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Obtaining a composite material based on quartz woven filler and pyrolysis matrix of organosilicon resin

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ABSTRACT

The relevance of the study is conditioned by the fact that the most popular and irreplaceable materials that have found wide application in the aerospace industry are composites based on quartz materials. These materials are distinguished by their high mechanical and electrical strength, chemical and corrosion resistance. In this regard, it is of interest to obtain a composite material that combines a low specific gravity, processability of polymers, and thermal stability of ceramics. The aim of this work was to study the effect of the temperature of thermal oxidative destruction of a polymer binder, which is a semi-finished product of a pyrolysis matrix, on the electrophysical parameters of a composite material. The paper investigates a composite material based on woven quartz material with a pyrolysis matrix of an organosilicon binder and functional additives. This composite was considered as a material for creating an electric rocket engine chamber. Thermogravimetric analysis was used to evaluate the effect of the temperature of thermooxidative degradation of the polymer binder on the electro-physical parameters of the obtained material. The tests were carried out according to standard test methods on an Instron 5969 universal testing machine with Bluehill software until the samples failed. In the course of the study, it was found that the processes that occur up to 400°C are mainly associated with the course of the reaction for non-entered functional groups, the telomerisation reaction, intramolecular rearrangement of macromolecules and the removal of low-boiling substances. According to the results of the study, the obtained characteristics of the test material turned out to be suitable for its use in structural elements of electric propulsion engines.

Keywords: Temperature, Dielectric strength, Composite material, Electric propulsion

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1. Introduction

Quartz materials have found wide application in the composition of composite materials [1-5]. In terms of a number of functional indicators (transparency in the infrared, ultraviolet and visible spectra of optical radiation, high specific mechanical and electrical strength, chemical and corrosion resistance), composites based on quartz materials are in demand and indispensable in the aerospace industry. Quartz materials can act as a matrix and as a filler for glass-ceramic and ceramic composites [6-9]. Fiberglass plastics with a polymer matrix, where quartz fibres act as a filler, are also used as high-temperature radio-transparent composite materials for antenna fairings and other aircraft products [10-12].
Fiberglass plastics with a polymer matrix based on organic and organosilicon binders are characterised by high transparency for radio waves and electrical strength. They have high dielectric properties and high specific mechanical strength. The disadvantage of fiberglass is the low operating temperature (not higher than +300°C), which is due to the beginning of the destruction of the polymer matrix of the composite material

[13-17].
Earlier, a composite material based on an organosilicon binder and a woven quartz material was investigated [18-23], which was proposed for the manufacture of dielectric assemblies of an electric rocket engine (ERE). EREs are widely used in space technology. With the help of ERE, stabilisation and correction of geostationary satellites is carried out [24-28]. The pulse of a high-frequency ion engine, one of the most demanded types of ERE, is formed when the ionisation products of the working medium plasma are accelerated in the electromagnetic field of the HF generator (Figure 1).

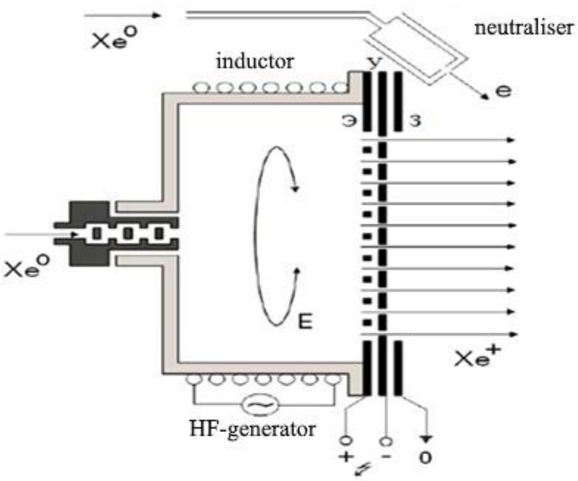

Figure 1. Schematic diagram of the operation of a high-frequency ion engine.

Ionisation of the working gas is carried out in the internal volume of the gas-discharge chamber (GDC). One of the essential requirements for the GDC material is a high dielectric strength of 15-20 V/mm. As shown by earlier tests of GDC [29-35], these values of dielectric strength correspond to organosilicon rubbers and ceramic composite materials based on silicon nitride and aluminium oxide [36]. However, polymeric materials do not meet the operational requirements of the GDC in terms of thermal stability at an operating temperature of the GDC above +400°C. Ceramic materials, due to their high density, significantly increase the mass of gas discharge tubes (in comparison with polymeric ones). In addition, these materials have low manufacturability and vibration resistance, especially in the manufacture of gas discharge tubes with a diameter of 300 mm and more [37-42].
In this regard, it is of interest to obtain a composite material that combines a low specific gravity, processability of polymers, and thermal stability of ceramics. Such a composite material would be represented by a matrix obtained as a result of pyrolysis of an organosilicon binder with a quartz woven filler [43-48]. Thus, the purpose of this work was to study the effect of the temperature of thermal oxidative destruction of a polymer binder, which is a semi-finished product of a pyrolysis matrix, on the electrophysical parameters of a composite material.

2. Materials and methods

The initial polymer was obtained by partial cohydrolysis in the heterosopolycondensation reaction of tetramethoxysilane with methyltrimethoxysilane, phenyltrimethoxysilane in acetonitrile solution [49]. The finished product represented 22-25% of the mass solution in acetonitrile, with a viscosity average molecular weight of 2700-3500 g/mol. Interesting results were obtained in [50]. As a reinforcing filler, we used quartz woven material of the TS-8/3-K brand, produced by the Research and production association "Stekloplastik". The properties of silica fibres are presented in Table 1.

Table 1. TS-8/3-K quartz fabric indicators

Weaving type	Mass, g/m^2	Gauge, mm	Tensile strength, N (kg*s)		Threads per unit length	
			warp-wise	filling-wise	warp-wise	filling-wise
satin 8/3	290	0.28	1078	588	36	20

As a functional additive, we used silicon nitride whiskers (α-Si_3N_4), with a purity of 99.99 wt %, obtained in the SHS process [1]. The prepreg was obtained by impregnating a quartz fabric with a solution of organosilicon resin (23-27 wt %) and a silicon nitride powder suspended in it (20 wt %). The impregnated fabric was laid out in layers and dried at a residual pressure of 1-3 mm Hg and a temperature of 150°C. The composite material was obtained by curing in an autoclave at 270°C and a pressure of 6 Atm. The curing process was carried out for 2 hours. The study of structural transformations occurring during heating of materials was carried out using the methods of synchronous thermal analysis on an STA 449 F3 Jupiter device from NETZSCH (Germany) (see Figure 2) in the differential scanning calorimetry (DSC) mode in conjunction with the thermogravimetric analysis (TGA) mode [51-56].

Figure 2. STA 449 F3 Jupiter device from NETZSCH (Germany).

The samples were heated to 900°C in open Al_2O_3 crucibles with a volume of 0.085 ml at a linearly increasing furnace temperature at a rate of 4°C/min. The crucibles were placed in the cylindrical recesses of the upper part of the DSC/TG sensor made of Pt-Rh alloy. The argon flow rate through the measuring cell (sample and standard) during the experiment was 50 ml/min. An empty crucible was used as a standard. Sample and reference temperatures were measured using built-in S-type thermocouples made of Pt-Rh alloys. The temperature measurement accuracy was ±0.3°C. The change in the mass of the samples was recorded with an accuracy of 1 μg. The isothermal drift of the balance in the entire temperature range did not exceed 10 μg/h. Data collection and calculations, as well as control of the device operation were performed using the NETZSCH Proteus Software (Germany) running under the MS-Windows shell [57].

Thermal destruction of CM samples was carried out in a chamber electric furnace LE 4/11/R6 (Nabertherm, Germany) with a working volume of 4 L in air under conditions of gradual heating at a rate of 5°C/min. Weighed samples in corundum crucibles were loaded into a furnace at room temperature, heated and kept on an isotherm (in the range of 270...1000°C) for a specified time, removed from the furnace and, after complete cooling in air, were reweighed. Determination of open porosity and apparent density of carbon-carbon composite materials (CCCM) was carried out by the method of hydrostatic weighing in accordance with GOST R ISO 12985-2-2014 (ISO 12985-2: 2000). The samples were weighed on a GR-202 analytical balance (AND, Japan) with an accuracy of 10–4 g [58].

3. Results and discussion

Strength tests were carried out based on GOST R 56810-2015 (ASTM D790, ISO 178). Polymer composites. Bending test method for flat specimens. The tests were carried out according to standard test methods on an Instron 5969 universal testing machine with Bluehill software. The loading rate was taken equal to 1 mm/min. The tests were performed until the destruction of the samples. For each tested batch, a stress-strain curve was plotted, and the following were determined: strength in bending, modulus of elasticity in bending, ultimate deformations [59; 60].

The choice of studying the mechanical characteristics by bending testing is primarily due to the high requirements for the vibration resistance of the GDC material, as well as the possibility of studying the nature of destruction and determining the apparent interlayer shear strength by the bending test method using the short beam method. However, when testing based on standards GOST 32659-2014 (ISO 14130: 1997) and

ASTM D2344, no interlayer shear failure was obtained. Only plastic shear was recorded on the tested samples. Therefore, it is accepted to perform tests in accordance with GOST R 56810-2015 (ASTM D790, ISO 178) [61].

Figure 3 presents a graph of DTA in air for a composite material sample filled with Si3N4. The process of thermooxidative destruction is accompanied by the oxidation of methyl, aromatic substituents at the silicon atom [62].

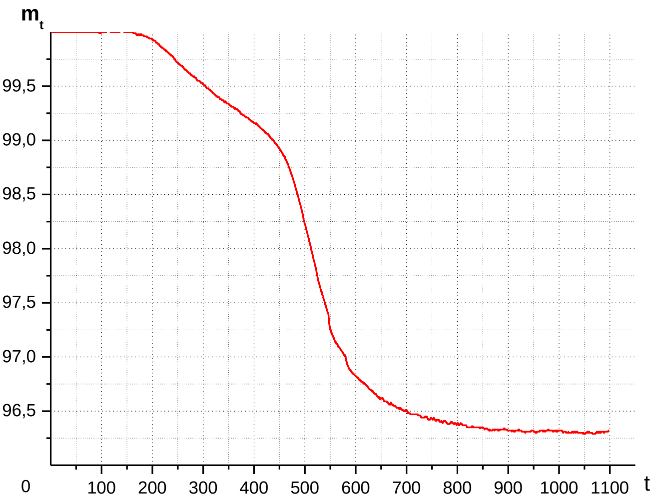

Figure 3. Temperature dependence of weight loss during DTA of the product of partial hydrolysis of Si(OMe)4, MeSi(OMe)3, PhSi(OMe)3 in air.

As a result, amorphous silicon oxide is formed [8]. The yield of the pyrolysis residue is more than 95%. Weight loss up to 400°C is associated with the condensation of end methoxyl groups and the release of methanol. In this region, the molecular weight of the polymer increases slightly. The increase in weight loss above 450°C is due to the initiation of the oxidation process of the side framing of the polymer chain. Thus, the processes that take place up to 400°C are mainly associated with the course of the reaction on non-entered functional groups, the telomerisation reaction, intramolecular rearrangement of macromolecules and the removal of low-boiling substances. Table 2 presents data on the change in density and porosity depending on the temperature of thermal oxidative destruction [63-67].

Table 2. Data on the change in density and porosity depending on the temperature of thermal oxidative destruction

T, °C	Apparent density,g/cm^3	Porosity, %	Specific mass, g/cm^3
270	1.5457	0.17	1.5483
400	1.5502	0.39	1.5562
500	1.4989	1.64	1.5238
650	1.4256	2.96	1.4690
750	1.3744	3.73	1.4276
900	1.3513	3.90	1.4061
1000	1.3456	4.11	1.4032

Despite the process of curing the organosilicon binder in an autoclave at 6 Atm, the porosity of the obtained fiberglass is 0.17-0.2%. Probably, this value of porosity is due to the hydrolysis of methoxyl groups at the silicon atom during hydrostatic weighing. The proportional increase in porosity versus temperature is consistent with the weight loss data (Figure 3). This circumstance indicates the absence of closed pores in the structure of the initial composite. Quantitatively, the process of thermal destruction is completed at a temperature of about 800°C. However, according to the data given in Table 2, there is a decrease in density and an increase in porosity. The reason for this phenomenon lies in the formation of the crystallobalite phase, as a result of the α-SiO2 phase transition of quartz fibres of the woven filler. This is also indicated by the data on the change in tensile strength with temperature, presented in Figure 4 [68].

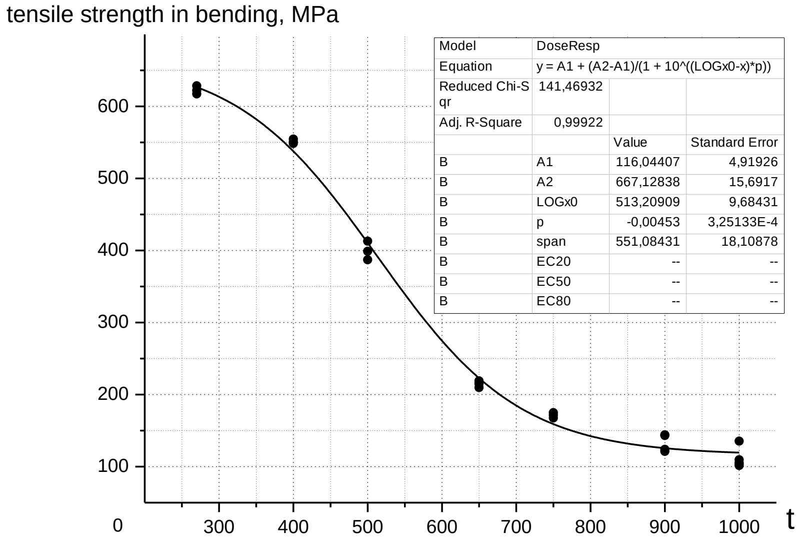

Figure 4. Dependence of the ultimate bending strength of a composite material depending on the temperature of thermo-oxidative destruction.

According to the data presented, degradation of mechanical properties is observed after oxidation of the polymer matrix of the composite. Since the strength properties of composite materials are mainly determined by the mechanical strength of the filler, it can be assumed that the softening of quartz fibres is due to the polymorphic transformation (density 2.56...2.53 g/cm^3) into the crystallobalite phase. This is also indicated by a decrease in the density of the composite above 750°C, which correlates with a decrease in the SiO_2 density after two successive second-order phase transitions from quartz (density 2.56...2.53 g/cm3) to tridymite and then to crystallobalite (whose density is 2.33...2.34 g/cm^3). When comparing the DTA data and the degradation of mechanical strength, the co-directionality of the dependences under consideration is clearly traced. As a rule, the dependence of the bending strength on the temperature of thermal oxidative destruction is exponential, but in this case, the dependence of the ultimate strength on the temperature of thermal oxidative destruction (Figure 4) contains two conjugate regions [69].

In the process of condensation along the terminal methoxyl groups, up to a temperature of 400-450°C, the change in mechanical properties is due to structural changes in the polymer matrix at the interface. Above 450°C, the oxidation of the polymer binder leads to disruption of the contact interaction at the matrix-filler interface. With a further increase to a temperature of 800-850°C, filler-quartz fibres undergo significant degradation as a result of a phase transition [70].

4. Conclusion

Within the framework of this study, the authors investigated the effect of the temperature of thermal oxidative destruction on the properties of a composite material with a pyrolysis matrix. The processes that proceeded up to 400°C were mainly associated with the progress of the reaction on non-entered functional groups, the telomerisation reaction, intramolecular rearrangement of macromolecules and the removal of low-boiling substances. The proportional increase in porosity as a function of temperature is consistent with the weight loss data, which indicates the absence of closed pores in the structure of the original composite. At a temperature of 800°C, a decrease in density and an increase in porosity are observed. In this case, the dependence of the ultimate strength on the temperature of thermooxidative destruction contains two conjugate sections.

Based on the cumulative experimental data presented in Figs. 3-4, it can be concluded that at a temperature of about 800-850°C, the strength of the composite material with a pyrolysis matrix is about 100 MPa, and this, most likely, is sufficient for the use of this material in the structural elements of an electric propulsion engine. Thus, the investigated material can be considered as the basis for the manufacture of the GDC ERE.

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