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CARBON PLASTICS FOR REUSABLE HYPERSONIC FLIGHT VEHICLES

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The development of hypersonic unmanned aerial vehicles (UAVs) for aerospace systems presents ambitious challenges for scientists and engineers. Extreme flight conditions, such as ultra-high speeds and significant aerodynamic heating, necessitate the creation of new materials capable of withstanding such loads. One of the most promising materials for constructing hypersonic UAVs is carbon fiber-reinforced polymer based on bisphenol nitrile. This material exhibits high thermal resistance, chemical stability, and excellent mechanical properties. Utilizing bisphenol nitrile combined with carbon fibers has enabled the production of composite materials that can operate at temperatures exceeding 300^{0}C, far surpassing the capabilities of traditional polymer matrices. To assess the suitability of the developed carbon fiber-reinforced plastic for hypersonic UAV applications, comprehensive studies of its physical, mechanical, and thermal characteristics were conducted across a wide temperature range from 20 to 300^{0}C. The obtained results provided a detailed characterization of the composite and allowed for comparisons with other high-temperature composite materials. The developed carbon fibre-reinforced plastic based on bisphenol nitrile binder shows great promise for constructing hypersonic UAVs. Its high thermal resistance, combined with excellent mechanical properties, makes it suitable for use in the extreme temperature conditions typical of hypersonic flight.

Keywords: aerospace and rocket technology, design parameters, hypersonic unmanned aerial vehicle, heat-resistant binders, carbon fiber-reinforced plastic, polymer composite materials, physical, mechanical, and thermal characteristics.

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Introduction

For regular space flights, the use of aerospace systems based on hypersonic unmanned aerial vehicles and single-use space rockets is cost-effective. The launch of payloads using hypersonic unmanned aerial vehicles and a single-use space rocket involves an aerial launch and landing on an aerodrome runway, which significantly reduces the cost of operating the system.

For a single-use space rocket, structural and heat-protective materials traditional in rocket and space technology are used. The creation of a reusable hypersonic unmanned aerial vehicle requires the use of new materials capable of repeatedly withstanding extreme thermal loads.

Based on the analysis of the parameters of thermal effects, it is established that the temperatures on the surface of a hypersonic unmanned aerial vehicle vary from 100 to 1350^{0}C [1–3]. The elements of structures according to their operating temperatures can be divided into several groups, which are shown in Table 1.

Temperatures of 100^0C are realized on fuel tanks of hypersonic unmanned aerial vehicles. Carbon plastics have the highest specific characteristics of all known materials, so it is reasonable to manufacture fuel tanks from this material. Materials, providing requirements of operation of structures working for a long time in other temperature ranges, require further development [1–3].

The main parts of the airframe undergo temperatures from 500 to 600^0C, and it is these structures that contribute significantly to the weight parameters of the UAV. Therefore, the selection and creation of materials with high values of specific strength, operable in this temperature range is an important scientific and technical task.

Thus, this study was aimed at selecting and developing materials intended for using in UAV with high specific strength at temperatures up to 600^0C.

Since carbon fiber-reinforced plastics have the highest specific strength characteristics, the weight of structures made of them is significantly lower than when using traditional metal alloys. Their main disadvantage is low operating temperatures of application, not exceeding 100^0C. At temperatures up to 600^0C carbon plastics can be used only with external heat protective coating.

Creation of new carbon composites with operating temperatures of 300^0C and more will reduce the thickness of the thermal protective coating and significantly reduce the weight of UAVs. Since the heat resistance of carbon fiber-reinforced plastics is determined by the operating temperatures of the binder, it is necessary to develop a new heat-resistant binder.

The use of reinforced composites for modern aerospace applications encourages the search for the high-temperature binders that are easy to handle and have high thermal and oxidative stability. Epoxy resins show temperature limitations when operating above 200^0C. The main problem with the polyimide system is chemical activity due to the volatile components. Other problems with both polyimides and epoxy resins include their brittleness, tendency to absorb water, and poor technical reliability.

Recently there has been a growing interest in the creation of polymer composite materials based on thermoplastic matrices. This is due to the appearance of a new class of heat-resistant linear polymers – polyether ketones. The main advantages of reinforced thermoplastics in comparison with polymer composites based on thermosetting binders are as follows: high resistance to fracture, crack resistance and post-impact strength; increased heat resistance (in comparison with traditional epoxy resins); resistance to aggressive media; unlimited long viability of prepregs; high speeds of technological cycles; and possibility of secondary processing and local elimination of defects.

Phthalonitrile resins are superior in physicochemical properties to epoxy resins, polyimides and other polymers as matrices for carbon composites. The main advantage of phthalonitrile resins over other polymers is their ability to withstand temperatures in excess of 300^0C for extended periods without damage to coatings, plastics or composites made from them. Resins with the presence of flexible bonds between aromatic rings minimise or significantly reduce brittleness and increase ductility. A number of phthalonitrile materials have been developed by experts in the USA [3–5]. The object of these inventions are compositions and methods for the preparation of phthalonitrile resins, which have high thermal and oxidation resistance and have good mechanical properties at temperatures above 300^0C. However, polymerization and synthesis of these monomers generally require heating for several days at very high temperatures.

Binders of this class are produced in the form of powders, whose melts have low viscosity, which allows the application of pressure impregnation technologies (RTM, RFI, VARTM) for the manufacture of products from polymer composites. Expansion of application areas of phthalonitrile binders and polymer composite materials based on them is possible due to their high

Table 1

Classification of structural elements by operating temperature

Maximum operating temperatures, ^{0}C	Structural elements of the UAV
100	materials of fuel tank
500–600	wing trim, wing power components, rudder trim, rudder power components, nacelle trim, nacelle power components, fuselage trim and power components.
600–1000	materials of cladding and power components of the fuselage conical section
1000–1200	force elements and air duct shell of the air intake duct of a direct-flow air-jet engine
1350	fuselage nose, fuselage leading edge, wing nose, air intake edges, nose of rudders and rudder nose

mechanical properties. Phthalonitrile binders are also used together with other thermosetting components.

Bisphthalonitrile binders (BFB) represent a new promising class of high-temperature binders for polymer composite materials based on phthalonitrile resins. BFB are non-combustible (the only class of materials to meet the US Navy's fire resistance standard), have high strength properties and very low moisture absorption. BFB is composed of oligomers containing phthalonitrile end fragments (Fig. 1).

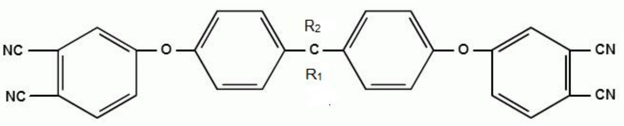


Fig. 1. Molecular structure of bisphthalonitrile

They are synthesized by nucleophilic substitution reaction between potassium salts of bisphenols and 4-nitrophthalonitrile. Polymerization of initial oligomers is carried out on nitrile groups with formation of heat-resistant cyclic structures, according to refs. [6,7].

The course of the BFB curing reaction depends on the type of hardener. Aromatic amines, phenols, metal complexes can serve as hardeners. The required amount of hardener is small compared to other 2-component systems. The reaction can be stopped by cooling the reaction mass. After that, it is started by heating and the conversion is brought to 100%, such polymer composite material can withstand peak loads up to 550⁰C, according to ref. [8].

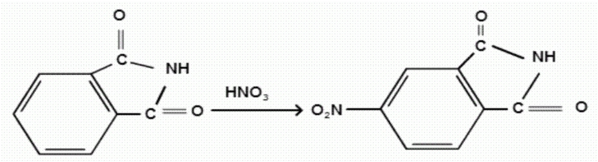
Based on the analysis of the world experience [9,10] and possibilities of synthesis and acquisition of initial components available in Ukraine, we developed in this study a binder based on bisphthalonitrile. It allows producing polymer composite material withstanding working temperature of 300–400⁰C with high physical and mechanical properties.

Experimental, results and discussion

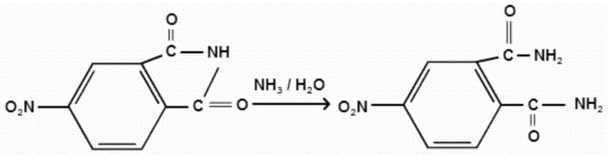
Synthesis of bisphthalonitrile monomer

Synthesis of bisphthalonitrile monomer includes the following steps [11]:

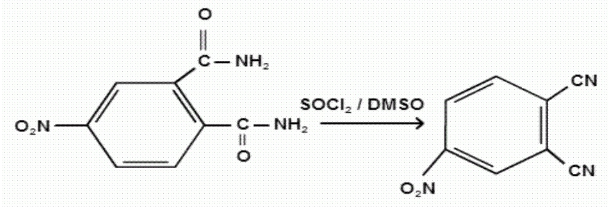
1. Preparation of 4-nitrophthalimide by nitration of phthalimide:



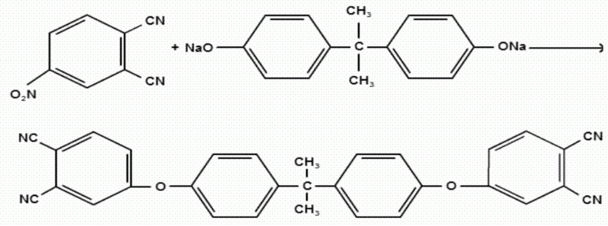
2. Hydrolysis of nitrophthalimide in ammonia water:



3. Preparation of 4-nitrophthalonitrile by interaction of nitrophthalimide with thionyl chloride in dimethyl sulfoxide solution:



4. Synthesis of bisphthalonitrile monomer by interaction of 4-nitrophthalonitrile with the sodium salt of bisphenol:



Production of a bisphthalonitrile binder

The production of bisphthalonitrile binder involves the following operations:

– introduction of heat-resistant polyfunctional novolac resin of DOW company (D.E.N.438). DOW's novolac epoxy resins have glass transition temperatures in the range of 120 to 255⁰C, which should provide high temperature resistance in cured bisphthalonitrile-based compositions and high adhesion to various substrates;

– use of hardener from the class of aromatic diamines (4,4'-diaminodiphenylmethane) and regulation of its content, which provides oligomer formation, but does not lead to premature cross-linking of the binder in the process of its preparation. The ratio (by weight) was established as follows: 1 g of hardener:(9.5÷18) g of bisphthalonitrile monomer;

– selection of methyl ethyl ketone and methylene chloride mixture as a solvent (we investigated solubility in methylene chloride, acetone, methyl ethyl ketone, dimethylformamide, etc.);

– use of Lewis acid-based complex catalyst;

– pretreatment of carbon fabric with an amine-containing silane appetite.

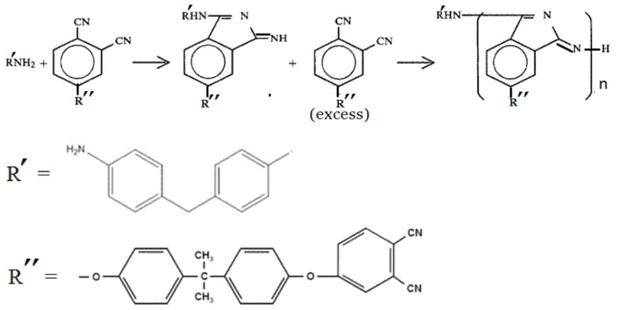
The use of a polyfunctional epoxy resin, a certain amount of hardener and catalyst, and targeted functionalization of the fabric surface with amino groups, are designed to increase the crosslinking density of the cured product (polymer matrix), adhesion between components, higher glass transition temperatures and protection against thermo-oxidative degradation. Bisphthalonitrile and epoxy react with each other during heat curing and both are cured with amino groups, which also increases the crosslinking density of the binder and its adhesion to the fiber.

The technology for obtaining BFB includes the following two stages:

1. Preparation of prepolymer

BFB is a melt or solution of the product of incomplete polymerization (prepolymer, resin) of bisphthalonitrile monomer with 4,4'-diaminodiphenylmethane (hardener).

The prepolymer is synthesized by adding the hardener 4,4' diaminodiphenylmethane (melting point of 156 to 158^{0}C) to a melt of bisphthalonitrile monomer (melting point 194 to 200^{0}C) at a specified ratio (by weight): 4,4' diaminodiphenylmethane:bisphthalonitrile monomer=1:(9.5÷18):



The mixture is kept at 220^{0}C with vigorous stirring for 3 hours. Then the temperature is increased to 240–270^{0}C and incubated for 2 to 4 hours, additionally incubated for 4 to 6 hours at 270 to 300^{0}C and 4 to 10 hours at 300 to 315^{0}C.

The prepolymer obtained by cooling the melt to room temperature can be stored at room temperature indefinitely.

2. Preparation of the binder

To prepare the binder, the prepolymer is suspended in an organic solvent (mixture of methyl ethyl ketone and methylene chloride) to obtain a solution of the required concentration of 10 to 60 wt.%.

The conditional viscosity of the binder based on methyl ethyl ketone according to viscometer VZ 246 (nozzle diameter of 4 mm) at temperature 25±2^{0}C is as follows:

– 12 seconds at the prepolymer concentration of 10 wt.% ;

– 17 seconds at the prepolymer concentration of 60 wt.%.

The binder viability in a closed hermetic container is at least 12 hours.

Thus, BFB is one-component and is a product of incomplete polymerization (prepolymer) of bisphthalonitrile monomer with a hardener, 4,4'-diaminodiphenylmethane. To obtain the required viscosity of the binder in the manufacture of polymer composites, it is suspended in a mixture of methyl ethyl ketone and methylene chloride and cured in the temperature range of 220–300^{0}C.

Experimental determination of binder heat resistance

The cured BF binder was investigated by thermogravimetric analysis on a derivatograph at a temperature rise rate of 10^0/min in air in the temperature range from 20 to 1000^{0}C (Fig. 2). Five samples weighing 100 mg each were investigated.

The thermal characteristics of the cured binder samples are given in Table 2. BFB samples were obtained by curing bisphthalonitrile monomer with hardener 4,4'-diaminodiphenylmethane with a set ratio (by weight) of 10:1 by heating under the above regime in the temperature range of 220–315^{0}C.

Based on the analysis of thermal characteristics of BFB binder, it was found that the weight loss of BFB at temperatures up to 367^{0}C is 5%. At temperature

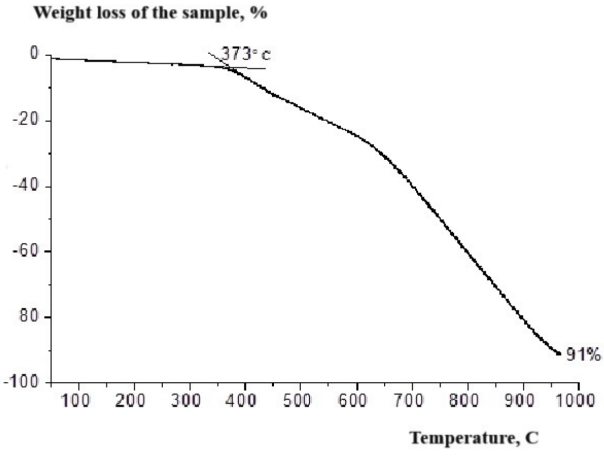


Fig. 2. Typical thermogravimetric curve of the developed BFB

Table 2

Thermal characteristics of cured binder samples

Weight loss temperature, ^{0}C			Coke residue at 965^{0}C, %
5%	10%	50%	
367	431	715	12.4

increases up to 430^{0}C, it reaches 10%. This confirms the high thermal stability of the developed binder and ensures its performance up to temperatures of about 350^{0}C.

Manufacturing carbon plastics based on bisphthalonitrile binder

BFB-based carbon fiber-reinforced plastics products are manufactured by the lay-up method. For the technological plate from carbon fiber-reinforced plastic with BFB as a filler, we used carbon fabric of 3606 mark (made by Porcher company).

Preparation of technological plates from carbon fiber-reinforced plastic was carried out according to the following scheme:

1. Preparation of the binder by suspending the BPS prepolymer in an organic solvent (mixture of methyl ethyl ketone and methylene chloride) to obtain a solution with a concentration of 60 wt%.
2. Impregnation of carbon fabric with 60% prepolymer solution in organic solvent (mixture of methyl ethyl ketone and methylene chloride).
3. Cutting the impregnated fabric into blanks.
4. Laying out the package of the required thickness, placing it in a vacuum cover.
5. Curing of carbon fiber-reinforced plastic according to the following regime:
   - heating the package to a temperature of 220^{0}C;
   - holding in vacuum at 220^{0}C for 4 hours;
   - creation of specific pressure of 3–4 kgf/cm^2;
   - switching off vacuum and heating up to 240^{0}C;
   - holding under pressure at 240^{0}C for 4 hours;
   - heating plates to 280^{0}C;
   - pressure holding at 280^0 C for 4 hours;
   - heating to a temperature of 300^{0}C;
   - pressure holding at 300^{0}C for 1 hour;
   - switching off electric heating and natural cooling under pressure to room temperature.

The quality of the finished plates was controlled by visual inspection of the surface and determination of their continuity. If non-destructive control does not reveal unglued areas and other defects, then the quality of the external surface of carbon fiber-reinforced plastic is good.

Experimental determination of the strength characteristics of carbon fiber-reinforced plastic on the obtained plates made of five layers of 3606 and BFB fabric was carried out at normal and elevated temperatures. The samples were tested in the warp direction.

Determinations of physical and mechanical characteristics of the obtained specimens were carried out on a universal high and low temperature testing machine Instron 5582, which is equipped with a special high-temperature chamber [12]. The results of tests of carbon fiber-reinforced plastic based on BFB at 20 and 300^{0}C are presented in Table 3.

At temperature of 20^{0}C, the value of maximum bending loading of the material is 837 MPa; maximum loading at tension and at compression being 890 MPa and 494 MPa, respectively. The obtained strength values exceed the characteristics of epoxy-bonded carbon fiber-reinforced plastic in bending by 20% and in compression by 50%. In tensile strength of carbon fiber-reinforced plastics based on both types of binder, the values of breaking stress are close.

The maximum bending loading of BFB-based carbon fiber-reinforced plastic at 300^{0}C is 285 MPa, which exceeds this characteristic for carbon fiber-reinforced plastic based on epoxy binder more than 50 times. The maximum tensile loading at a temperature of 300^{0}C reaches 550 MPa, which is 20% higher than that of epoxy-bonded carbon fiber-reinforced plastic. The maximum compressive loading at 300^{0}C is approximately 142 MPa, which is 20 times higher than that of epoxy carbon fiber-reinforced plastic.

Thermophysical characteristics of carbon fiber-reinforced plastic based on fabric 3606 and BFS binder are given in Table 4. Thermal conductivity of carbon fiber at temperatures of 20 and 300^{0}C was determined using the IT-λ-400 thermal conductivity meter. The specific heat capacity of the material under study at temperatures of 20 and 300^{0}C was measured using the IT-S-400 device.

Measurements of thermophysical parameters of carbon fiber-reinforced plastic based on BFB revealed that at 20^{0}C its heat capacity is 0.91 kJ/(kg·K) and thermal conductivity is 0.23 W/(m·K), while at 300^{0}C these characteristics are equal to 1.90 kJ/(kg·K) and 0.49 W/(m·K), respectively. Thus by its thermophysical characteristics, carbon fiber-reinforced plastic based on BFB is superior to carbon fiber-reinforced plastic with epoxy matrix.

Table 3

Results of bending tests of carbon fiber-reinforced plastics at normal and elevated temperatures (in the base direction)

Type of test	Type of binder	Maximum loading, MPa		Fall, %
		20^{0}C	300^{0}C	
bend	BFB	837	285	66
stretching		890	550	38
compression		494	142	71
bend	Epoxy	690	5	99
stretching		900	450	50
compression		230	5	98

Table 4

Results of determination of thermophysical characteristics of carbon fiber-reinforced plastics

Type of test	Type of binder	Value	
		20^0C	300^0C
Specific heat capacity according to Martens, kJ/(kg·K)	BFB	0.91	1.90
Thermal conductivity, W/(m·K)		0.23	0.49
Specific heat capacity according to Martens, kJ/(kg·K)	Epoxy	0.40	1.65
Thermal conductivity, W/(m·K)		0.39	0.65

Conclusions

We reported a substantial reduce of the weight of hypersonic unmanned aerial vehicles by creating structural carbon fiber-reinforced plastics with operating temperatures of more than 300^0C. The heat resistance of carbon composites is determined by the properties of the binder. Bisphthalonitrile binders represent a new promising class of high-temperature binders for polymer composites.

We developed a laboratory technology for obtaining bisphthalonitrile binder for structural carbon fiber-reinforced plastics. Based on the analysis of thermal characteristics of the binder BFB, its high temperature resistance and workability at temperatures up to 375^0C have been established.

The physical and mechanical characteristics of the material at temperatures of 20^0C and 300^0C have been determined experimentally. It is shown that the maximum loading of the obtained heat-resistant carbon fiber-reinforced plastic at various types of loading exceeds the characteristics of epoxy carbon composites by 20–50% and by 20–50 times at room temperatures and at the temperature of 300^0C, respectively.

By its thermophysical characteristics carbon fiber-reinforced plastic based on BFB exceeds carbon fiber-reinforced plastic with epoxy matrix.

The disadvantages of the heat-resistant binder include high temperatures of prepolymer synthesis and carbon fiber-reinforced plastic production (up to 315^0C), which causes the need for re-equipment of production with special equipment.

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ВУГЛЕПЛАСТИК ДЛЯ БАГАТОРАЗОВИХ ГІПЕРЗВУКОВИХ ЛІТАЛЬНИХ АПАРАТІВ

М. Казакевич, І. Гусарова, В. Казакевич, Т. Манько, В. Хорошилов, К. Козіс, Г. Осіновий, І. Суха, О. Зайчук

Розробка гіперзвукових безпілотних літальних апаратів (БПЛА) для аерокосмічних систем ставить перед науковцями та інженерами амбітні завдання. Екстремальні умови польоту, такі як надвисокі швидкості та значний аеродинамічний нагрів, вимагають розробки нових матеріалів, здатних витримувати такі навантаження. Одним з найбільш перспективних матеріалів для створення гіперзвукових БПЛА є армований вуглецевим волокном полімер на основі бісфенолнітрилу. Цей матеріал характеризується високою термостійкістю, хімічною стійкістю та високими механічними властивостями. Використання бісфенолнітрилу в поєднанні з вуглецевими волокнами дозволило отримати композитні матеріали, здатні працювати при температурі понад 300^0С. Це значно перевищує можливості традиційних полімерних матриць. Для оцінки придатності розробленого вуглепластику для використання в гіперзвукових БПЛА були здійснені комплексні дослідження його фізико-механічних і теплофізичних характеристик в широкому температурному діапазоні від 20 до 300^0С. Отримані результати дозволили детально охарактеризувати композит і порівняти його з іншими відомими високотемпературними композиційними матеріалами. Розроблений вуглепластик на основі бісфенолнітрильного зв'язуючого є перспективним матеріалом для створення гіперзвукових безпілотних літальних апаратів. Висока термостійкість у поєднанні з хорошими механічними властивостями дозволяє використовувати цей матеріал в екстремальних температурних умовах, характерних для гіперзвукового польоту.

Ключові слова*:* авіаційна та ракетно-космічна техніка, проєктні параметри, гіперзвуковий безпілотний літальний апарат, термостійкі зв'язуючі, вуглепластик, полімерні композиційні матеріали, фізико-механічні та теплофізичні характеристики.

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The development of hypersonic unmanned aerial vehicles (UAVs) for aerospace systems presents ambitious challenges for scientists and engineers. Extreme flight conditions, such as ultra-high speeds and significant aerodynamic heating, necessitate the creation of new materials capable of withstanding such loads. One of the most promising materials for constructing hypersonic UAVs is carbon fiber-reinforced polymer based on bisphenol nitrile. This material exhibits high thermal resistance, chemical stability, and excellent mechanical properties. Utilizing bisphenol nitrile combined with carbon fibers has enabled the production of composite materials that can operate at temperatures exceeding 300^0C, far surpassing the capabilities of traditional polymer matrices. To assess the suitability of the developed carbon fiber-reinforced plastic for hypersonic UAV applications, comprehensive studies of its physical, mechanical, and thermal characteristics were conducted across a wide temperature range from 20 to 300^0C. The obtained results provided a detailed characterization of the composite and allowed for comparisons with other high-temperature composite materials. The developed carbon fiber-reinforced plastic based on bisphenol nitrile binder shows great promise for constructing hypersonic UAVs. Its high thermal resistance, combined with excellent mechanical properties, makes it suitable for use in the extreme temperature conditions typical of hypersonic flight.

Keywords: aerospace and rocket technology; design parameters; hypersonic unmanned aerial vehicle; heat-resistant binders; carbon fiber-reinforced plastic; polymer composite materials; physical, mechanical, and thermal characteristics.

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