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**INTRINSIC CHARACTERISATION OF
CONTINUOUS CARBON FIBRE THERMOPLASTIC
COMPOSITES—3. FATIGUE CRACK GROWTH**

(Technical Report)

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Intrinsic characterisation of continuous carbon fibre thermoplastic composites—3. Fatigue crack growth (Technical Report)

Abstract: This paper reports some fatigue crack growth results obtained on unidirectional laminates of AS4/PEEK. Crack growth is intralaminar and the study has included the influence of laminate thickness, method of consolidation and level of applied stress level. Presentation of data is by a Paris Law type approach, where crack growth per cycle is plotted against a stress field intensity factor. The measurements have involved four different Laboratories and therefore it has been possible to attempt to embrace the likely scatter involved in the collection of fatigue data commensurate when several Laboratories conducting seemingly similar measurements.

1 INTRODUCTION

This work is part of a larger study relating to the intrinsic characterisation of continuous carbon fibre reinforced composites conducted by IUPAC Working Party IV.2.1. The other sections of the programme describe toughness (1), deformation (2) and structure (3) of these continuous carbon fibre reinforced thermoplastic composites. The specific material subjects of the work relate to AS4 carbon fibres impregnated with the thermoplastic matrix polyether ether ketone (PEEK). Materials in the form of unidirectional laminates are consolidated from AS4/PEEK prepreg by both autoclave and compression moulding methods. Full details of the materials and processing histories are described in an earlier paper (1).

This particular study concerns fatigue crack growth measurements of unidirectional laminates of AS4/PEEK supplied by ICI Materials (APC-2/AS4). Four laboratories have participated in this section of the work:-

Laboratory 1. ICI Materials, Wilton Research Centre, England (D R Moore, R S Prediger, A Kitano).

Laboratory 2. Polymeric Composites Laboratory, University of Washington, Seattle, USA (Professor J C Seferis, A Kitano (now with Toray, Japan).

Laboratory 3. Montefluos S P A, Research Centre of Bollate, Italy (G Castiglioni, G Paolucci, G Ajroldi).

Laboratory 4. School of Industrial Science, Cranfield Institute of Technology, Bedford, England (Professor C B Bucknall).

The performance of such materials under cyclic loading conditions is of particular interest if these composites are to operate successfully as engineering materials. Moreover, a knowledge of the manner in which cracks may grow in such materials is an especially relevant aspect if the materials are to seek application in primary aerospace structures. However, fatigue crack growth is a wide topic and only a limited approach is attempted in this work.

The initiation and propagation of cracks in composites can follow many forms. In particular, it has been shown that numerous failure mechanisms may occur (4) and these include:-

- (i) Interlaminar fracture where cracks propagate through the matrix material in the inter-ply region; this is also known as delamination fracture.
- (ii) Intralaminar fracture where the crack propagates in the matrix material between the fibres, but not in the inter-ply region; this is also referred to as between fibre crack growth.
- (iii) Translaminar fracture where the crack propagates by breaking fibres.
- (iv) Debonding fracture where the crack propagates in the interfacial material that surrounds the fibre.
- (v) Fibre bridging fracture where the crack propagates in the matrix material but in so doing it removes a bundle of fibres away from the laminate; this is often a sub-mechanism of intralaminar and/or interlaminar fracture.

Many of these mechanisms occur simultaneously although some control is possible by selection of the lay-up of the laminate and the direction of applied stress relative to the direction of the fibres.

The principal mechanism that we intend to study in this work relates to intralaminar fracture. This was

achieved by measuring crack growth in unidirectional laminates with the applied stress directed at right angles to the direction of the fibres. Consequently the crack should grow between the fibres in an intralaminar manner. Crack growth was energised by a cyclic stress field and thus fatigue crack growth was measured.

There are two approaches that can be used to investigate fatigue crack growth. First, it may be analysed in terms of a strain energy release rate function (G) which can be determined from the rate of change of specimen compliance with crack growth. Second, it can be articulated in terms of a stress field intensity factor (K) which is the approach that has been used in this work. Our choice has more to do with practical convenience than scientific resolution, in fact, there is a strong school of thought that believes that presentation in terms of G is preferable.

Fatigue experiments on continuous fibre reinforced composites are intricate because they permit several likely failure mechanisms but also because the number of experimental parameters can be large. These might include stress level, load-waveform type and frequency, test geometry, laminate size (ie the number of plies in the laminate); in addition, there are many possible methods for analysing and presenting the results. Inevitably, such choice can lead to confusion in understanding fatigue crack growth characteristics. Therefore, one of our aims in this study was to compare results where common data collection practices were used together with similar analytical and presentational approaches.

The process of generating intralaminar fatigue crack growth behaviour of unidirectional composites fundamentally involves the collection of crack growth data as a function of number of cycles under load. Unfortunately, the presentation of results in the form of crack growth versus number of cycles seldom leads to helpful characterisation of the fatigue behaviour of the composite. For a start, it does not accommodate the experimental variable of stress. An empirical approach developed by Paris (5) suggested a power law analysis for normalising such data:-

$$\frac{da}{dn} = A(\Delta K)^m \dots\dots\dots 1$$

Where da is an incremental crack growth in an interval of cycles dn . A and m are constants.

ΔK is the change in applied stress field intensity factor, defined as follows:-

$$\Delta K = \Delta\sigma_R Y a^{\frac{1}{2}} \dots\dots\dots 2$$

Where $\Delta\sigma_R$ is the change in remote applied stress.

Y is a geometry function related to specimen type and size.

a is the crack length.

The term remote applied stress is defined in terms of the force applied to the test specimen and therefore it is necessary to define the specimen geometry. We used a compact tension geometry in this study as shown in Figure 1 where the dimensions a , W and B are shown. Remote applied stress, or more precisely the change in stress, can now be defined:-

$$\Delta\sigma_R = \frac{\Delta P}{BW} \dots\dots\dots 3$$

$$\Delta P = P_{max} - P_{min} \dots\dots\dots 4$$

Where P_{max} is the maximum applied force. And P_{min} is the minimum applied force.

There is at least one common alternative to these stress definitions, namely the use of net stress. Applied to our compact tension specimen this would lead to:-

$$\Delta\sigma_{net} = \frac{\Delta P}{(W-a)B} \dots\dots\dots 5$$

In the conduct of nominal "zero-tension" fatigue experiments it is expected that the applied force on the specimen will vary from some tension value to zero. In a practical sense this can lead to buckling instabilities as the inertia of the specimen is not precisely in phase with the control actuator on the machine. This practical problem is overcome by cycling the force between tensile values where the minimum force is usually small compared with the maximum force. This ratio is commonly defined by the fatigue R value:-

$$R = \frac{P_{min}}{P_{max}} \dots\dots\dots 6$$

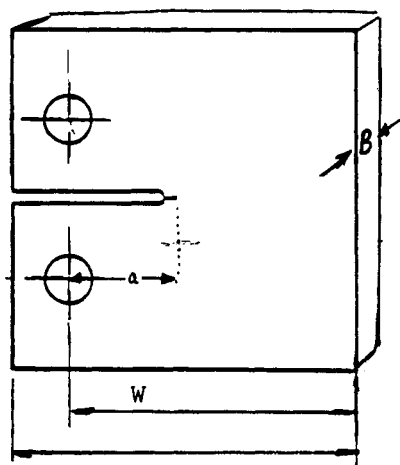


Fig. 1 Compact tension specimens for fatigue testing.

It is possible that some laboratories equate the minimum value of force to "zero", whilst others use a precise magnitude in the definition of minimum stress. This can be confusing since it will influence the calculation of the stress field intensity factor. In addition and for completeness, it is also helpful to indicate that some laboratories express applied forces in terms of mean force:-

$$P_{mean} = \frac{P_{max} + P_{min}}{2} \dots\dots\dots 7$$

These ramifications alone can lead to differences in results from a comparative fatigue crack growth study. Therefore we have adopted a common approach in defining the stress field intensity as used in the results of our inter-laboratory study, namely:-

$$\Delta K = \Delta \sigma_R Y a^{\frac{1}{2}} \dots\dots\dots 8$$

Where $\Delta \sigma_R = \frac{(P_{max} - P_{min})}{BW} \dots\dots\dots 9$

And $Y = \frac{(2+\beta)}{\{\beta(1-\beta)^3\}^{\frac{1}{2}}} (0.886 + 4.64\beta - 13.32\beta^2 + 14.72\beta^3 - 5.6\beta^4) \dots\dots\dots 10$

Where $\beta = \frac{a}{W}$

The geometry factor for isotropic materials (Y) is defined in Brown and Srawley (6) for a compact tension specimen in terms of a/W . Of course, unidirectional laminates are anisotropic. However, we have shown that the isotropic value for Y is sufficiently accurate at least for intralaminar fracture of unidirectional continuous carbon fibre reinforced composites (1).

With this background in mind there are four principal aims in this inter-laboratory study.

- (i) To measure fatigue crack growth in several laboratories and to compare these data by using identical analytical and presentational approaches.
- (ii) To conduct these measurements in such a manner as to accommodate a study of the influence of stress.
- (iii) To study fatigue crack growth in terms of different numbers of plies in the laminates and by different methods for consolidating the laminates (autoclaving and compression moulding).
- (iv) To provide an understanding of what fatigue crack growth measurements may indicate about the fatigue performance of these materials.

2 MATERIALS AND EXPERIMENTAL METHODS

AS4/PEEK laminates were prepared following the manufacturer's recommendations and full details of processing are described elsewhere (1). Four laminate types were prepared as follows:-

Unidirectional laminates composed of 8, 20 and 40 plies by autoclaving; these laminates are designated:- $(0)_8/A$, $(0)_{20}/A$, $(0)_{40}/A$

Unidirectional laminates composed of 8 plies prepared by a compression moulding method; this laminate is designated:-

$(0)_8 / CM$

Fatigue crack growth measurements were conducted on compact tension (CT) specimens as shown in Figure 1. The square plaques were machined from the laminates using a diamond-ended cutter and a sharp tool was used for introducing the notch. The dimensions of the outline CT specimens were similar for each laboratory and varied in the range 55 mm to 68 mm. Laboratories 3 and 4 started their fatigue test at 2Hz and at relatively high loads in order to start a small amount of crack growth and then they reduced the waveform frequency to 0.5 Hz and applied the appropriate stress level. Laboratory 1 selected the required applied load from the outset and used a waveform frequency of 1 Hz throughout. All laboratories used a sinusoidal waveform at 23°C using load control where the R ratio was 0.1.

Laboratories 3 and 4 measured crack growth with a travelling microscope; Laboratory 1 used a "Kraak" gauge attached to the side of the specimen which was connected to a "Fractomat" amplifier and this enabled continuous crack length monitoring. Laboratory 3 transcribed the output from their travelling microscope to a linear variable differential transducer (LVDT) enabling the crack growth signal to be connected to a potentiometric recorder for continuous monitoring. These various procedures enabled the crack length versus number of cycles functions to be monitored from tests conducted on servo-hydraulic machines (eg Instron 8031).

The da/dn data were obtained from the derivatives of the a versus n functions. Laboratories 3 and 4 fitted polynomials to the a versus n functions in order to obtain the derivatives. For example, Laboratory 3 used the methods described by Lanczos (7) using a least squares procedure based on a 2nd order polynomial. Laboratory 1 used a different approach based on acquiring an appropriate sample rate for the (a, n) data and then determining the derivative from a three-point analysis. The data sampling rate was computer controlled in order to ensure that new crack length data were monitored only when a significant change in crack length occurred. This improved the accuracy and balance in the definition of the da/dn versus n function. The process of determining da/dn however is always associated with some experimental scatter.

Laboratory 3 conducted some scanning electron microscopy (SEM) of the fracture surfaces in order to identify the failure mechanisms.

In order to ensure a similar analysis and presentational style for the results from all the participants, Laboratory 1 used a Microvax based database software package called "MEDOC" to collate all results from which analysis and presentation could be conducted.

3 PRESENTATION AND DISCUSSION OF RESULTS

3.1 Crack growth measurements

The crack length versus number of cycles functions for four fatigue crack growth experiments are shown in Figure 2; the associated remote stresses are also included. This form of presentation limits interpretation as discussed in the Introduction. Part of the limitation concerns the stress field at the tip of the crack which is

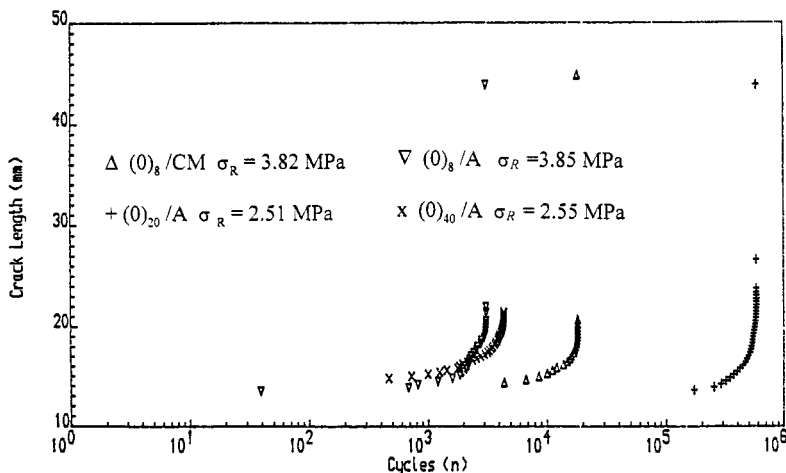


Fig. 2
Crack growth versus log
 n at 23 °C.

largely responsible for how the crack grows. The expression for a as defined in the Introduction can be seen to have two terms, namely stress and crack length. Moreover, the Y function also changes with crack length. Therefore, plots of crack length versus number of cycles even at different stress levels do not easily unite the inter-dependent parameters that govern crack growth. With these limitations in mind, it has been conventional to present results in the form of "Paris law" plots of $\log (da/dn)$ versus $\log a$.

3.2 Fracture morphology

It is helpful to make some observations on the nature of failure during the crack growth process. These observations can be generalised by resolving the fracture into two parts; an initial period of crack growth (10 mm to 15 mm of crack extension) where the fracture surface gives an impression to the naked eye of a "stress-whitened" region and a second region some >5mm beyond the "stress-whitened" region where faster fracture is occurring. This description is compatible with the nature of the crack growth plots of Figure 2. SEM micrographs taken from the $(0)_{40}/A$ materials are shown in Figures 3 and 4.

The features that occur during the "slow crack growth" region associated with "stress-whitening" are shown in Figure 3 at three different levels of magnification. The lowest level of magnification (Figure 3

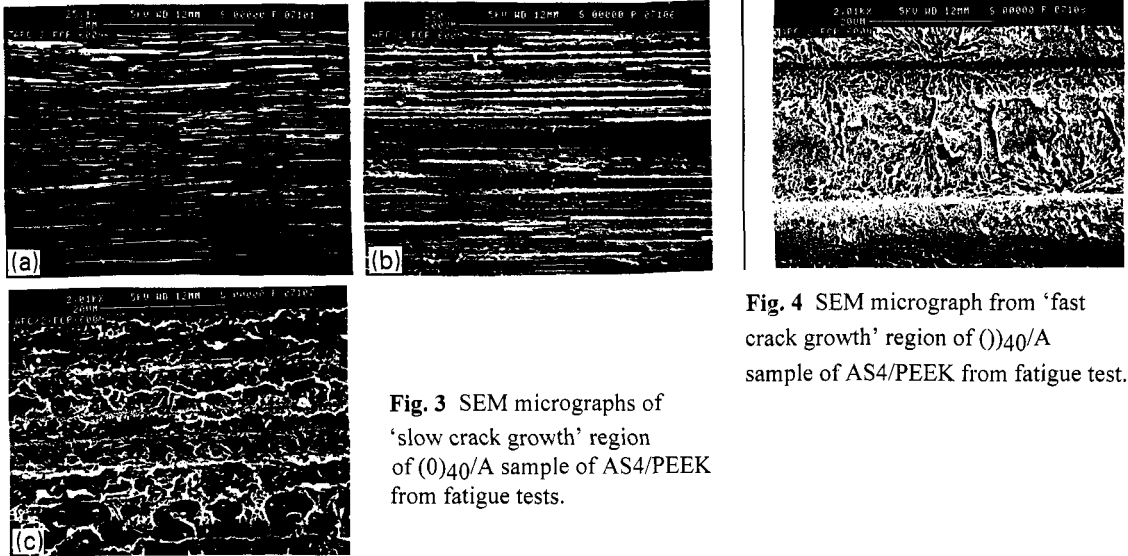


Fig. 3 SEM micrographs of 'slow crack growth' region of $(0)_{40}/A$ sample of AS4/PEEK from fatigue tests.

Fig. 4 SEM micrograph from 'fast crack growth' region of $(0)_{40}/A$ sample of AS4/PEEK from fatigue test.

$\Delta (0)_8/A \sigma_R = 3.85 \text{ MPa}$ $\nabla (0)_8/A \sigma_R = 3.95 \text{ MPa}$
 $+ (0)_{20}/A \sigma_R = 2.97 \text{ MPa}$ $\times (0)_{20}/A \sigma_R = 2.51 \text{ MPa}$

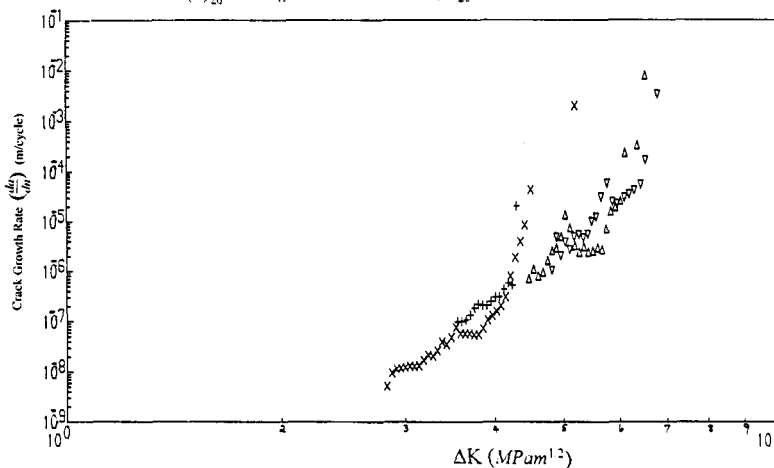


Fig. 5 $\log (da/dn)$ versus $\log \Delta K$ from laboratory 1.

(a) shows that the crack propagates in more than one plane whilst Figure 3(b) shows that fibre fracture and fibre-bundle bridging are also occurring. These relate to the anticipated failure mechanisms discussed earlier. The highest magnification micrographs (Figure 3(c)) indicates the nature of the matrix fracture for the intralaminar crack growth, where large matrix ductility is observed. Therefore, in summary, it can be concluded that in the "slow crack growth" region the fatigue crack propagation is intralaminar accompanied by fibre bridging and with some fibre fracture (although this is just an end-point of the bridging mechanism).

The failure process that occurs in the "fast crack growth" region is similar on a macroscopic scale, but the nature of the matrix fracture changes. This is shown in Figure 4 at a similar magnification to Figure 3 (c). In Figure 4 the extent of the matrix ductility has significantly reduced although small scale ductility is still clearly present.

Overall, the failure mechanisms do not include any debonding process, because all fibres in the fracture surfaces are fully covered with the PEEK matrix material. Therefore, crack growth is intralaminar with some fibre bridging. At both crack growth speeds it is possible to observe matrix yielding in the fracture surface, however, the extent of plastic deformation is significantly higher in the "slow crack growth" fracture surfaces.

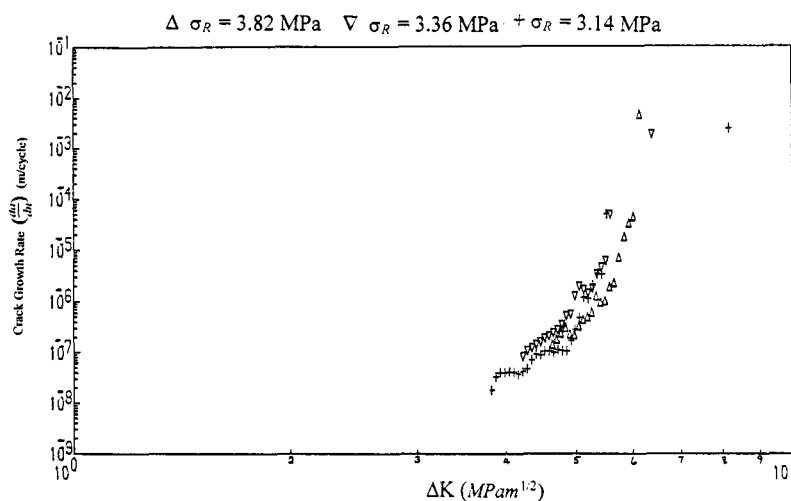


Fig. 6
Log (da/dn) versus log ΔK from laboratory 1 for $(0)_8/CM$.

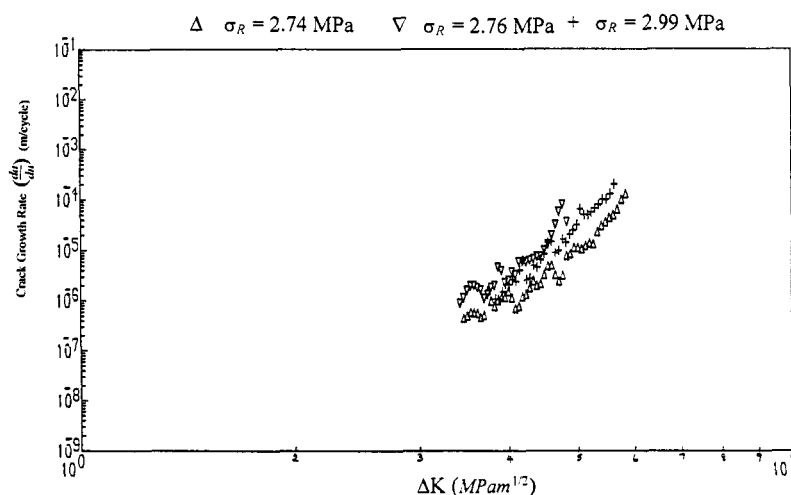


Fig. 7
Log (da/dn) versus log ΔK from laboratory 3 on $(0)_{40}/A$.

3.3 Paris law presentations

The plots of $\log(da/dn)$ versus \log will be presented for each of the laboratories first. Figure 5 shows these curves for the 8 and 20 ply unidirectional laminates consolidated in an autoclave $\{(0)_8/A \text{ \& } (0)_{20}/A\}$ where it can be seen that all data fit a similar curve except for those parts of the test near to complete fracture. It was observed that near complete fracture of the specimen some bending of the specimen took

place and therefore the more credible crack growth data relate to the lower values of stress field intensity factor. Figure 6 shows these plots for the 8ply compression moulded material where again similar comments and observations relate to data collected near complete fracture.

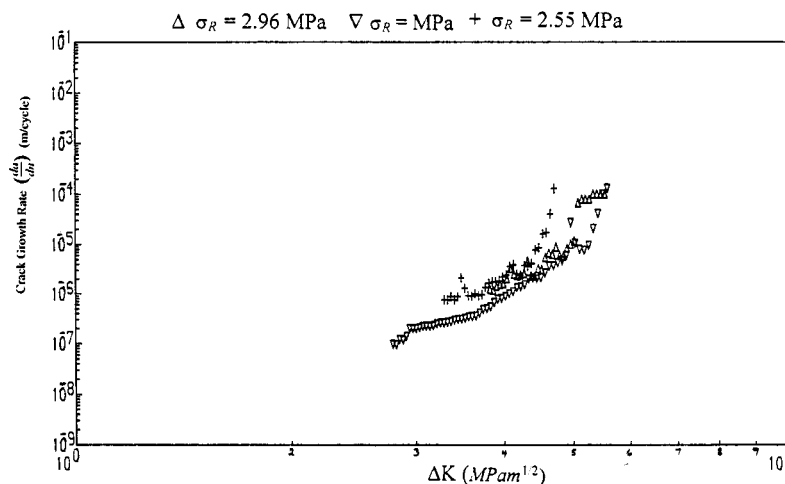


Fig. 8

Log (da/dn) versus log ΔK
from laboratory 3 on $(0)_40/A$.

Figures 7 and 8 show the Paris law plots for Laboratory 3 for the 40 ply autoclaved laminates. The results are spread over two plots because six different stress levels were used in the data collection. Their spread of remote stresses only covers a small band, namely 2.19 MPa to 2.99 MPa. If Figures 7 and 8 are superimposed it can be seen that the spread of data on the plots of $\log(da/dn)$ versus $\log \Delta K$ is relatively small; commensurate with the small spread in applied stress. Figure 9 shows the plots for Laboratory 4 again for 40 ply laminates. Their data cover a large stress range but a smaller amount of crack growth.

Superposition of all these plots has been conducted in order to investigate the influence of applied stress on crack growth rate. Such an analysis was unresolving. Therefore, since the better quality data have been shown to occur at the smallest levels of stress field intensity, it is possible to extract values of da/dn at a value of $4.0 \text{ MPa m}^{1/2}$. These specific values of da/dn are then plotted against remote stress in order to compare all the results and to investigate the influence of stress level; such a graph is shown in Figure 10 using a log-linear scale. The cross plotted data in Figure 10 also contain all the different laminate thicknesses and the different methods used for consolidation. With reference to Figure 10 it is useful to first consider the results for the 40 ply autoclaved laminates. For a given level of remote stress there is just over a decade of scatter in the values of da/dn . This may be interpreted in one of two possible ways. Either there is a source of variability in these data (for example, this may have been introduced by the different notching procedures used in the various Laboratories) or such scatter is typical for fatigue data. Further study would be necessary to resolve this point.

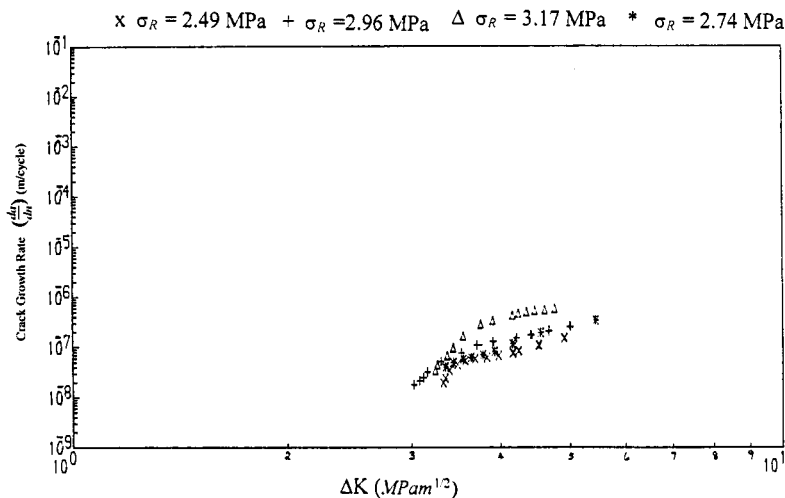


Fig. 9

Log (da/dn) versus log ΔK
from laboratory 4 on $(0)_40/A$.

If the results for the thinner laminates (whether autoclaved or compression moulded) are now added to this picture, then it is plausible to believe that these lie within the same experimental scatter. The question therefore arises as to whether there is evidence to believe that intralaminar fatigue crack growth is independent of stress level, number of plies and processing method for the manufacture of the laminate. Unfortunately, it is difficult to be convinced of this possible conclusion because of the lack of confidence in whether the data have unknown sources of experimental error, as mentioned above.

It could be argued that the degree of fibre bridging that accompanies the intralaminar crack growth should be affected by the laminate thickness. For example, it might be expected that more fibre bridging will occur with a thicker laminate. The results in Figure 5 do not support this view because the slope of the Paris law plot for the 8 and the 20 ply laminates can be seen to be similar.

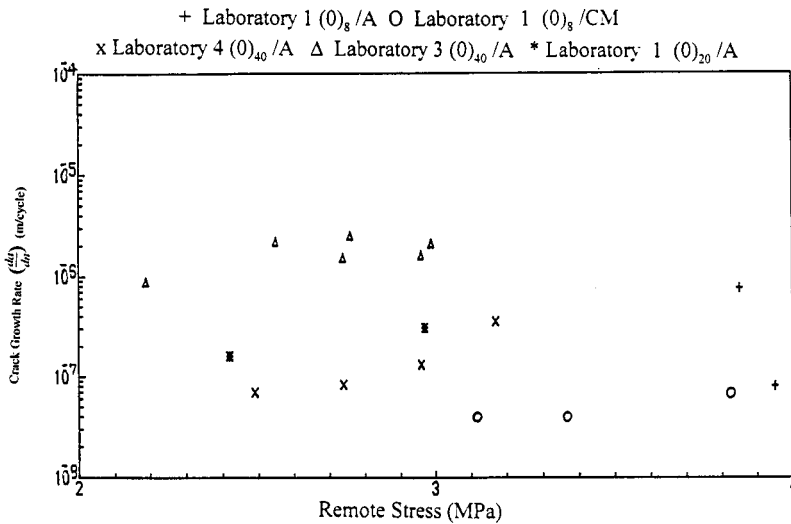


Fig. 10

Log (da/dN) versus remote stress (log (da/dN) defined at $\Delta K = 4.0 \text{ MPam}^{1/2}$).

4 CONCLUDING COMMENTS

Four laboratories have combined to measure intralaminar fatigue crack growth on unidirectional laminates of AS4/PEEK with different thicknesses and prepared by different methods of consolidation. Special care has been given to ensure that the quality of the data are accurately obtained, within the usual variations in collecting fatigue result, and that the data are analysed and presented in identical fashions. We have also attempted to discuss some vagaries that can arise if such care is not applied.

It has been possible to identify the failure mechanisms that contribute to the intralaminar process when growing fatigue cracks in these materials. In addition, it could be possible that fatigue crack growth is independent of the level of applied stress, the number of plies in the laminate and the method of consolidation. An inevitability in conducting such measurements is experimental scatter in the results. Our experience suggests that about a decade of crack growth scatter is possible. This observation would need to be either accommodated in future fatigue work, particularly that aimed at materials comparison, or that further studies should be aimed at identifying the source of this scatter.

5 REFERENCES

- 1 D R Moore, J C Seferis *Pure & Appl. Chem.*, 63, 11 (1991), pp1609-1625
- 2 A Cervenka, D R Moore, J C Seferis "Intrinsic characterisation of continuous fibre thermoplastic composites; 2 Pseudo-elastic constants for APC-2" *Pure & Appl. Chem.* 64,11, (1992) p1801
- 3 J C Seferis, G Zachmann, D R Moore "A collaborative study of the structure and morphology in continuous fibre reinforced PET and PEEK" *Pure & Appl. Chem.* 65, 7 (1993) p1581
- 4 D R Moore "Fatigue of Thermoplastic Composites" Chapter 10 of "Thermoplastic Composite Materials" ed L A Carlsson Elsevier Science Publishers B V 1991.
- 5 P C Paris PhD Dissertation (Lehigh University) September 1962.
- 6 W F Brown, J E Srawley "Plane Strain Crack toughness Testing of High Strength Metallic Materials" ASTM STP 410 1966.
- 7 C Lanczos "Applied Analysis" Prentice Hall, Englewood Cliffs N.J 1961.