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Increase of the resistance to high-temperature effects of carbon composite materials

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Abstract. The article considers the possibility of increasing resistance to high-temperature effects of carbon composite polymeric materials by combining the components of the binder and its modification by silicon carbide powder. The technology for producing a single-layer composite is described. According to the results of the experiments, the composition and amount of the binder and SiC additives leading to a significant reduction in mass loss during high-temperature tests were revealed.

1. Introduction

Modern aircraft designs are aimed at the transition to high power and flight speeds, which in turn leads to an increase in operating temperatures and aggressiveness of working units and parts of aerospace engineering. Accordingly, the requirements for materials used are toughened: for example, temperature resistance, resistance to thermal shock, and resistance ablation, erosion, oxidation, strength characteristics, weight and size parameters, etc. Therefore, obtaining and improving high-temperature materials used as heat-protective coatings (HPC) in aircraft structures is an urgent task.

Carbon fiber-based polymer composite materials (PCMs) have a high density and greater processability compared to high-temperature ceramics.

Some groups of polymers such as phenol-aldehyde, organosilicon, polyimide and their compositions can be considered in role of matrices.

Phenol-formaldehyde resins and organosilicon compounds are of the greatest interest for the creation of thermoelectric heaters operating at temperatures above 1500 °C.

Resol phenol-formaldehyde resins (PF) have high thermal stability and are able to withstand high temperatures in the range of 1000-1500 °C for several seconds [1,2]. PF are widely used to produce carbon-carbon materials through high-temperature pyrolysis. For example, a matrix based on carbon fibers is impregnated with a phenol-formaldehyde resin and then pyrolyzed at 2000 °C to form a strong coke residue, which remains until the carbon sublimation temperature [1].

Thus, FP can be a promising material as a PCM matrix developed for products subject to short-term high-temperature effects in the range of 1300-3600K.

Organosilicon polymers have elasticity, heat resistance, heat resistance, weather resistance, resistance to gases at elevated temperatures.

One of the most important properties is the formation during high-temperature degradation of silicon and its compounds (carbides, nitrides, oxides, etc.) [3,4], which have higher melting and sublimation temperatures. This property is widely used to obtain ceramic matrices in composite materials that effectively protect against external influences. For example, under the influence of high temperatures, the healing properties of SiO₂ are used, which is formed as a result of oxidation of the SiC matrix or Si and restricts the access of oxygen to the fiber surface; such a matrix works for up to 1650-1700 °C for several hours. [4] Organosilicon is widely used for heat-shielding materials in China and Japan [4], active development is underway in the Russian Federation: for example, a quasi-plastic composite material reinforced with carbon fibers UKN-5000 LU-24P and organoelement polymers with a temperature resistance of up to 1500 °C; the Steklarm is a material with a glass-ceramic matrix obtained from polymer



precursors (polysilosanes, polycarbosilanes, polyborethoxysilanes), it have high-strength heat-resistant at 1500 ° C[6,7].

Such properties of organosilicon polymers as the elasticity in its original form and the formation of a ceramic component under the influence of heating of the working medium under operating conditions can be successfully implemented to protect products that must be flexible, withstand harsh high-temperature environmental conditions.

Fillers and catalysts, modifying additives, flame retardants, such as metals and their compounds, silicon-based compounds, carbon, calcium fluorides, phosphorus-based flame retardants, ceramic powders, and other various sizes and quantities are actively used to increase operational characteristics [8-11].

For example, it is possible to use finely dispersed fillers, including finely dispersed silicon carbides, and thermally expanding graphite as a high-temperature additive in elastomeric matrices [10].

The introduction of flame retardants into a polymer binder can have a significant effect on increasing the heat resistance of polymer composites used for parts operating in oxidizing environments.

In this work we consider layered composite materials based on carbon fibers with a matrix obtained from a mixture of phenol-formaldehyde resin and an organosilicon polymer with inorganic additives to create thin-walled HPC.

2. Materials and methods

The studied materials are single-layer composite materials based on carbon fibers. The polymer matrix consists of phenol-formaldehyde resin in the form of a powder, silicone and hardener. Micron powder of silicon carbide was used as a modifying additive.

Composite materials are made by traditional technological methods of liquid-phase combination of filler and matrix.

The PF powder or a solution of PF in isopropyl alcohol is mixed with silicone and then subjected to intensive mixing using the Ultraturex apparatus for 2-3 minutes. After that, silicon carbide powder and condensation agent are added to the mixture.

The samples are dried at 60 ° C for 4 hours and cured at 120 ° C for 4 hours after uniformly application a binder to the carbon fabrics layer.

To determine the optimal composition of the samples, fire resistance tests are carried out in the flame of an acetylene burner for 60 s. After the test, the relative weight loss is determined.

3. Results and discussions

To determine the ratio of the main components, a series of samples was prepared with matrices containing parts by weight of components: silicone without PF, silicone: PF (3: 1), silicone: PF (2: 1), silicone: PF (1: 1), silicone : PF (1: 2), silicone: PF (1: 3). PF was dissolved in isopropyl alcohol. The test results in the burner flame are shown in Figure 1.

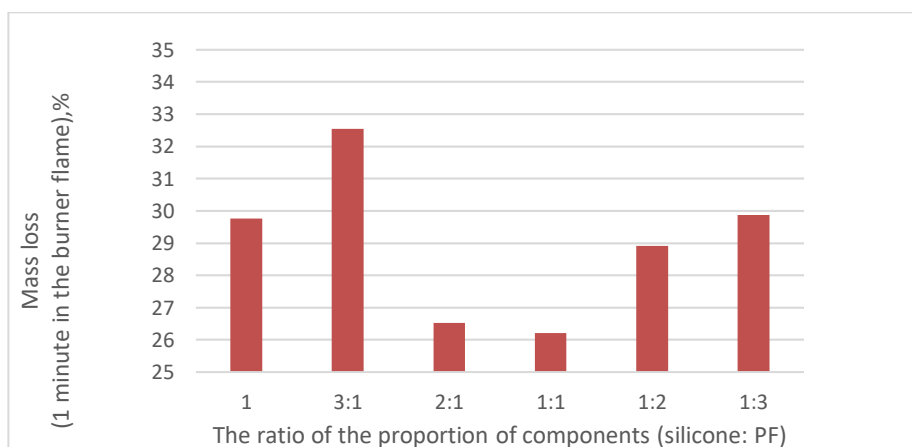


Figure 1.Dependence of mass loss after exposure to flame for a minute on the ratio of the proportions of polymer binders.

The smallest weight loss is exemplified by the compositions: silicone: FFS (2: 1) - 26.52%, silicone: FFS (1: 1) - 26.21%. However, the latter has a higher viscosity, therefore, the polymer composition silicone: FFS (2: 1) was chosen as the basis for further tests.

To assess the effect of silicon carbide particles on the fire resistance of polymer composites, samples were prepared based on silicone and phenol-formaldehyde resin powder (2: 1) and silicon carbide additives in amounts of 1, 3.5% of the mass.

Photographs of the original structure are shown in Figures 2-5.



Figure 2. Microstructure of samples based on silicone and phenol-formaldehyde resin powder (2: 1) without silicon carbide additives (x100).

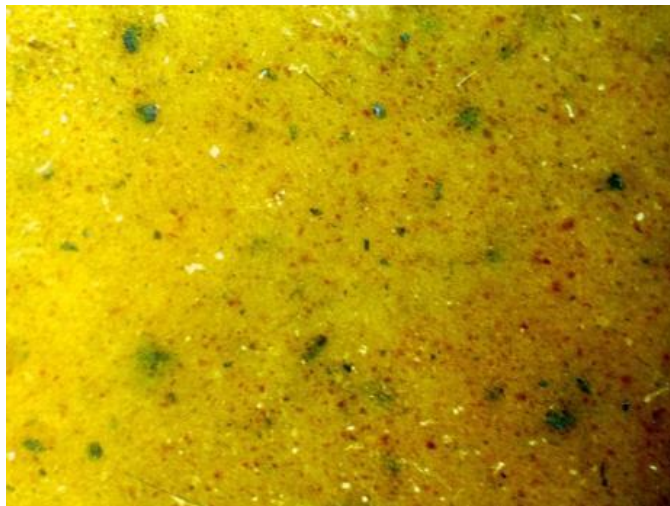


Figure 3. Microstructure of samples based on silicone and phenol-formaldehyde resin powder (2: 1) with the addition of silicon carbide in an amount of 1% mass (x100).



Figure 4. The microstructure of samples based on silicone and phenol-formaldehyde resin powder (2: 1) with the addition of silicon carbide in an amount of 3% by mass (x100).

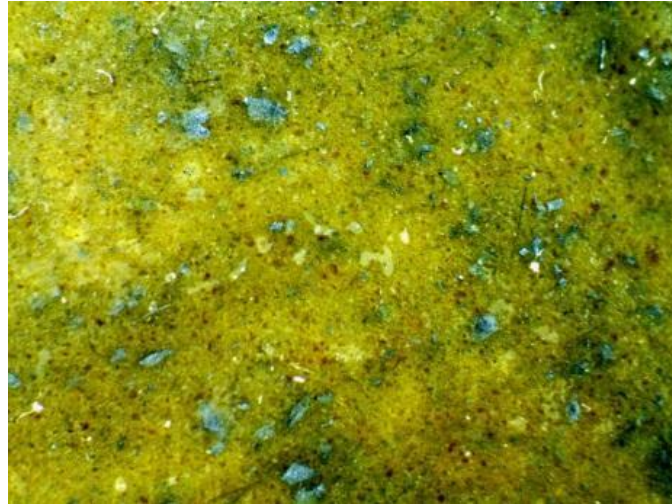


Figure 5. Microstructure of samples based on silicone and phenol-formaldehyde resin powder (2: 1) with the addition of silicon carbide in an amount of 5% by mass (x100).

The microstructure of the polymer matrix shows continuity, uniformity, the added silicon carbide particles are distributed evenly throughout the binder.

Burner flame test results are shown in Figure 6.

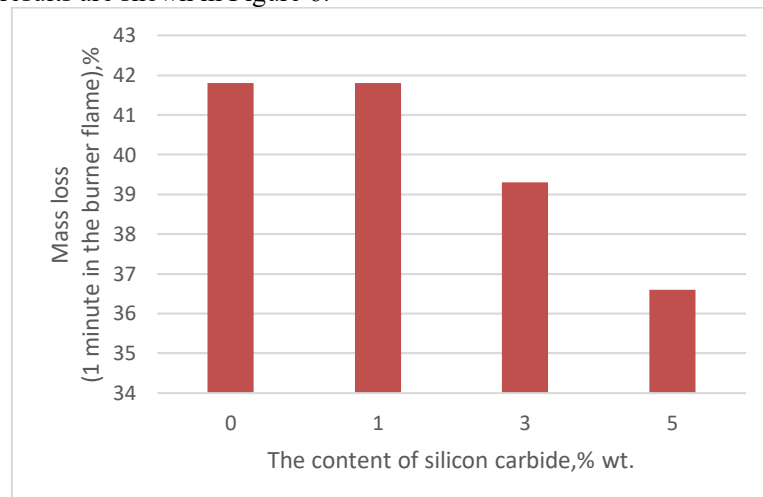


Figure 6. Effect of the addition of silicon carbide on weight loss after exposure to flame for a minute

According to the results of the introduction of the additive, there is a tendency to reduce weight loss during combustion. The best result is shown by a sample with the addition of 5% SiC. The relative change in sample weight decreased from 41.8% for samples without additives to 36.6% for samples containing 5% SiC. A 15.2% reduction in losses indicates the effectiveness of the modification of the binder with a ceramic filler.

Samples after testing are shown in Figures 7-10.

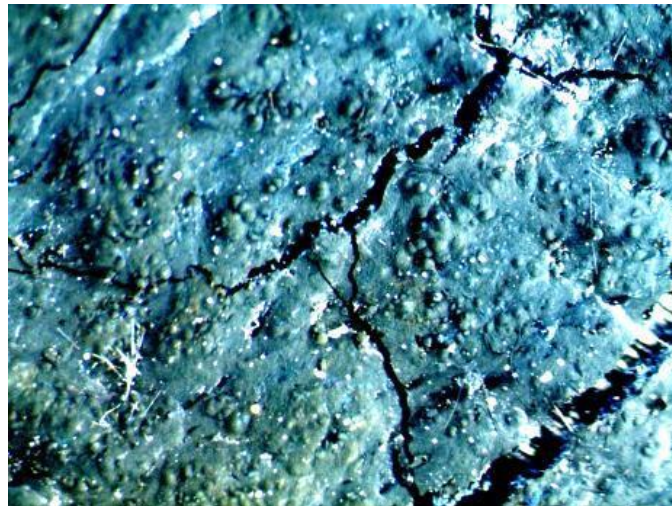


Figure 7. The surface after testing in a flame of a burner of samples based on silicone and phenol-formaldehyde resin powder (2: 1) without additives of silicon carbide.

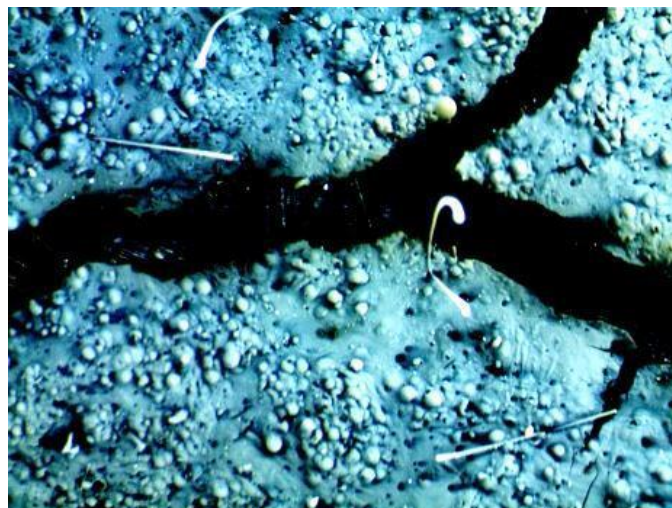


Figure 8. The surface after testing in a flame of a burner of samples based on silicone and phenol-formaldehyde resin powder (2: 1) with the addition of silicon carbide in an amount of 1% mass (x100).

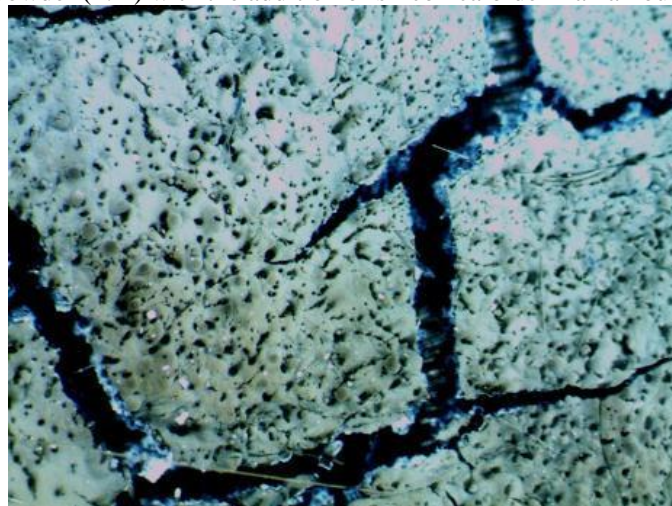


Figure 9. The surface after testing in a flame of a burner of samples based on silicone and phenol-formaldehyde resin powder (2: 1) with the addition of silicon carbide in an amount of 3% by mass (x100).

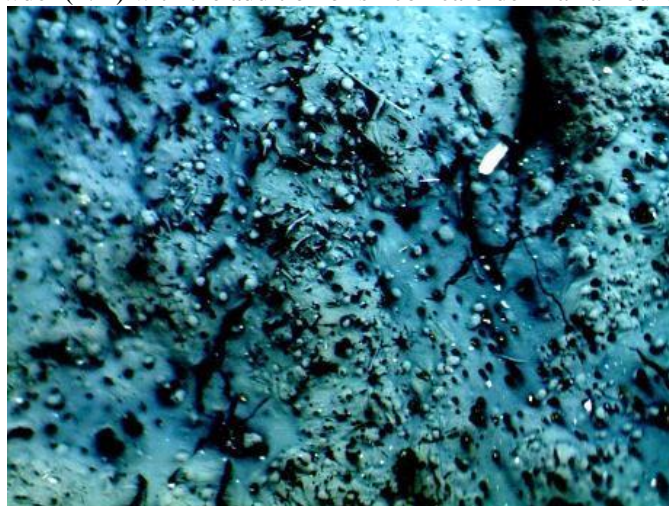


Figure 10. The surface after testing in a flame of a burner of samples based on silicone and phenol-formaldehyde resin powder (2: 1) with the addition of silicon carbide in an amount of 5% by mass (x100). In the process of flame exposure, a carbon residue forms on the surface of the samples, which protects the carbon fibers from oxidation. The presence of spherical oxides is observed.

4. Conclusions

Thus, a silicone-phenol-formaldehyde resin-silicon carbide system composite has been proposed as heat-protective thin-walled composite materials. Based on the results of the experimental data, the composition of the binder silicone was selected as the basis: phenol-formaldehyde resin with respect to parts 2: 1 by weight. The results of the experiments revealed the effect of inorganic additives of silicon carbide on reducing weight loss. The most effective is the addition of 5% SiC. Samples with this content show a loss reduction of 15.2% relative to unfilled binder. Thus, the combination of the considered polymers and the modification of the mixture with the addition of silicon carbide leads to an increase in the resistance to high temperatures of the material in an oxidizing environment. This composite can be recommended as a heat-shielding material for parts and components of aviation and space technology.

References

- [1] Sabadaha E N, Prokopchuk N P, Schutova A L and Globa A I 2017 Thermostable composites//*Works of BGTU. Series 2 Chemical technologies, biotechnology, geoecology*. **2**(2) Page 108-115.
- [2] Pakhomova SA and Povalyayev A I 2019 Silicon nitride-based ceramic composite materials for corrosion-resistant rolling bearings // IOP Conf. Series: Materials Science and Engineering 683 012040. DOI:10.1088/1757-899X/683/1/012040
- [3] Rudakova T A, Perov N S and Ozerin A N 2011 Fire protection elastic coatings on organosilicon binder//*News of UFU. Technical sciences..* Page 215-219.
- [4] Garshin A P and Kulik V I 2017 Oznaya directions of increasing corrosion and heat resistance of refractory fibre-reinforced ceramic-matrix composites//*Scientific research and development.. T. 12. Page 49-59.*
- [5] Kraev I D and other. 2017 Prospects for the use of organosilicon elastomers in the development of modern polymer materials and coatings for various purposes // *Proc. VIAM*. **12** C. 5–5.
- [6] Suntsev S S 2010 High-temperature composites and coatings based on glass and ceramics for aerospace engineering//*Ros. chemical. G. (J. Ros. chemical. D.I. Mendeleeva).. T. 1. Page 25-33.*
- [7] Gunyaev G M and Gofin M J 2013 Carbon-carbon compositions//*Aviation materials and technologies..* Page 62-90.
- [8] Polonik V D and Prokopchuk N R 2013 Properties of elastomeric compositions with fluorine-containing additive//*Works of BGTU. Series 2 Chemical technologies, biotechnology, geoecology* **4** Page 141-143.

- [9] Kablov V F, Novopoltseva O M, Kochetkov V G, Lapina A G and Zvada S S 2015 Study of the influence of fine swelling systems of flame retardants on fire resistance of elastomeric compositions//*Journal of Technological University*. **18(14)** Page 1-2.
- [10] Patent RU № 2594014. Method of producing binder based on phenol-formaldehyde resin of rubber type for laminar material, binder and laminate based on binder and reinforcing fibrous base. Federal State Budget Educational Institution of Higher Education "Kazan National Research Technology
- [11] Shvets N I et al. 2013 Phenol formaldehyde binder of reduced combustion//*Fire and explosion safety* **22(5)** Page 26-32.