The Chemical Vapor Deposition of Carbon in Open-Ended Capillary Tubes

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Abstract. Chemical vapor deposition of carbon was studied in uniform bore capillary tubes using CH_4 as deposition gas. The relation between CVD parameters and the resultant deposition profile, rate, and optical microstructure were examined. Higher temperature, pressure and flow rate produce higher deposition rate and optical activity. Lower temperature, pressure, but high flow rate yield better deposition uniformity along the capillary.

Introduction

The chemical vapor deposition of carbon in the interior of textile products often produces poor deposition uniformity, improper structure and low deposition rate. The understanding of the carbon deposition process in textile preforms is complicated by the complex pore system. Jacklowski [1] used open-ended, uniform bore capillaries to model carbon deposition. This paper reports the effects of pressure, temperature, gas flow rate, capillary diameter, and contact time on the deposition of carbon in capillary tubes. A mechanism of deposition will be discussed.

Experimental

The graphite capillaries were 38 mm long, different diameters and open at one end only. Capillaries were located in a graphite block 220mm long at levels L-1 and L-2, Fig. 1. Ultra high purity methane was used as a source gas. Deposition conditions were studied over the following ranges:

Temperature 1100-1400°C
Pressure 2-50 Torr
Gas Flow Rate 90-700SCCM
Capillary Diameter 430-1030$_{\mu m}$

Reaction time was adjusted to minimize any epitaxial surface effects and change in capillary cross-section. The coating thickness was generally about 50$_{\mu m}$ at the mouth of the capillary. Optical microscopy was used to determine deposit thickness and microstructure. Optical activity was measured under polarized microscope using a photometer, and is presented as an apparent rotation angle.

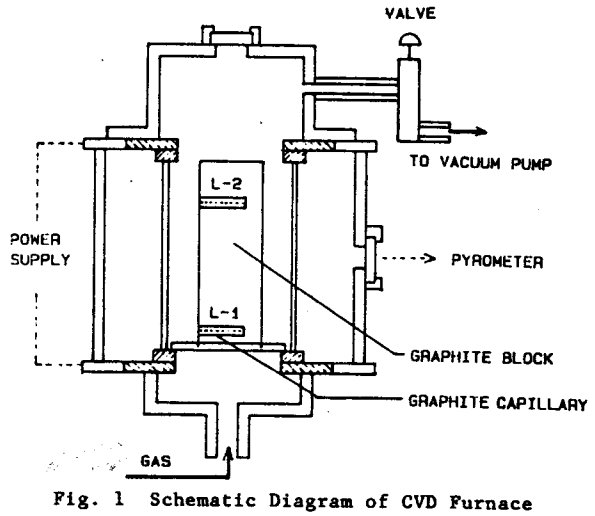

Fig. 1 Schematic Diagram of CVD Furnace

Results and Discussion

The deposits along the capillary were examined for microstructure and the thickness was measured. The maximum deposition rate, (k_{max}) whether at the capillary mouth or inside the capillary was half order over the pressure range of 2-20 Torr (Fig. 2). The activation energy for k_{max}

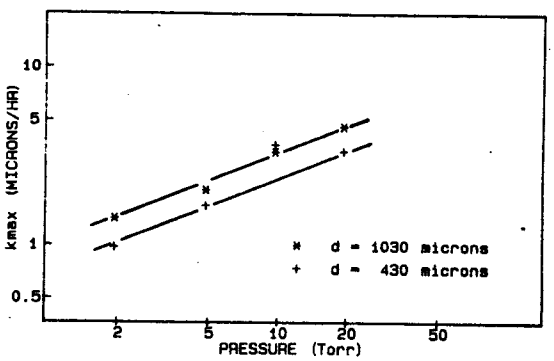

Fig. 2 Effect of Pressure
1300°C, CH_4 270 SCCM, L-1

was 54.0 kcal/mole. Lower temperature and particularly lower pressure produced better penetration within the capillaries (Fig. 3 and 4). Higher flow rate past the capillary increased k_{max}, but did not improve penetration at higher pressures (Fig. 5). In general, capallaries with large diameter produce deeper penetration. In Fig. 6, deposition profile at different capillary diameters is plotted against DEPTH/diameter ratio which suggests surface/volume ratio of a capillary is one of the factors which could determine penetration profile. Longer contact time (L-2) gives higher rate at mouth of a capillary; on the other hand, shorter contact time (L-1) shows a better profile (Fig. 7). The amount of deposition species has not reached to give maximum deposition rate at the mouth of L-1.

Finally, higher orientation (apparent rotation angle of 7-12 degrees) was observed at high temperature and high flow rates.

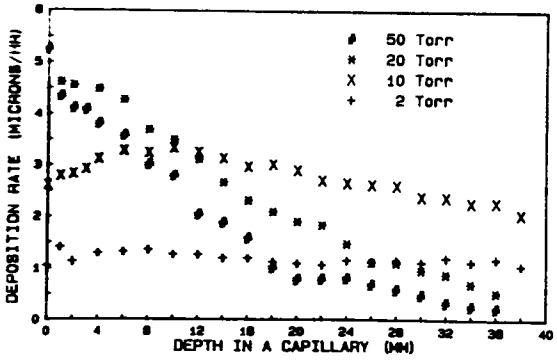

Fig. 3 Effect of Pressure
1300°C, CH_4 270 SCCM, d=1030

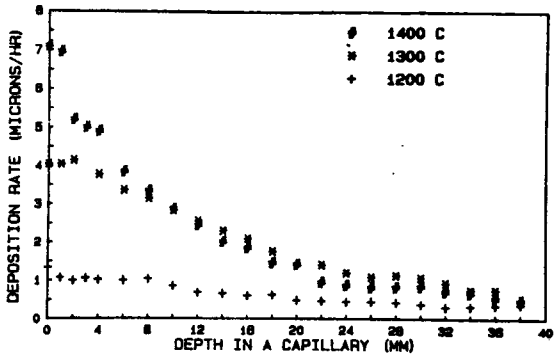

Fig. 4 Effect of Temperature
20 Torr, CH_4 270 SCCM, d=1040 microns, L-2

References

1. T. Jacklowski and R.J. Diefendorf, 15th Biennial Conf. on Carbon, 1981, 284-285.

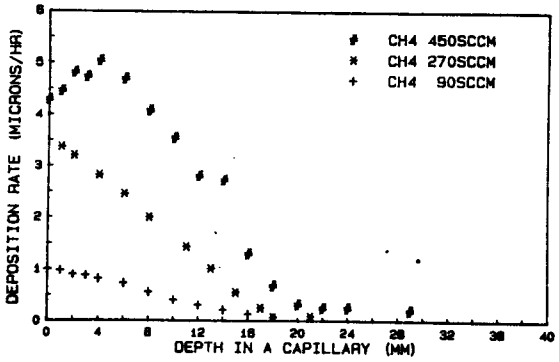

Fig. 5 Effect of CH_4 Flow Rate
1300°C, 20 Torr, d=430 microns, L-1

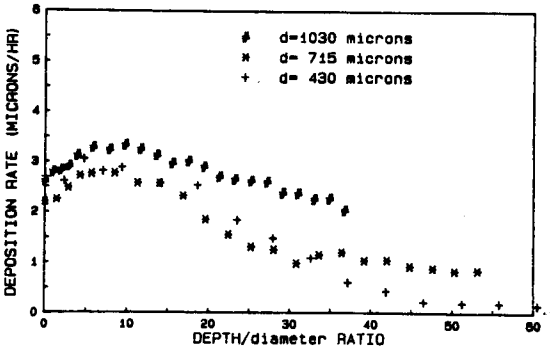

Fig. 6 Effect of Capillary Diameter
1300°C, 10 Torr, 270 SCCM, L-1

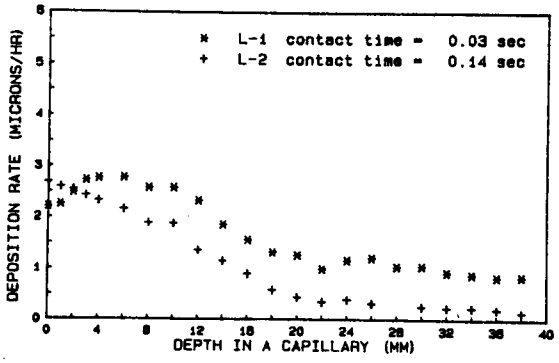

Fig. 7 Effect of Contact Time
1300°C, 10 Torr, CH_4 270 SCCM, d=715 microns