The Charpy Impact Fracture Behavior of Phenolphthalein Polyether Ketone*

YANCHUN HAN,† YUMING YANG, BINYAO LI, XUHUI WANG, and ZHILIU FENG

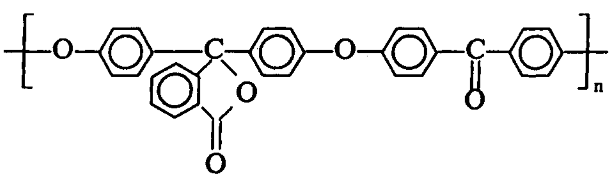
Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun 130022, People's Republic of China

SYNOPSIS

The Charpy impact fracture behavior of notched specimens of phenolphthalein poly(ether ketone) (PEK-C) has been studied over a range of temperature using a JJ-20 Model instrumented impact tester. For PEK-C, there exist two temperature regions which distinguish the fracture mechanism, and the brittle fracture was preferentially governed by slip or shear bands at relatively high temperatures, but by crazes at low temperatures. The temperature dependence of the ductility index (DI) shows similar peaks to the tan δ loss.

INTRODUCTION

Phenolphthalein poly(ether ketone) (PEK-C) is an amorphous material with a high T_g at about 215°C. Its molecular weight is 3.26×10^5. The chemical structure of PEK-C is



PEK-C can be used as an engineering thermoplastic and matrix of composites. As PEK-C is being increasing used in engineering applications, there is a need to understand the temperature effect on the impact properties. The aim of this article was to use an instrumented impact tester to investigate the temperature effect on Charpy impact properties of PEK-C.

EXPERIMENTAL

The polymer used in this study was phenolphthalein poly(ether ketone) supplied by Xu Zhou Engineering Plastics Co., China. Its reduced viscosity in chloroform at 25°C is 0.49 dL/g. The specimens were injection-molded bars that were 55 mm long (L), 6 mm wide (B), and 4 mm thick (D) and of about $\frac{1}{3}D$ crack length (a).

Charpy impact tests were carried out on a JJ-20 Model instrumented impact tester (made by Changchun Institute of Applied Chemistry, Chinese Academy of Sciences and the Intelligent Instrument and Apparatus Institute of Changchun) with single-edge notched specimens in the temperature range from 15 to 220°C. The span S was 40 mm, the capacity was 20 J, and the striking velocity was 3.8 m/s. Tests at elevated temperature involved heating the sample to the required temperature for at least 10 min in order to reach the thermal equilibrium. The specimens were then quickly mounted on the specimen holder and impacted within 2–4 s of removal from the oven. The crack length, a, was measured using a traveling microscope on the fractured specimen of the test. Five specimens were tested at each condition, and the averages were reported. The error was within 10%.

RESULTS AND DISCUSSION

Load–Time and Energy–Time Curves

With the recent introduction of an instrumented impact test,[1–3] as was employed in the present study,

* Key Project of the National Natural Science Foundation of China.
† To whom correspondence should be addressed.
Journal of Applied Polymer Science, Vol. 56, 1059–1063 (1995)
 CCC 0021-8995/95/091059-05

a load–time history for a standard Charpy impact test may be obtained. The amount of energy dissipated at the various stages of fracture may then be observed. The maximum load which the specimen can sustain is also available.

Figure 1 shows typical load–time and energy–time curves which were obtained from the instrumented Charpy impact test of PEK-C at room temperature. The monotonically increasing curve represents the energy input to the test specimen up to a given time. It is obtained by integrating the load–time trace after the load–time trace has been converted to a load–deflection trace. The time axis (the horizontal axis) may be easily converted to represent the midpoint deflection of the test specimen by assuming that the velocity of the striking pendulum is essentially constant during the fracture of the specimen. (The event is quite fast, and the error of this assumption is negligible.) The midpoint deflection is found by multiplying the time representing a particular point in the fracture process by the velocity of the pendulum at impact. The portion of the load–time trace up to the maximum or peak load is assumed to be the elastic portion of the impact event. The portion of the curve following the first peak load represents the postfracture process.

If the assumption is made that the area under the load–deflection curve to the point of the maximum load is the energy required to initiate fracture of a polymer, and if it is further assumed that fracture does not initiate until this point of the maximum load is reached, then the total strain energy stored within the specimen should equal the energy integrated beneath the load–deflection curve to the point of maximum load. It must also be pointed out that even for a "ductile" material, in which substantial amounts of energy are dissipated after maximum load has been reached, the initial fracture can also be analyzed in a similar manner.

The failure of a material when subjected to an impact load involves a complex fracture process.[4,5] Fracture usually occurs as follows: When the applied load exceeds a certain level, shear or slip bands initiate at the notch root. Being developed by further loading, they become curved and the form of a logarithmic spiral becomes evident. The extension of the plastic zone including slip bands increases the stress at the elastic–plastic boundary due to the plastic constraint to initiate a cracklike flaw, i.e., a fracture nucleus propagates in a brittle manner to lead to a final fracture. In other words, the fracture process passes through three stages as follows: (a) initiation and extension of shear bands from the notch, (b) nucleation and slow growth of the fracture

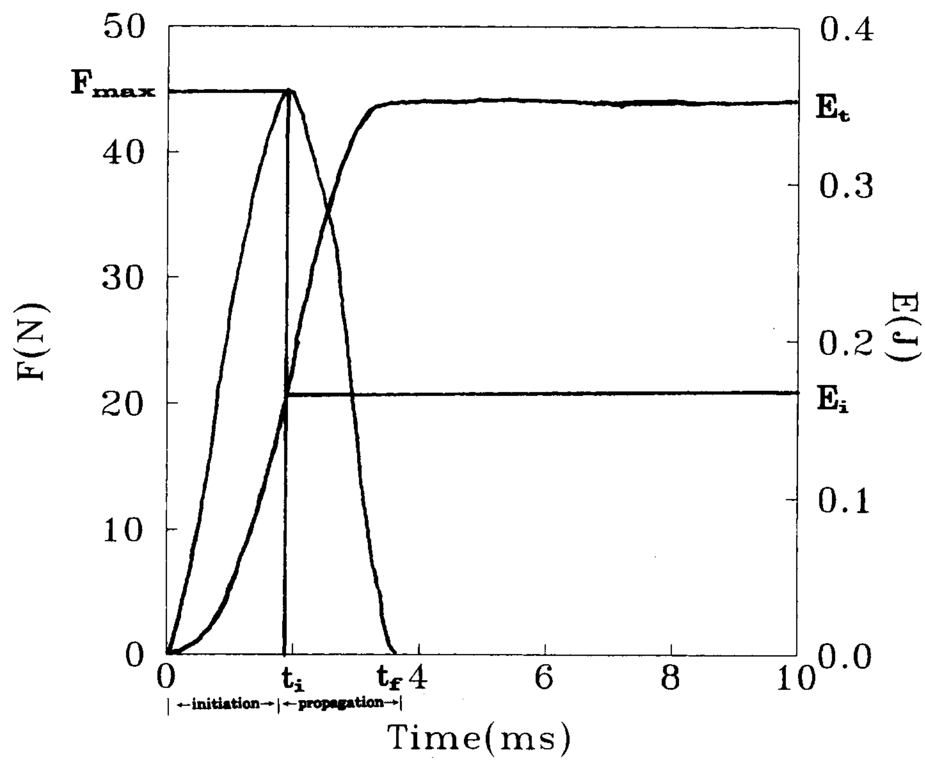


Figure 1 Load–time and energy–time curves of PEK-C at RT.

nucleus at the elastic–plastic interface, and (c) rapid propagation of the nucleus.

Temperature Dependence of the Maximum Load

The effect of temperature on the maximum load (F_{max}) is shown in Figure 2. In the temperature range tested, the maximum load first increases with increasing temperature and then decreases with increasing temperature. At a certain temperature, there is a sharp change in the maximum load; it represents the transition from fracture to general yielding. Above this temperature, general yielding occurs before fracture.

The observations made by an unaided eye show that a disk-shaped region (fracture nucleus) is nucleated at a small distance from the notch root, grows slowly, and suddenly propagates to lead to a final fracture. The load at which the fracture nucleus initiates is slightly smaller than the maximum load.

Temperature Dependence of the Maximum Stress

The maximum stress of a notched sample is given by

$$\sigma_{max} = 3F_{max}\, S/2B(D - a) \qquad (1)$$

where S is the distance between the supports; D, the thickness of the sample; B, the width of the sample; a, the crack length; F_{max}, the maximum load; and σ_{max}, the maximum stress.

For PEK-C, the temperature dependence of σ_{max} is shown in Figure 3, which can be divided into two regions: In region I, σ_{max} increases with increasing temperature, but in region II, σ_{max} decreases with increasing temperature. Next, region I is considered. The yield stress is higher than the crazing stress,

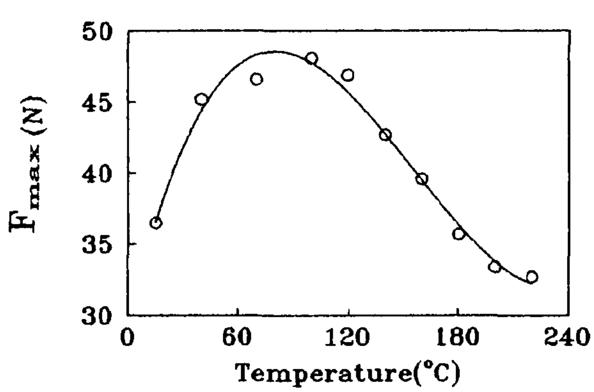


Figure 2 Temperature dependence of the maximum load of PEK-C.

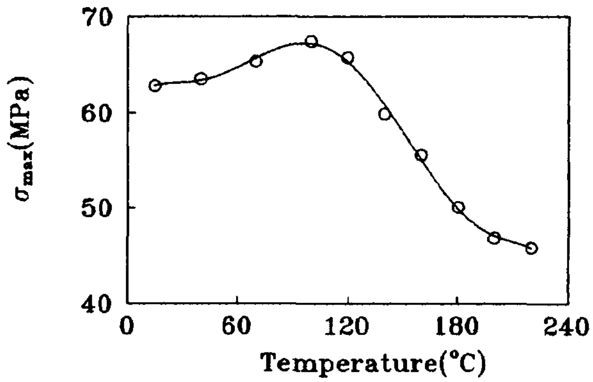


Figure 3 Temperature dependence of the maximum stress of PEK-C.

and then crazes initiate nearly in an elastic state. The plastic constraint is not required for craze initiation. The transition from region I to region II occurs at a temperature where the yield stress becomes equal to the crazing stress. For PEK-C, at relatively high temperatures, the internal craze away from the notch root, which is nucleated by the stress, increases due to the plastic constraint of the shear bands and becomes the fracture origin, and at low temperatures, the surface crazes associated with or without slip bands at the notch root become a direct precusor of the fracture. At any rate, how crazes initiate may determine the characteristic temperature region.

Temperature Dependence of Initiation Energy, Propagation Energy, Total Fracture Energy, and Ductility Index

As shown in Figure 1, the load–time history can be divided into two distinct regions—a region of fracture initiation and a region of fracture propagation. In the initiation region, as the load increases, elastic strain energy is accumulated in the specimen which it acquires on contact with the striking head of the pendulum. In this region, no gross failure takes place, but a failure mechanism on a microscale does. When a critical load is reached at the end of the initiation phase, the specimen may fail, either by a tensile or by a shear failure depending on the relative values of the tensile and interlaminar shear strengths. At this point, the fracture propagates either in a catastrophic "brittle" manner or in a progressive manner, continuing to absorb energy at smaller loads. The total impact energy, E_t, as recorded on the energy–time curve on the oscilloscope, is, therefore, the sum of the initiation energy, E_i, and the propagation energy, E_p. Since a high

Table I e_t, e_i, e_p, t_f, t_i, and t_p **of PEK-C as a Function of Temperature**

T (°C)	e_t (kJ/m²)	e_i (kJ/m²)	e_p (kJ/m²)	t_f (ms)	t_i (ms)	t_p^* (ms)
15	17.50	8.75	8.75	3.60	1.95	1.65
40	20.63	11.25	9.38	3.65	2.18	1.47
70	21.88	13.13	8.75	3.63	2.00	1.63
100	23.13	13.13	10.00	3.66	2.14	1.52
120	22.50	13.13	9.37	3.66	2.09	1.57
140	20.00	11.88	8.12	3.58	2.08	1.50
160	18.75	10.00	8.75	3.54	1.98	1.56
180	16.88	9.38	7.50	3.54	2.04	1.50
200	15.63	10.00	5.63	3.53	2.08	1.45
220	14.38	8.13	6.25	3.55	2.03	1.52

[a] $t_p = t_f - t_i$.

strength brittle material, which has a large initiation energy but a small propagation energy, and a low strength ductile material, which has a small initiation energy but a large propagation energy, may have the same total impact energy, knowing that the value of E_t alone is not sufficient to properly interpret the fracture behavior of the material. The values of the total impact energy, E_t, and initiation energy, E_i, can be divided by the cross-sectional area of each specimen in order to obtain their normalized values. Thus,

$$e_t = E_t/B(D - a) \quad (2)$$

$$e_i = E_i/B(D - a) \quad (3)$$

The propagation energy per unit area, e_p, is given by

$$e_p = E_p/B(D - a) = E_t/B(D - a) - E_i/B(D - a) \quad (4)$$

or

$$e_p = e_t - e_i \quad (5)$$

Corresponding to the total fracture energy, fracture initiation energy, and fracture propagation energy, the time of total fracture (t_f), fracture initiation (t_i), and fracture propagation (t_p) can also be obtained (as shown in Fig. 1). The values of e_t (impact strength), e_i, e_p, t_f, t_i, and t_p of PEK-C at different temperatures are listed in Table I.

Another proposed characteristic of the material that can be obtained from these relationships is the ductility index (DI),[6] which is a dimensionless parameter and is defined as the ratio of propagation energy to the initiation energy. Thus,

$$DI = E_p/E_i \quad (6)$$

$$DI = e_p/e_i \quad (7)$$

For PEK-C, the temperature dependence of E_p,

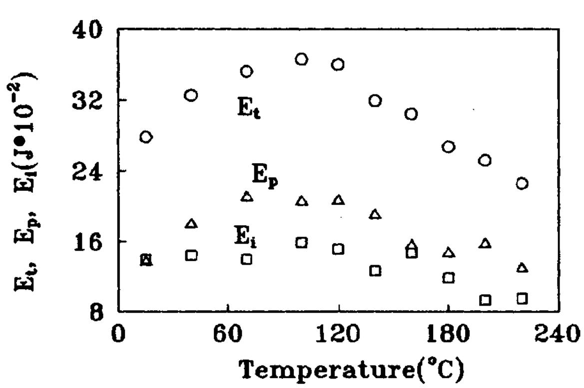


Figure 4 Temperature dependence of the total impact energy, initiation energy, and propagation energy of PEK-C.

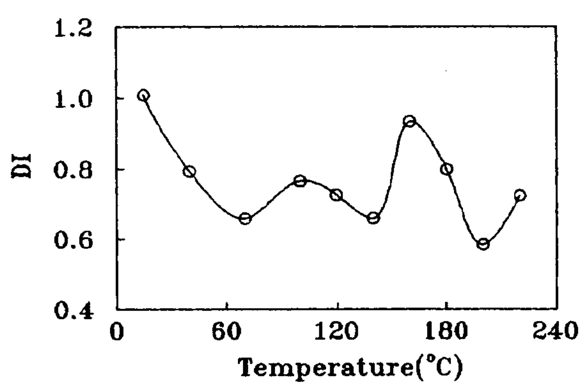


Figure 5 Temperature dependence of DI of PEK-C.

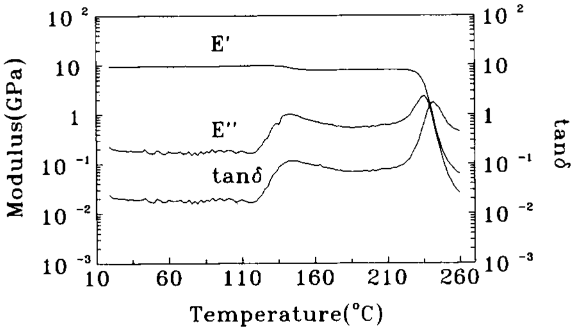


Figure 6 Elastic (E'), loss (E'') moduli, and tan δ of PEK-C as a function of temperature at 3.5 Hz.

E_i, E_t, and *DI* is shown in Figures 4 and 5, respectively. From Figure 5, we can see that the temperature dependence of *DI* has two peaks, which is similar to the tan δ loss (Fig. 6). This suggests that the tan δ loss has some effect on the ductility although the peak temperatures in the *DI*–temperature curve are lower than those in the tan δ–temperature curve. The reason for this is that the impact tests and the viscoelastic experiments were done under different conditions. The impact tests were done with standard impact testing equipment, and the relaxation tests were typically done with standard dynamic mechanical experiments. The different testing configurations typically involve different specimen sizes, different loading conditions, and different loading rates. For a comparison of *DI* and tan δ to be valid, both quantities have to be calculated at the same effective frequency. The impact event involves a wide range of frequencies. The most important frequencies in a Charpy impact test is about 1000 Hz; the tan δ curve was obtained at 3.5 Hz, a factor of about 100 less than the highest frequencies in the impact event. So, the tan δ curve would have to shift to higher temperature to match the *DI* curve.

CONCLUSIONS

The Charpy impact fracture behavior of notched specimens of phenolphthalein poly(ether ketone) (PEK-C) has been studied over a range of temperature using a JJ-20 Model instrumented impact tester. For PEK-C, there exist two temperature regions which distinguish the fracture mechanism; the brittle fracture was preferentially governed by slip or shear bands at relatively high temperatures, but by crazes at low temperatures. The temperature dependence of the ductility index (*DI*) shows similar peaks to the tan δ loss.

REFERENCES

1. A. J. Hemingway, A. D. Channell, and E. Q. Clutton, *Plast. Rubber Compos. Process. Appl.*, **17**, 147 (1992).
2. Y. Nakamura, M. Yamaguchi, and M. Okubo, *Polym. Eng. Sci.*, **33**, 279 (1993).
3. A. J. Kinloch, G. A. Kodokian, and M. B. Jamarani, *J. Mater. Sci.*, **22**, 4111 (1987).
4. N. Isikawa, I. Narisawa, and H. Ogawa, *J. Polym. Sci. A-2*, **15**, 1971 (1977).
5. M. Kitagawa, *J. Mater. Sci.*, **17**, 2514 (1982).
6. P. Beaumont, P. Riewald, and C. Zweben, *Methods for Improving the Impact Resistance of Composites Materials*, ASTM STP 568, 1974, p. 134.

Received July 15, 1994
Accepted November 12, 1994