DIRECT MEASUREMENT OF CARBON THERMAL DIFFUSIVITIES USING OPTICAL BEAM DEFLECTION (OBD)

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Introduction

Several authors have reported measurements of the thermal diffusivities of carbon films and fibers [1-3]. In this paper we propose an alternative and direct method to measure the thermal diffusivity of carbon materials. This method is OBD.

Experimental

OBD is a nondestructive, non-contact method of measuring the thermal diffusivity of materials [4-6]. Figure 1 shows the arrangement of the components of the OBD instrument. A chopped beam of light focused on the surface of a material generates heat pulses. The pulses travel outward along the surface of the material and heat the layer of air immediately above the sample. A time-varying gradient in the index of refraction of the air layer is produced. A laser beam propagating in this layer of air is deflected by each passing heat pulse.

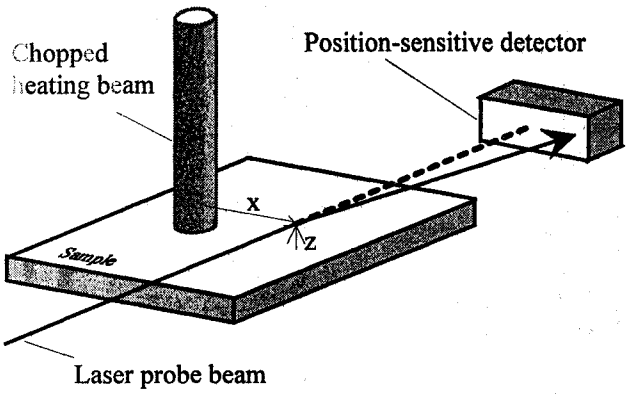

Figure 1. Arrangement of OBD experiment.

The deflections of the probe beam are very small, and the experiment must be done on a platform isolated from vibrations. In addition, the probe beam must propagate close to the surface of the sample, so the surface must be as planar as possible.

The phase ϕ of the probe beam is plotted versus the position x of the beam. A different set of data is obtained for each frequency of the chopped heating beam. The wavelength λ of the thermal wave is determined from the gradient of the phase of the signal, $\Delta\phi/\Delta x$, which is found experimentally:

$$\lambda = \frac{360^\circ}{\left(\frac{\Delta\phi}{\Delta x}\right)}$$

where ϕ is measured in degrees. Finally, the thermal diffusivity α is calculated from the wavelength and the frequency f:

$$\alpha = \frac{\lambda^2 f}{4\pi}.$$

Sample Preparation

Isotropic and anisotropic carbons were made by chemical vapor deposition (CVD). In each case the carbon material was recovered from the surface of a crucible after thermal cracking of a hydrocarbon.

Two polyimide carbon films of 30 μm and 120 μm thicknesses were used in this study. They were cured and carbonized by raising the temperature at a rate of 4 C°/min to a temperature of 1000 °C and maintaining that temperature for 1 hr.

A 90 μm carbon film was made by stacking 3 of the 30 μm films before curing and carbonization. This stack was subjected to a pressure of 500 lb/in^2 for 30 minutes and allowed to cool. The pressure was removed, and this film was cured and carbonized in the same manner as the other films.

Results

The thermal diffusivities of five carbons were studied. Samples of each carbon were heat treated at six temperatures from 1000 °C to 2600 °C. The thermal diffusivity of each sample was measured after each heating.

Figure 2 shows the thermal diffusivities of anisotropic and isotropic carbons as a function of temperature of heat treatment. After heat treatment at the highest temperature, the thermal diffusivity of anisotropic carbon rises signifi-

cantly compared to the essentially unchanged value of isotropic carbon.

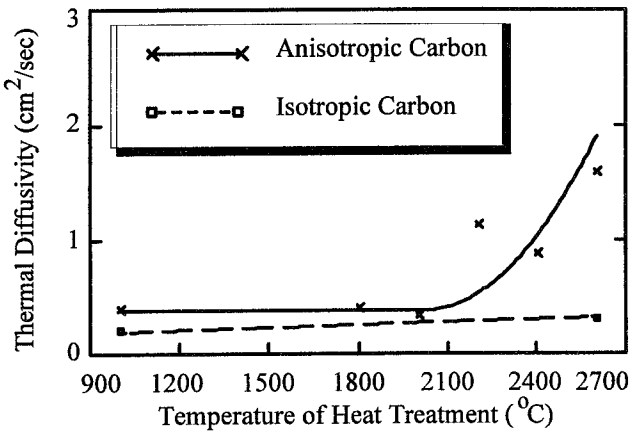

Figure 2. Thermal diffusivities of anisotropic and isotropic carbon as a function of the temperature of heat treatment.

Figure 3 shows the thermal diffusivities of the carbon films as a function of the temperature of heat treatment. After treatment at the highest temperature, the thermal diffusivities are all higher than for lower temperatures.

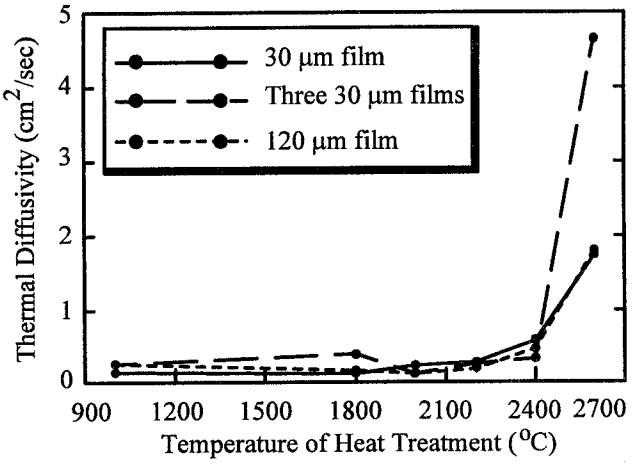

Figure 3. The thermal diffusivites of three carbon films as a function of the temperature of heat treatment.

Although the polyimide carbon films studied are thin, the effect of the scattering of thermal waves from the rear surface and backing material are negligible. This is because the thermal diffusivity measured along the graphitic planes is approximately 200 times that measured across the planes.

The data on the isotropic and anisotropic CVD carbons show the effect of increased crystallinity. The higher heat treatment temperatures increase the crystallinity which results in an increase in the thermal diffusivity. As is the case for graphitic material, having larger and more perfect crystallites and fewer sites for phonon scattering increases the thermal diffusivity. This is the case for anisotropic CVD carbon which consists of large graphitic grains compared to isotropic carbon. Its graphitizability is expected to be greater. This would lead to the increased thermal diffusivity observed after heat treatment at graphitizing temperatures.

Conclusions

OBD has been proven to be a successful method for monitoring the changes in the thermal diffusivity of graphitizable carbons as a function of heat treatment.

The thermal diffusivities of polyimide carbon films behave like anisotropic CVD carbons when heat treated.

The 30 μm and the 120 μm polyimide carbon films show the same increase in the thermal diffusivity. However, the sample made by stacking 3 of the 30 μm polyimide carbon films shows a larger increase in the thermal diffusivity. This sample was made under pressure. It's grain size is much larger. It confirms the possibility of making a highly orientated pyrolytic thin polyimide film under pressure.

References

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