matrix;

– selective corrosion of the matrix/filler interface;

– corrosion of matrix defects in the composite.

During composites processing a reaction between the filler particles and the matrix can occur with the formation of new phase. If the redox potential of this phase differs markedly from that of the matrix, the composite may undergo selective corrosion in the area of contact between the new phase and the matrix.

Corrosion of aluminium alloys is essentially a micro galvanic process between these phases and the matrix alloys [40]. Corrosion occurs rapidly only when the

Corrosion resistance of composite samples

Material	Testing time, h	m_0, g	Δm, g	V_k, g/(m^2·hour)	P mm/year	Score according to GOST 9.908-85
Al + 0 % Cr_3C_2	192	1.4042	0.0108	0.1377	0.4646	6
Al + 4 % Cr_3C_2		1.4151	0.0101	0.1288	0.4224	6
Al + 7 % Cr_3C_2		1.4451	0.0087	0.1110	0.3500	6
Al + 10 % Cr_3C_2		1.4502	0.0041	0.0523	0.1591	6
Al + 5 % Cr_3C_2 + 5 % Mg		1.4551	0.0089	0.1135	0.1246	6
Al + 5 % Cr_3C_2 + 10 % Mg		1.3802	0.0083	0.1058	0.3657	6
Al + 0 % Cr_3C_2	336	1.4042	32.5	0.2369	0.7994	7
Al + 4 % Cr_3C_2		1.4151	22.5	0.1640	0.5378	7
Al + 7 % Cr_3C_2		1.4451	15.9	0.1359	0.4285	6
Al + 10 % Cr_3C_2		1.4502	13.7	0.0998	0.3037	6
Al + 5 % Cr_3C_2 + 5 % Mg		1.4551	49.5	0.3608	1.2113	8
Al + 5 % Cr_3C_2 + 10 % Mg		1.3802	84.6	0.6167	2.0698	8
Al + 0 % Cr_3C_2	504	1.4042	0.0485	0.2375	0.8014	7
Al + 4 % Cr_3C_2		1.4151	0.0447	0.2189	0.7114	7
Al + 7 % Cr_3C_2		1.4451	0.0443	0.2169	0.6839	7
Al + 10 % Cr_3C_2		1.4502	0.0266	0.1303	0.3964	6
Al + 5 % Cr_3C_2 + 5 % Mg		1.4551	0.0849	0.4157	1.3512	7
Al + 5 % Cr_3C_2 + 10 % Mg		1.3802	0.1395	0.6831	2.3612	7

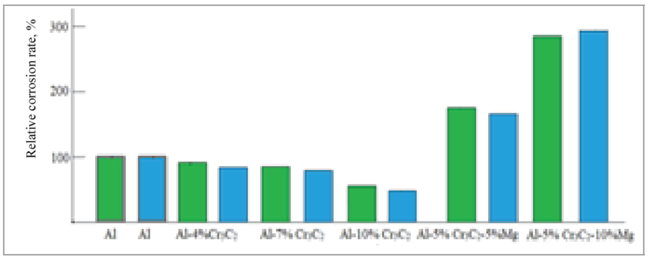

Fig. 4. Relative corrosion rate of specimens based on 504 h tests: 🟩 V_k, g/(m^2·h); 🟦 P, mm/year

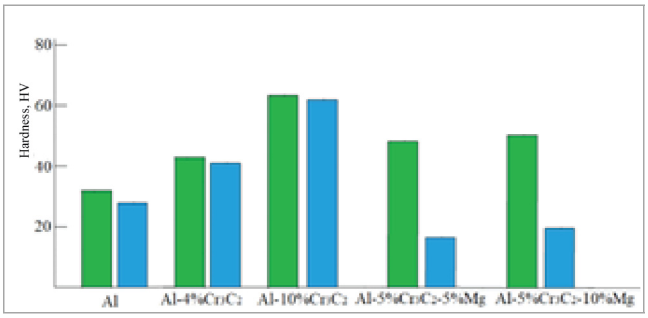

Fig. 5. Hardness of samples before 🟩 and after 🟦 corrosion resistance tests

passive film is destroyed, and this is mainly influenced by pH and Cl- ion concentration. When this occurs, corrosion with hydrogen release proceeds relatively easily. Pitting corrosion in the presence of aggressive ferric chloride is localised. The pits are initiated in weak spots in the oxide film during chloride attack. Pit initiation involves the absorption of Cl- ions on defects in the surface oxide film followed by chemical reaction. The presence of Cl- ions interferes with repassivation. Inhomogeneities in the alloy or composite play a major role in the development of pitting.

In our case, all samples show (Fig. 6) that there is no any sign of pitting corrosion on their surface. Continuous uniform corrosion is observed for all tested samples, and in all cases uniform distribution of corrosion damage to the metal surface is characteristic.

The formation of a thin layer of aluminium-based corrosion products (oxides or oxyhydroxides) was observed. The presence of chlorine can be attributed to both NaCl ions and Cl- ions adsorbed on the passive film. The micrographs (see Fig. 6) show solid particles inside the pits where the AlClx dissolution product led to the formation of a solid salt; this is consistent with previous studies [40–43], and the salt film may have stabilised some of the pits.

Formation of a protective layer of aluminium oxide has been mentioned in many research works, e.g. in [42, 44], and the presence of Al and Cl- can be related to $AlCl_3$ formed by the interaction of $Al(OH)_3$ film with Cl- ions, which occurs after the breakdown of the passive state of Al in the salt medium.

The addition of magnesium to the composite increased the corrosion rate. Aluminum demonstrated a pseudo-passivation process, but on the other hand, Al-Mg alloys corroded without forming protective compositions at the interface. Therefore, such elements as Zn, Mg and Hg are added to pure aluminum for cathodic protection. The inclusion of magnesium in the composite led to its dissolution in a corrosive environment.

It can be concluded that the addition of chromium carbides to aluminum-based alloys increases the corrosion resistance of the alloys, while the addition of magnesium to the composite does not improve alloys' corrosion resistance.

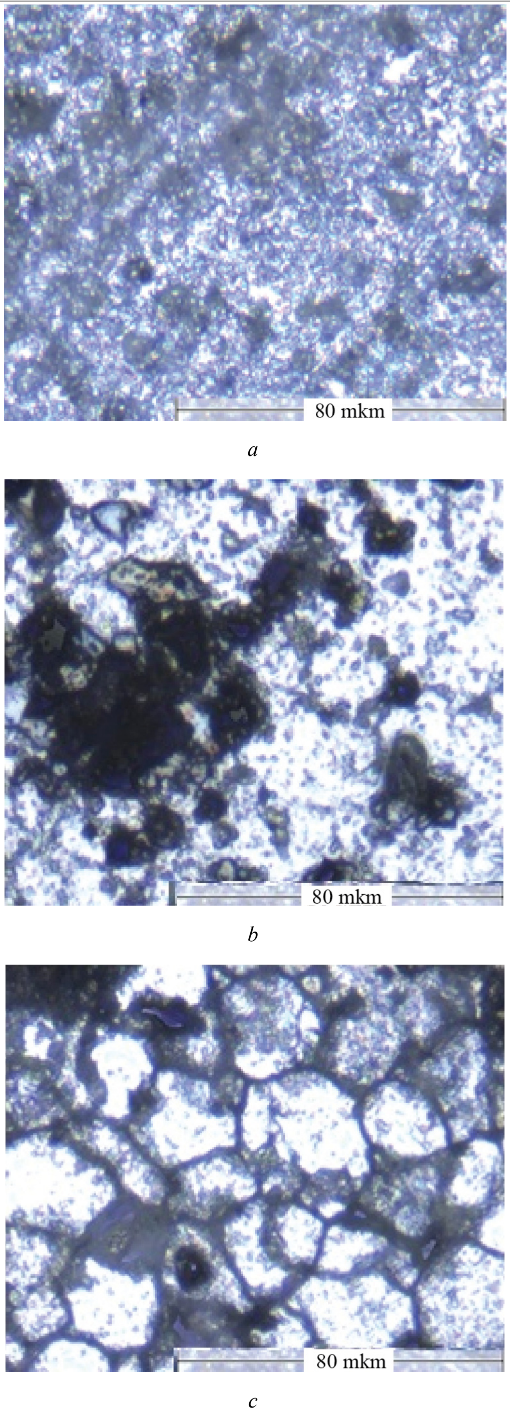

Fig. 6. Surface morphology of specimens after corrosion tests during 504 h: *a* – Al; *b* – Al + 10 % Cr_3C_2; *c* – Al + 5 % Cr_3C_2 – 10 % Mg

Conducted investigations of AMg6, D16-T cast alloys and 08Kh17 steel in seawater showed low weight loss, 100 % corrosion damage to the surface, but the absence of intergranular corrosion [45]. Tests of aluminum alloy castings in basic salt and acid media showed their low corrosion resistance. Pitting, uniform, fatigue and intergranular corrosion was observed in Al-Mg alloys [46, 47].

Conclusion

The necessity to protect the equipment of oil producing and processing enterprises from corrosion damage is associated with significant material costs. Thus, the technological support of both oil and natural gas production processes requires the development of modern materials capable of prolonged operation in an aggressive environment. Due to the increased demand for materials with improved physico-mechanical and physico-chemical properties, the use of composite materials in oil and gas industry is promising.

New aluminum matrix composites reinforced with chromium carbide particles are successfully produced by powder metallurgy. Microstructure of obtained composites is homogeneous, with low content of porosity, low defect with uniform distribution of reinforcing phase in aluminium matrix. No secondary intermetallic phases formed at the interface, the matrix and reinforcement showed good adhesion without any signs of reactivity. The corrosion behavior of the resulting composites appears to be mainly controlled by matrix corrosion. The addition of chromium carbide to the aluminum matrix increases the hardness and corrosion resistance of the material, while additional magnesium alloying reduces the corrosion resistance of the material. The manufacture of parts from accessible aluminum-matrix materials allows you to increase the service life of equipment without the use of expensive and unavailable metals.

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