INVESTIGATION ON ABLATION BEHAVIOR OF CFRC COMPOSITES PREPARED AT DIFFERENT PRESSURE

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Introduction

Low thermal conductivity and high thermal stability are desirable for the materials due to their characteristic roles in ablation resistance applications (*e.g*., reentry parts and renewable rocket nozzles) [1]. Ablation is defined as an erosive phenomenon with a removal of material by a combination of thermomechanical, thermochemical and thermophysical factors due to the high temperature, pressure and velocity of combustion flame [2]. A rocket nozzle, a typical ablation material is often experienced to severe environment. Under these extreme conditions it is obvious that the superior properties of CFRC composites can take advantages and it has been proved the effectiveness of the composites on ablation resistance [3].

Due to the inconvenience of CFRC composites preparation as mentioned, high pressure is often applied during impregnation to increase the density of the composites: an increase in densification efficiency [4]. It was reported that the microstructures of carbon matrix are predominately affected by pressure [5]. The role of the matrix in composite materials on ablation resistance has been discussed [6] but it has not been reported yet how these microstructural changes affect the ablation resistance of the composites. The objectives here are to assess the effects of microstructural changes of CFRC composites on ablation resistance. To increase the certainty of the composites performance, a liquid rocket engine was employed and the composites sample was placed in the nozzle part of the engine. The changes of the carbon fibers in the composites for different ablation period will also be discussed.

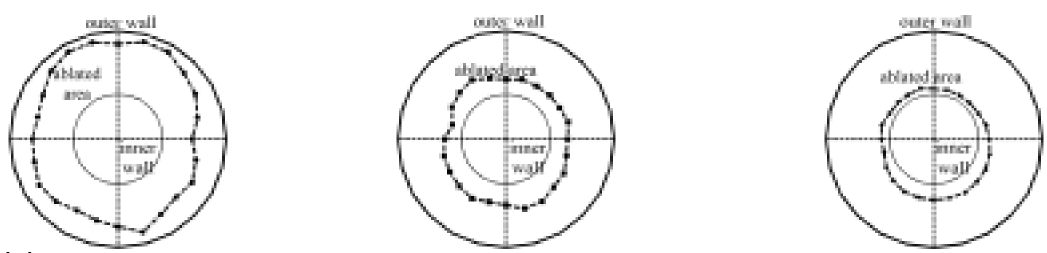
Experimental

CFRC composites were prepared using PAN-based carbon fibers (Taekwang Co., Korea) as the reinforcement and a coal-tar pitch (Jungwoo Co., Korea) as the matrix. Preparation of 4D CFRC composites requires many repeated heat treatment cycles, whose objective is to achieve maximum densification and thus minimize both open and closed porosity development. The preform was impregnated with coal-tar pitch before heat treatment. Then the preform was employed in a series of densification cycles; it was cut to 3 pieces, impregnated at 650 °C under different pressure (0.1, 60 and 90 MPa), carbonized at 1000 °C and graphitized at 2300 °C. The densification was repeated three times to remove the voids and consequently, to increase the density of the composites.

The ablation test was performed in a liquid rocket engine using kerosene and liquid oxygen as combustion fuels. The composite samples were machined and placed in a nozzle part of the rocket engine. The flame temperature was monitored by an optical pyrometer and estimated to be *ca*. 3000 °C. The ablation period was varied from 30 to 120 seconds to evaluate the structure changes of the composites when the sample was exposed to the flame for different period. The erosion rates were given by weight and length changes per unit time. Scanning electron microscopy (SEM, JSM-840A, JEOL Co.) was used to examine the morphological features of the sample before and after the tests.

Results and Discussion

Fig. 1 presents the ablated area of the composites prepared under different pressure. As explained the sample was a cylindrical shape with 10 cm of inner diameter, 20 cm of outer diameter and 20 cm of length. It is clearly shown here that the ablated area of the sample decreases as the pressure increases. In the sample prepared at 0.1 MPa, more than 60 % of the samples were removed and the pressure of fuels in the combustion chamber was also decreased, suggesting that the reaction was extensively being progressed. The ablated area of the composites at 90 MPa was quite small and evenly distributed around the peripheral of the inside circle of the sample. The pressure of the chamber was also kept almost constant, implying that the composites are appropriate for the use of rocket nozzle.



(a) (b) (c)

Fig. 1. Effect of pressure in the ablated area of the CFRC composites nozzle; (a) 0.1 MPa, (b) 60 MPa and (c) 90 MPa.

The average erosion rate of each sample was calculated in terms of changes in weight and length per unit time, and was presented in Table 1. As noticed in Fig. 1 the rates did decrease as the pressure increases. But the rates were quite higher than other studies [7] due to mainly ablation conditions; an arc-plasma torch and the heat flux of the torch were quite lower than that of the combustion engine used here. Another difference can be noticed from the fuel used. Unlike using argon and hydrogen [7], liquid oxygen and kerosene were used here as fuels; excess oxygen in the fuel resulted in combustion of the composites (excessive oxidation).

Table 1. Average erosion rate of the CFRC composites prepared at different pressure.

Pressure	Erosion rate (g/sec)	Erosion rate (mm/sec)
0.1 MPa	9.06 ± 0.52	0.63 ± 0.51
60 MPa	1.88 ± 0.31	0.21 ± 0.08
90 MPa	0.47 ± 0.04	0.05 ± 0.04

A comparison of the erosion rates between the samples is quite interesting. The difference of the rates between the samples at 0.1 and 60 MPa is quite acceptable but that of the rates between the composites at 60 and 90 MPa is larger than expected from density difference between the samples. Heat treatment at high pressure is known to increase the density of carbon [4] and the ablation resistance of CFRC composites is also known to depend on the density of the composites [8]; the higher the density, the higher the ablation resistance. Here the density of CFRC composites was measured by a densimeter based on ASTM D-4018: 1.50, 1.83 and 1.88 g/cm^3 for 0.1, 60 and 90 MPa, respectively. Although the difference in density was only 0.05 g/cm^3 between the samples at 60 and 90 MPa, the erosion rate at 60 MPa was almost 4 times higher than that of 90 MPa. This difference in erosion rate seems to be related with the change of microstructures, and it will be explored in great detail.

Figs. 2 and 3 present a comparison of the ablated carbon matrix area in the composites prepared at 60 and 90 MPa, respectively. The area inside the broken line in Figs. 6(a) and 7(a) was enlarged and presented in (b) at higher magnification. The ablation period was 60 seconds. The role of carbon matrix in the composites during ablation is thought to be the absorption of heat from flame [6].

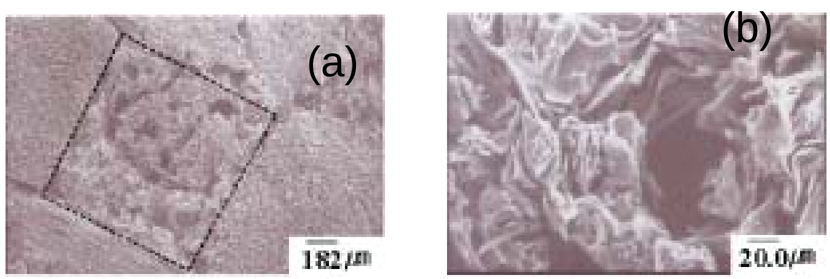


Fig. 6. SEM of the ablated carbon matrix in the CFRC composites prepared at 60 MPa.

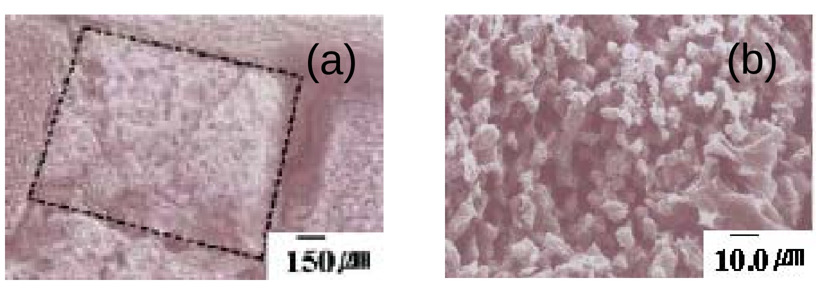


Fig. 7. SEM of the ablated carbon matrix in the CFRC composites prepared at 90 MPa.

The matrix in Fig. 2 does still maintain lamellar structures even after ablation but the orientation of the layers seems to be quite disrupted and corrugated; meanwhile, the coarse grain structures from high pressure (90 MPa) in Fig. 3 do not have any noticeable changes except the grains become smaller and more spherical shape. An examination at higher magnification reveals different ablation mechanisms of the two composites. In the composites at 60 MPa, a part of the layer is disconnected and removed by thermochemical, thermophysical and thermomechanical reactions with flame during ablation; in contrast, relatively small parts of grains only are removed from the matrix by ablation reaction in the sample prepared at 90 MPa. It seems to be that each coarse grain at 90 MPa does dissipate the heat generated from flame, resulting in only small portion of volume is removed: low erosion rates.

Conclusions

The microstructures of the matrix in CFRC composites prepared at different pressure were changed; anisotropic lamellar structures were developed at 60 MPa but coarse grain structures appeared at 90 MPa. These different microstructures did affect the ablation resistance of the composites in different ways.

It is truth that the ablation resistance increases as the density of the composites increases. But the difference of the erosion rates between the composites prepared at 60 and 90 MPa was quite noticeable despite that of the density between the composites was relatively small. This discrepancy here seems to be from the change of microstructures; each coarse grain in the composites at high pressure does dissipate the heat generated from flame, resulting in only small portion of volume is removed: low erosion rates. Some parts of lamellar layers in the composites at 60 MPa were removed by ablation; in contrast, coarse grains at 90 MPa did only become smaller and more spherical after ablation reaction.

References

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