

## PROPERTIES OF ABLATIVE COMPOSITES BASED ON BISMALEIMIDE RESIN REINFORCED WITH GRAPHITE FELT

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*This paper presents a study regarding the obtaining, characterization and testing of a new ablative type composite material, composed of simple and nanofilled bismaleimide resin with various mass concentrations of nanometric silicon carbide particles (nSiC), reinforced with graphite felt. After the obtaining process parameters were established, the materials were tested in terms of mechanical, thermo-mechanical (HDT), tribological and thermal properties (thermal shock conditions) and they were characterized from morphostructural point of view (optical and scanning electron microscopy). The experimental results indicated an improvement of the performance of the nanofilled material samples compared to the control sample in the tested conditions.*

**Keywords:** Ablative composites, bismaleimide resin, mechanical testing, friction coefficient, thermal shock properties

### 1. Introduction

The harsh conditions that are encountered in the aerospace field lead to the need to develop new complex composite materials to cover the requirements and to meet the high needs in this field. In the most scientifically and technologically developed countries, the research development stages have been overcome, composite materials with special thermal resistances (such as ablative composites, carbon-carbon composites, thermal shields) being current solutions in top technical fields. The development of research originated in the use of these

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materials in aerospace vehicles such as heat shields, propulsion nozzles and guides. Remarkable mechanical and tribological properties have led to their use in aircraft braking systems (Concord, Boeing, Phantom, Suhoi), resistors in the steel industry and even insulators in the high temperature industry (1000  $\div$  3000  $^{\circ}\text{C}$ ) [1].

Thermal protection systems (so-called thermal shields) consist of several layers of material applied to the surface of vehicles to maintain the temperature within certain limits during the period of re-entry into the atmosphere. Thermal shields are generally made of advanced composites with phenolic or mesophasic matrix and preforms of materials with ablative properties with low thermal conductivity (such as cork, carbonic felt, etc.). The assembly formed by progressive pyrolysis has the role of preventing/ preventing the transfer of extreme temperature during thermal shock from the moment of re-entry, generated by ultra-fast compression of air, to the metal layer with high thermal conductivity of the vehicle. The concept behind consumables (so-called ablatives) is that they burn slowly, in a controlled manner, so that heat is directed near the wall of spacecraft by the gases generated during the ablation process and the material in the state. The remaining solid has the role of insulating the vehicle from overheated gases [2-4]. Ash-forming ablative consumables are often used in combination with sublimating or melting reinforcing materials (eg silica, nylon). Carbon fiber-reinforced phenolic composites were often used as heat shields in high-load vehicles [5], and were also used in tests for the Galileo probe [6]. Reinforcements such as carbon fibers offer superior mechanical strength, both to withstand environments with high shear phenomena and to function as structural thermal protection materials.

There are many technologies that can be applied to thermal shields, but the final selection depends on many main criteria, such as the speed of the vehicle at re-entry, its geometry and size, the atmosphere of the planet, etc.

The matrix or polymeric binder that underlies the development of ablative composite materials is made of polymers resistant to high temperatures. Among the polymers used as matrices in composites with special thermal resistance are; phenolic, polyamide, polyimide resins and certain variants of epoxy resins [8].

Poly (bismaleimide) resins (BMI) are thermoreactive plastics obtained by polyaddition of aromatic diamines to bismaleimides. The manufacture of these resins involves 3 stages: the synthesis of bismaleimide, the manufacture of the prepolymer and its hardening. Bismaleimide resins are a class of thermosetting resins that have gained ground in the industry due to the fact that they incorporate a number of stable physical properties at high temperatures and in humid environments, as well as stable electrical constants over a wide range of temperatures and fire resistant. Some BMI resins achieve higher working performances in the 230-290  $^{\circ}\text{C}$  range, respectively [9]. BMI resins are mainly

used in aerospace applications (engine and rocket components) that require higher temperature capabilities than epoxy resins (approximately 230 °C), with BMI resins having excellent thermal stability [10].

Bismaleimide resins are quite fragile materials and bismaleimide composites can experience micro-cracks during thermal aging, and due to volatile compounds, the process of processing impregnation of fibers with polyimides is a difficult one. Therefore, there is a need to improve their hardness to provide a wider range of applications at high temperatures [11]. One of the most used inorganic ceramic fillers is micrometric (SiC) or nanometric (nSiC) silicon carbide used to improve the thermo-oxidative properties [12] and wear resistance [13,14] of ablative materials.

Silicon carbide is a ceramic with high hardness, high wear resistance, thermal stability and chemical inertia [15,16], properties that recommend the use of SiC in micrometric or nanometric form to obtain polymeric composites and not only with improved properties. SiC and/or nSiC has been used in numerous studies to improve the properties of thermoreactive matrices (epoxy or phenolic) [16,18], but also thermoplastics such as polyolefins [19] or polystyrene [20,21]. One of the most known applications of silicon carbide is for the production of C/SiC brake pads, developed by Porsche [22].

G. Pulci et al., have developed an ablative material for re-entry space vehicles based on an insulating material (graphite felt preform) made of graphite fibers and a carbon binder. It is nominally suitable for temperatures up to 3000 °C and can be made in the form of a plate or cylinder. Its properties are affected by the precursor, the type and amount of binder, the compression ratio and the heat treatment [23]. The graphite felt from the ASTERM™ component, presented notable performances from a thermomechanical point of view in hybrid thermal protection structures (ablatives/ ceramic composites), structures aiming at spatial applications [24].

Although nSiC is a largely used nanofiller, literature survey identified the existence of few studies involving nSiC addition into bismaleimide resin [25, 26], and no studies using graphite felt as reinforcement material for ablative type targeted applications. Most known studies use phenolic resin as matrix of ablative/carbon-carbon type protection materials for high temperature application [27-29].

Based on the results of national research activities on advanced materials, the technical objectives of the paper focus on the development of multifunctional composite structures with resistance to extreme temperatures with superior mechanical, tribological, thermal and mechano-thermal properties [7]. The composite structures will be based on a matrix of thermoreactive resin (bismaleimide) with high thermal resistance, modified by the addition of various mass concentrations of silicon carbide of nanometric dimensions and will be

reinforced with graphite preform. The novelty of the study consists in the development of ternary composites with high performance as ablative type materials replacing the widely used phenolic resin matrix with bismaleimide matrix in graphite felt reinforced materials, and modifying it with silicon carbide nanoparticles for thermal and tribological performance improvement.

## 2. Experiments

### *Materials*

The polymer matrix used was Homide 250L resin, purchased from HOS-Technik. This is a high performance duroplastic resin, which is intended for use in applications involving high temperatures, such as those in the aerospace, military and military industries, due to advantages such as good dimensional stability, excellent fire resistance, chemical resistance and excellent properties thermo-oxidative and mechanical which are maintained even after 250 °C [30].

Table 1

Properties of BMI Homide 250L resin [25]	
Homide BMI 250L	Specification
Appearance	Brown liquid
Curing Temperature	180-200 °C
Post-curing Temperature	200-220 °C
Glass temperature	300 °C
Softening range	90-125 °C

The filler used to improve the properties of the BMI matrix was  $\beta$  silicon nanocarbide (nSiC), purchased from Nanostructured& Amorphous Materials Inc., USA, with 97.5% purity, specific surface area 34-40 m<sup>2</sup>/g and density of 3.22 g/cm<sup>3</sup> [31].

The addition of nSiC to the liquid BMI resin was achieved by dispersing three mass percentages (0, 1 and 2%) mechanically homogenizing for 3- 5 minutes and then ultrasonication with the help of the Bandelin Sonopuls Probe for 15 minutes (three steps of 5 min each).

The reinforcement material used was the graphite felt preform, heat treated at approximately 2200 °C and with a low impurity content <300 ppm, purchased from SGL Group - The Carbon Company which provide preforms of various thicknesses and weights per unit surface [32].

### *The obtaining of composites with high thermal protection*

The crosslinking of the composites materials with high thermal protection was performed following the temperature curve shown in figure 1. When reaching 120 °C, the force of 5 kg/cm<sup>2</sup> was applied for an optimum compaction. Cooling took place under pressure to room temperature.

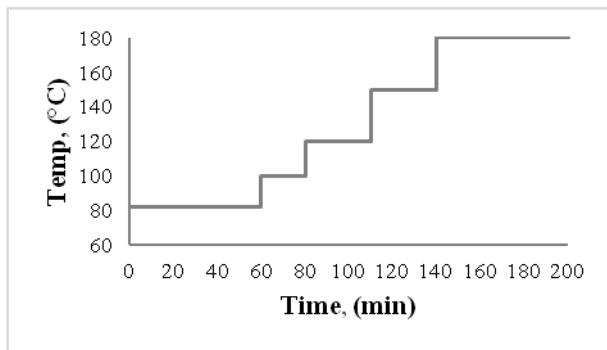


Fig. 1. Time-temperature curve for obtaining materials with high thermal protection with BMI resin matrix

After crosslinking, three types of materials were obtained (fig. 2.) (Felt/BMI; Felt/BMI+1% nSiC and Felt/BMI+2% nSiC), of which specimens with specific dimensions, mentioned in the international standards, were cut for each characterization test.



Fig. 2. Composites with high thermal protection (Felt/BMI/nSiC)

### **Materials testing and characterization methods**

The obtained materials were subjected to different tests and analyses in order to evaluate their performance as potential thermal protection materials.

*Density determination* was performed experimentally, according to the indications of the ASTM D 792 standard [33]. The method involves weighing the sample in air and then weighing the sample immersed in water, both measurements being performed with a prediction of 0.1 mg, the results leading to the determination of the specific gravity.

*Morphostructural analysis* consisted of optical microscopy and scanning electron microscopy analysis (SEM) on high thermal protection composites developed. Optical microscopy images were captured using the MEIJI 8520 microscope equipped with video camera at 100x magnification. SEM micrographs were captured using the SEM Quanta 250 electron microscope.

*Mechanical testing* consisted of 3-point bending and compression, performed at room temperature using the INSTRON 5982 mechanical test system, equipped with 10 and 100 kN force cells. The 3-point bending test was performed according to the international standard SR EN ISO 178 ("Plastics. Determination of bending properties") at speeds of 2 mm/mm, using rectangular specimens and the nominal distance between supports (16 x specimen thickness). The conventional value of the deformation was set (as 1.5 of the thickness of the specimen), but the tested materials have a fragile/ brittle nature, which determined the occurrence of rupture before reaching the established deformation [34]. Compression test was performed in accordance with the conditions recommended by the international standard ASTM 1074 [35], using a test speed of 1.3 mm/min. *Tribological testing* focused on friction coefficient measurements, that were determined using the CETR UMT 3 (Universal Macro Materials Tester) equipped with the roller-shoe type test module. Tribological tests were performed using a 10N pressure load at 1000 rpm test speed of for 900 sec. The tests were performed under dry friction conditions.

*Thermal shock testing* was performed in air, in a Nabertherm calcination/ carbonization furnace, where the samples were introduced from room temperature directly at 1100 °C and they were subjected to this temperature for three different time intervals (30, 60 and 120 seconds). Mass loss evaluation was performed comparing the specimen mass before and after testing.

*Thermo-mechanical testing* consisted in the determination of thermal stability under mechanical load or the determination of the deflection temperature (HDT). Qualitest HDT1 Tester equipment was used for HDT measurement, equipped with a silicone oil bath, thermoregulator, the heating speed being 2 °C/min, and the pressing load was 1.8 MPa. Three specimens from each sample were tested according to the international standard ISO 75 [36].

### 3. Results and discussions

#### *Density determination*

Determining the density of the obtained materials is a necessary step to obtain some basic characteristics, the density being an important parameter in the case of composites intended for applications in the space industry. The sample density was calculated using the relation 1 [33]:

$$D \left( \frac{\text{kg}}{\text{m}^3} \right) = \text{Specific gravity} \cdot 997.5 \quad (1)$$

Also, the density was calculated mathematically, using the classical formula (relation (2)), in order to be able to make a comparison between the theoretical and the experimental density.

$$D \left( \frac{kg}{m^3} \right) = \frac{m(kg)}{V(m^3)} \quad (2)$$

Table 2

The density of Felt / BMI / nSiC materials determined via experimental method via mathematical method

Specimen	d measured [g/cm <sup>3</sup> ]	d calculated [g/cm <sup>3</sup> ]
Felt/BMI	0.60	0.56
Felt/BMI+1%nSiC	0.77	0.70
Felt/BMI+2%nSiC	0.85	0.76

From table 2 it can be seen that the materials obtained have a very low density (less than 1 g / cm<sup>3</sup>), which includes them in the category of light-weight materials, representing a major advantage for the applications concerned. As expected, it is observed that the addition of nSiC causes an almost insignificant increase in density, due to the increased density of nSiC, which, however, is added in extremely small mass percentages.

#### *Morphostructural analysis*

Optical microscopy investigation was performed prior to scanning electron microscopy, as although it uses lower magnification levels, it allows the analysis of the entire sample (without the need to cut smaller specimens from the material).

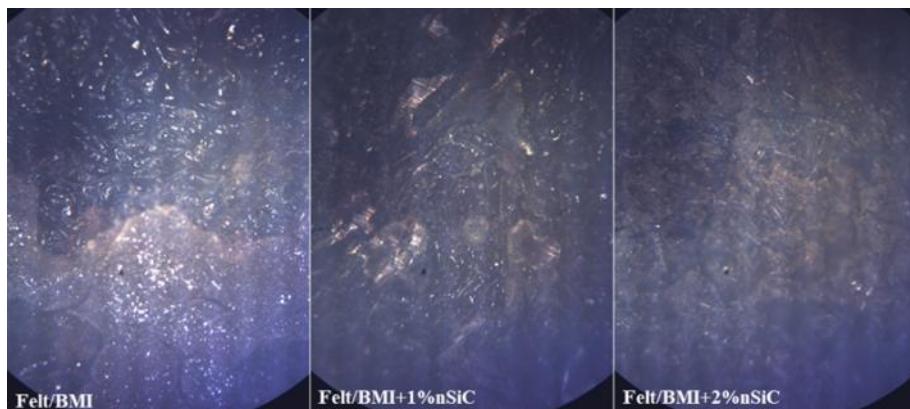


Fig. 3. Optical photomicrographs of Felt/BMI/nSiC composites

Optical photomicrographs of graphite felt samples (Fig. 3) illustrate the uniform appearance both in the case of the control sample and in the case of samples with the addition of nSiC. In order to verify the absence of smaller agglomerations, the samples were also investigated with the aid of scanning electron microscopy (SEM) which allows to achieve much more advanced magnifications.

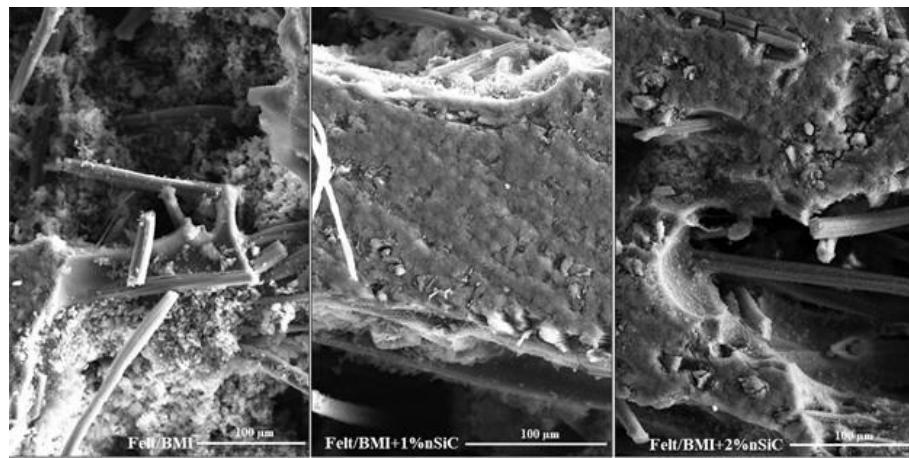


Fig. 4. SEM photomicrographs of Felt/BMI/nSiC composites

SEM photomicrographs (fig. 4.) of felt-based composites provide a much clear picture of the structure of these materials compared to that offered by optical microscopy. Thus, the graphite fibers that make up the graphite felt can be observed, these being generally embedded in the BMI type matrix. This appearance suggests that the interconnection between the matrix and the reinforcing felt is a strong one, providing a mechanically efficient interface in the composite.

#### ***Mechanical 3-point bending test***

The average results of the Young modulus of elasticity, bending strength and elongation were added in Table 3.

Table 3

#### **Mechanical properties at 3-point bending test of Felt/BMI/nSiC composites**

Sample	Modulus [MPa]	Strength [MPa]	Elongation [%]
Felt/BMI	840	12.65	2.03
Felt/BMI + 1% nSiC	1050	15.19	1.78
Felt/BMI + 2% nSiC	900	13.28	1.6

The results presented indicate higher values for samples with nanometric addition of nSiC compared to non-additivated samples. Samples with 1% nSiC, show values of bending strength 20% higher, and the values of the Young modulus of elasticity increase by 25%, compared to the values of the control sample. In the case of elongation, the values decrease with increasing nSiC concentration.

#### ***Mechanical compression testing***

The mechanical compression test aimed at measuring the modulus of elasticity, the resistance and elongation at compression, the mediated values being presented in table 4.

Table 4

**Mechanical properties at 3-point bending test of Felt/BMI/nSiC composites**

Sample	Modulus [MPa]	Strength [MPa]	Elongation [%]
Felt/BMI	280	65.46	59.55
Felt/BMI + 1% nSiC	300	66.58	62.69
Felt/BMI + 2% nSiC	260	62.54	66.60

This time, the mediated values of the mechanical properties for the studied composites present close values between the nanomodified samples and the control sample.

For samples with 1% nSiC, the values of bending strength increase by only 2%, and the values of the modulus of elasticity increase by only 7%.

**Tribological testing**

A set of 3 specimens was used for each sample, the final friction coefficient (fig. 5.) being calculated as the average of the 3 values obtained.

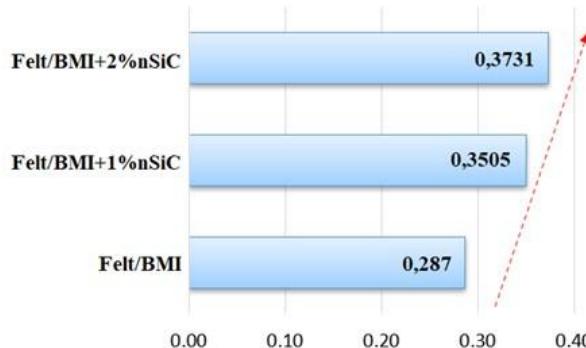


Fig. 5. Friction coefficient of Felt/BMI/nSiC composites

The abrasive nature of nSiC significantly increases the values of the coefficient of friction, as it can be seen in fig. 5. The percentage of 1% nSiC generated an increase of 22% and in the case of the concentration of 2% nSiC the increase was 30% of the coefficient of friction compared to the control sample.

**Thermal shock testing**

The thermal shock test aimed at the mass loss evaluation after each period of maintenance at the critical temperature and the evolution of the integrity of the material after testing [7].

Figure 6 illustrates the appearance of the samples before testing at 1100 °C and after each maintenance period at this temperature, highlighting the consumption (mechanical ablation) of each type of material. After macroscopically examining the samples, it can be observed that all samples maintained their overall integrity, but the samples that have nanometric nSiC in their structure show compact shapes, while the control sample is visibly carbonized.

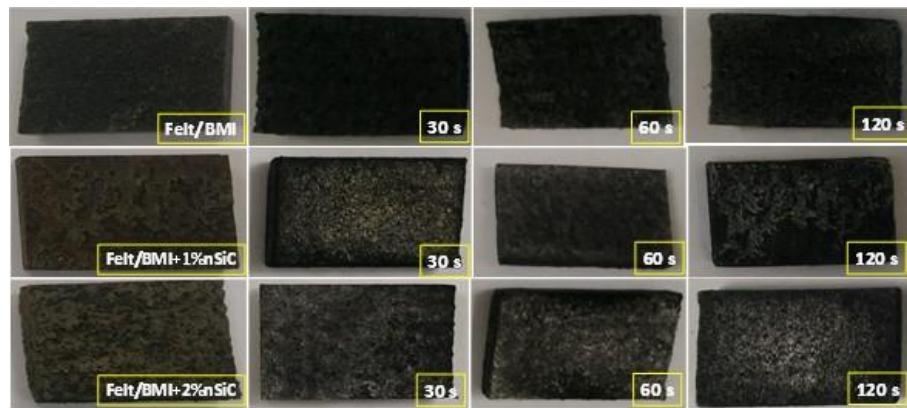


Fig. 6. Felt/BMI/nSiC composites before and after heat shock treatment at 1100 °C for 30, 60 and 120 sec, respectively

Figure 7 illustrates the mass loss percentage of the composite material samples tested at thermal shock (Felt/BMI (control); Felt/BMI+1% nSiC and Felt/BMI+2% nSiC) after the 3 maintenance periods at 1100 °C.

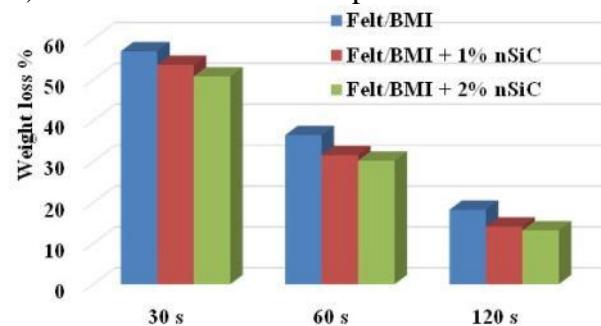


Fig. 7. Loss of mass recorded by Felt/BMI/nSiC composites after thermal shock testing

It is observed that the increase of the heat treatment time leads to the accentuated decrease of the sample mass in the first-time interval, the one of 30s, due to the decomposition of the organic compounds in the composition. Analysis of samples with the addition of nSiC shows that the mass loss decreases with increasing percentage of nSiC in the sample, due to the excellent thermal resistance of this compound.

#### **Thermo-mechanical testing**

Thermo-mechanical testing measured the heat deflection temperature of the obtained materials. The average values of the HDT reached by the tested specimens are illustrated in figure 8.

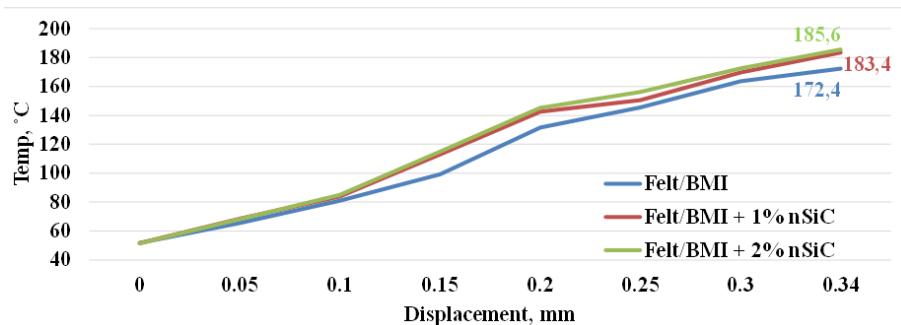


Fig. 8. Deflection-temperature curves, recorded during the test for HDT determination of Felt / BMI / nSiC materials

As expected, the values of the deflection temperature results increase with increasing mass concentration of nSiC. Thus, it can be seen in the graph in fig. 8 a slight change of the HDT curves, the percentages of 1% nSiC generated an increase of 6.4% and in the case of the concentration of 2% nSiC the increase was of 7.7% of the value of the deflection temperature compared to the control sample.

#### 4. Conclusions

In this work there were obtained composite materials based on polymeric matrix bismaleimide resin simple and with various mass concentrations of nanometric silicon carbide (1 and 2% respectively) and graphite felt preform reinforcement.

The density of the developed materials was calculated and they were characterized from mechanical, thermal, tribological and mechano-thermal point of view. They were also investigated from a morphostructural point of view, where a good compaction and homogeneous dispersion of the nanoparticles was observed on the entire analyzed area.

Overall, nSiC improves the values of mechanical properties both in 3-point bending and compression testing. Samples with mass addition of 1% nSiC showed the most representative values compared to the control samples.

From a tribological point of view, the abrasive nature of silicon carbide nanopowder substantially increases the values of the coefficient of friction with increasing nSiC concentration.

Following the thermal shock tests, all materials showed an adequate behavior, however the samples with nSiC in their composition showed macroscopically compact shapes while the control sample is visibly carbonized. Analysis of samples with nSiC addition shows that the mass loss decreases with increasing percentage of nSiC in the sample, due to the excellent thermal resistance of this compound.

As expected, also in the case of thermal stability tests under mechanical load, small increases in results are observed with increasing nSiC concentration.

As a general conclusion, the presence of nSiC has the role of protecting the organic matrix and also the preforms that underlie the ablative composite materials, increasing their mechanical and tribological resistance but especially their resistance to thermal degradation under various conditions. The experimental results indicate that the developed materials have potential for thermal protection materials applications, the BMI matrix based composites showing a similar behavior to well-known phenolic matrix based composites [37].

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