EFFECT OF TEMPERATURE AND RESIDENT TIME IN CARBON INFILTRATION BY PULSE-CVI

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Introduction

Chemical vapor infiltration(CVI) is a method of densing composites. There are several kinds of CVI such as isothermal CVI, thermal gradient CVI, pressure gradient CVI and pulse CVI. Isothermal CVI has the advantage of simultaneous treatment of numerous components of different size and shape, but has the disadvantages of nonuniformity and long processing time[1]. Therefore, Pulse CVI offers the potential for overcoming these problems[2]. The pulse CVI process is characterized by repeat cycles of evacuation of the chamber, introduction of the source gas, holding to allow infiltration and re-evacuation. When the chamber is re-evacuated, the unreacted gas is also evacuated and the inside of the preform reaches a state of low pressure. Then the gas is flowed into the chamber and rapidly penetrates throughout the preform. So the preform is densified uniformly in a short time. Sugiyama *et al.* densified various preforms with BN or SiC using pulse CVI[3]. They could get highly densified and uniformly infiltrated composites.

In this study, carbon/carbon composites were densified by pulse CVI. To determine the effects of temperature of the susceptor, propane vapor pressure and residence time on densification of carbon /carbon composites, each parameter was varied with the other parameters being held constant. The fast optimum deposition conditions were found through these experiments.

Experimental

The substrate material(preform) used in this study was a 2-D carbon/carbon composite. It was carbonized at 1000℃ for 2 hours after stacking carbon cloth(8-harness satin), made of PAN-based carbon yarn(package of fibers), with phenolic resin. Most pores had a size lying between 1 and 7 ㎛. The size of the specimen with 6㎜ penetrating hole was 20×20×20㎜. The preform was heated by susceptor using an induction heating apparatus. The density and porosity were measured by water immersion method (ASTM-C20-87). The infiltration rate was measured by the ratio of the mass gain of carbon per unit volume of preform in process of time. Hitachi S2150S and Autopore II 9220 were used for SEM observation and measurenent of pore size distribution, respectively.

Results and Discussion

The PAN-based carbon/carbon samples untreated by pulse CVI have a bulk density of 1.41±0.01g/㎤ and a porosity of 22.1±0.6%.

The previous work reported that the most important processing parameters in pulse CVI to densify the carbon/carbon composites were temperature, introduction time of gas and number of pulses[4]. Moreover, in reducing the processing time, the important parameters are residence time and vapor pressure(P) of gas and evacuated pressure(P_{eva}). After conducting carbon infiltration according to varying residence time by pulse CVI, the infiltration rates are shown in Fig.1. In these experiments, the density increased to 1.57 - 1.60g/㎤ and the porosity decreased to about 5%. The density increment is larger for experiment conducted with residence time of 45sec than that of 60sec. The reason may be attributed to deposition of pyrocarbon at the entrance of the pore(or surface) with the lapse of residence time.

Regarding mass transfer, a pulse is composed of two stages. First, during a few tenths of second, mass transfer occurs by forced convection[5]. Then during the rest of the pulse diffusion generally occurs between the gas phase (chamber dead volume and pores of preform) and the preform surface in a furnace. In this work, however, the pyrolysis of

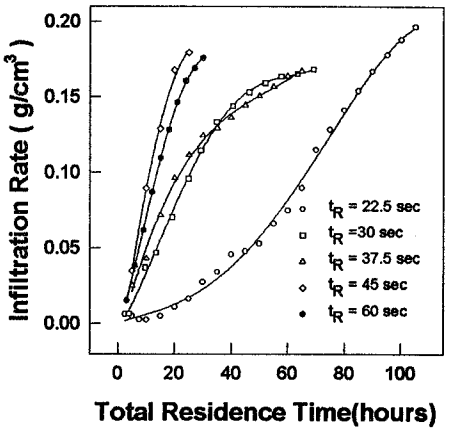

Figure 1. The infiltration rate as a function of total residence time obtained from pulse CVI.(Propane vapor pressure: 200Torr, Temperature of susceptor: 724℃, Pressure of evacuation: 0.06Torr)

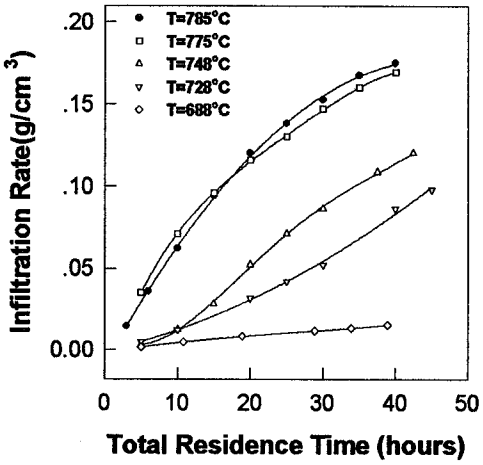

Figure 2. The relations of infiltration rate with temperature as a function of residence time by pulse CVI(Propane vapor pressure: 200Torr, Evacuated pressure: 0.06Torr, Residence time: 45sec)

propane occured mainly in pores, preform surface and susceptor surface by use of the induction heating aparatus. Therefore, it was possible to infiltrate a pyrocarbon over the preform without soot at a long residence time. But at a high propane vapor pressure and long residence time, it will be impossible to obtain a dense carbon/carbon composites becuase of filling up the entrance of pores with the deposited carbon.[6]

Fig.2. shows an infiltration rate dependence on different temperatures of susceptor at the same residence time and gas vapor pressure. As the temperature increases, the infiltration rate increases without soot under the gas vapor pressure of 200Torr. At low temperature, the infiltration rate is limited by chemical kinetics and increased exponentially with temperature. In order to achieve a dense carbon/carbon composite in a short time, the temperature of susceptor must be higher than 775℃.

Conclusions

The fast effective processing parameters of pulse CVI on densification of carbon/carbon composites are temperature, vapor pressure of reactant gas, residence time and evacuation pressure. Upto now the optimum experimental condition is thought to be 775℃ of susceptor temperature and 45sec of residence time at P= 200Torr and P_{eva}= 0.06Torr.

References

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