CARBON FIBER FELTS DENSIFIED BY CVI PROCESS

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Introduction

Carbon Fiber Reinforced Carbon (CFRC) composites Composites are thermo-structural materials characterized by a low density and retention of strength and modulus at high temperatures. These are good reasons to make them practically the ideal material for use in aerospace applications, such as rocket nozzles, exhaust cones, heat shields [1].

The properties of CFRC composites, similarly to fiber reinforced plastics, is fiber dominated and a variety of fiber preforms can be used to obtain a monolithic piece. One of the fiber preforms that can be used is carbon fiber felts. Carbon fiber felts are obtained by standard textile methods, that is, carding and needling fibers, leaving a network of open spaces suitable for densification. The more the needling the denser is the carbon felt [2]. Also, carbon fiber felts are the base substrate to conduct any experiment to obtain preliminary data on composite densification because their low cost.

The purpose of this work is show can densification preliminary laboratory densification experiments using chemical vapor infiltration (CVI) can be performed on a porous substrate. The well know CVI process produces a highly anisotropic carbon matrix and is conduced in temperature range of 900 - 1100°C [3]. A number of variables have to be considered in this process, such as deposition temperature, pressure, gas composition, properties of substrate among others. A carbon bearing gas is usually utilized to diffuse in the pores network of the substrate and deposit carbon on the available surface area. Consequently, pore volume will progressively reduce with increasing densification time [3].

This work describes experiments on densification of carbon fiber felts in a laboratory scale during various intervals of time and gas composition in order to observe trends on densification efficiency.

Experimental

A random carbon fiber felt was used in this work as a preform and it is made of oxidized polyacrylonitrile (PANox). The oxidation is usually conduced at 200 - 250°C in air. To convert this PANox felt into carbon fibers it was carbonized in inert atmosphere up to 1000°C at 20°C/min. A weight loss of ~35% and an apparent density of ~0.12 g/cm^3 was obtained after carbonization.

The experiments were carried out on isothermal mode at 1000°C using a tubular furnace, hot zone ~20 cm, see Figure 1, under atmospheric pressure of ~94 kPa. The gas mixture was composed of methane, as carbon bearing gas and nitrogen as the carrier and diluent gas. The gas mixture was dehumidified before entering the furnace. Care was taken to avoid a direct contact of the flowing gas mixture directly with the felt substrate by using a deflector after the gas entrance.

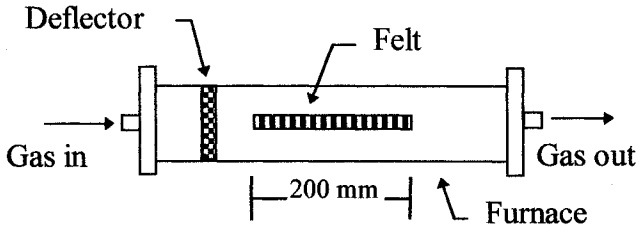

Figure 1 - Experimental set up for CVI processing.

The densification experiments were conduced in two ways:

- the carbon fiber felt was CVI processed during a period of 24 hours and then flipped over to another period of 24 hours of densification. This procedure was repeated until 144 hours of densification was completed. The N_2/CH_4 gas ratio was 12:1.

- the carbon fiber felt was CVI processed during a period of 24 hours and then flipped over, as before, for a second 24 hour period, using different N_2/CH_4 gas ratios. After each 48 hour densification period samples were cut at regular interval along the length of the substrate in order to determine apparent density.

Flipping the carbon fiber felt is expect to provide a more uniform deposition throughout the length of the substrate. The total flowing gas volume (N_2 + CH_4) was maintained at ~1 liter/min.. The partial pressures of the mixture N_2/CH_4 was calculated according to standard physical chemistry equations for gases and they are shown on the Table 1. Table 2 shows the apparent density along the sample length for different volume ratios of N_2/CH_4.

Table 1. Partial pressures of N_2 and CH_4

N_2/CH_4 Ratio	Partial pressures (kPa)	
	N_2	CH_4
12:1	6.1	87.9
10:1	7.4	86.6
6:1	11.3	82.7

Table 2. Apparent density (g/cm^3) for different N_2/CH_4 ratios along the sample.

N_2/CH_4	Position in the substrate				
Ratio	**0**	**5**	**10**	**15**	**20**
12:1	0.300	0.412	0.420	0.396	0.295
10:1	0.347	0.476	0.558	0.517	0.338
6:1	0.503	0.711	0.748	0.728	0.528

Figure 1 shows a plot of apparent density as function of CVI processing time. From an initial density of ~0.12 g/cm^3 the carbon fiber felt has been densified to an apparent density or ~1.18 g/cm^3 after 144 hours of carbon deposition, giving a rate of deposition of 40 mg/min. If we consider that an acceptable final apparent density for the carbon fiber felt composite is around 1.90 g/cm^3 and maintaining the same rate of deposition a total of ~250 hours would be necessary to achieve the total densification.

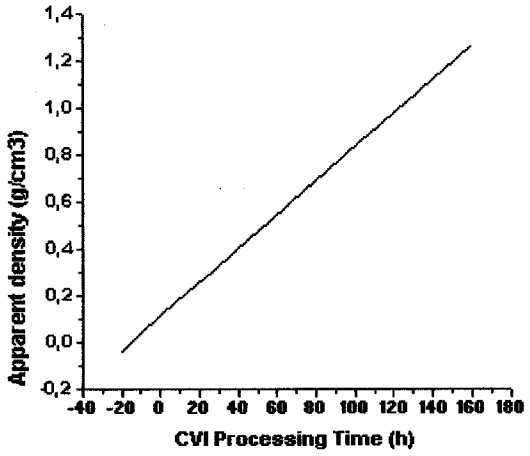

Figure 1. Apparent density as a function of CVI processing time. it increases with CVI processing time. N_2/CH_4 volume ratio: 12:1.

Figure 2 shows the plot of Table 2, i.e., apparent density as a function of sample length. A bell shape curve is found for any of the N_2/CH_4 ratios. The more diluted is the flowing gas stream the higher is the amount of carbon deposition in the substrate and consequently a higher density is achieved. This can be explained bearing in mind that a higher amount of collisions from CH_4 with the fibrous substrate could happen and possibly at higher concentration soot can be formed in the gas stream.

The variation in apparent density from the ends to center of each densified sample is around 30%. The temperature profile of the furnace has its maximum temperature in its center this explains the small difference in the apparent density profile.

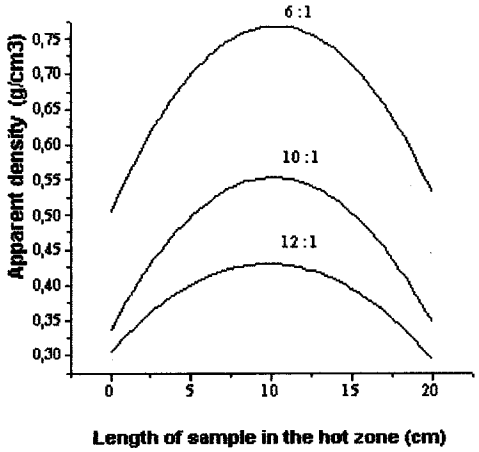

Figure 2. Apparent density as a function of length of sample The variation of apparent density along the sample length as function of different N_2/CH_4 ratios.

Conclusion

This study presents results on densification of carbon fiber felts. The ratio of N_2/CH_4 corresponding to 6:1 exhibit the highest apparent density achieved by the densified composite. A bell shaped curve is found for any ratio of N_2/CH_4 used in the experiments due to differences in the temperature profile of the CVI furnace. Laboratory scale experiments can be used to obtain preliminary data on CVI process.

References

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3. Kotlensky, W. V. ,Deposition of Pyrolytic Carbon in Porous Solids, Chemistry and Physics of Carbon, vol 9, edited by P.L. Walker, Jr. and P. A. Thrower, 1973, pp. 173.