JOURNAL OF MATERIALS SCIENCE 36 (2001) 4637–4641

Creep crack growth in a short glass fibres reinforced polypropylene composite

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In this paper the creep crack propagation in a short glass fibre reinforced polypropylene composite has been investigated at various temperatures in the range from 32 to 60°C. Creep crack speed (da/dt) resulted initially decreasing till a minimum value, and then gradually increasing up to instability and fracture. Both initial and minimum crack speed values were found to strongly increase as test temperature increased. Moreover, isothermal curves of the applied stress intensity factor K_{appl} as a function of the crack speed (da/dt) were obtained at various temperatures. Portions of these curves in the stable crack acceleration region were hence shifted along the da/dt, axis according to a time-temperature reduction scheme, thus allowing the construction of a creep crack propagation master curve. The shift factor values, a_T, for the creep crack propagation master curve appeared to be higher than those obtained, in the same temperature range, from dynamic mechanical measurements in a linear viscoelastic regime.

1. Introduction

Because of its characteristics of low density, good processability and environmental resistance, isotactic polyropylene (iPP) is considered one of the best thermoplastic candidates for many industrial applications, like appliances [1], geotextiles and geomembranes [2], components for the automotive market [3] cables [4], fibres [5], etc... In order to improve its performances, iPP is frequently added with fillers and/or reinforcements [6]. Short glass fibres are generally incorporated within iPP to enhance the mechanical properties, such as stiffness and fracture resistance, without substantially impairing the good processability of the material. Injection-moulded structural components having good mechanical performances can be thus obtained. In particular, short glass fibres embedded within iPP aid to limit the propensity of this material to deform under creep conditions, and then contribute also to prevent creep failure in load-bearing applications.

Creep crack propagation phenomena in thermoplastic polymers have been prevalently studied for materials intended for pressure pipes, like polyethylene (PE) [7–13] and unplasticized polyvinyl chloride (uPVC) [14], and for adhesive joints [15]. The present investigation has been conducted in the framework of a comprehensive experimental study regarding the fatigue/creep crack propagation in short glass fibres reinforced iPP composites [16–18]. In the previously published papers the effect of the presence of glass fibres on the crack growth process have been investigated for various fiber weight fractions (0, 10, 20 and 30%). In particular the fatigue crack propagation resulted to markedly decrease as the fibre content increased [17, 18]. Moreover, the influence of the fibre orientation on the crack propagation rate was also considered [18]. In particular, the fatigue crack propagation rate of either neat and fiber reinforced iPP resulted tremendously dependent on the crack propagation direction, with lower values for crack propagating transversely to the melt flow direction in the molded plaques. In these studies, the analysis of data obtained at various frequencies and applied mean loads suggested that crack propagation under cyclic loading conditions could mostly due to viscoelastic creep processes at the crack tip, especially at low frequencies.

In the present work crack growth under pure creep conditions in iPP reinforced with 10 wt% of short glass fibres was experimentally investigated at various temperatures between 32°C and 60°C.

2. Experimental

The material studied was iPP reinforced with 10 wt% of short glass fibres and it was supplied by Montell Polyolefins SpA (Ferrara, Italy) in the form of injection moulded square plaques with dimensions 127 × 127 × 2.7 mm. The matrix was a commercial grade iPP with a melt flow index of 3.5 dg/l, whereas the glass fibres were Owens Corning (R34B) E-type with a 14 μm diameter and an initial average length of 4.5 mm. In order to improve fiber/matrix adhesion the short E-glass

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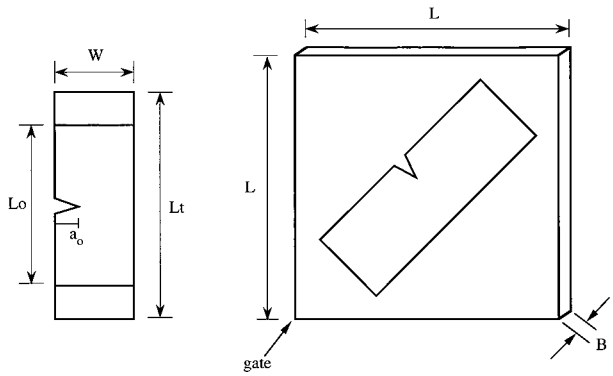


Figure 1 Shape and size of SENT specimens and position from which test pieces were cut from polypropylene/glass fibres injection moulded plaques. Dimensions are: $L = 127$ mm, $B = 2.7$ mm, $L_t = 120$ mm, $L_0 = 62$ mm, $W = 27$ mm, $a_0 = 3$ mm.

fibres were treated with a polypropylene compatible coating (Hercoprime HG 201). After compounding and injection-moulding the manufacturer evaluated an average fibre length of about 0.5–0.7 mm.

Single edge notched tension (SENT) specimens, with dimensions as reported in Fig. 1, were cut from the injection-moulded plaques always in same position (see Fig. 1). An initial sharp notch of about 3 mm in length was introduced in the specimens by means of a razor blade mounted on a saw cutter. Creep crack growth at different temperatures in the range between 32°C and 60°C was obtained by applying to the specimens a constant load of 1200 N by an Instron machine, mod. 4502, equipped with a thermostatic chamber. The crack length during fracture propagation was evaluated by using a video-camera, a video-recorder, and an image analyser system. Dynamic mechanical tests were performed in bending mode by a DMTA (Dynamic Mechanical Thermal Analyser), model MKII by Polymer Laboratories Ltd, on rectangular $60 \times 10 \times 2.7$ mm samples. Temperature was scanned from −50°C to 130°C at a heating rate of 0.5°C/min and 7 different test frequencies, between 0.1 Hz and 100 Hz, were selected.

3. Results and discussion

The results of creep crack growth, $a(t)$, reported in Fig. 2 for the various temperatures explored, clearly show that temperature accelerates the creep fracture

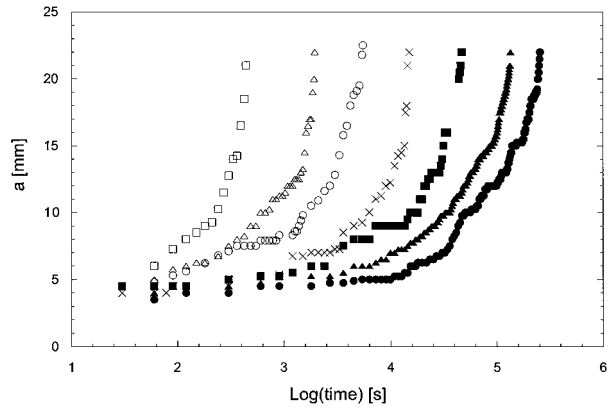


Figure 2 Crack length vs. time for creep tests performed at various temperatures. Symbols refer to: (●) 32°C, (▲) 35°C, (■) 40°C, (×) 45°C, (○) 50°C, (△) 55°C, and (□) 60°C.

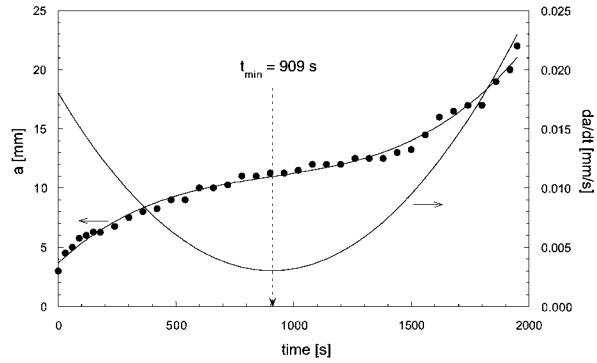


Figure 3 Crack length and crack speed vs. time for a creep test performed at 55°C.

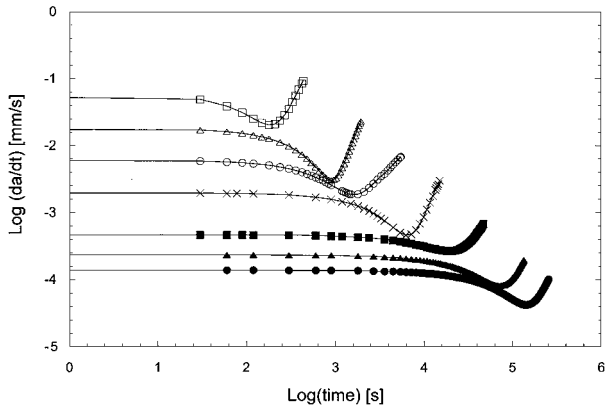
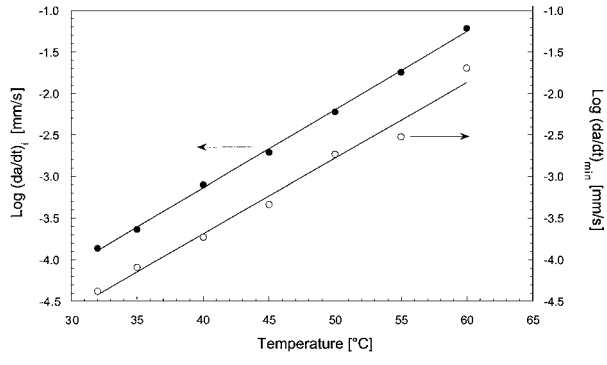
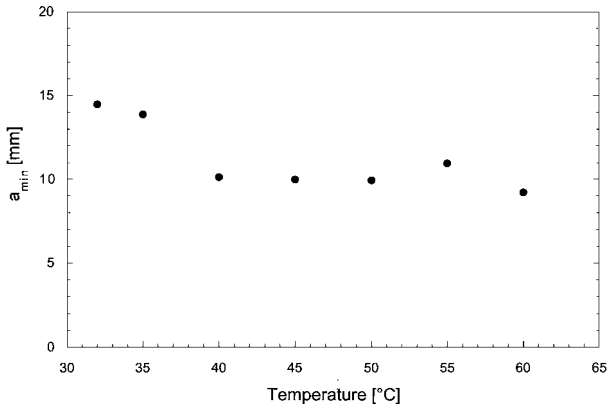


Figure 4 Crack speed vs. time for creep tests performed at various temperatures. Symbols as in Fig. 2.



(a)



(b)

Figure 5 (a) Initial (●) and minimum (○) values of creep crack speed as a function of temperature. (b) Crack length corresponding to the minimum value of the crack speed as a function of temperature.

process. Complete fracture of a test-piece is reached in some hundred seconds at 60°C, while some hundred thousands seconds are needed at 32°C. By plotting $a(t)$ using a linear time scale it can be observed that, at each temperature, the fracture propagation is typically constituted by a first stage where crack decelerates (i.e. the slope of $a(t)$ is decreasing), followed by a stage of stable crack acceleration (i.e. the slope of $a(t)$ is increasing) which ends with fracture instability. This phenomenology is clearly evidenced in Fig. 3 for a test performed at 55°C. Some discontinuous crack growth could also be appreciated. A similar behaviour

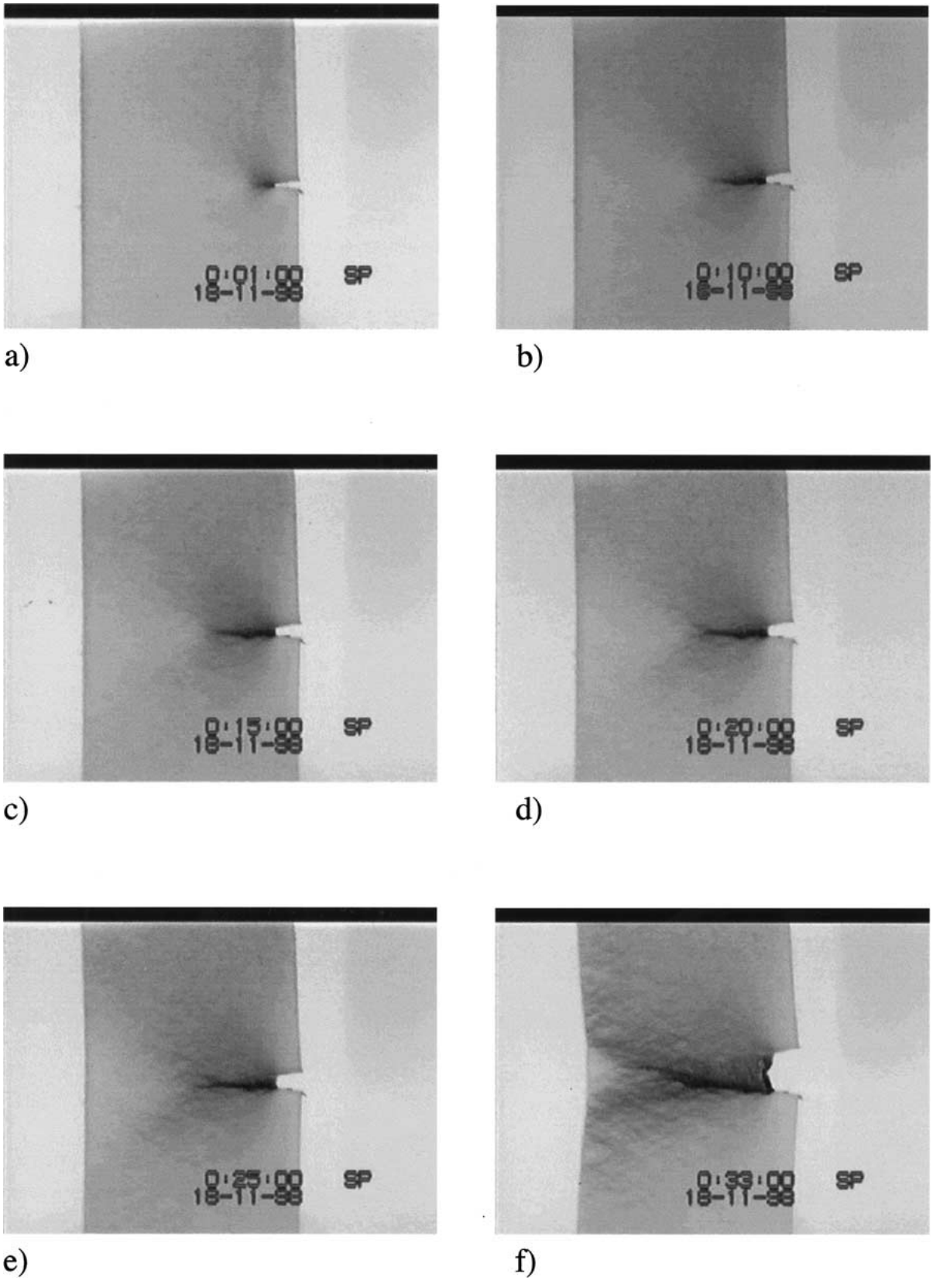


Figure 6 Videocamera frames sequence of a crack creep propagation test performed at 55°C. Frames are taken after (a) 60 s, (b) 600 s, (c) 900 s, (d) 1200 s, (e) 1500 s, and (f) 1980 s.

has been already observed for the fatigue crack propagation of the same materials [16–18] and also for similar composites [6, 19].

The overall fracture kinetics is brought into evidence by plotting the crack speed, da/dt, as a function of time. It is worth noting that da/dt has been evaluated by interpolating the crack length versus time curves with a best fitting 3rd order polynomial function. Plots of da/dt vs time are reported in Fig. 4 for the various temperatures investigated. The similarity of the fracture kinetics at different temperatures clearly appears, the effect of increasing temperature consisting substantially in a shortening of the whole fracture process. The crack speed remains fairly constant at an initial value $(da/dt)_{\mathrm{i}}$, then it decreases to a minimum value, $(da/dt)_{\min}$, and thereafter increases up to instability.

As shown in Fig. 5a, both the initial and minimum crack speeds exponentially increase as temperature rises, thus evidencing the thermally activated nature of the fracture process. On the other hand, the crack length in correspondence of the minimum speed is almost constant with temperature, as evidenced in Fig. 5b.

A creep experiment performed at $T = 55^{\circ}\mathrm{C}$ is shown in Fig. 6 at various stages of the crack propagation process. Images of the specimen surfaces, recorded during crack propagation, provide indication that crack deceleration could be associated to a development of a damaged zone at the crack tip. When this zone is fully developed crack starts to accelerate. Extension and shape of the damaged zone do not seem to be affected by temperature. Some micro branching of the crack itself can also be observed. For as concern the experimental difficulties of the crack length measurement due to damage and branching, we based our observations on the fact that a "prevalent crack" could always be detected during creep crack propagation experiments and on the assumption that the failure behaviour of the material is mainly governed by this "prevalent crack", being all other damage processes (including branching) of secondary importance.

The kinetics of the crack growth reflects also in the behaviour of the crack speed as a function of the applied stress intensity factor, K_{appl}, as reported in Fig. 7. As shown in Fig. 8, the portion of the curves related to the stable crack acceleration range can be satisfactory interpolated by a power law equation [20] in the following form:

$$K_{\mathrm{appl}} = A\left(\frac{da}{dt}\right)^{n} \tag{1}$$

It is worth pointing out that the exponent n remains quite constant with temperature being in the range 0.9–1.4, whereas the term A decreases as temperature increases.

It has been shown [21–27] that, due to the viscoelastic nature of the crack propagation process, a time-temperature reduction approach holds for $K_{\mathrm{c}}(da/dt)$ being the crack speed a variable related to time. On this basis the curves of Fig. 8 were horizontally shifted to best superposition as shown in Fig. 9, providing the values of a shift factor a_{T}, referred to a temperature of 32°C, reported in Fig. 10. No vertical shift was adopted. The following shift factors were also determined:

$$b_{\mathrm{T}}(T) = \frac{(da/dt)_{\mathrm{i}}(T)}{(da/dt)_{\mathrm{i}}(T_0)}$$

$$c_{\mathrm{T}}(T) = \frac{(da/dt)_{\min}(T)}{(da/dt)_{\min}(T_0)}$$

As reported in Fig. 10, the values of $b_{\mathrm{T}}(T)$ and $c_{\mathrm{T}}(T)$, also referred to 32°C, are very close to those of $a_{\mathrm{T}}(T)$, showing that the overall fracture kinetics scales with temperature according to the same shift factor. On a theoretical basis it has been proposed that a_{T} should

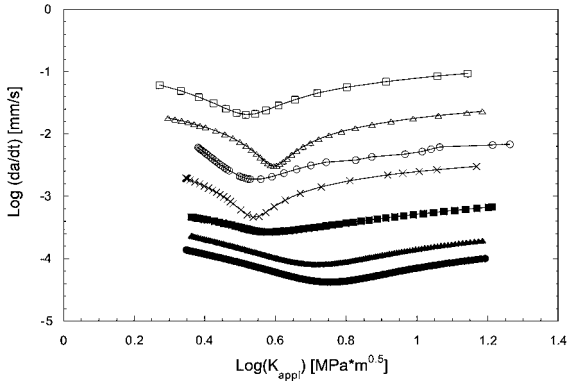


Figure 7 Crack speed vs. the applied stress intensity factor for creep tests performed at various temperatures. Symbols as in Fig. 2.

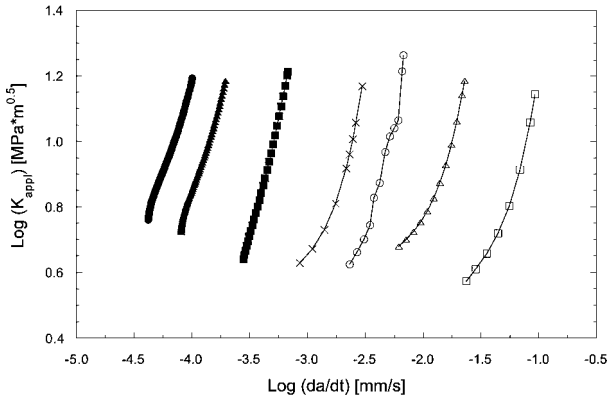


Figure 8 Stress intensity factor vs. crack speed in the stable acceleration range for creep tests performed at various temperatures. Symbols as in Fig. 2.

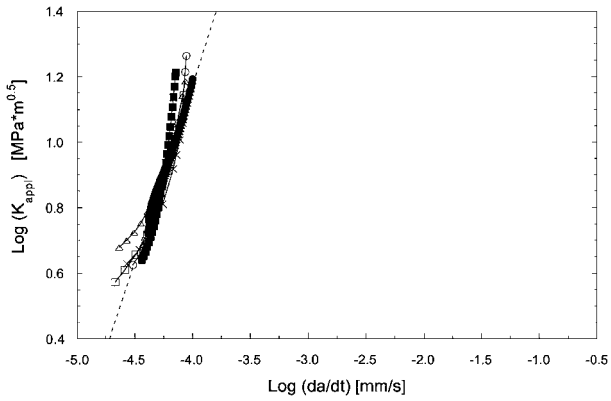


Figure 9 Creep crack propagation master curve referred to $T_0 = 32^{\circ}\mathrm{C}$. Symbols as in Fig. 2.

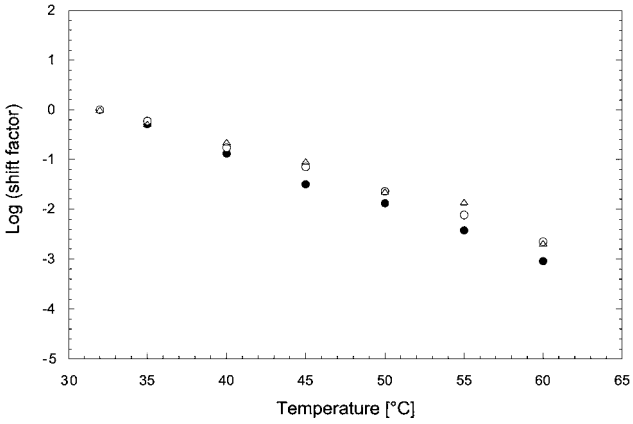


Figure 10 Shift factors as a function of temperature. Symbols refer to: (●) a_T, (○) b_T, and (△) c_T.

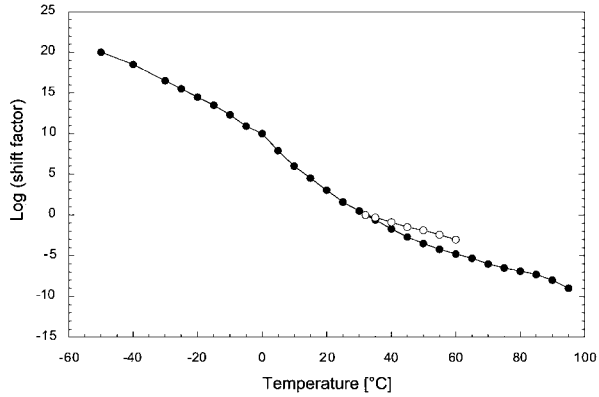


Figure 11 Temperature dependence of shift factor a_T for the creep crack propagation (○) and dynamic mechanical storage modulus master curves (●), respectively.

coincide with the shift factor linked to the creep compliance [20, 27]. The time-temperature superposition principle, applied to data of the dynamic storage modulus $E'(\nu)$ obtained in linear viscoelastic regime at different temperatures, provided the shift factor reported against temperature in Fig. 11, where the crack speed-temperature shift factor, a_T, is also shown. It appears that this last has higher values than those concerning the storage modulus in the linear viscoelastic range. This discrepancy can be attributed to the fact that the two shift factors are associated to markedly different strain levels: lower than 0.07% for dynamic tests and higher than 1.5% (overall specimen deformation) in the fracture tests. In fact, this behaviour is in agreement with the observations reported by Mariani *et al.* [26] who found that the time-temperature shift factor of a viscoelastic function, such as the stress-relaxation modulus, increases by increasing the strain level.

4. Conclusions

The present results show that in a polypropylene composite reinforced with short glass fibres under creep conditions crack speed is initially decreasing to a minimum value and then steadily increasing up to fracture, similarly to the case of fatigue crack propagation. Crack speed is strongly influenced by the test temperature, being the overall fracture kinectis exponentially accelerated by temperature. Isothermal curves of applied stress intensity factor K_{appl} as a function of the crack speed (da/dt) were obtained at various temperatures. Part of these curves, regarding the stable crack acceleration region, were shifted along da/dt axis according to a time-temperature reduction scheme thus allowing the construction of a creep crack propagation master curve. The shift factor values, a_T, so evaluated appear to be higher than those obtained, in the same temperature range, from dynamic mechanical measurements in linear viscoelastic regime.

Acknowledgements

This work was partially supported by Consiglio Nazionale delle Ricerche, CNR, Rome. Montell Polyolefins SpA (now Basell), Ferrara, Italy, is kindly acknowledged for the provision of the materials. The authors would like to thank Dr. A. Dimauro for his contribution to the experimental work.

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Received 21 September 2000
and accepted 21 May 2001