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Novel phenolic impregnated 3-D Fine-woven pierced carbon fabric composites: Microstructure and ablation behavior

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1. Introduction

PICA known as a novel thermal protection system (TPS) material has been developed for heat-shield application in the field of aerospace and planet missions. Conventional TPS materials (such as carbon phenolics composites) had high densities and thermal conductivities, yielding a TPS mass fraction that exceeded mission constraints [1–3]. PICA has the advantages of low density (about 0.2–0.4 g/cm³) coupled with efficient ablative capability at high heat fluxes that made PICA an enabling technology for efficient heat shielding material [4,5].

PICA is highly porous fibrous carbon substrates that is partially impregnated with phenolic resins, and used for heat fluxes greater than 400 W/cm². It was chosen as the heat shield materials for Stardust Sample Return Capsule's forebody and is a candidate for future outer planet missions (to Jupiter, Saturn, Neptune, and their moons) that are anticipated toward the end of the current decade [6–8].

Experimental and theoretical results showed that PICA is also a typical thermal insulating material with anti-ablation performance [9–11]; it may be considered that lowering the thermal conductivity of a composite of interest is primarily more important than maintaining its mechanical strength. Lowering the thermal conductivity of PICA may be successfully achieved by controlling the pore content and density variation and it is also effectively managed through a variety of impregnated process [12–14]. However, because the microstructure controlling and ablation mechanism of such a

ABSTRACT

The processing, microstructure and ablative properties of novel phenolic impregnated 3-D Fine-woven pierced carbon fabric ablator (PICA) with different bulk density were investigated. The density of PICA material ranges from 0.352 to 0.701 g/cm³ that having uniform resin distribution within the fibrous substrate. An oxyacetylene torch was used to explore the ablative characteristics in terms of linear/mass ablation rate and microscopic pattern of ablation. Surface and in-depth temperatures during ablation were measured by using optical pyrometers and thermocouples. The experimental results showed that the linear ablation rate varied between 0.019 and 0.036 mm/s and the mass ablation rate increased from 0.045 to 0.061 g/s for the tested PICA composites. It suggests that the PICA composites with lower density may significantly contribute to improving the thermal insulation and ablative properties.

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lightweight ablator is not fully understood compared with carbon/ carbon (C/C) composites infiltrated with carbide, boride and organic resin [15–20], the evaluation of such heat shielding performance is difficult. Meanwhile compared with phenolic or polymer based composites reinforced with SWNTs, carbon fiber and glass fiber [21–28], the mechanical and thermal properties for PICA have fundamental differences although the chemical composition is similar to some extent. Therefore, we believe that a deep understanding of the ablation behavior for such a lightweight ablator is prerequisite in order to use it in a future space mission of interest. Published data in this area is limited as many patents or AIAA meetings have been filed due to this significance of immediate application. However, most of the patents reported complex composite processes leading to complex infrastructure requirements.

In this work efforts were made to develop simpler method to fabricate PICA materials and improve the ablation resistance of PICA composites. The aims of this paper are: (i) to study the effect of density of carbon fabric on the ablation resistance, mechanical properties and thermal conductivity of PICA; (ii) to investigate the microstructure of the ablated specimens to understand the mechanisms operating during ablation.

2. Experimental procedure

2.1. Preparation of bulk PICA composites

T700 PAN-based carbon fiber (Density of 1.70 g/cm³, Yixing Tianniao High Technology Co., Ltd., Jiangsu, China) was employed



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to fabricate the integrated felt. The felt was fabricated by alternatively stacked weftless piles and short-cut-fiber webs by a needle-punching technique. Two successive weftless plies were oriented at an angle of 45°. The fiber volume content of the felt was about 10%. The as-prepared bulk needled carbon fiber felts (0.185 g/cm³) were used as reinforcements for PICA composites. The thermosetting phenolic resin (Density of 1.25 g/cm³, Xi'an Taihang Polymer Co., Ltd., Shanxi, China) was used as the infiltrants. Viscosity of the resin was controlled in the range of 250–300 cp (measured by a Book-Field viscometer). Certain mechanical loads were applied during infiltration to assure that the carbon fiber was coated with phenolic resin homogenously. After infiltration, redundant resin was removed to control the final density by evaluated mechanical pressures.

The bulk needled carbon fiber felts were impregnated in a phenolic solution for 0.5 h and the impregnated felts were dried at room temperature for 24 h. Then the dried PICA performs were placed in an electric furnace. The performs were heat treated at 150 °C for 2 h with an identical curing cycle in an autoclave, followed by 4 h curing at 180 °C and 5 bar pressure. The mass of phenolic in the carbon felt was calculated by weighing the carbon felt before the procedure of impregnation and after heat treatment.

2.2. Ablation behavior

The ablation test was carried out in oxyacetylene torch with plate specimens (40 mm \times 40 mm \times 20 mm) (as shown in Fig. 1). The parameters of ablation test are shown in Table 1. The inner diameter of the oxyacetylene gun tip was 2 mm and the distance between the gun tip and the specimen was 10 mm. During the test, the flame temperature was estimated to be approximately 3000 °C. The specimen, fixed in a water-cooled copper concave, was exposed to the flame for 200 s. The flame width was about 5–6 mm, and it was less than the specimen length and width. So, there was indeed a thermal gradient between the center and the borders of the specimen, which rendered the ablation rate very non-uniform throughout the surface. Therefore, the linear ablation rate measurement was performed only at the central ablation regions and was different from the evaluation of mass ablation rate. The linear/mass ablation rate was calculated by thickness/mass change before and after ablation test of each specimen. The ultimate ablation rates of PICA composites were those of three specimens on average.

2.3. Characterization

Thermal conductivity for PICA is one of the most parameters to characterize the thermal performance. In order to investigate heat

Specimen under

oxyacetylene torch

Just tested specimen

Fig. 1. Composite specimen testing under oxyacetylene torch.

Table 1

Ablation parameters	of oxyacetylene	torch for PICA	materials.
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Flu	ux (L	/s)	Pressure (MPa)		Heat flux (MW/m ²)	Ablation angle (°)
02	2	C_2H_2	02	C_2H_2		
0.4	44	0.37	0.5	0.09	4.5	90

transfer through the porous matrix and obtain the effective thermal conductivity of insulation sample exactly and reliably, an apparatus was built to measure the steady-state effective thermal conductivity. The radiant heat source is a graphite radiant heater and the sample is placed between a septum plate and a watercooled plate maintained at room temperature. Thermocouples are installed at strategic locations to monitor the change of temperature distribution throughout the specimen during heating. Heat flux gauges located on the water-cooled plate are used to measure the flux of heat energy flowing through the sample. The environmental condition in this study is at atmosphere pressure. Using the measured heat flux, the hot side temperature, the cold side temperature and the sample thickness, the effective thermal conductivity can be calculated from Fourier's law of heat conduction: $K = qL/(T_1 - T_2)$, where q is the measured heat flux, L is the insulation thickness, and T_1 and T_2 are the measured temperatures on the hot side and the cold side, respectively.

The compressive tests were performed on a universal testing machine using cylindrical specimen ($\Phi 6 \text{ mm} \times 9 \text{ mm}$) to determine the mechanical properties. The specimen was placed between two steel platens. The tests were carried out with a constant speed of 0.05 mm/min at room temperature. In the present work, for each kind of test, three specimens (about $\Phi 6 \text{ mm}(\pm 0.1 \text{ mm}) \times 9 \text{ mm}(\pm 0.1 \text{ mm})$) were machined from each type of the as-received composites. The measured density of the specimen was the apparent density (overall mass-to-volume ratio). The phase composition and morphology of the composites were investigated by X-ray diffraction (XRD, X'Pert Pro MPD), scanning electron microscopy (SEM, JSM6460) combined with an energy dispersive spectroscopy (EDS).

3. Results and discussion

3.1. Microstructure and properties

Fig. 2a shows the photograph of a typical sample of PICA composites. No noticeable defects, such as cracking or delamination, were found on the sample surface. The samples for microstructure analysis are selected from the intermediate position of the fabricated product. It revealed that the phenolic resin was adhered to carbon fiber discontinuously and most of carbon fiber was coated by the resin (Fig. 2b–d). Due to the high porosity for PICA, the determined density for PICA-1, PICA-2 and PICA-3 ranges from 0.352 to 0.701 g/cm³ (shown in Table 2), which are lighter than traditional carbon–phenolic composites (often ranging from 1.2 to 1.7 g/cm³).

The content of phenolic resin after infiltration was variable and quite lot amounts of phenolic were extruded from the fiber belt due to the needed density controlling (i.e., 11.1 vol.% for PICA-1 and 35.5 vol.% for PICA-3). However, the content of carbon fiber also increased slightly from 12.5 to 14.2 vol.% after impregnation although the initial fiber volume content of the felt was about 10% (Table 2). It is because more phenolic resin existed in carbon fiber fabric probably led to some volume shrinkage of fiber belt during producing process. For actual application, PICA are often densified on the surface, while the inner interior of the samples remains porous structure. As is known, that, the insulative materials often contain high porosities, which is helpful to thermal



Fig. 2. Macrostructure and microstructure of fabricated PICA (a) Typical macroscopic sample; (b) PICA-1; (c) PICA-2 and (d) PICA-3.

Table 2Measured properties of fabricated PICA materials.

Sample description	Specimen density (g cm ⁻³)	Content of phenolic (vol.%)	Content of carbon fiber (vol.%)	Thermal conductivity (W/m K)
PICA-1	0.352	11.1	12.5	0.094
PICA-2	0.565	26.9	13.4	0.125
PICA-3	0.701	35.5	14.2	0.198

insulation and density decrease. Due to the partial infiltration for phenolic resin into carbon fiber felt, resulting controllable porosities for PICA inside the prepared samples.

3.2. Ablation behavior

Fig. 3 shows the measured surface temperature history and indepth temperatures history under oxyacetylene torch. In these investigation, surface temperature was measured by pyrophotometer with the maximum measurement value of 3000 K, and the indepth temperature was examined through several thermocouples embedded in the sample with different depth. The surface temperature increases rapidly and the nearly constant value of 1800 °C during the testing. The in-depth temperature at 5 mm increases slowly during the testing compared with that for the surface temperature and the in-depth temperature becomes at 1000 °C, although a small discontinuity is seen probably due to an experimental noise. From the in-depth temperature at 15 mm, the temperature is about 300 °C at most during 200 s. This demonstrates the fairly good insulative properties of PICA, and indicates that most of the heat is rejected by a reradiation mechanism rather than stored in heat conduction [29].

As is indicated in Table 2, the fabricated PICA in this work has a rather low thermal conductivity (i.e., ranging from 0.094 to 0.198 W/m K). Here, the loading of the phenolic resin in the virgin PICA also reduces the pore size of carbon fiber belt and thus reduces the internal radiation effect. Additionally, the combination



Fig. 3. Measured surface and in-depth temperatures history under oxyacetylene torch.

of the excellent high-temperature-capability of the carbon fabric substrate, due to its good network structure and insulative properties, with a virgin coated phenolic resin gives PICA the capability to provide protection against severe thermal environments.

The linear and mass ablation rate listed in Table 3. With increasing PICA density, the linear ablation rate and mass ablation rate increases linearly. The linear ablation rate ranged from 0.019 to 0.036 mm/s and mass ablation rates of PICA varied between 0.045 and 0.061 g/s. It was found that the more phenolic resin infiltration, the higher thermal conductivity is obtained in this study. Therefore, in this heating range, the higher heat energy and oxygen atoms could easily diffuse into the boundary layer, which increases the oxidation rate and surface recession. It is imaginable that the ablation performances of the composites are dominated by the phenolic contents.

After ablation, slices were cut from central regions on cross-section for the ablation microstructure analysis. Variation in PICA morphology after ablation, as a function of depth, was evaluated.

Table 3

Linear and mass ablation rate for PICA-1, PICA-2 and PICA-3 specimens.

Specimen	Ablation time (s)	Linear ablation rate (mm/s)	Mass ablation rate (g/s)
PICA-1 PICA-2	200 200	0.019 0.028	0.045 0.052
PICA-3	200	0.036	0.061

Fig. 4a presents micrographs near the ablation surface, the carbon fiber were severely eroded due to oxidation were also detected on the ablation surface. Fig. 4b shows the morphology at known locations (about 5 mm in-depth) in the near stagnation core, including a typical charred PICA microstructure, where the two constituents, having carbon fibers and a high surface area phenolic phase, are easily identified.

Fig. 4c and d reveals the virgin-like microstructure at locations about 10 mm and 15 mm in-depth from ablation surface, which the carbon fiber is comparatively stable and phenolic is partially corroded here due to its relative low temperature (i.e., about 400 °C for 15 mm in-depth). Fig. 4e is the virgin microstructure which is almost not impacted during ablation test, showing that temperature in these regions is relatively low and heat transfer was held back.

Fig. 5 designates the ablation morphology of the central and outer regions on the upper ablation surface in a tested PICA-1, PICA-2 and PICA-3 specimen. The flame is expected to be most intensive near the central regions and least intensive near the edge regions. Through microscopic observations on the eroded face after the ablation test, the ablation pattern has been closely examined. It has been found that the central ablation regions of all the reinforcing fibers in the three PICA samples have a severe carbonized-pattern formed by simultaneous combination of thermomechanical, thermochemical, and thermophysical effects (Fig. 5a, c and e). However, PICA-1 in the central regions revealed that the carbon fiber retained relative good morphology and eroded minimally compared with PICA-2 and PICA-3.

In the edge region for PICA specimens (Fig. 5b, d and f), a number of fibers exist with a charred matrix, and carbon fibers are in blunt shape and no severe carbonization appears compared with that in ablated center. The fact suggests that the erosion around the ablated center is stronger than that of the edge region. It can be found that the alignment of the eroded fibers is not regular in the PICA-2 and PICA-3 composite but relatively regular in the PICA-1 composite. The matrix part between the cross-plies is also broken away showing small cracks and progressive erosion with the fibers, as seen in Fig. 5d and f. This suggests that the content of phenolic resin in the PICA composites somewhat affect not only



Fig. 4. Ablated PICA morphology as a function of in-depth on cross-section from the top surface (a) 0 mm; (b) 5 mm; (c) 10 mm; (d) 15 mm; (e) 20 mm.



Fig. 5. Ablation morphology of the central and edge regions on the upper surface for PICA specimen (a) Central region and (b) Edge region for PICA-1; (c) Central region and (d) Edge region for PICA-2; (e) Central region and (f) Edge region for PICA-3.

on the thermal insulation property but also on the ablation behavior with reduction of abnormal erosion pattern such as spalling, carbonization and/or ply-lifting.

3.3. Ablation mechanism

3.3.1. Mechanism I: char formation

The formation of the char layer is known as a phenomenon that when the pyrolysis gas pass through the porous char layer to the surface, the hydrocarbon species in the pyrolysis gas is chemically decomposed to lighter gaseous species; a solid carbon is produced in this chemical decomposition process; the solid carbon so produced is deposited onto the char layer and sometimes surface layer. During ablation, elimination of groups along polymer chains predominates over chain cleavage, leading to char formation. There are large numbers of porous carbon in the char layer during ablation, as can be seen in Fig. 6, the porous solid carbon supplies a good thermal insulation layer to backward the heat energy transfer to inner layer.

As the formation of char from phenolic resin is an endothermic process, heat dissipation during the ablation depends on the efficiency of char formation. Moreover, char that has formed on the surface during ablation dissipates a large fraction of incident heat through surface radiant emission. Complete charring of the resin, holding the char for maximum duration, gives better heat dissipation during ablation.

3.3.2. Mechanism II: porous insulation

The surface temperature at this heating range is extremely high, such that the carbon fibers at the surface, especially at ablated center, begin to sublime, thus contributing to the surface recession. Usually PICA has a very low thermal conductivity and heat capacity, and since the materials have high porosity (i.e., exceeding 76% porosity for PICA-1 according to Table 2); the heat is being rejected at the surface rather than stored in heat conduction. This can reduce the thermal conductivity of solid and gas phases, which resulted in the in-depth temperature decreased from the ablation surface. Therefore, most of combustion heat energy was blocked and the inner layer was protected, resulting rather low ablation rate.

3.3.3. Mechanism III: surface reradiation

The carbon substrate here has a very high melting temperature, so that an increase in surface temperature resulting from the formation of a char layer is not sufficient to cause the melting of the substrate. In addition, the carbon substrate has high surface emissivity ($\varepsilon \approx 0.9$), so that most of the energy absorbed at the surface is being reradiated. Another factor that contributes to the



Fig. 6. Formation of porous solid carbon in the char layer during ablation (a) low magnification and (b) high magnification.

remarkable thermal performance of PICA may be the high charring characteristics of phenolic; Reradiation coupled with boundarylayer blockage is the most efficient mechanism in rejecting heat at the surface. Surface reradiation seems to be the main mechanism for heat dissipation.

4. Conclusions

Phenolic impregnated 3-D Fine-woven pierced carbon fabric ablator (PICA) with controllable density was fabricated. The network distribution of carbon fabric filled with continuous phenolic resin can make the composites more resistant to the high aerodynamic forces and is expected to result in enhanced ablation resistance. With increasing PICA density, the linear and mass ablation rate increased linearly. The ablation resistance of PICA composites was greatly improved with the increasing of the phenolic contents. The linear ablation rate ranged from 0.019 to 0.036 mm/s and mass ablation rates of PICA varied between 0.045 and 0.061 g/s. After ablation, large numbers of porous solid carbon in the char layer supplies a good thermal insulation layer. Carbon fiber of PICA with lower density in the ablation center showed good morphology and eroded minimally compared to PICA with higher density. The ablation mechanism includes char formation, porous insulation and surface reradiation.

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