THE FRACTURE TOUGHNESS OF GLASSY CARBONS AT ELEVATED TEMPERATURES

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Abstract—The fracture toughness of three different glassy carbons heat treated at 1000, 2000 and 3000°C was measured at temperatures between 25 and 750°C. Young's elastic modulus varies inversely with measurement temperatures, whereas fracture toughness varies directly. Fracture characteristics of the glassy carbons are compared with those of float glass and graphites.

1. INTRODUCTION

Glassy carbons, resembling inorganic silicate glasses, are noted for their low gas permeability, glass-like isotropic properties, high temperature stability and chemical inertness[1]. They are produced under a variety of trade names by various manufacturers. Even though much research work has been carried out to determine the physical and mechanical properties of glassy carbons[2–8], little work has been done on their fracture characteristics[9–10]. The purpose of this work is to investigate the fracture characteristics of glassy carbons and to determine how measurement temperature affects Young's elastic moduli and fracture toughness.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

The glassy carbons studied were produced by Tokai Electrode Manufacturing Co., using proprietary processes. The samples GC-10, GC-20, and GC-30 has maximum HTT of 1000, 2000 and 3000°C, respectively. Subcritical crack growth of these carbons in water was previously studied in this laboratory[10].

The properties of these glassy carbons are listed in Table 1. As received samples were in the form of plates 3mm thick, 25 mm wide and 75 mm long. Fracture specimens were cut, using a diamond saw, to 3 mm thick, 3 mm wide and 25 mm long. Final test surfaces were polished using a No. 600 grit wheel.

2.2 Elastic moduli

Young's elastic moduli were determined by the sonic technique[11] using a commercial acoustic spectrometer made by Nametre Corp. This method consists of measuring the resonant frequencies of test specimens and computing the elastic moduli from these frequencies. The flexural resonant frequencies

were measured to estimate Young's elastic moduli (E) and the torsional resonant frequencies were measured to estimate the shear moduli (G). An iteration method was used to determine precisely the elastic moduli and Poisson's ratio (v)[12], that is by assuming a value for Poisson's ratio and calculating a tentative value for Young's elastic modulus. From the shear modulus and the tentative value for Young's elastic modulus, Poisson's ratio is calculated by means of the relation

$$v = (E/2G) - 1. \qquad (1)$$

This value is compared with the assumed value. If a discrepancy exists, the Young's modulus is recalculated with the new value for Poisson's ratio. This was repeated until the value of Poisson's ratio finally calculated agreed within 0.005 with the values used in the final calculation of Young's modulus.

The coefficient of thermal expansion was measured and used to calculate Young's moduli at elevated temperatures by[13]

$$E_T = E_0 f_T^2 / f_0^2 (1 + \alpha \Delta T) \qquad (2)$$

where the subscripts T and 0 refer to temperatures T and room temperature, respectively, α is the coefficient of thermal expansion, ΔT is the difference between temperature T and room temperature, and f is the resonant frequency of the first mode of the torsional vibration.

2.3 Fracture toughness

The controlled surface microflaw technique was used to measure the fracture toughness K_{Ic} in air[14]. Knoop microhardness indentations were placed in the centers of the specimens using a 3600 g load. The long diagonal of the indentation was aligned perpendicular to the direction of tensile stress in subsequent flexural tests. In order to remove the residual stress at the vicinity of the indentation, the surface of each indented specimen was ground down about 76 μm. This condition was chosen after a series of preliminary experiments to determine the

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Table 1. Properties of the glassy carbons

Property		GC-10	GC-20	GC-30
Density	g/cm^3	1.47	1.51	1.48
Bending Strength (R.T.)	kg/cm^2	903	1266	1122
Elastic Modulus (R.T.)	$10^4 MN/m^2$	2.89	3.19	2.31
Shear Modulus (R.T.)	$10^4 MN/m^2$	1.25	1.35	1.03
Poisson's Ratio (R.T.)		0.15	0.17	0.12
Fracture Toughness (R.T.)	$MN/m^{3/2}$	0.93	1.17	1.19
Critical Strain Energy Release Rate (R.T.)	J/m^2	29.9	43.3	61.9
Coefficient of Thermal Expansion (25-750°C)	$10^{-6}/°C$	4.4	3.6	5.4

extent of grinding required to remove the effect of grinding depth on the flexural test measurement. Measurements were made in a vertical tube furnace. With the furnace at temperature, the specimen was quickly placed in the hot zone and tested immediately upon reaching thermal equilibration, that is, within 5 min. Three-point bending tests were performed over a 19 mm span on an Instron machine at a crosshead speed of 0.05 mm/min. The fracture toughness K_{lc} was calculated from

$$K_{lc} = 2.06\sigma_f(a/\pi)^{1/2} \qquad (3)$$

where a is the depth of the flaw and σ_f is the bending strength[15]. At least four specimens were tested at each temperature. Results are reported as the average. In all cases at the 95% confidence limit, values fell within ±2.5% of the average value.

3. RESULTS AND DISCUSSION

3.1 Elastic properties

Changes in Young's elastic moduli of glassy carbons as a function of measurement temperature are shown in Fig. 1. Elastic moduli decrease essentially linearly with temperature over the range 25–750°C, showing decreases

ranging from 3–5%. The extent of change in moduli with temperature is essentially independent of the HTT of the glassy carbon, even though there are large differences in the absolute values of moduli for samples heated to different temperatures. Moduli appear to be very sensitive to differences in density of the glassy carbons as expected; the density peaks for GC-20 as do the moduli. The elastic moduli of float glass also decrease with increasing measurement temperature, the decrease being particularly marked above 400°C[16].

3.2 Fracture toughness

Changes in fracture toughness of glassy carbons with measurement temperature are shown in Figs. 2–4. At all measurement temperatures up to 750°C, fracture toughness increases with increasing HTT of the glassy carbon. The dependence of fracture toughness on measurement temperature is different for the three glassy carbons, as is the extent of the total change in fracture toughness in going from 25–750°C. For GC-10, no change in fracture toughness is found up to 300°C, followed by an abrupt large increase with further increases in measurement temperature. For both GC-20 and GC-30, fracture toughness rises immediately with increasing measurement temperature, with the rate of increase increasing monotonically with temperature rise. The overall fractional increase in
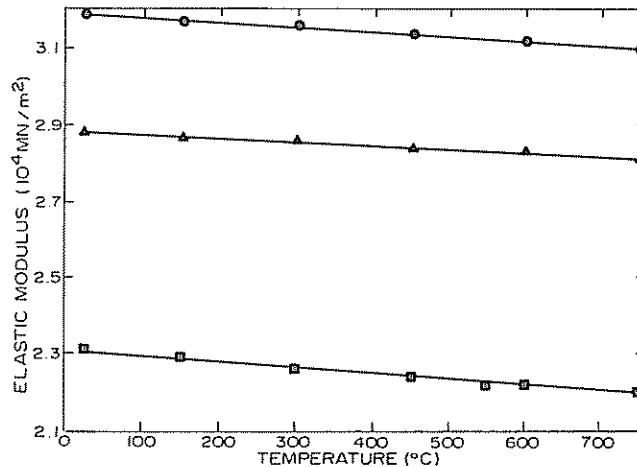

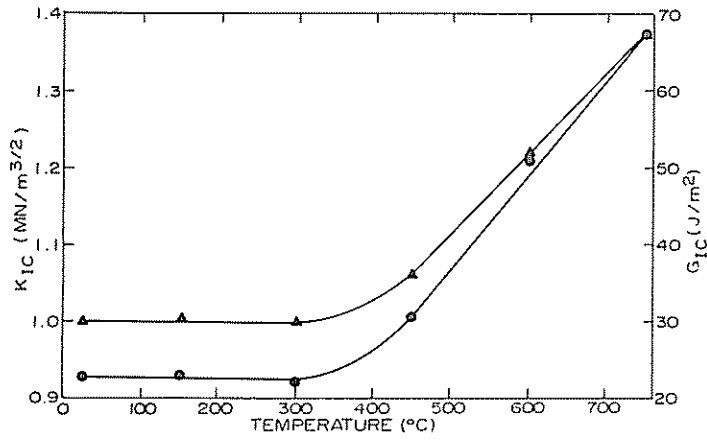Fig. 1. Young's elastic moduli of glassy carbons as a function of measurement temperature. ▲, GC-10; ◉, GC-20; ▣, GC-30.

Fig. 2. Fracture toughness ($\bullet$, K_{Ic}) and critical strain energy release rate ($\blacktriangle$, G_{Ic}) of GC-10 as a function of measurement temperature.
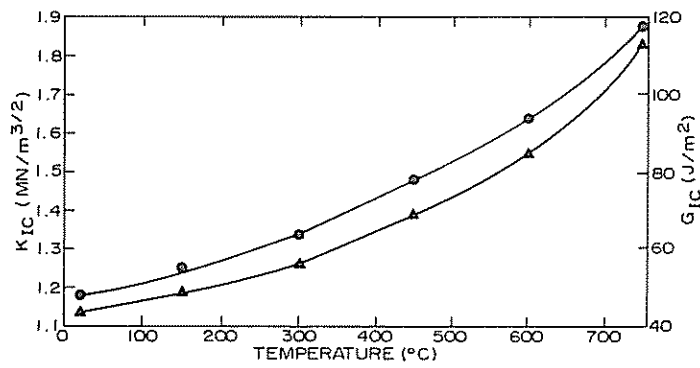

Fig. 3. Fracture toughness ($\bullet$, K_{Ic}) and critical strain energy release rate ($\blacktriangle$, G_{Ic}) of GC-20 as a function of measurement temperature.
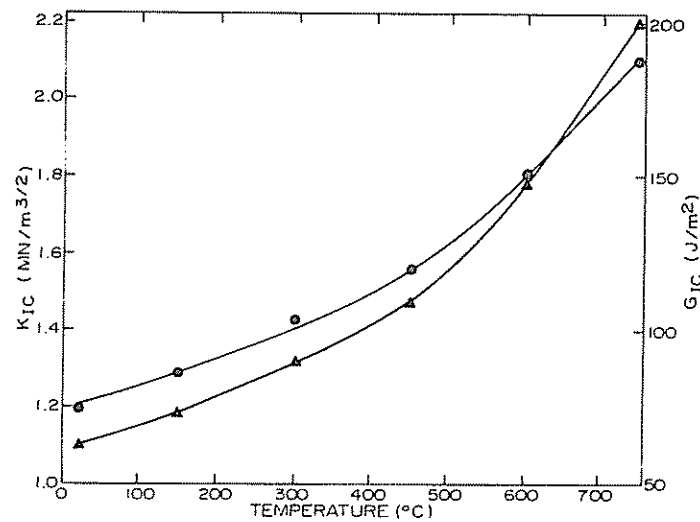

Fig. 4. Fracture toughness ($\bullet$, K_{Ic}) and critical strain energy release rate ($\blacktriangle$, G_{Ic}) of GC-30 as a function of measurement temperature.
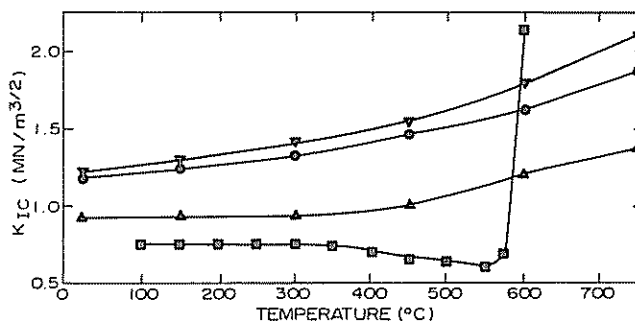
Fig. 5. Comparison of fracture toughness of float glass[16] with glassy carbons between 25 and 750°C. ▲, GC-10; ◉, GC-20; ▼, GC-30; ▣, float glass.

fracture toughness between 25–750°C is greatest for the glassy carbon of highest HTT.

Figure 5 compares the fracture toughness of float glass with that of glassy carbons between 25 and 750°C. At temperatures up to 550°C the fracture toughness of float glass is less than that for the glassy carbons. However, between 550 and 600°C, the fracture toughness of float glass increases very sharply and at 600°C exceeds values for the glassy carbons. The rapid increase in fracture toughness of float glass above about 550°C is attributed to a transition from elastic to viscous flow. At about 550°C viscous flow at the crack tip becomes significant; it escalates rapidly with increasing temperature, leading to blunting of the crack tip. Thus, results on glass dramatically show the importance of reducing stress concentrations at pores and other defects on increasing σ_f and thus K_{lc}. Increases in K_{lc} and G_{lc}, with measurement temperature, for glassy carbons are attributed to a similar mechanism.

3.3 Critical strain energy release rate

Knowing how the elastic modulus and K_{lc} change with temperature, one can calculate the change in critical strain energy release rate G_{lc} with temperature[17] from

$$G_{lc} = \frac{K_{lc}^2}{E} \qquad (4)$$

Values of G_{lc} are given in Figs. 2–4. G_{lc} changes essentially as K_{lc}^2 changes because of the small change in E with temperature.

G_{lc} reflects the total energy required for creating 1 m² of primary crack surface. It includes all of the energy needed and consumed by all phenomena occurring in the process zone surrounding the advancing crack tip. For the glassy carbons studied, room temperature values of G_{lc} ranged from 30–62 J/m². These values are substantially higher than those for graphite single crystals[18, 19] when creating crack surface parallel to the basal plane (~ 0.15 J/m²) or perpendicular to the basal plane (6 J/m²), but lower than artificial polycrystalline graphite values[17], which range from 85–160 J/m².

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