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Effect of frequency and orientation on fatigue crack propagation in polyamide-12

R. Boukhili, F. X. de Charentenay and T. Vu-Khanh

Fatigue crack propagation has been studied in injection moulded polyamide-12, and the influence of test frequency, material orientation, load waveform and specimen type on fatigue crack propagation and fracture mechanisms are reported. Specimen orientation and thickness effects were found to depend on test frequency. At the same time the effect of test frequency was found to vary according to the frequency range (high or low) and type of specimen (single-edge notched or compact tension). The results are interpreted by considering the contribution of the highly oriented skin regions. The importance of large-scale hysteretic heating and material orientation in conjunction with the high strain rate at the vicinity of the crack tip are emphasized.

Key words: fatigue; fatigue crack propagation; frequency effect; material orientation; far-field stress; hysteretic heating; polyamide-12

Most investigations into the fatigue crack propagation (FCP) behaviour of polymeric solids to date have been concerned with the effects of internal and external variables on FCP rate. (These topics were extensively discussed in a book by Hertzberg and Manson which covers much of the literature up to 1980.[1])

The internal variable which most affects the mechanical properties (such as FCP) of polymers is the molecular weight,[2-4] and a viscoelastic model was proposed recently to describe this effect.[5]

The highly viscoelastic nature of polymers makes the test frequency one of the most important experimental variables. It was found to have a strong effect on the FCP in several polymers;[6,7] in general the FCP rate decreased as the frequency increased. Hertzberg *et al*[7] showed that the sensitivity of FCP rate to frequency is related to the β transition of the viscoelastic damping of polymers, and explained the decrease in FCP rate with increasing frequency by the blunting of the crack tip due to localized hysteretic heating. However, in the highly toughened nylon 66, it was found that the FCP rate increases with increasing frequency. This inverse effect of frequency was explained by the hysteretic heating on a large scale of the ligament.[8]

An investigation of the frequency effect in another engineering thermoplastic, polyamide-12 (PA12), showed that FCP rate can either increase or decrease depending on the frequency range, and much attention was given to the role of highly oriented skin regions.[9] It should be noted that the skin layer of injection mouldings can affect both fatigue behaviour, as shown by Crawford *et al* who reported that the skin layer inhibits the fatigue crack initiation of an acetal copolymer,[10] and monotonic loading, as shown by Jang *et al* who reported that it is difficult to initiate crazes in the highly oriented skin of an injection moulded polypropylene.[11]

The work described in this paper extends the investigation reported in Reference 9 on injection moulded PA12, which shows high FCP resistance and large-scale heating. The effects of specimen orientation and thickness are discussed as functions of the test frequency. Load waveform and specimen geometry are investigated to isolate the effect of both strain rate and localized or generalized hysteretic heating. To complete this study, the effect of the far-field stress (gross stress) is examined using specimens with different crack lengths.

Experimental details

The AMVO grade of polyamide-12 from ATO CHEM was used in this study. Standard single-edge notched (SEN) and compact tension (CT) specimens were cut from injection moulded plates with the notch, made by a razor blade, either parallel or perpendicular to the flow direction, as shown in Fig. 1.

FCP tests were run at constant load range with $R = P_{min}/P_{max} = 0.1$ on a closed-loop, servohydraulic Instron 1341 testing machine. Crack length was monitored using a travelling microscope. Log–log plots of crack growth rate (da/dN) *vs* stress intensity factor range (ΔK) were determined making use of the following relationships:

For SEN specimens

$$\Delta K = (Y \Delta P \sqrt{a})/(BW)$$

where $\Delta P = P_{max} - P_{min}$ is the load range, B is the specimen thickness, W is the specimen width, a is the crack length and Y is a geometric factor given by:[12]

$$Y = 1.99 - 0.41\,(a/W) + 18.70(a/W)^2 - 38.48(a/W)^3 + 53.85(a/W)^4$$

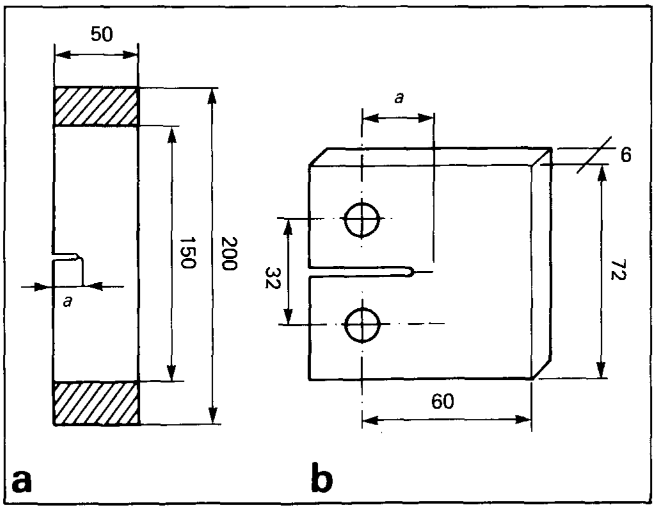

Fig. 1 Geometry and dimensions (in mm) of: (a) single edge-notched and (b) compact tension specimens

For CT specimens

$$\Delta K = (Y\Delta P)/(BW^{1/2})$$

where Y is given by:[12]

$$Y = 29.6(a/W)^{1/2} - 185.5(a/W)^{3/2} + 655.7(a/W)^{5/2} - 1017(a/W)^{7/2} + 638.9(a/W)^{9/2}$$

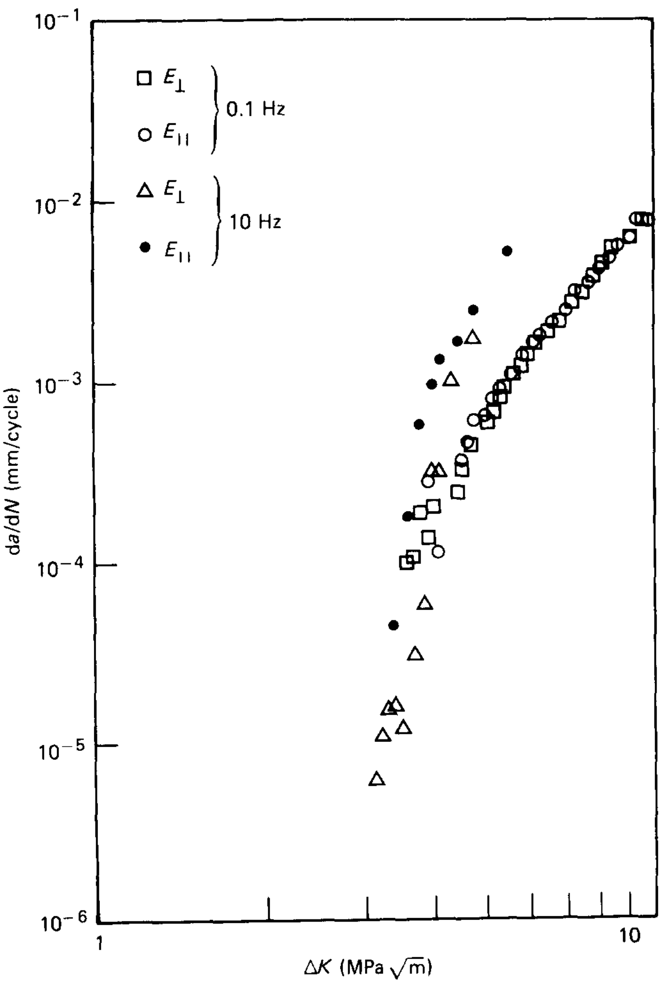

Fig. 2 Effect of specimen orientation (crack parallel ($E_{\parallel}$) or perpendicular ($E_{\perp}$) to the flow direction) on FCP rate in PA12 at low and high test frequencies

Fracture surfaces were examined using a Cambridge model Stereoscan 100 scanning electron microscope.

Unless otherwise specified, all fatigue tests were performed on SEN specimens 4 mm thick with the crack perpendicular to the flow direction and with a sinusoidal load waveform.

Results

Effect of test parameters on FCP rate

The results show that for both low and high frequencies (0.1 Hz and 10 Hz) specimen orientation and thickness do not have any effect on FCP rates and fracture surfaces (see Figs 2 and 3). At the intermediate frequencies of 1 and 5 Hz, however, a strong effect of specimen orientation and thickness is observed (see Figs 4 and 5). FCP rates are lower in the thinner specimen or when the crack is perpendicular to the flow direction. This effect is more important at 1 Hz.

When load waveform was changed keeping the frequency at 1 Hz, the FCP rate was found to decrease when passing from a triangular to a sinusoidal and finally to a square waveform, as shown in Fig. 6.

During the investigation it was noted that, depending on the test frequency range (relatively low or high) and the specimen configuration (SEN or CT specimens), there could be a decrease, an increase or no change in the FCP rate. For SEN specimens in the frequency range 0.1 to 1 Hz, FCP rate decreased as the frequency increased (see Fig. 7). In the frequency range 1 to 10 Hz, FCP rate increased with increasing frequency. This effect was found to be more pro-

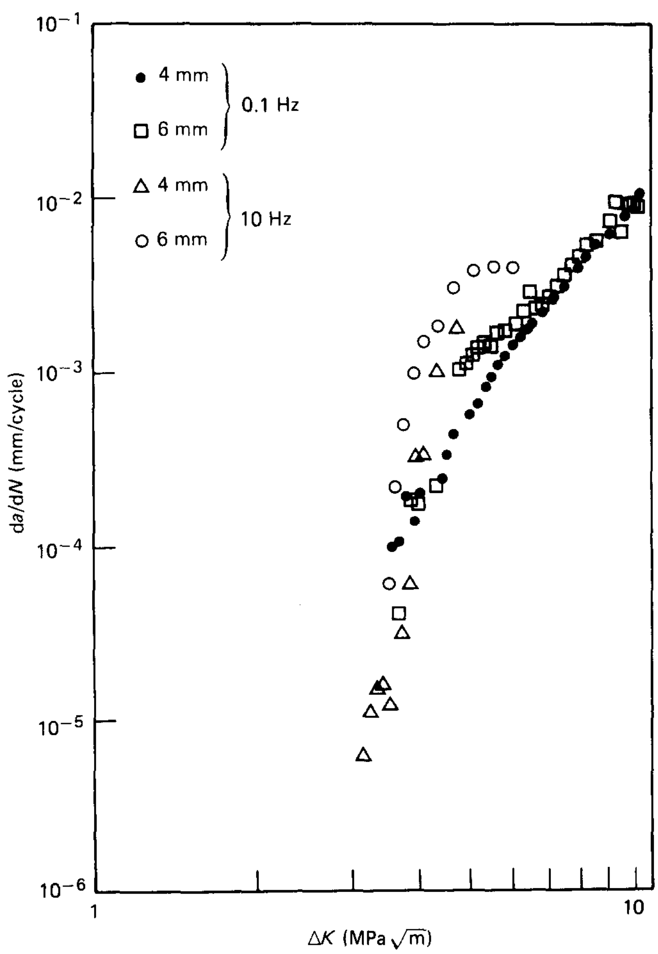

Fig. 3 Effect of specimen thickness on FCP rate of PA12 at low and high test frequencies

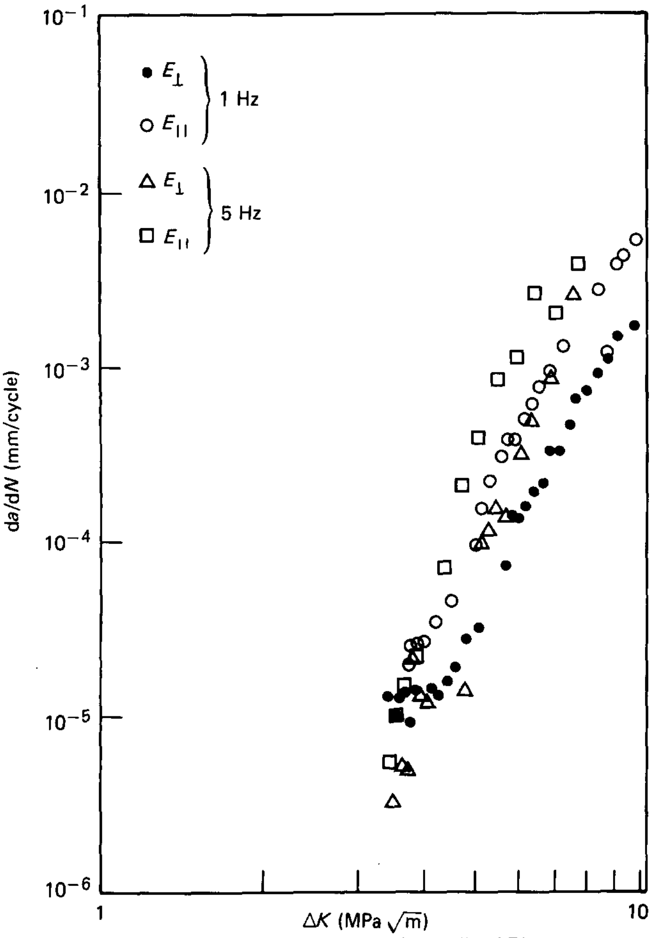

Fig. 4 Effect of specimen orientation (crack parallel ($E_\parallel$) or perpendicular ($E_\perp$) to the flow direction) on FCP rate in PA12 at intermediate test frequencies

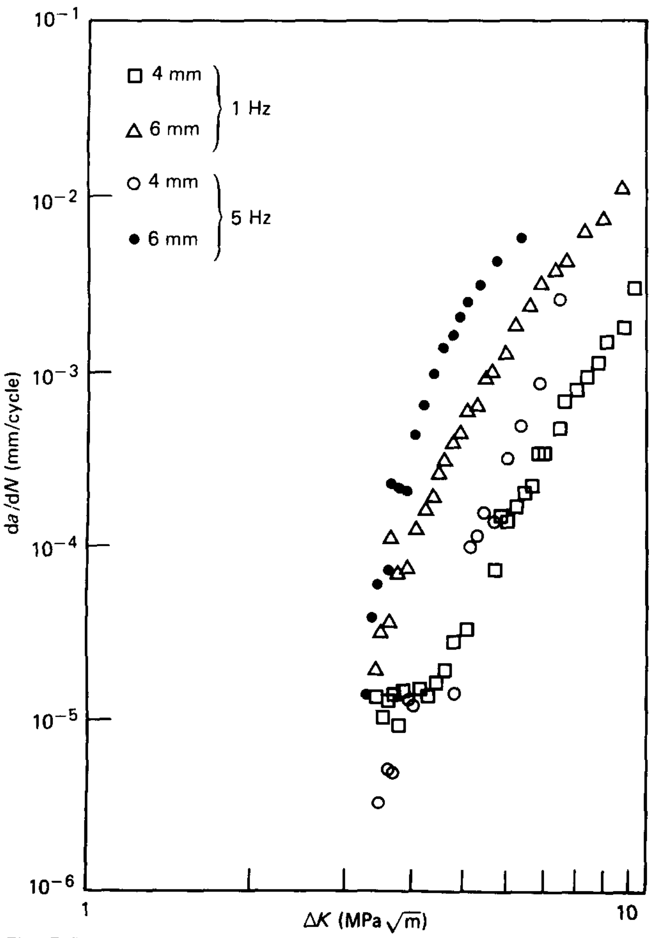

Fig. 5 Effect of specimen thickness on FCP rate in PA12 at intermediate test frequencies

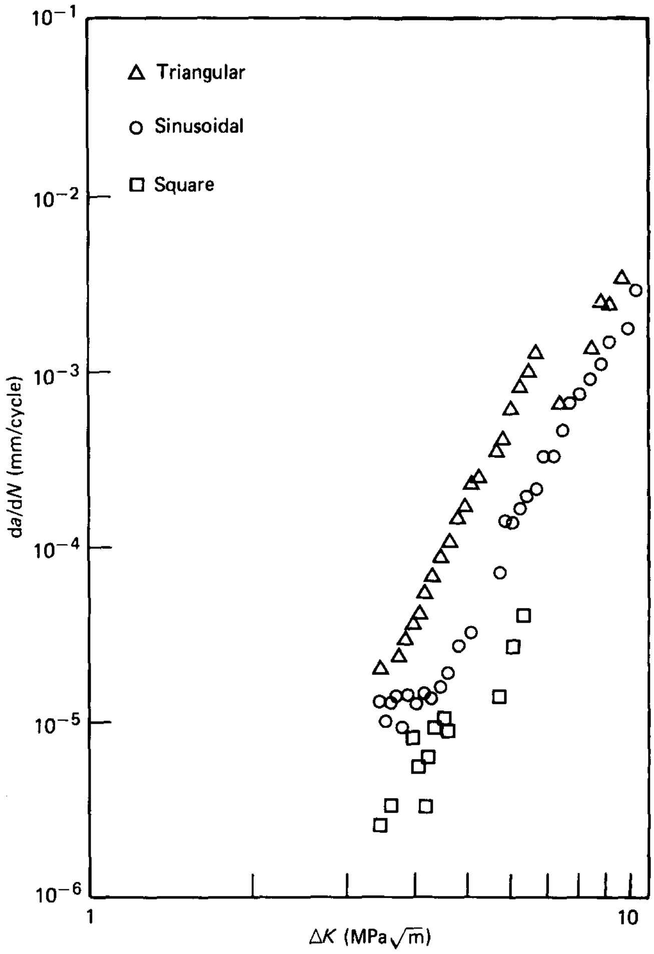

Fig. 6 Effect of load waveform on FCP rate in PA12

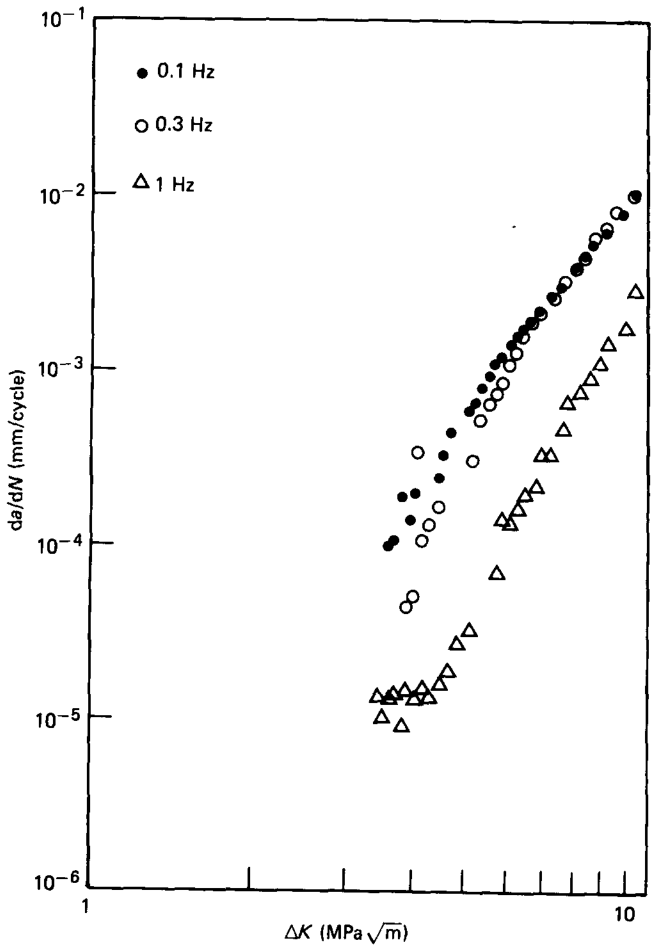

Fig. 7 Effect of test frequency (0.1–1 Hz) on the FCP rate in PA12

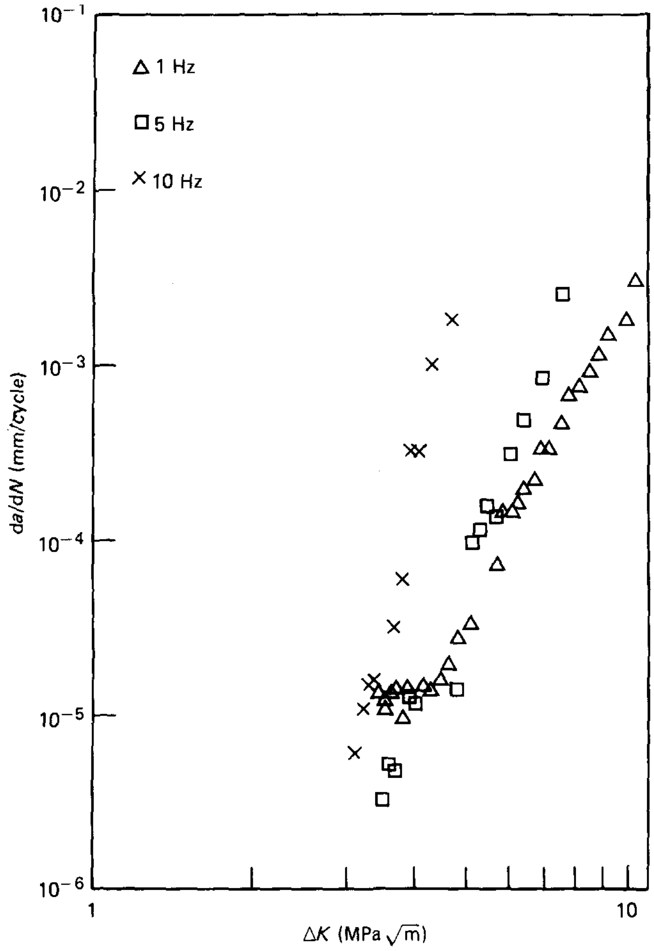

Fig. 8 Effect of test frequency (1–10 Hz) of FCP rate in PA12

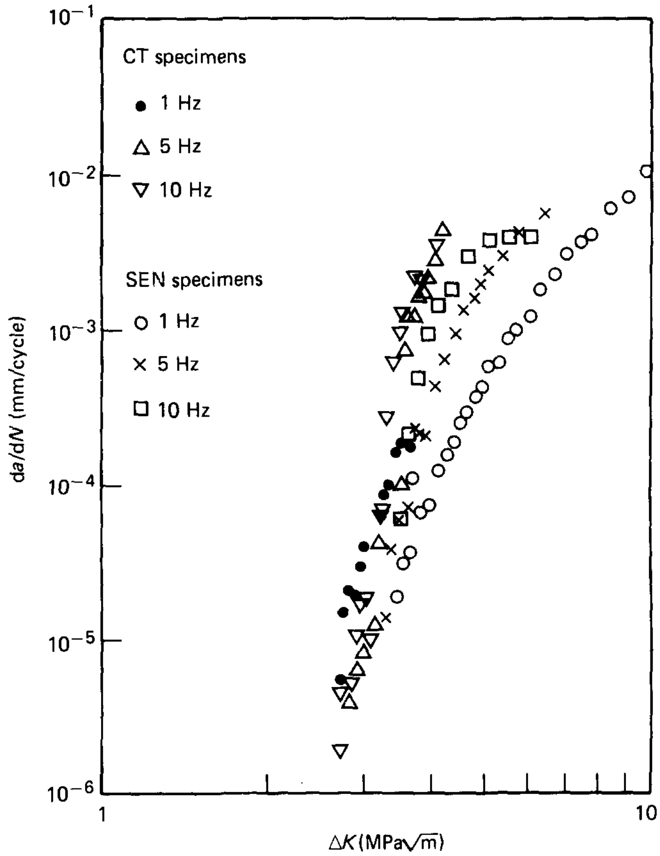

Fig. 9 Effect of specimen configuration on frequency sensitivity in PA12 from 1 to 10 Hz

nounced as ΔK increased (see Fig. 8). Fig. 9 shows that frequency (1, 5 and 10 Hz) has no effect on the FCP rate of CT specimens, and that FCP rates were higher in CT specimens than in SEN specimens.

General FCP features in polyamide-12

It has to be emphasized that during the FCP test the crack propagates at first in the core regions. This behaviour was confirmed by a test interrupted after a few millimetres of crack propagation. The crack tip is preceded by an elliptical whitened region, a consequence of voiding in plane strain.[9]

Examination of the fatigue fracture surfaces of PA12 reveals the following zones, see Fig. 10:

- zones A and B are the mechanical notch (saw and razor blade)
- zone C is a whitened region of cavitation and voiding
- zones D are ductile shear lips with a saw-tooth shape (SLST)
- zones C and D correspond to slow fatigue crack propagation
- zone E corresponds to fast fatigue crack propagation, followed by final necking.

The dimensions of regions C, D and E depend on the test parameters, as shown in Fig. 11. The SLST are larger when the crack is perpendicular to the flow direction and in thinner specimens. In addition, the width of the SLST increases when changing from a triangular to a sinusoidal and finally to a square waveform, and with increasing frequency.

When the skin of a 6mm thick SEN specimen is machined to give an SEN specimen 4 mm thick, the SLST disappear totally at 5 and 10 Hz (see Fig. 12).

Discussion

Explanation of results

Two effects can explain the crack propagation profile obtained; one is the plane strain/plane stress state and the other is the material heterogeneity induced by moulding.

It should be pointed out that, because PA12 is injection moulded, the spherulite sizes are larger in the core regions and decrease when approaching surfaces regions, giving rise

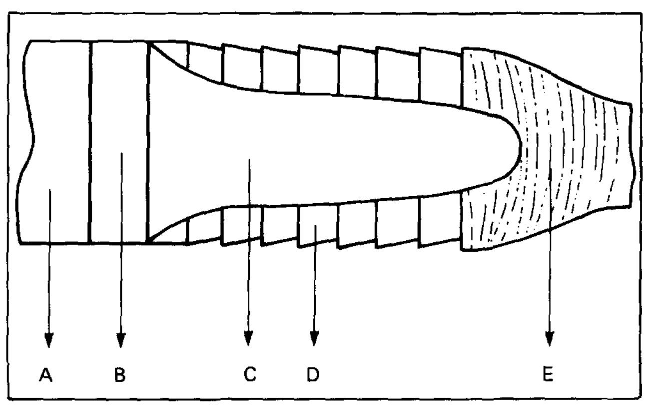

Fig. 10 Schematic representation of the general features of fracture surfaces in PA12: zones A and B are the mechanical notches (saw and razor blade); zone C is a whitened region of cavitation and voiding; zones D are ductile shear lips with a saw tooth shape; zone E corresponds to fast fatigue crack propagation

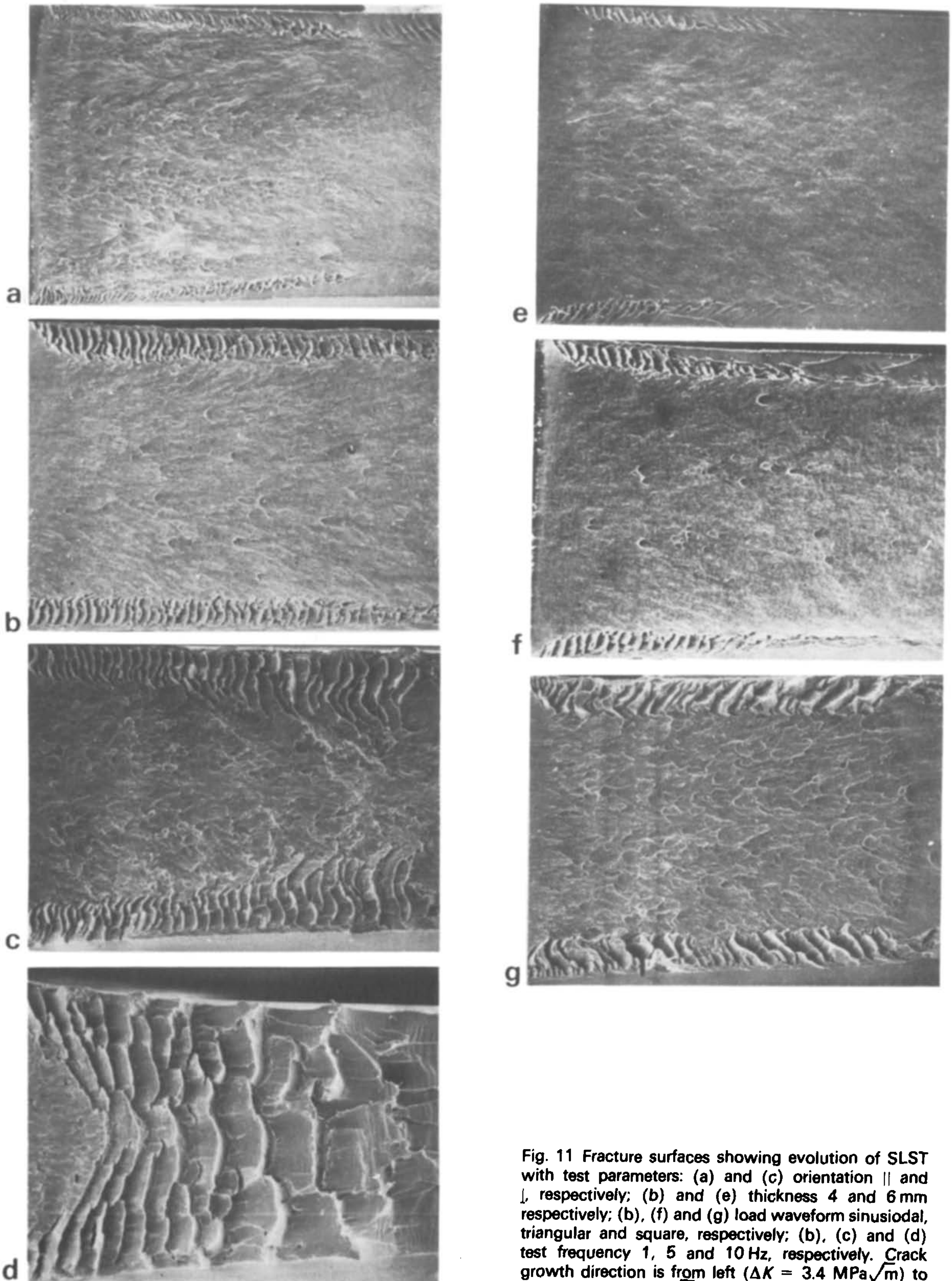

Fig. 11 Fracture surfaces showing evolution of SLST with test parameters: (a) and (c) orientation || and ⊥, respectively; (b) and (e) thickness 4 and 6 mm respectively; (b), (f) and (g) load waveform sinusiodal, triangular and square, respectively; (b), (c) and (d) test frequency 1, 5 and 10 Hz, respectively. Crack growth direction is from left (ΔK = 3.4 MPa$\sqrt{m}$) to right (ΔK = 5 MPa$\sqrt{m}$)

to an oriented structure which is more resistant to crack advance.[9] The material heterogeneity is then identified as a material orientation in the skin regions.

To explain the effect of specimen orientation and thickness for different frequencies, consider the following results and the information that is deduced from them.

- When the skin is machined the SLST disappear completely, as shown for the two frequencies 5 and 10 Hz in Fig. 12. Thus it can be concluded that the SLST result from the highly oriented skin regions.
- The highly oriented skin regions produce larger SLST when they are loaded in the flow direction (see Figs 11a and 11c).
- The SLST are larger for a square waveform than for

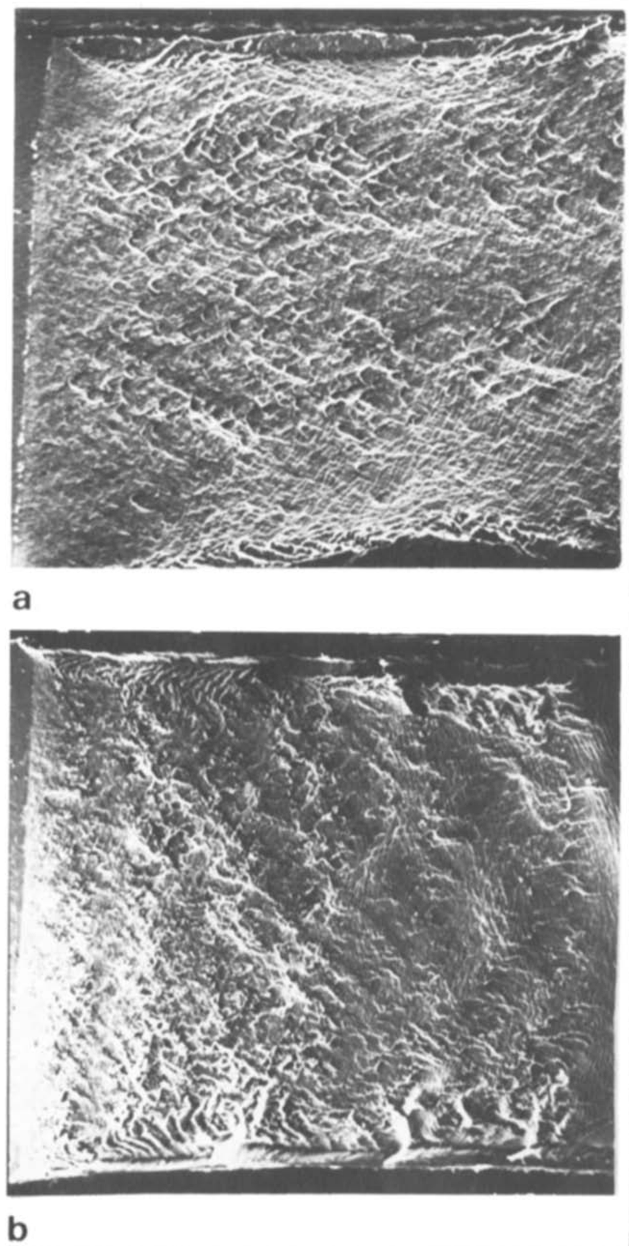

Fig. 12 Fracture surfaces of specimen with machined skin, tested at: (a) 5 Hz and (b) 10 Hz (to be compared with Figs 11c and 11d, respectively)

a triangular waveform. Thus increasing the strain rate leads to an increased contribution of the skin regions as if they had become larger. In other words, an increase in strain rate leads to an induced orientation in regions near the skin.

Thus the effect of specimen orientation is due to the contribution of the highly oriented skin regions induced by moulding. The effect of specimen thickness is also assigned to the contribution of the skin regions, which is greater in thinner specimens.

The fact that in low frequency tests (0.1 Hz) PA12 behaves like an isotropic material with regard to specimen orientation and thickness (the plots of FCP curves at 0.1 Hz for the two specimen orientations and thicknesses are superposed), and like an anisotropic material at intermediate frequencies (1 and 5 Hz), can be understood in terms of the higher strain-rate sensitivity of a specimen oriented in the flow direction. At 0.1 Hz the core and the skin regions behave in the same manner. But when strain rate is increased, the skin regions (which are oriented in the direction of applied stress) are modified faster than the core (which is oriented in the other direction), improving the material's FCP resistance by the formation of larger SLST.

At 10 Hz the temperature rise due to hysteretic heating leads to a generalized heating in the remaining ligament which causes a thermal fatigue failure. In this case the contribution of the skin regions is negligible. As a consequence of this situation, FCP curves at 10 Hz for the different specimen orientations (parallel and perpendicular) and thicknesses (4 and 6 mm) are superposed.

The first and third of the three results above also explain the decrease in FCP rate and increase in shear lips when increasing frequency from 0.1 to 1 Hz, because of the increase in strain rate.

The increase in FCP rate between 1 and 10 Hz is due to generalized heating in the unbroken ligament.

However, when using CT specimens no frequency effect was observed from 1 to 10 Hz, and FCP rates were found to be higher in CT specimens than in SEN specimens. These two results are not only in disagreement with the predictions of linear elastic fracture mechanics (LEFM), but also with the fact that stresses in the unbroken ligament of SEN specimens are higher (due to the stress intensity calibration factor Y in the expression $\Delta K = Y\Delta\sigma\sqrt{a}$), contributing to a greater creep component as reported by Hertzberg *et al*[13] in the case of nylon 6.6. The results obtained suggest that the difference in far-field stress profiles (due to specimen configuration) plays a beneficial role in the case of SEN specimens, and this behaviour will be confirmed in the next section.

Another consideration which must be taken into account is the larger bending moment in the CT specimen, which can play an important role in the case of a ductile material. Indeed, during a monotonic tensile test on SEN specimens, the maximum stress intensity factory at the failure point is around 11 MPa $\sqrt{\text{m}}$. In the case of the SEN bending test this value is around 3.7 MPa $\sqrt{\text{m}}$. These results emphasize the influence of the bending moment for this type of material. In bending, the plastic zone is more restricted than in a SEN tensile test.[9]

Effect of far-field stress on the unbroken ligament

Considering the SEN specimen A shown in Fig. 13, after N cycles when the crack had reached the length $a(N)$, the remaining ligament ($W-a(N)$) was subjected to the far-field stress during N cycles; it is called the stressed ligament. To isolate the effect of far-field stress on the remaining ligament ($W-a(N)$), its FCP behaviour is compared with that of an unstressed ligament, represented by the SEN specimen B in Fig. 13, where the initial notch of length $a(N)$ is obtained by saw and razor blade. Three representative frequencies were chosen (0.3, 1 and 5 Hz) and the results are presented in Fig. 14.

When SEN specimens of type B are used, no effect of frequency was observed from 0.3 to 5 Hz. If FCP curves of specimens A and B are then plotted in the same diagram it may be noted that there is no effect of far-field stress at the frequency of 0.3 Hz, while a large effect is observed at 1 and 5 Hz. The fact that the stressed ligament is more resistant to FCP than the virgin ligament (specimen B) for

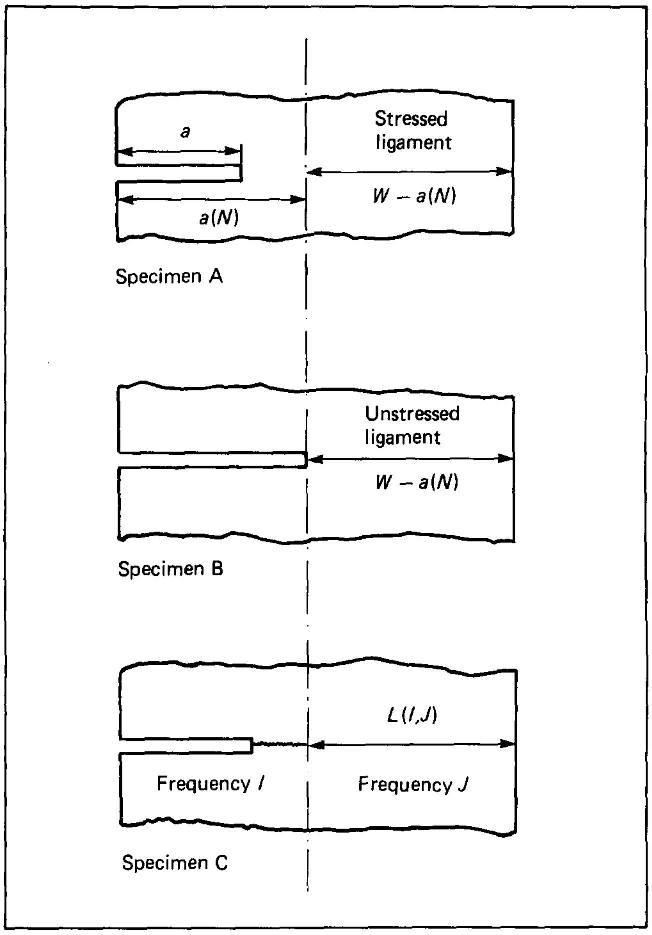

Fig. 13 Schematic representation of the SEN specimens used to investigate the effect of far-field stress on the remaining ligament

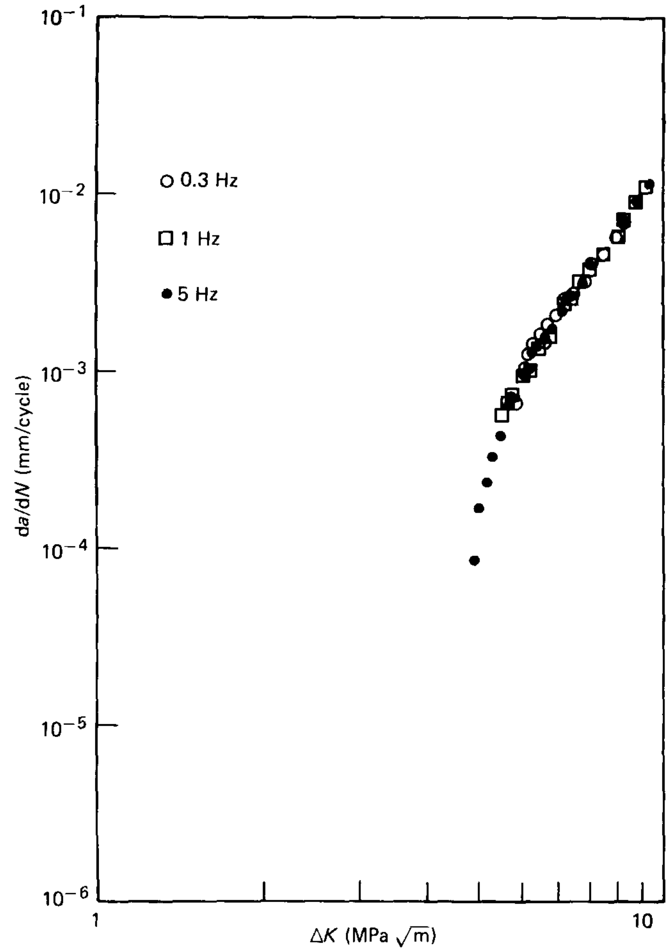

Fig. 14 Effect of frequency on the FCP curves obtained when using SEN specimen type B

1 and 5 Hz confirms the beneficial effect of far-field stress under these frequencies, as was suggested when the effect of specimen configuration was discussed.

This beneficial effect only occurred for intermediate frequencies, and this can be linked to the results which show that the effect of orientation and thickness only intervenes at intermediate frequencies. It was suggested that this behaviour is due to the prominent part of the orientation in this intermediate range of frequencies.

To confirm that only intermediate frequencies play an important role, the procedure used was to take two frequencies, I and J. The FCP test, conducted on SEN specimen A, is started under the frequency I until the dimension of the ligament is reached (see Fig. 13, specimen C) and then the FCP test is completed at a frequency J; this ligament is called $L(I,J)$. $L(I,J)$ undergoes the effect of the far-field stress at the frequency I and then the crack propagates at the frequency J. Particular cases are $I = J$, which corresponds to specimen A and $I = 0$ which corresponds to specimen B.

If this formulation is applied to the previous results, then:

- $L(5, 5)$ is more resistant to FCP than $L(0, 5)$
- $L(0.3, 0.3)$ and $L(0, 0.3)$ are superposed.

In the general case, when frequencies 0.3 and 5 Hz are combined to give $L(0.3, 5)$ and $L(5, 0.3)$, the result of these combinations can be anticipated, because there is no effect of far-field stress at 0.3 Hz contrary to at 5 Hz.

On this basis $L(0.3, 5)$ is expected to superpose with $L(0, 5)$ and this is verified experimentally in Fig. 15. The expected behaviour for $L(5, 0.3)$ is that obtained for $L(5, 5)$, but Fig. 16 shows improved FCP resistance for $L(5, 0.3)$; this difference can be explained if hysteretic heating, which leads to an increase in the specimen temperature, is taken into account. Indeed in $L(5, 5)$ the temperature continues to increase by heat accumulation, while for $L(5, 0.3)$ the decrease in frequency from 5 to 0.3 Hz allows a decrease in specimen temperature thus conserving the beneficial effect of far-field stress and removing the deleterious effect of heat accumulation.

Conclusions

1. Injection moulded polyamide-12 was found to exhibit high FCP resistance, compared with other thermoplastics.
2. The orientation induced by moulding has a strong effect on the FCP resistance of the material.
3. The highly oriented skin regions produce larger, saw-tooth shaped shear lips when they are loaded in the flow direction.
4. The width of the saw tooth increases with increasing strain rate (by increasing frequency or by changing load waveform). This seems to indicate that induced orientation occurs in the boundaries of the oriented skin layer.
5. The influence of frequency and type of specimen on FCP rate can be explained by the evolution of the ligament properties under cyclic far-field stress.

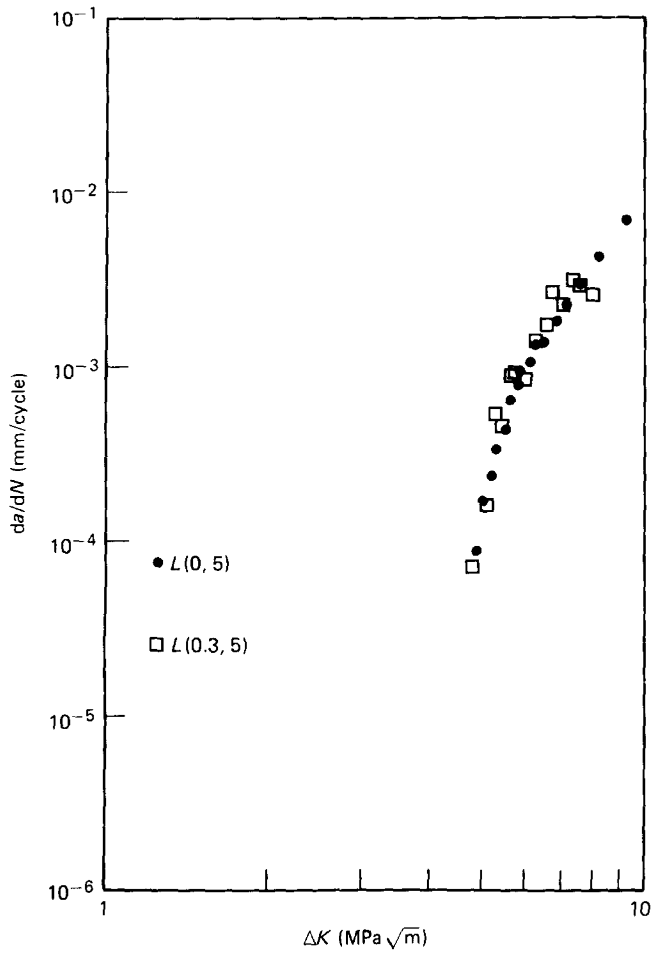

Fig. 15 FCP curves of the ligament $L(0, 5)$ corresponding to specimen B and the ligament $L(0.3, 5)$ corresponding to specimen C

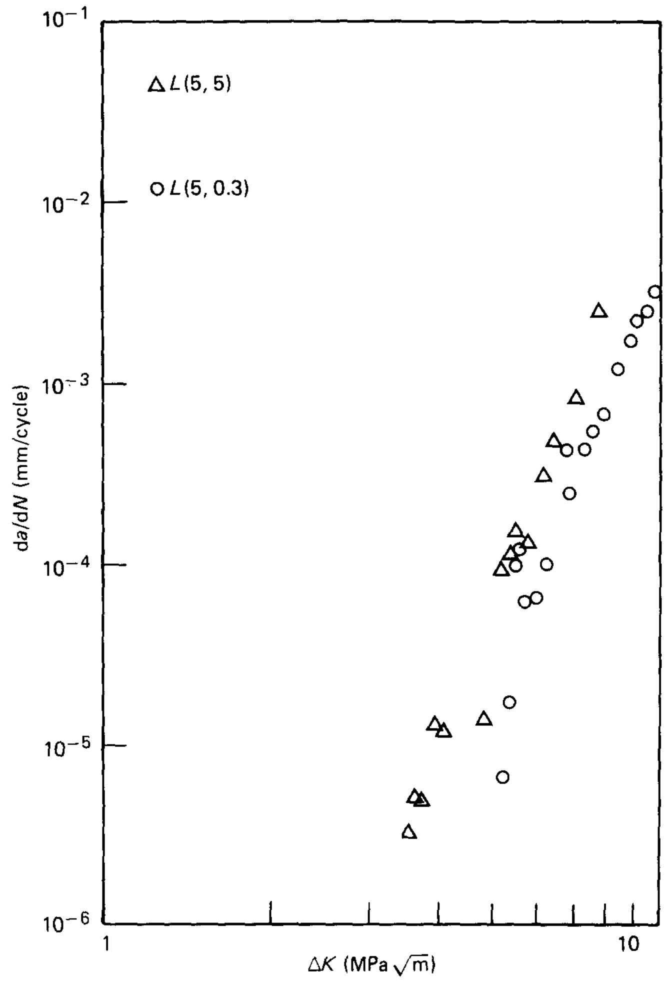

Fig. 16 FCP curves of the ligament $L(5, 5)$ corresponding to specimen A and the ligament $L(5, 0.3)$ corresponding to specimen C

6. Thus LEFM cannot be applied in the FCP studies of semi-crystalline polymers such as polyamide-12 (AMVO).
7. The da/dN *vs* ΔK plot is not a material parameter and should be considered taking account of other parameters.

Acknowledgements

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Authors

R. Boukhili and F. X. de Charentenay are with the Universite de Technologie de Compiegne, Department de Genie Mecanique, Division Polymeres et Composites, BP 233, 60200 Compiegne, France. T. Vu-Khanh is with the Institut de Genie des Materiaux, 75 Boulevard de Mortagne, Boucherville, Quebec, Canada, J4B 6Y4.