CARBON/CARBON COMPOSITES BY PULSE CHEMICAL VAPOR INFILTRATION

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INTRODUCTION

Chemical vapor infiltration(CVI) has received considerable attention over the last 15 years as a process for the fabrication of fiber reinforced composites.[1-4] In this process, a source gas is flowed through a fiber preform and the specific material is deposited as a matrix between the fiber. The largest advantages of CVI are near net shaping and excellent adhesion of matrix to fiber. However, the process requires a lengthy operation time due to blockage of the flow paths and generation of byproduct gas from the deposition site.

A unique process of "pulse CVD" was proposed by Bryant.[5] The process is characterized by repeat cycles of evacuation of the vessel, instantaneous filling with source gas, holding to allow deposition and re-evacuation. Sugiyama et al.[6-8] applied this pulse CVD process to CVI expecting prominent reduction of operation time.

In the present paper, the pulse CVI was studied for the preparation of carbon/carbon composites from a gas of propane.

EXPERIMENTAL

The preform samples were prepared by liquid impregnation of phenolic resin to carbon cloth and followed by carbonization at 1000°C for 2hr in N_2 atmosphere. The only once impregnated and carbonized carbon/carbon preforms were used as the sample of this pulse CVI. The phenol based carbon/carbon samples were infiltrated with carbon from a gas system of propane and nitrogen. The sequential steps of gas filling and evacuation were controlled by programmed controller via electromagnetic valve.

In order to determine the important parameters in this pulse CVI process, robust experimental design method was adapted.

The density and porosity were measured by water immersion method(ASTM-C20-87). Hitachi S2150 and Autopore II 9220 were used for SEM observation and measurement of pore size distribution, respectively.

RESULTS AND DISCUSSION

The phenol based carbon/carbon samples untreated by pulse CVI have a apparent density of 1.34 g/cm^3 and a porosity of 21.0%. After conducting matrix experiment of pulse CVI, the density and porosity of the carbon/carbon composites are shown in Fig.1. In experiment #2, the density increased to 1.51 g/cm^3 and the porosity decreased to 10.1%, which shows the best results. The density increment is larger for experiments conducted at 900°C than those of at 1100°C. The reason may be attributed to the slow deposition rate or some desorption at higher temperature.

In order to measure the infiltration uniformity more detailed, the density of seven slices of each sample was measured and showed very uniform infiltration. As a result of analysis of variance of density in carbon/carbon composites obtained from pulse CVI matrix experiments, the most important processing parameters in pulse CVI to density of the carbon/carbon composites are temperature, introduction time of gas, and number of pulses. The interaction of introduction and evacuation time and concentration of gas are also considered as somewhat important processing parameters for pulse CVI process.

Fig.2. shows a deposition rate dependence on sample temperature exemplified by the infiltration of carbon from propane gas. Two deposition

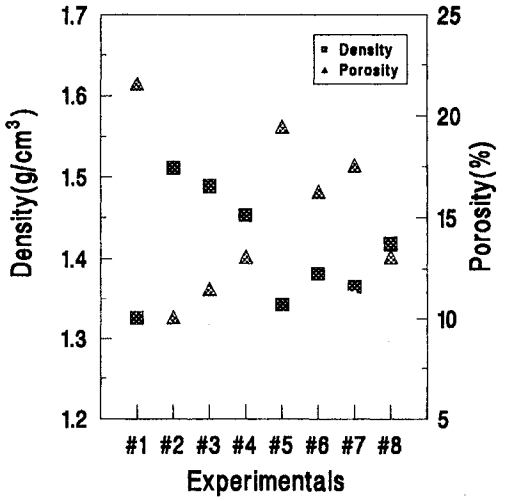

Fig.1. The apparent density and porosity of the carbon/carbon composites obtained from matrix experimentation.

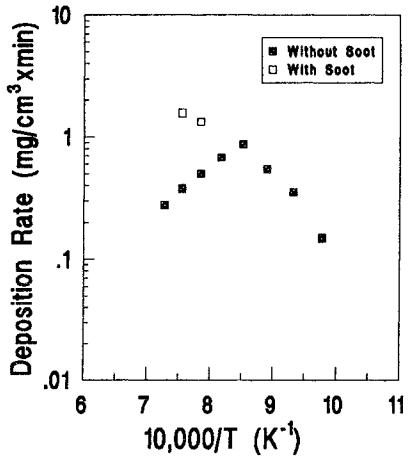

Fig.2. Deposition rate dependence on sample temperature in pulse CVI.

temperature regions are apparent. At low temperature region between 800 and 900°C, the deposition rate is limited by chemical kinetics and increased exponentially with temperature. Since the rate is limited by chemical kinetics, infiltration can be achieved by minimizing temperature variations. At high temperature region, after removal of soot, the deposition rate decreases with increasing deposition temperature. It may be due to an increased decomposition of propane and the pressure of the chamber exponentially. So the diffusion of carbon particle from pyrolysis of propane was not easy. Therefore, the carbon particle collided many active carbons before deposit on chamber wall or specimens and the carbon particle grew to soot. In order to achieve maximum density of carbon/carbon composites, the experimental conditions of pulse CVI were set up based on the results of robust design analysis and of kinetic study. The density increment of 20% was obtained at this study.

SUMMARY

The most effective processing parameters of pulse CVI on densification of carbon/carbon composites are temperature, introducing time of reactant gas, number of pulse, and interaction of introducing and evacuating time of gas. The optimum experimental condition in this study is thought to be 850°C of temperature, 100% of propane gas, 5sec of gas introducing time, 20sec of evacuation time and 10,000 of pulse number.

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