

Restorative Epoxy-Composite Materials with Micro-Nano-Dispersed Carbide Fillers (Si, Ti, Zr, Cr, Mo and Nb-carbides)

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Abstract

A practically important parameters of strength and chemical resistance of polyepoxide based on resin-analogue ED-20 with PEPA hardener, filled with microdispersed powders of Si, Zr, Cr, Nb, Ti, Mo carbides, have been experimentally studied in this article. The microstructure of the composites is characterized by a relatively uniform dense distribution of filler agglomerates, which can be seen in the example of compositions with 50 wt% TiC and ZrC. The absence of a significant effect of carbides on the temperature ranges of oxidative degradation of composites was established. Swelling method established that filling can radically increase the resistance of composites to acetone organic solvents. At the same time, resistance in an acidic environment is noticeably reduced (for example, nitric acid). It was found that the introduction of 50 wt% carbide fillers gives a significant increase in the strength and modulus of elasticity during compression of the composites. There is also a decrease in shrinkage and an increase in adhesion to steel (with normal tearing).

Keywords: Polyepoxide, compressive strength, bending strength, modulus of elasticity, microhardness, adhesion, SEM, swelling.

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INTRODUCTION

Epoxy- and ether-acrylate oligomers remain the most popular in the engineering plastics market [1]. As popular fillers for investigation, can use the carbon [2-3] and silica nanodispersions [4], gypsum / cement [5], vegetable fillers [8, 9], chromites [10], metal carbides and nitrides [5, 6, 11-19].

Silicon and metal carbides are a widely used practical group of chemical compounds for materials science and applied chemistry. Structural features make it possible, on their basis, to obtain wear- and heat-resistant coatings, solid materials and products for high-load applications.

The aim of the work is to obtain compositions "epoxy resin - carbide", which could expand the applied value of epoxy composites, - primarily by increasing their strength and durability by simple filling. This task is not so simple: according to many basic characteristics of strength/resistance, a pure epoxy polymer is better

than a filled one. This includes, for example, such important application properties as compressive strength, flexural strength, adhesion, thermal oxidation resistance. Resistance to aggressive solvents, oxidants, and acids is seen as important properties of such composites. At the same time, the initial compositions for the production of polymers should be well moldable and curable by standard techniques.

Silicon and metal carbides are a widely used practical group of chemical compounds, the basis of a number of segments of materials science and applied chemistry. Structural features make it possible, on their basis, to obtain wear and heat-resistant coatings, solid materials and products for high-load applications.

Zirconium carbide ZrC — is an interstitial-phase with a wide homogeneity region with (38–50 at.% Carbon, ZrC_{0.62} - ZrC) [19]. He is refractory (melt. 3532 °C), hard (to Mohs 8.5) and high-density (6.73 g / cm³) powder, is considered a "cermet" material. Can be

used as a refractory coating in nuclear reactors. Zirconium carbide is actively used as a coating for uranium dioxide and thorium dioxide in nuclear fuel.

Chromium (II) carbide Cr_3C_2 , is an ordered phase with a very narrow homogeneity region with a carbon content of 13.3 wt%. There are other chromium carbides: gray Cr_{23}C_6 & Cr_3C_2 , “silver” Cr_7C_3 — and (except for Cr_3C_2) they are insoluble even in aqua regia. Their reactivity increases at 800-1000 °C. When they can interact with metals (zinc, Cr_3C_2), into each other (at 800 °C, Cr_7C_3 carbide turns into Cr_{23}C_6).

Molybdenum carbide is an inorganic compound of molybdenum metal and carbon with the formula MoC , black crystals, insoluble in water.

Silicon carbide (carborundum) SiC is the most used and well-known carbide compound (of course,

after welding calcium carbide). Silicon carbide is a rare substance on Earth, but it is widespread in space - in particular, outside the solar system (masses of SiC grains are formed near carbon-rich giant stars). But on Earth, he occurs naturally in the form of an extremely rare mineral, moissanite (see Figure 1). The actual silicon carbide powder was obtained in 1893 [19]. It is used as an abrasive, semiconductor, in microelectronics (including as a detector and in power-plants of electro-cars), as a substitute for diamond in jewelry, in the production of the first LEDs (experiments by D. Round in 1907 and O. Losev in 1923). Titanium carbide TiC is a compound of light gray color with a metallic luster, is inert - but above 2500 °C it begins to react with nitrogen, and decarburizes when interacting with hydrogen [19]. Refractory, highly dense (melt p 3150 °C, 4930 g / cm^3), very hard (according to Mohs 8.5).

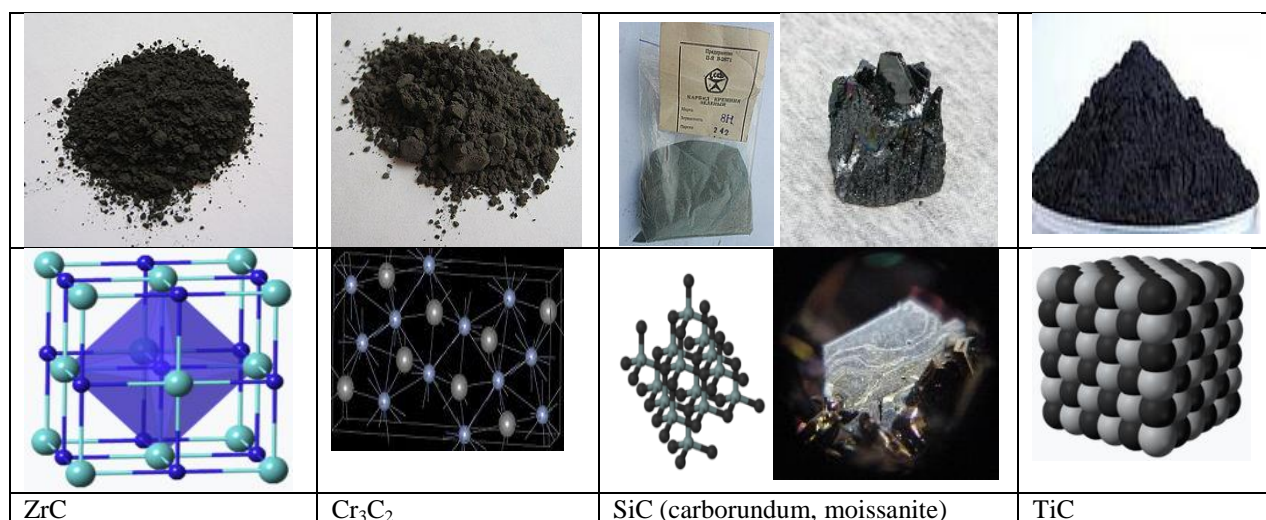


Fig 1: Visual data on powders or crystals of carbides (pictures from [19])

The structure and properties of carbides are conducive to the search for such optimal compositions - and this search is constantly being conducted in the scientific world [1, 11-18]. Epoxy compositions with carbides studies regularly, and over the past decades, the activity of such studies has not decreased [5, 6, 11-16]. In this field are working an eminent researchers: Mostovoy & Nurtasina [10], Yan & Zhang [11], Shen & Yu [12], Zhou & Xiong [13], Nhuapeng & Tunkasiri [14], Park & Yoon [15], Luo [16]; Poornima, Thomas & Huczko [18].

Article [11] shows that the addition of Ti_3C_2 /graphene compositions reduces the wear of epoxy coatings by almost half (by 82%), while maintaining good anti-corrosion and a sharp increase in tribological properties.

A significant improvement in thermal-conductivity was found after the introduction of only 3 wt% SiC nanofibers (“... a 106% enhancement as compared to neat epoxy”) [12]. Interesting graphic

material is also presented here, which we considered necessary to reproduce for completeness of the perception of the topic. Thus, the SEM and TEM presented here perfectly visualize both microparticles and nanofibers of carbides (for example, SiC). Looking ahead, we can say that these figures are in relatively good agreement with our SEM photos of the corresponding systems (see below).

According to Russian patent [17], the introduction of boron carbide into the epoxy resin makes it possible to obtain a heat-resistant polymer composition. However, it loses its strength (in compression and tension) after 15-50 days of heat aging at 200-250 °C.

In [10], dispersed chromite (a mineral with 40-45% CrO and 20-25% CaCO_3) was used as an effective filler - that improves the physicochemical and mechanical properties and thermal stability.

As is known [1-17], the introduction of micro- and nanofillers into epoxy-resin does not always give a dramatic increase in the basic strength characteristics (in compression, bending), as well as resistance (in water, acids, solvents). The selection of the type and percentage of filler content sometimes allows to optimize a composition with increased performance. Carbides could act as such optimal fillers - however, this must be verified experimentally. This work is devoted to this.

EXPERIMENTAL PART

1. Methods and reagents

Used Czech epoxy resin "Epoxy520" (refined analogue of the Soviet ED-20), cured with polyamine PEPA in a ratio of 5: 1. The filler was added immediately after stirring these components, after which the composition was homogenized and immediately formed into samples (Fig 3).

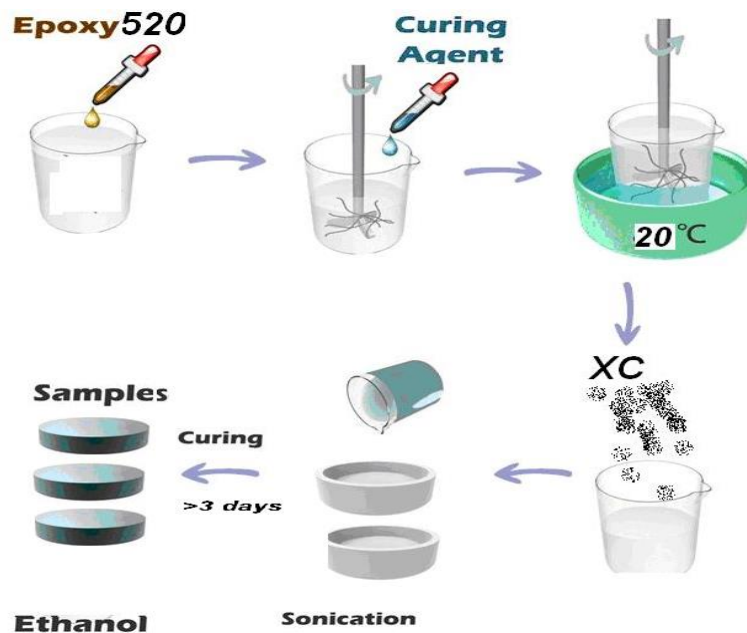


Fig 2: A typical scheme for producing epoxy-carbide composites (the graphical elements from Mostovoy., [20] are used)

Optical microscopy was performed on the optical microscope Dresser. SEM photos were obtained using a JSM-35 JEOL scanning electron microscope

For bending tests (GOST 56810-2015), plates with a size of $6 \times 1 \times 0.2$ cm were made. Their bending fracture was carried out on the basis of $L = 3$ cm of a

DI-1 bending testing machine. Based on the test results, the strength and elastic modulus were calculated.

Tests for adhesion (GOST 14760-69) with normal tearing were carried out on cylinders 5 cm^2 (Fig 3).



Fig 3: Films of epoxy-carbide adhesives in tearing-tested cylinders

For compression tests (GOST 4651-2014, ISO 604:2002), cylindrical samples $d=6.5 \text{ mm}$, $h=12 \pm 1 \text{ mm}$ were taken, compressed on a press-machine LouisShopper until complete destruction. Based on the results of the tests, the strength was calculated: $f = P/s$ (P is the load in kgf, s is the area equal to 0.332 cm^2) and the modulus $E: E=f/e$ (e is the relative elongation).

Brinell microhardness (GOST 9012-59, ISO 6506-1: 2005) was measured as resistance load when a steel hemisphere ($d=3 \text{ mm}$) was immersed in a sample-plate ($b=1.5 \text{ mm}$) to $10\text{-}60 \text{ }\mu\text{m}$.

Samples were tested in aggressive environments using the standard swelling method (GOST 4650-2014, ISO 62: 2008). Tablets $1 \times 1 \times 0.2$

cm were placed in an aggressive environment. The change in their weight was periodically recorded, which was converted into % of swelling.

RESULTS AND DISCUSSION

1. The morphology of the compositions, according to microscopy data

The microstructure of the filled composites is characterized by a relatively uniform dense distribution of filler agglomerates, which can be seen in the example of compositions with 50 wt% TiC and ZrC (Fig 4). This fact, together with the absence of pores, suggests that carbides can enhance the strength of the polyepoxide. This is possible, for example, by forming its own framework with the compacted polymer phase surrounding them.

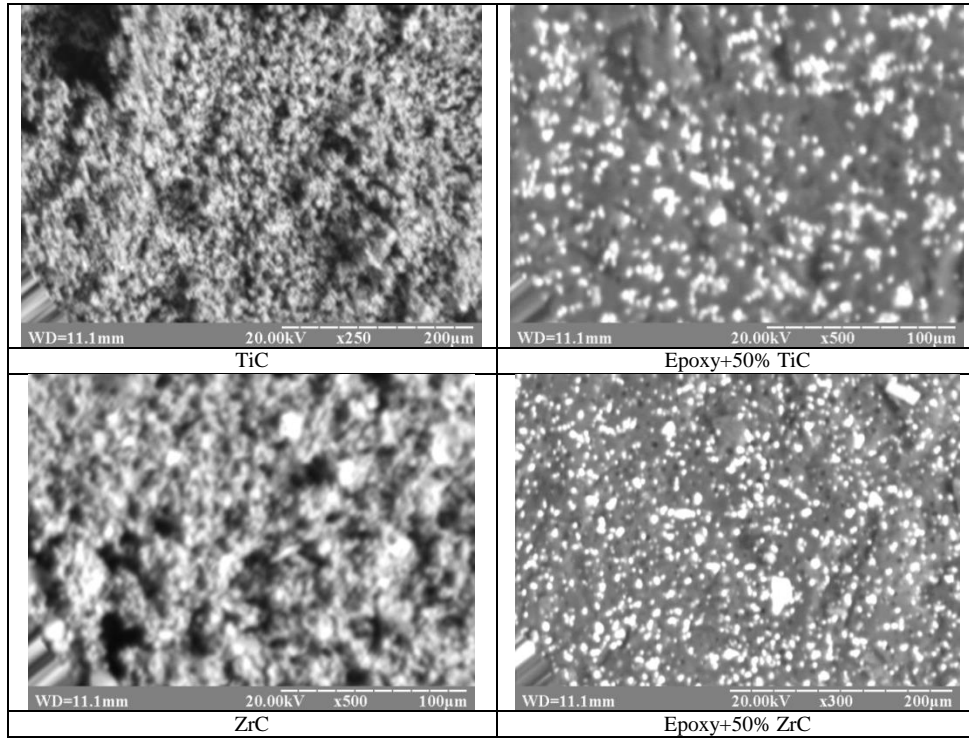
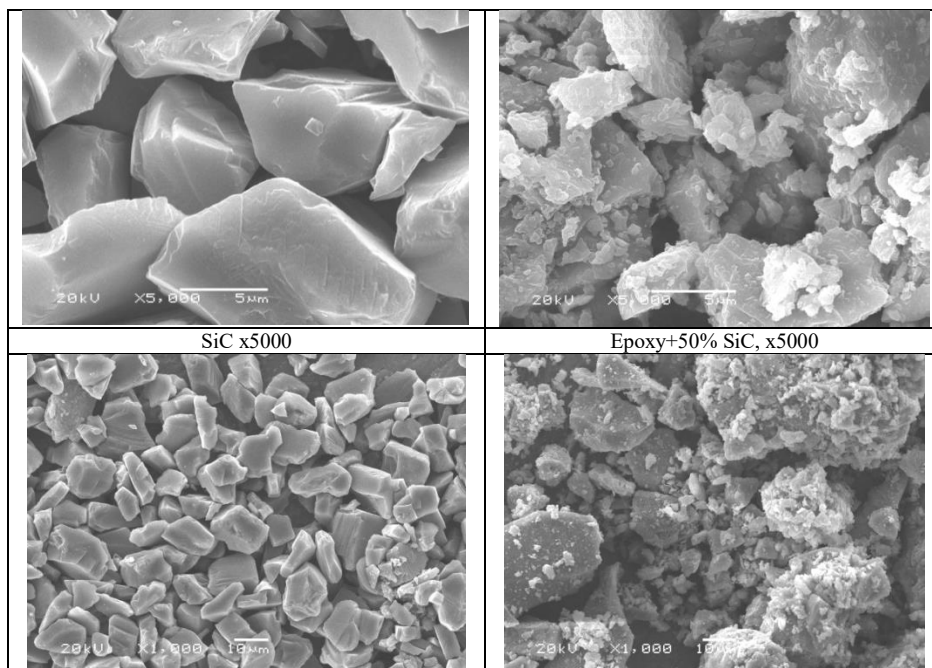


Fig 4: SEM photo of the initial Ti and Zr carbide powders and the native surface of the polymerized composites

SEM of SiC and a composite with it (Fig 5) shows that after the introduction into the epoxy resin,

SiC particles undergo a certain structural rearrangement.



SiC x1000	Epoxy+50% SiC, x1000
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Fig 5: SEM images of initial SiC and epoxy composites with it

It can be seen that very small SiC particles appear in the composite (which are absent on the SEM of dry carbide, Fig 5), and they “stick together” into a framework structure inside the polymer. This indicates an improvement in the physical and mechanical properties of the final composite.

2. Strength of composites

From Table 1 follows that 50 wt% of any taken carbides significantly increases the compressive strength (C) (Elastic Limit) and the modulus E. Note that the compressive strength C is one of the most conservative characteristics, which is very difficult to enhance by simple filling. According to our previously published data, as a rule unmodified fillers (nanosilica,

graphene, gypsum, cement, quartz / sand etc [3-9] allow increasing C only at 5-15%.

In the case of our carbides, an increase in C by 20-30% is achieved without much effort, by simple filling. This, however, does not apply to the value of the Total Destruction Limit (is indicated by an index in Tables 1 and 2), which is usually higher for unfilled polymer. Total Destruction Limit suggests how long an already deformed composite product will resist the load (which is also important). However, in mechanical tests, this characteristic does not have such a universal value as C, which gives the actual resistance of the product to the load.

Table 1: Compression load C^P of the Limit C of elastic resistance and limit ^P of total destruction (if it is fixed), for cylindric sample d=6.5 mm, h=11⁺¹ mm. Thermal mode - 55 ° C, then 105 ⁺⁵ ° C. Lagging values (not taken into account in averaging and statistical processing) are in *italics*

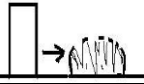



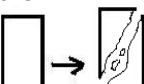


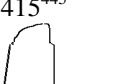

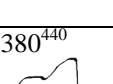

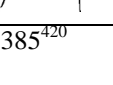
Sample	C ^P (kgf), all values obtained in the order of growth of C.	Average C ^P and type of diagrams	Modulus E * 1000.Kgf / cm ² , all values and average E _{av} (% to E _{unfilled})
Unfilled 	340 – 340 ⁴²⁰ – 345 ⁵⁰⁵ – 350 ⁴²⁵ – 350 ⁵¹⁰ – 360 ⁴⁵⁰ Brittle-plastic fracture, with a low threshold of elastic resistance	350 ⁴⁷⁰ 	8,7 – 10 – 11 – 12,7 – 13 E _{av} = 12.2 (100%)
ZrC 	380 ⁴⁰⁰ – 385 ⁴⁵⁰ – 400 ⁴³⁰ – 400 ⁴⁸⁵ – 410 ⁴¹⁵ – 415 ⁴²⁰ – 440 <i>Elastic-brittle fracture into several parts</i>	410 ⁴⁴⁰ 	11,9 – 12 – 14,1 – 14,1 – 14,6 – 16 E _{av} = 14,2 (116,4%)
SiC 	400 – 430 – 450 – 450 ⁴⁶⁰ – 490 Brittle fracture in-two, with a diagonal crack (destruction along the Chernov-Luders Diagonal)	450 ⁴⁶⁰ 	11,8 – 11,9 – 13,6 – 14 – 16,4 E _{cp} = 14,0 (115%)
TiC 	390 ⁴⁰⁰ – 400 ⁴²⁰ – 400 ⁴²⁰ – 430 – 430 ⁴⁹⁰ Brittle fracture, with a diagonal crack (destruction along the Chernov-Luders Diagonal)	415 ⁴⁴⁵ 	14,2 – 14,4 – 14,6 – 15,1 E _{cp} = 14,8 (121%)
CrC 	370 ⁴²⁵ – 375 ⁴⁶⁰ – 375 ⁴⁶⁰ – 390 ⁴⁰⁰ – 400 ⁴³⁰ <i>Elastic-brittle fracture into several parts</i>	380 ⁴⁴⁰ 	10,6 – 11,6 – 16,9 – 18,8 – 19 E _{cp} = 16,6 (136%)
Mo ₂ C 	370 – 370 – 400 ⁴³⁰ – 400 ⁴¹⁰ <i>Elastic-brittle fracture into several parts</i>	385 ⁴²⁰ 	10,9 – 13,2 – 14 – 14,2 E _{cp} = 13,8 (113%)

Table 2: Compression load C^P of the Limit C of Elastic Resistance (averaged over 3-5 tests of identical specimens, at S_{av.square} = 0.97 and α = +-2%) and Limit ^P of Complete Destruction (if it is fixed), and average Modulus E of elasticity (α = +-6%) with a load on a post with a diameter of 6.5 and a height of 11⁺¹ mm. In brackets - values for similar composites without exposure to water. Thermal mode - 55 ° C, then 105 ⁺⁵ ° C, after which the samples were kept in distilled water (1 month with daily heating to 55 ° C)

Sample and its appearance after destruction	Average C ^P and type of diagrams	Modulus E * 1000, Kgf / cm ²
Unfilled	305 ⁴²⁰ (350 ⁴⁷⁰)	8.2 (12,2)
ZrC	350 ³⁹⁵ (410 ⁴⁴⁰)	11.3 (14,2)
SiC	370 ³⁸⁰ (450 ⁴⁶⁰)	12.5 (14,0)
TiC ₂	355 ⁴¹⁰ (415 ⁴⁴⁵)	12.5 (14,8)
CrC	350 ³⁶⁰ (380 ⁴⁴⁰)	12.0 (16,6)

Mo ₂ C	330 ³⁴⁰ (385 ⁴²⁰)	12.6 (13,8)
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Table 2 clearly shows the reinforcing effect of carbides on resistance of polyepoxide's strength to aging in water. The unfilled polymer loses its strength by ≈15% (from 350 to 305 kgf, Table 2), and the Modulus decreases by ≈1.5 times. This is a noticeable loss, which in some cases will prevent the use of epoxy plastics in applied areas (for example, for waterproof supports of dams and embankments, waterproof self-leveling floors, etc.).

For filled composites, decrease of compressive strength after water-treatment go in the same way – by ≥15-17% or ≤ 10% (for CrC) (Tab.3). The drop in the elastic modulus is much less pronounced than for the unfilled polymer (Table 3). As a rule, filled composites conserves a high strength C of Elastic Limit (≥350 kgf, Tab.3) and Modulus E (≈12±0.5, Tab.3), comparative with C & E of initial (non-hydrated) unfilled epoxy polymer. But Limit of Complete Destruction is weakened after filling (Table 3, see index ^P).

Table 3: Strength parameters of composites (* - estimated)

	H	ZrC	SiC	TiC	CrC	Mo ₂ C
Flexural strength $i, \text{kg} \setminus \text{mm}^2$	8	5,5	7,5	8,5	6	5*
Flex.Modulus $I * 1000 \text{kg} \setminus \text{cm}^2$	18,5	14*	26	23	27,4	24
Weight, mg at $h = 11\text{mm}, d = 6.5\text{mm}$	385	635	530	575	630	645
Shrinkage, mm	0,9	0,6	0,5	0,4	0,4	0,5
Abrasion, mm	60	90	90	80	95	85
Fire resistance, sec	1	2,5	3	3,5	3	3
Adhesion ^{max.value} , kgf	80 ¹⁰⁰	105 ¹¹⁰	150 ²²⁰	-	80	210 ²⁵⁰

Flexural strength is a very sensitive characteristic to filling, which most often drops by 2 ± 0.5 times after filling [4-8]. In the case of carbides, the flexural strength is almost preserved or even increases (for TiC, Table 3). The elastic moduli in bending increase significantly - by about 1.5 times.

Fire resistance naturally increases with filling, since the fire resistance of epoxy plastic is much lower than that of incombustible carbide fillers (Table 3).

Adhesion to steel increases very noticeably after filling (except for chromium carbide) - in some cases by 2-2.5 times (for zirconium and molybdenum carbides). This important practical result is apparently due to the strengthening of the structure of the adhesive film. And possibly a mechanical factors takes place -

for example, the incorporation of filler particles into irregularities in the steel surface.

The microhardness after filling increases markedly (Table 4), the result is expected (carbides are highly solid substances), and very important from an applied point of view. It can be seen that ordinary unfilled polyepoxide produces moderate values of microhardness load (M) at small punch depressions (10-20 microns), and already at 50-60 microns the sample is brittle. Filling with any of the carbides taken increases M immediately by 2-2.5 times at small immersions and by 1.3-1.8 times at 30-50 microns immersion. Of all fillers, only composite with 50 wt% CrC is more brittle than unfilled polymer; the rest of carbide fillers impart a superior resistance to composite's cracking (especially SiC).

Table 4: Microhardness (in Newtons, N) at different immersion depths. The "t" index denotes cases of cracking of individual samples during the test

	Immersion of a hemisphere, μm					
	10	20	30	40	50	60
Non-filled epoxy polymer	50	120	250	350	400 ^m	500 ^m
SiC	90	250	400	500	600	650
TiC	120	300	420	500	600	750 ^m
ZrC	120	250	350	420	490	560 ^m
CrC	130	250	330	390 ^m	450 ^m	500 ^m

3. Resistance to aggressive influences

Epoxy plastics (based on ED20 and analogs) are very resistant to aggressive liquid and gaseous media. However, there are a number of solvents and media that are very corrosive to polyepoxides, and enhancing resistance to them remains an important practical task. These include acetone and its solutions (including ethyl acetate - nail polish remover), nitric

acid (the more aggressive, the stronger), chlorocarbons, peroxides [13-15].

Figure 6 shows that carbide-filled composites are less stable in nitric acid than conventional polymer. An exception is NbC, which does not change the nature of the swelling of the composite.

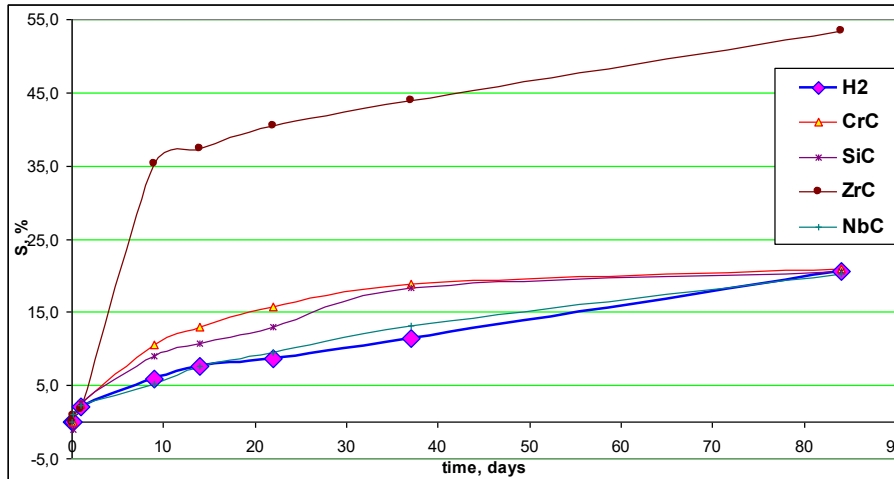


Fig 6: Swelling curves of composites in 20% HNO₃, H2 - unfilled polymer, the rest - with 50 wt% of the corresponding carbides

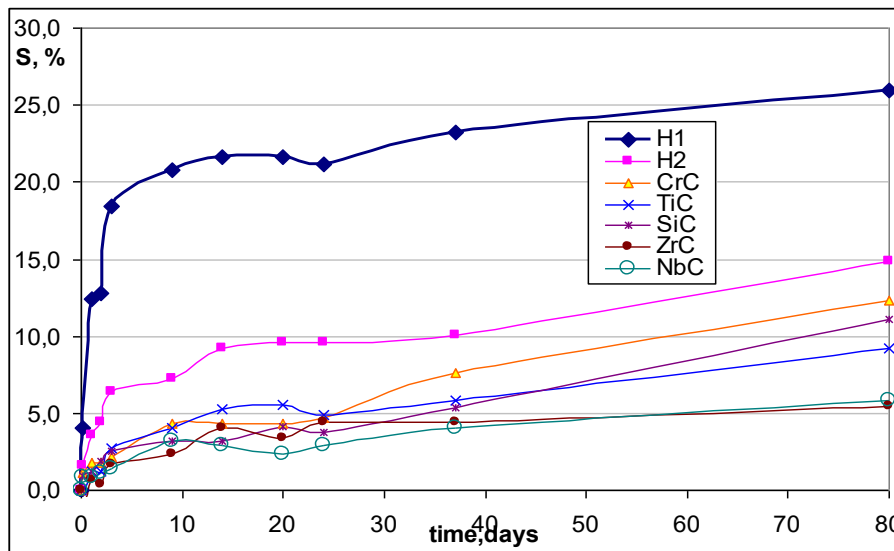


Fig 7: Swelling curves of composites in acetone. H2 - unfilled polymer, the rest - with 50 wt% of the corresponding carbides

Carbides can significantly enhance the resistance in acetone: the swelling activity is reduced by 3-4 times (Fig 7). The effect of various carbides is \approx the same. Sometimes there is some correlation with behavior in HNO₃. Thus, samples with NbC (it is the most stable in HNO₃) and ZrC (the least stable in HNO₃) are characterized by the smallest swelling. This suggests that the resistance in these media depends not only on the structure of the polymer phase in the filled

composites, but also on the resistance to acetone and HNO₃ of the fillers themselves.

4. Thermooxidative destruction

Table 5 shows the manifold increase in fire resistance of polyepoxide after filling with carbides. Table 5 shows a clear multiple increase in the fire resistance of polyepoxide after filling with carbides. composites have the ability to delay combustion up to 6-7 seconds after the fire is brought up.

Table 5: Start time and character of burning, at open flame exposure. S/In - Self-ignites, S/D - self-extinguishing after inflammation

	Unfilled	SiC	ZrC	TiC	CrC	Mo ₂ C	NbC
Time, sec	1-2	4-5	4-5	5-6	5-6	4-5	4-5
Character of burning	S/In	S/D	S/D	S/D	S/D	S/D	S/D
Time after which the composite self-ignites, sec	1-2	5-6	5-6	6-7	6-7	5-6	6-7

From Fig 8 it can be seen that the thermal oxidative destruction after filling changes its character

only in the segment of high-temperature burnout (“burnout of the coke residue” [10]).

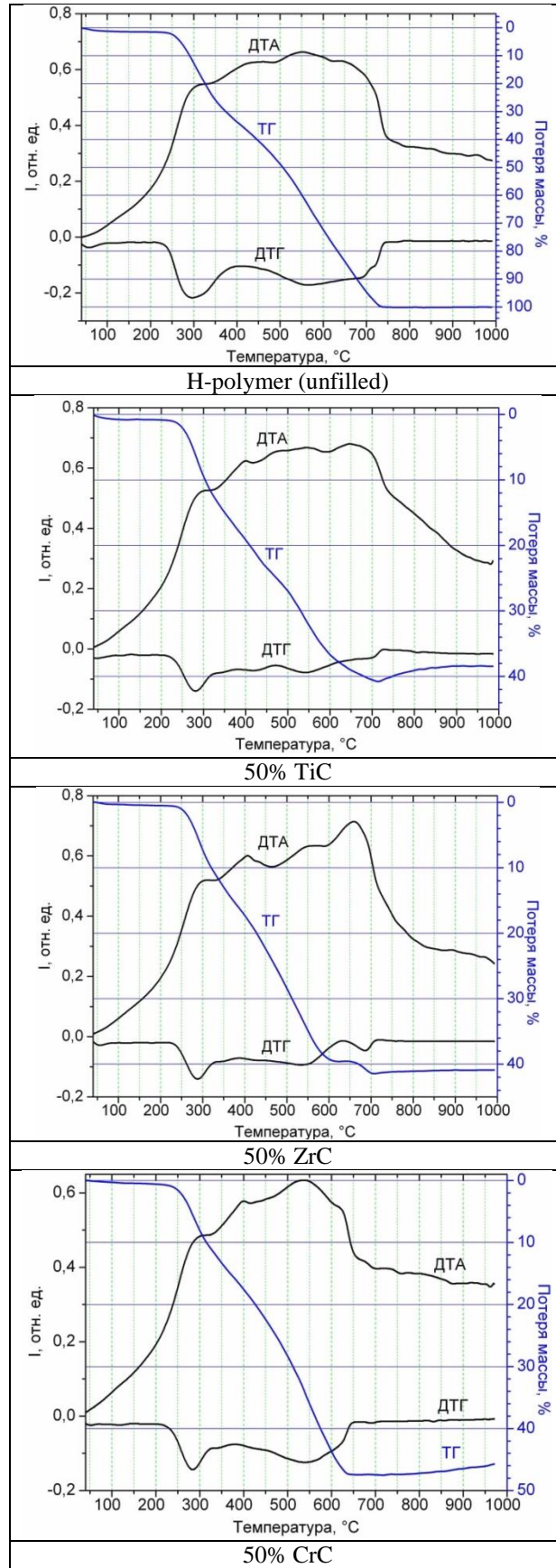


Fig 8: Thermograms of oxidative destruction of the studied polyepoxides. Right Y-axis – Loss of masse, %; Left Y-axis – I, relative units

The process of primary decomposition (PDR) at 250-300 °C (in which the polyepoxide is carbonized and loses its strength/resistance) in all cases retains a similar appearance. Filling by carbides (refractory materials) does not increase the temperature of the PDR maximum (see position of DTG-curve's minimum, Fig 8). On the contrary, its peak shifts "to the left" - from 280-290 °C for H-polymer to 270-280 °C for filled ones (Fig 8). The differences are only in quantitative estimates: after 300 °C, the H-polymer immediately loses up to 35-40% of its mass, and the filled ones - only 10-15% (Fig 9). This allows say that the temperature of 10% weight loss increases significantly after filling: from 290 °C for the H-polymer to 300 °C for TiC and up to 320 °C for ZrC and CrC (Fig 8).

Also, the weight of the completely burned-out mass is significantly reduced: from 100% for unfilled H-polymer, to 35-45% for 50 wt% filled composites. Note that 35-45% is noticeably less than 50 wt%, i.e. filling allows you to keep 5-15% of the polymer mass in the unburnt residue. In an applied aspect, this shows the important fire protection value of filling for industrial, restorative and domestic epoxy composites.

CONCLUSIONS

1. The introduction of 50 wt% of all investigated carbides (Si, Ti, Zr, Cr, Mo & Nb) makes it possible to obtain viscous masses, which, after curing, transform into well-formed composites of moderate viscosity.
2. The morphology of the compositions reflects the aggregate distribution of microparticles, which are relatively well incorporated into the supramolecular structure of the epoxy resin. In this case, the composition contains both nanomicro- and mesofragments of the filler.
3. Filling increases the most of the investigated strength characteristics - compressive strength, modulus of elasticity in compression and bending, abrasion resistance, adhesion to steel. An important consequence of filling is a noticeable increase in strength C (yield point) and modulus in compression in all cases. Compressive strength C increases by 15-30% (especially for SiC and TiC₂), - and this tendency persists after holding the samples in water (when C decreases slightly). The elastic modulus also grows in the same way (by 15-30%, for the case with CrC - by 36%), which is especially noticeable after exposure in water (an increase of 40-55%).
4. After filling, the resistance of polyepoxide to aggressive liquids can increase (for example acetone) or decreases (in 20% HNO₃). Also, after filling, the fire resistance of composites increases by 1.3-2 times.

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Absence of conflict of interest

The authors declare that they have no conflicts of interest requiring disclosure in this article.

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