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Investigation of the properties of composite materials based on epoxy resins with microsilica additives

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ABSTRACT

The outcomes of studying epoxy-based composite materials supplemented with microsilica are provided in the article. Microsilica was used as a filler. The samples were produced on the epoxy ED-20 basis supplemented with 2, 5 and 10 mas. % of microsilica. The structure and size of finely dispersed filler particles were defined. The obtained composites were tested for resistance to the effect of variable temperatures, corrosive, and abrasion. The study outcomes proved that samples supplemented with 2% of microsilica are more resistant to acid and alkali as well as to petrol than those ones supplemented with 25% of microsilica. Besides the amount of the filler from 2 to 10% doesn't sufficiently affect the resistance to variable temperatures. When microsilica is added to epoxy resin, it causes scuff resistance increase. The conducted testing proved that the developed composite materials are resistant to the effect of variable temperatures, corrosive, and abrasion. This enables to use these materials as coatings and anti-corrosion protection during machine maintenance.

Keywords: epoxy resin, microsilica, filled composite material, chemical resistance, composite material, corrosive, abrasion, coating.

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Introduction

Technology for applying hardening coating including composite ones plays an important role for upgrading farming machinery, as they contribute to durability of individual elements, parts, and entire machines during their maintenance. Polymer composite materials are used during machines maintenance for restoring the size of worn-out parts, for filling cracks and holes, for reinforcing threaded joints and stationary fits, for anti-corrosion protection, for bonding parts and materials, and also for parts production.

Increasing demands to the quality and rate of equipment repair operations require a wide range of highly-efficient systems capable to restore metal surfaces and different structures damaged from wear, abrasion, blows and corrosion. In this connection, production of composite materials based on epoxy oligomers that are highly adhesive to many materials is relevant and demanded in practice in different spheres.

Polymer matrix for composite material is chosen taking into account the conditions of maintenance. Composite properties depend on the matrix of polymer material. These properties are resistance to corrosive, thermal resistance, strength, moisture resistance, as well as the method of item production.

Epoxy binders take an important place in the world industry due to their production output and consumption level. They are widely used in different fields of application [1-6]. At the same time, they have some negative properties: high fragility, fire risk, low resistance to climatic factors [4-9].

One of the basic directions for development of epoxy composites with improved operational properties is to introduce different fillers into their compositions (basalt, talcum, chromite, metallic oxides, technogenic wastes from industrial enterprises). The fillers enable to decrease cost price of finished products considerably due to decreasing the cost of expensive binders and to impart different functional properties [2-9, 10].

Technogenic wastes from industrial enterprises are often used as dispersed fillers of epoxy resins. The authors of works [11-13] suggest to use fly ash which is a by-product of coal combustion at thermal power plants, as a filler for epoxy composites. The filler content ranged from 10% to 50%. The conducted researches proved that when fly ash is added into epoxy composite, tension strength increased as the amount of the filler was increased to the critical point (30%), and then decreased considerably.

Composite materials supplemented with microsilica have improved strength characteristics in comparison with polymer composites without the filler [14]. Eastomers introduction into epoxy resin increases their strength characteristics [15-16]. Literature analysis proved that due to fillers addition [17] into epoxy resin it is possible to improve some of its parameters.

The goal of the research is to study the impact of dispersed microsilica addition on chemical resistance of ED-20 epoxy-based polymer composite material, as well as on abrasion capacity and resistance to variable temperatures of ED-20 epoxy-based composite materials.

Experimental procedure

Epoxy-diane resin ED-20 (GOST 10587-84) was used as a binder for the produced compositions, as this resin is characterized by stability of dielectric and physical and chemical properties. Along with high thermal resistance, ED-20 is very fragile that can be improved by introducing special modifiers and solidifiers into composition. Polyethylenepolyamine (TU 2413-010-75678843-2012) produced by research and production company "Astat" (Dzerzhinsk, Russia) was used as a solidifier. It is intended for solidifying epoxy resins at room and low temperatures under humid conditions. Solidifier appearance is as follows: a liquid, color is from light-yellow to dark-brown, without mechanical impurities.

To fill polymer compositions, microsilica was used («Tau-Ken Temir» Silicon Plant, Karaganda). Microsilica is formed in the process of silicon alloys (ferrosilicium) smelting. After oxidation and condensation some silicon monoxide form extremely small product in the shape of round particles with high content of amorphous silica [14].

Microsilica in the amounts 2, 5, 10 mas. % was added into epoxy resin and thoroughly blended for 5-10 minutes until uniform mass. Then the solidifier was added (*resin: solidifier* ratio is *1:10*) and blended for 2 minutes. The resulted mass was applied by several thin layers on substrate in order to avoid bubbles in the samples. The check sample was made without adding the filler. It was solidified at room temperature for 24 hours. All the tests were carried out on samples of steel substrate 70×150 mm in size. Coating thickness was defined in compliance with ST RK GOST R 51694-2007. (Coating materials. Definition of coating thickness). Inaccuracy is ± 2 μm. Table 1 provides coating samples thickness data.

Table 1 - Coatings thickness

№	Sample name	Thickness, μm
1	ED-20	16
2	ED-20 + 2% of microsilica	18
3	ED-20 + 5% of microsilica	17
4	ED-20 + 10% of microsilica	18

Chemical and elemental composition was defined on an x-ray fluorescent spectrometer produced by PANalytical, model Axios Max (Rh 2.4kW). Filler surface morphology was studied with scanning election microscope TM 3030 (Hitachi). Filler particles size was defined with laser particle size analyzer Mastersizer 3000 with attachment Hydro MV (120 ml), water was used as dispersive medium.

X-ray diffraction was measured with the X-ray diffractometer SmartLab produced by Rigaku Corporation. X-radiation source is x-ray tube Cu Kα-radiation (1.54059). Current and voltage of the tube are set at 50 mA and 40 kW respectively. One-dimensional detector (D/teX Ultra, Rigaku) with filter Kβ. Step-and-shoot technology was used for measurements under conditions that measurement angles range is 2θ = 5-90°, pace (Δ2θ) = 0,1° scanning rate - 2°/min. Phases are identified by comparing the obtained data with reference database, the most complete one is maintained by ICDD.

Samples resistance to corrosive impact was defined in compliance with GOST 9.403-80 method A. Decorative and protective properties [18] of coatings are defined after the tests in acid solutions (25% H_2SO_4), alkalies (25% NAOH) and petrol. Samples resistance to corrosive impact was defined in compliance with GOST 20811-75 method A. Samples resistance to variable temperatures impact was defined in compliance with GOST 27037-86 in a climatic chamber BINDER of MK series for testing with heating and cooling in the range from -40°C to +60°C for 60 minutes.

Discussion of the results

The researches enabled to find out that microsilica mainly consists of Si, K, Ca, Fe oxides, and contains a small amount of impurities such as Mg, Mn oxides (Table 2).

Table 2 - Chemical composition of microsilica

Element	Concentration	Unit
Mg	345.4	ppm
Si	39.677	%
K	0.294	%
Ca	0.857	%
Mn	204.8	ppm
Fe	0.126	%

*1ppm = 0.0001%

Fraction composition of microsilica is characterized by biomodal distribution and represented by particles from 0.1 to 100 µm with average particles size 2-6 µm and 7-14 µm (Figure 1), which is also proved by SEM data (Figure 2).

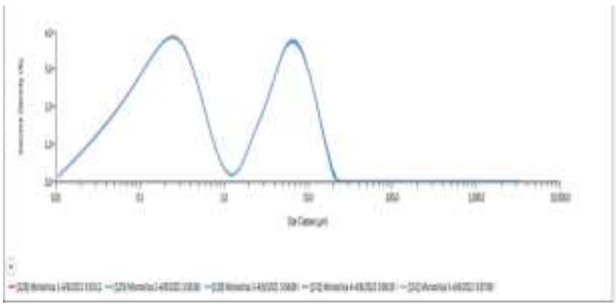


Figure 1 - Distribution of microsilica particles size

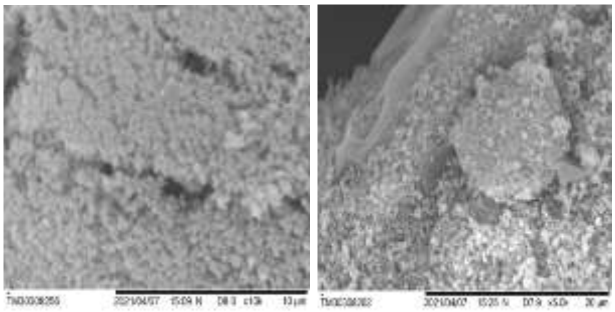
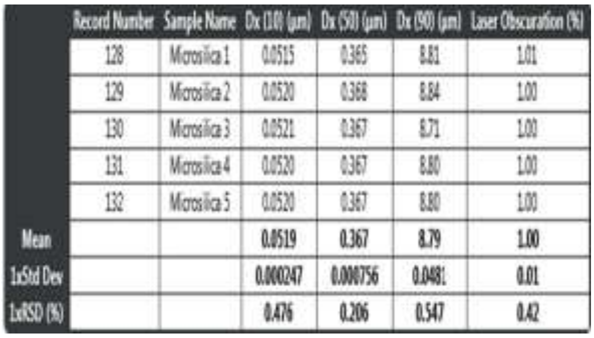


Figure 2 - SEM images of microsilica

It can be seen from Figure 2 that microsilica particles have smooth surface and spherical shape. The powder practically consists of loose agglomerates of silicon with very low bulk density.

Laser particle size analyzer data shows that 9 µm microsilica particles volume ratio is 90% (Figure 3).



	Record Number	Sample Name	Dx (10) (µm)	Dx (50) (µm)	Dx (90) (µm)	Laser Obscuration (%)
	128	Microsilica 1	0.0515	0.365	8.81	1.01
	129	Microsilica 2	0.0520	0.368	8.84	1.00
	130	Microsilica 3	0.0521	0.367	8.71	1.00
	131	Microsilica 4	0.0520	0.367	8.80	1.00
	132	Microsilica 5	0.0520	0.367	8.80	1.00
Mean			0.0519	0.367	8.79	1.00
1xStd Dev			0.000247	0.000756	0.0481	0.01
1xRSD (%)			0.476	0.206	0.547	0.42

Figure 3 - Distribution of microsilica particles size

According to XRF data, microsilica is represented by two structures: Fe_2O_3 - 88% and SiO_2 - 12% (Figure 4). XRF of particles showed that they have orthorhombic lattice structure.

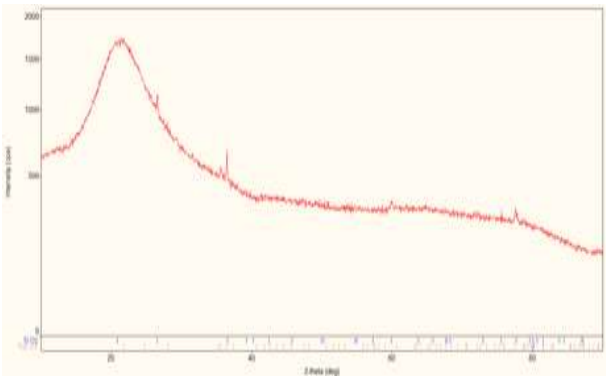


Figure 4 - X-ray diffraction pattern of microsilica (XRD)

One of possible application areas of the developed compositions is automobile industry where materials are affected by different aggressive factors: high temperatures, moisture, coldness, freeze-thaw temperatures, chemical agents (acid, alkaline solutions in the compound of cleaning means etc.).

So it was critical to study resistance of the developed materials to the impact of variable temperatures, corrosive and abrasion. To study chemical resistance, samples were dipped into 25% solutions of H_2SO_4, NaOH and petrol at temperature (20±2) °C for 24 hours. The following characteristics were defined as resistance criteria for the tested epoxy coatings: the change of bond strength of coating to metal, appearance and kind of samples damage. Testing of resistance to corrosive showed that the samples supplemented with 2% of microsilica are more resistant to acids and alkalies impact in comparison with the check sample (Figure 5).

a) b)

Figure 5 - Epoxy resin samples supplemented with microsilica after holding in 25 % solutions for 24 hours:
a) NAOH (left-to-right 0, 2, 5, 10%),
b) H_2SO_4 (left-to-right 0, 2, 5, 10%)

After holding the samples in petrol for 24 hours there were no visible changes in them. When they were examined, surface staining and splotches were recorded. (Figure 6).

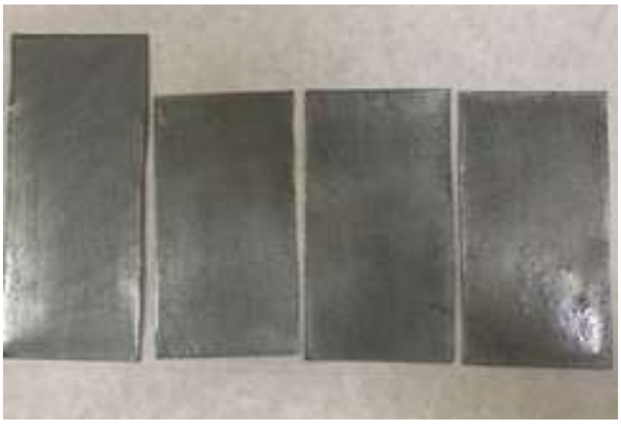


Figure 6 - Epoxy resin samples supplemented with microsilica after holding in petrol for 24 hours (left-to-right 0, 2, 5, 10%)

Table 3 provides the results of epoxy resin chemical resistance indexes. Epoxy resin has different mass content of filler. Coating thickness reduction wasn't found out after corrosive impact.

Table 4 provides the results of coating resistance to abrasion indexes and indexes of resistance to epoxy resin variable temperatures impact. Epoxy resin has different mass content of filler.

Table 3 - Indexes of chemical resistance of epoxy resin with different mass content of filler

№	**Epoxy resin name**	Chemical resistance		
		Resistance to static effect at t = (20±2)°C for 24 hours in 25% solution H_2SO_4	*Resistance to static effect at t = (20±2)°C for 24 hours in 25% solution NAOH*	*Resistance to static effect at t = (20±2)°C for 24 hours in petrol*
1	ED-20	Surface dimmed, splotches are observed	Valid flaking is observed on edges no more than 10 mm, surface dimmed	Splotches are hardly observed, surface dimmed
2	ED-20 + 2% of microsilica	Splotches are observed, surface dimmed	Splotches are hardly observed, surface dimmed, valid flaking is observed on edges no more than 10 mm	Surface dimmed, splotches are observed
3	ED-20 + 5% of microsilica	Splotches are hardly observed, surface dimmed	The sample surface became dull, the defect of porosity and coating flaking has been found out	Splotches are observed, surface dimmed
4	ED-20 + 10% of microsilica	Splotches are observed, surface dimmed	Surface dimmed and became dull, coating flaking is observed	Splotches are observed, surface dimmed

Table 4 – Indexes of coating resistance to abrasion and resistance to variable temperatures impact of epoxy resin with different mass content of filler

№	**Epoxy resin name**	Coating resistance to abrasion	Resistance to variable temperatures impact
1	ED-20	0.056	Brightness and colour variations, flaking from the surface at temperature from (40±2)℃ below zero up to (60±2)℃ above zero (for 60) minutes haven't been found out
2	ED-20 + 2% of microsilica	0.044	Brightness and colour variations, flaking from the surface at temperature from (40±2)℃ below zero up to (60±2)℃ above zero (for 60 minutes) haven't been found out
3	ED-20 + 5% of microsilica	0.052	Brightness and colour variations, flaking from the surface at temperature from (40±2)℃ below zero up to (60±2)℃ above zero (for 60 minutes) haven't been found out
4	ED-20 + 10% of microsilica	0.036	Brightness and colour variations, flaking from the surface at temperature from (40±2)℃ below zero up to (60±2)℃ above zero (for 60 minutes) haven't been found out

The results provided in Table 4 indicate that appearance changes of epoxy resin samples with different mass content of fillers were not found out during their examining after variable temperatures impact: coating on the substrate is even, there is no flaking from the substrate, no visible defects and cracks. The test on coating resistance to abrasion showed that the samples supplemented with 2% of microsilica are mostly resistant to abrasion.

Conclusions

As it can been seen from the obtained data, after corrosive impact the samples supplemented with 2% of microsilica got the best indicators of preserving appearance and change of bond strength of coating to metal. Besides the amount of the filler from 2 to 10 mass % is insufficiently reflected in the resistance to variable temperatures impact. When tests were completed, appearance changes weren't observed during epoxy resin samples examination after variable temperatures impact: homogeneous mass, coating on the substrate is even, there is no flaking from the substrate, no visible defects and cracks. When microsilica is added into epoxy resin, it causes the improvement of resistance to abrasion.

Thus, microsilica usage as a filler enables to produce epoxy materials with good chemical resistance, with high resistance to abrasion and variable temperatures impact.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

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Микрокремнезем қоспалары бар эпоксидті шайырлар негізіндегі композициялық материалдардың қасиеттерін зерттеу

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ТҮЙІНДЕМЕ

Мақалада эпоксидті шайыр негізіндегі композициялық материалдарды зерттеу нәтижелері келтірілген. Толтырғыш ретінде микрокремнезем қолданылды. Үлгілер ЭД-20 эпоксидті шайыр

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құрамына 2, 5, 10 мас. % микрокремнезем қосу арқылы жасалды. Ұсақ дисперсті толтырғыш бөлшектерінің құрылымы мен өлшемдері анықталды. Алынған композициялар ауыспалы температураға, агрессивті ортаға және үйкеліске төзімділікке тексерілді. Зерттеу нәтижелері көрсеткендей, 2% микрокремнезем қоспалары бар үлгілер 25% қышқыл мен сілтінің әсеріне, сондай-ақ бензиннің әсеріне төзімді. Бұл жағдайда толтырғыштың мөлшері 2-ден 10 мас. % болғанда айнымалы температураның әсеріне, төзімділікке айтарлықтай әсер етпейді. Эпоксидті шайырға кремнеземді енгізу үйкеліс төзімділігін арттырады. Өткізілген сынақтар әзірленген композициялық материалдардың ауыспалы температураға, агрессивті ортаға, үйкеліс әсеріне төзімділігін көрсетті, бұл оларды машиналарды жөндеу кезінде жабын және коррозияға қарсы қорғаныс ретінде пайдалануға мүмкіндік береді.

Түйін сөздер: Эпоксидті шайыр, микрокремнезем, толтырылған композициялық материал, химиялық төзімділік, композициялық материал, агрессивті орта, үйкеліс, жабын.

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Исследование свойств композиционных материалов на основе эпоксидных смол с добавкой микрокремнезема

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АННОТАЦИЯ

В статье приведены результаты исследований композиционных материалов на основе эпоксидной смолы. В качестве наполнителя использовали микрокремнезем. Образцы были изготовлены их эпоксидной смолы ЭД-20 с добавлением 2, 5 и 10 мас. % микрокремнезема. Определены структура и размеры частиц мелкодисперсного наполнителя. Полученные композиции исследовали на устойчивость к воздействию переменных температур, агрессивных сред и прочности к истиранию. Результаты исследований показали, что образцы с добавками 2% микрокремнезема более стойки к воздействию 25%-ных кислоты и щелочи, а также к действию бензина. При этом количество наполнителя от 2 до 10% мас. несущественно отражается на устойчивости к воздействию переменных температур. Введение микрокремнезема в эпоксидную смолу приводит к росту стойкости к истиранию. Проведенные испытания показали устойчивость разработанных композиционных материалов к воздействию переменных температур, агрессивных сред, стойкости к истиранию, что дает возможность использовать их в качестве покрытий и антикоррозионной защиты при ремонте машин.

Ключевые слова: эпоксидная смола, микрокремнезем, наполненный композиционный материал, химическая устойчивость, композиционный материал, агрессивная среда, истирание, покрытие.

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