Mechanical Properties of Isotropic Pyrolytic Carbon Deposited in a Tumbling Bed

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Abstract. The relations between the coating conditions and mechanical properties of the isotropic pyrolytic carbons deposited in a tumbling bed have been investigated. The mechanical properties were affected by the soot inclusions in the deposits which had a non-load bearing character and behaved as cracks under load. By alloying silicon with the carbon, the fraction of soot inclusions was not varied, and the fracture strength increased linearly with the SiC content.

Introduction
Stationary beds, fluidized beds and tumbling beds are usually employed to deposit pyrolytic carbons. For pyrolytic carbons deposited in a fluidized bed, the correlations between deposition variables, structures and mechanical properties are rather well established. But the correlations for carbons deposited in a tumbling bed are not known at all. The objective of this work is to establish the dependence of the mechanical properties of pyrolytic carbons deposited in the tumbling bed on structures and process variables.

Experimental
A tumbling bed apparatus used to deposit pyrolytic carbons has been shown elsewhere. The carbon samples were prepared at 1220 °C of deposition temperature using argon-propane gas mixture. The concentration of propane and the amount of bed particles were varied and related to the mechanical properties. For silicon-alloyed pyrolytic carbons, the contents of silicon in the deposits were controlled by varying the argon flow rate through the bubbler containing liquid methyltrichlorosilane (CH₃SiCl₃). The density of the deposited carbon was measured by the sink-float method and the apparent crystallite size by X-ray diffraction. The mechanical properties of the pyrolytic carbons were measured by three-point bend test.

Result and Discussion

Pure Pyrolytic Carbon
The densities of pure pyrolytic carbons measured are in the range 1.90 - 1.96 g/cm³.

This variation of densities is, however, not significant enough to affect the mechanical properties greatly. The crystallite sizes vary having no relations to the variations of the propane concentrations and the amount of bed particles.

Fig. 1 shows that the Young's modulus, the fracture stress and the strain energy to fracture all increase as propane concentration decreases and/or the amount of bed particles increases. In this figure, we modified the measured fracture stresses to take into account the size differences of mechanical test specimen using Weibull's statistical method in order to compare the mechanical properties obtained in this work with those obtained in the fluidized bed.

Kaae has related the mechanical properties of pyrolytic carbons deposited in a fluidized bed with structural properties, i.e., the density and the apparent crystallite size. The large strength changes with a small increase of density and with the irregular changes of crystallite sizes as shown in Fig. 1 cannot be explained by the same argument. Moreover, the absolute values of the mechanical properties of pyrolytic carbon obtained in the tumbling bed are generally lower than those deposited in a fluidized bed.

Fig. 2 shows soot inclusions in the deposited layers and Fig. 3 indicates that the fraction of soot inclusions, which was analyzed by means of the point counting method, increases with increasing the propane concentration and decreasing the amount of bed particles.

The soot inclusions cannot bear much load, thus reducing the effective area. This effect lowers the Young's modulus. They also reduce the fracture stress and strain energy to fracture...
because they act as flaws. According to the Griffith theory, the fracture strength ($\sigma_f$) and the strain energy to fracture ($\varepsilon_f$) are represented as

$$\sigma_f = (Y/C)^{1/2}$$

$$\varepsilon_f = (\sigma_f^2/2E) = Y/C$$

where $Y$ and $C$ denote effective work of fracture and critical flaw size, respectively, and $E$ is Young's modulus. As soot inclusions act as flaws, they decrease the fracture strength and, more significantly, the strain energy to fracture.

Silicon-Alloyed Pyrolytic Carbon

Silicon in the pyrolytic carbon exists as $\beta$-SiC. As the content of SiC increases, the carbon matrix density is almost constant while the apparent crystallite size decreases. The fraction of the soot inclusions are independent of the silicon content. Fig. 4 depicts the changes of mechanical properties with the weight fraction of SiC. As the amount of SiC increases. Young's modulus and fracture strength increase while the strain energy to fracture remains constant. These

Figure 1. Variation of mechanical properties of pure pyrolytic carbons. (a) the dependence on propane conc.: $\circ$ 6% of bed particles (measured), $\bullet$ 6% (modified), $\triangle$ 3% (measured), (b) the dependence on bed particle wt. $\circ$ 50% of propane conc. (measured), $\bullet$ 50% (modified), $\triangle$ 90% (measured).

Figure 2. Microstructures of pure pyrolytic carbons deposited at various conditions; the propane conc. and the wt. of bed particle are (a) 30%, 6g (b) 50%, 6g (c) 70%, 6g (d) 70%, 0g (e) 70%, 3g (f) 70%, 8g.

Figure 3. Dependence of the fraction of soot particles on propane conc. and the amount of bed particles.

Figure 4. Variation of mechanical properties of Si-alloyed pyrolytic carbons with the content of SiC. $\triangle$ 30% $C_3H_8$ conc., $\circ$ 50% $C_3H_8$ conc.
changes in mechanical properties can be explained by the two facts of lower $L_c$ and higher SiC content. $L_c$ cannot be, however, expected to affect the mechanical properties significantly at a high matrix density. The major influence on mechanical properties, therefore, appears to come from the SiC content.

**Summary**

Mechanical properties, Young's modulus, fracture strength and strain energy to fracture of pure pyrolytic carbon deposited in a tumbling bed are increased as the propane concentration decreases and the amount of bed particles increases. This is due to the soot inclusions. All mechanical properties improve as the fraction of soot inclusions decreases. In silicon-alloyed pyrolytic carbons, Young's modulus and fracture strength increases as the SiC content increases.

**References**