

Proceedings of the Seminar

THERMOPLASTIC COMPOSITES TECHNOLOGY

Cagliari, 11-12 October 1990

Fatigue life of graphite-peek laminates

P. Priolo', M. Sanna', Fan Jingbo' and F. Bertolino''

'Universita' degli studi di Cagliari - Dipartimento di Ingegneria
Meccanica

" Ecole Polytechnique Federale - Lausanne

Sommario: Numerose prove di fatica sono state svolte su laminati in grafite-Peek (polietereterchetone), in controllo di carico con rapporto di fatica pari a 0,1. Il danneggiamento e' stato seguito attraverso la registrazione del modulo di Young medio del ciclo di isteresi e dell'area del ciclo ed anche con l'osservazione durante la prova della superficie laterale del provino. Per laminati unidirezionali, cross-ply e quasi-isotropi l'entita' del danneggiamento e' molto scarsa. Per laminati angle-ply in asse e fuori asse il danneggiamento e' molto piu' accentuato ma concentrato quasi totalmente nei primi cicli. Oltre a questo, e' stato effettuato un tentativo di predire la curva di Wohler del laminato partendo da quella dello strato unidirezionale con l'applicazione di un criterio di resistenza. I migliori risultati si ottengono utilizzando pero' per i laminati piu' complessi curve S-N ricavate dai laminati cross-ply e angle-ply .

Abstract: A series of fatigue tests on graphite-peek (polyetheretherketone) laminates has been performed on standard specimens in a hydraulic test machine under load control with a fatigue ratio $R=0.1$. Damage has been followed during the tests by continuously monitoring the Young modulus and the hysteresis loop area and by optically observing the lateral surface of the specimen. In certain cases i.e. for unidirectional, cross-ply and quasi-isotropic laminates the amount of damage is very limited and the initial elastic properties preserved until ultimate failure. For angle-ply in-axis and off-axis loaded damage is mainly concentrated in the first cycles where a strong variation of initial properties occurs. An attempt has been made to predict fatigue Wohler curves of multiangle laminates starting from that of the unidirectional lamina, by making use of two known strength criteria. Better results were achieved introducing fatigue data drawn from angle-ply and cross-ply laminate .

1. Introduction

Graphite fibre reinforced polyetheretherketone (peek) has been recently introduced [1] by ICI as a thermoplastic high performance composite material for aerospace applications. The mechanical and impact behaviour in hot wet environment [2], in comparison with epoxy based materials, is a major factor encouraging the use of this composite in critical elements [3]. Furthermore, thermoformability and re-mouldability are properties that make thermoplastic structural composites a challenging opportunity not only in aerospace but also in automotive industry.

When long fibre graphite-peek laminates are tested under fatigue loads they generally do not show great amount of damage before failure. The maximum axial stiffness loss during laboratory tests on quasi-isotropic laminates is of order of 13%, which is small if compared with similar epoxy based laminates [4]. Optical observation of the lateral specimen surface during the test can provide useful information on fracture mechanism preceding ultimate failure [5] both of interlaminar and intralaminar type.

A complete set of experimental data on the strength of laminates with different stacking sequence can constitute the database to check the validity of known failure criteria [6]. The problem is to predict laminate strength starting from the unidirectional lamina. Among all the existent criteria two seem particularly efficient in correlating strength with observed fracture mechanism: the maximum stress criterion (M.S.C.) and the Hashin criterion (H.C.) [7]. An associated incremental damage accumulation technique is needed to predict the failure mechanism from the first damage in the weakest ply to the final failure [8].

2. Experimental details

Laminates, made of APC2/AS4 Hercules AS4 fibres and peek matrix were initially supplied by ICI and then by Fiberite. The first specimen shape for quasi-isotropic laminates was of the dog-bone type, but later the shape for all the specimens was unified to the parallel-side type. The specimen length was always between 200 and 250 mm and width equal to 20 mm. The end aluminium tabs (1.5 mm thick) were bonded with Ciba-Geigy Redux 410. Both 1 mm (8 plies) and 2 mm (16 plies) thicknesses were used. All static and fatigue experiments were performed with a servohydraulic testing machine in load control mode. Fatigue loading was sinusoidal with fatigue ratio $R=0.1$ and frequency ranging from 3 to 5 Hz. During the test the load cell and

the extensometer signals were recorded and processed to obtain the mean elastic secant modulus and the hysteresis loop area.

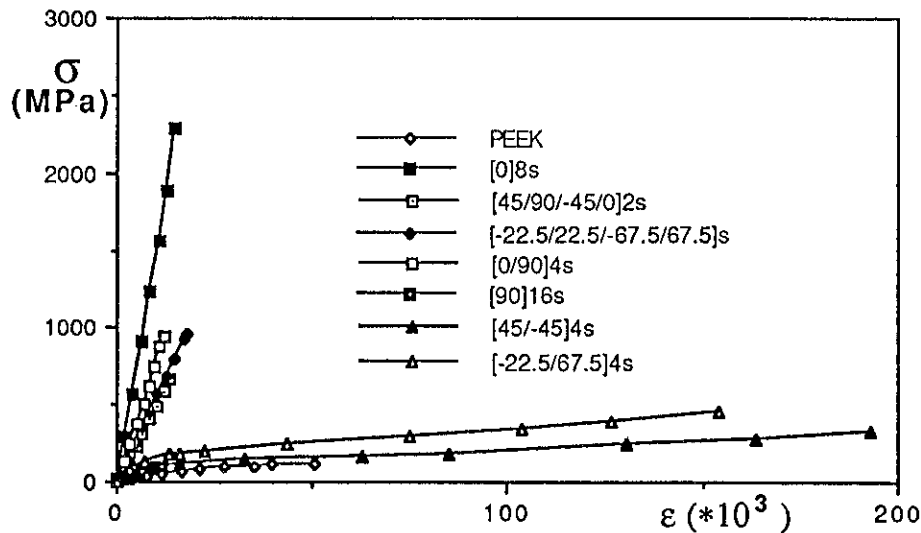


Fig.1 Static stress-strain curves for several laminates

An optical microscope was faced to the lateral specimen surface and photographs taken periodically to follow the damage mechanism.

Symmetric laminates with three basic stacking sequences were considered i.e. unidirectional [0], cross-ply [0/90]_{2s} and quasi-isotropic [45/90/-45/0]_{ns}. For each of them the load direction was varied and sometimes the ply order. In this way we have also tested the transverse [90], the angle-ply in-axis [45/-45]_{4s} and off-axis [22.5/-67.5]_{4s}, the quasi-isotropic off-axis [22.5/67.5/-67.5/22.5]_{ns}, the varied quasi-isotropic [0/45/-45/90]_{2s} and the off-axis [22.5/67.5/-22.5/-67.5]_{2s}. Some of the results were presented in a preceding paper [9] but for completeness they are also discussed here. Table 1 gives an idea of test variety.

LAMINATE	LOAD	NO. OF DATA
[0] _{4s,8s}	static,fatigue	19
[0/90] _{4s}	static,fatigue	10
[45/-45] _{4s}	static,fatigue	8
[-22.5/67.5] _{4s}	static,fatigue	8
[45/0/-45/90] _{2s,4s}	static,fatigue	37
[0/45/-45/90] _{2s}	static,fatigue	10
[-22.5/22.5/-67.5/67.5] _{2s}	static,fatigue	11
[22.5/67.5/-67.5/-22.5] _{2s}	static,fatigue	15
[90] _{16s}	static	3
peek	static	3

Table 1. Type of laminate, load and number of data for each case.

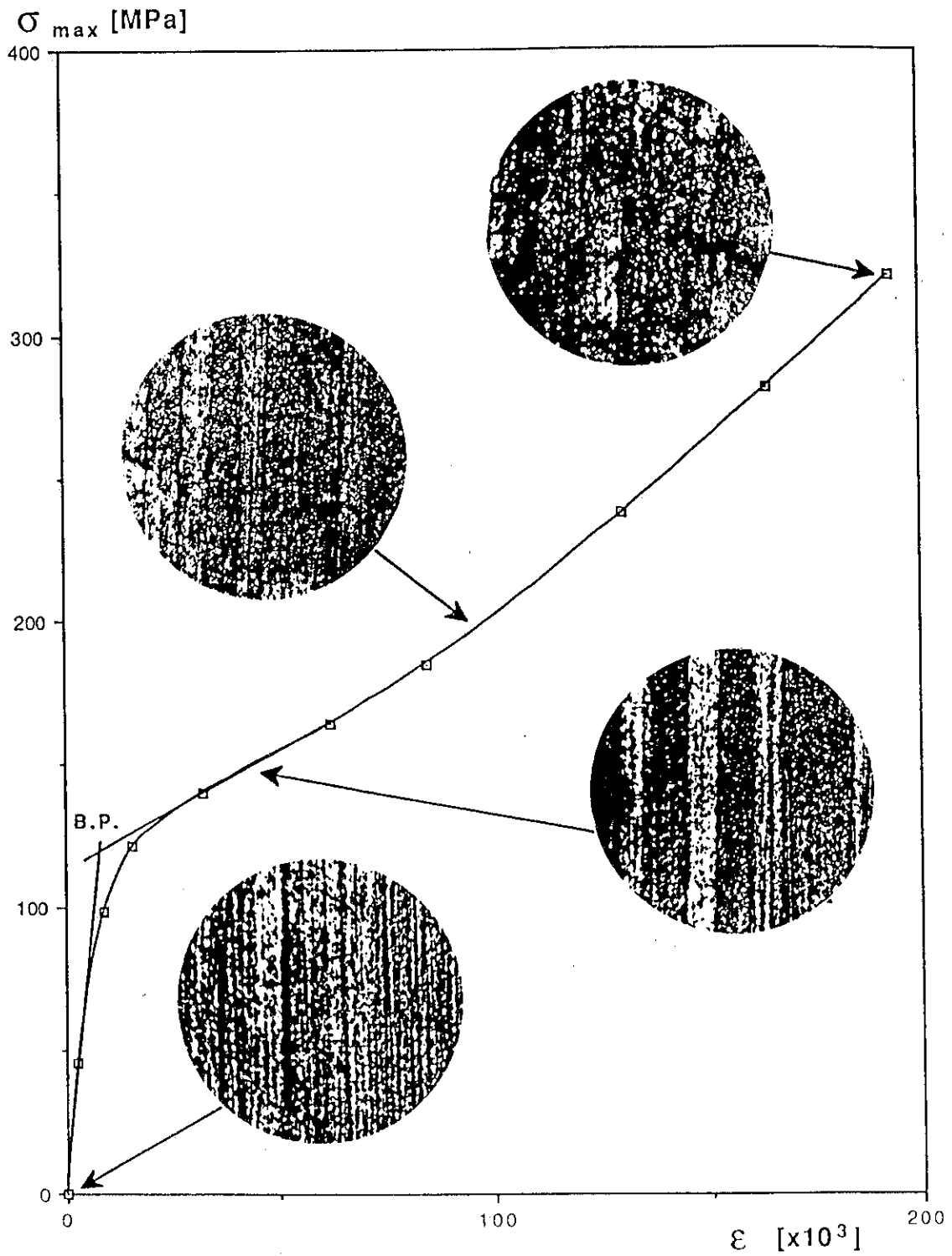


Fig.2 Static stress-strain curve for an angle-ply ($[45/-45]_4$) laminate with photographs taken during the test. After the break point (B.P.) matrix cracking and delamination become visible.

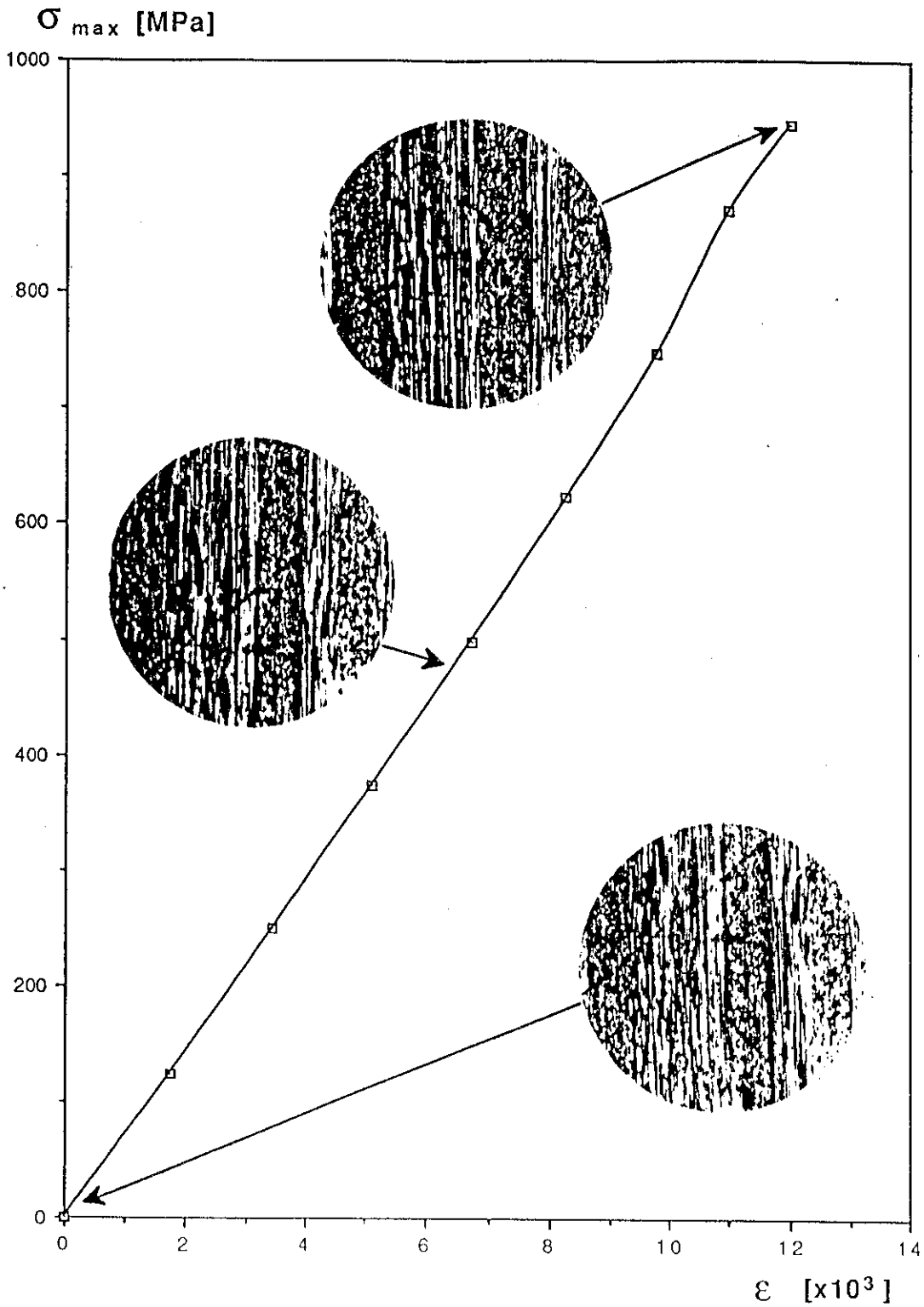


Fig. 3 Static stress-strain curve for a cross-ply ([0/90]4s) laminate. Damage is practically absent. Matrix cracking near the fracture region was observed only in some specimen.

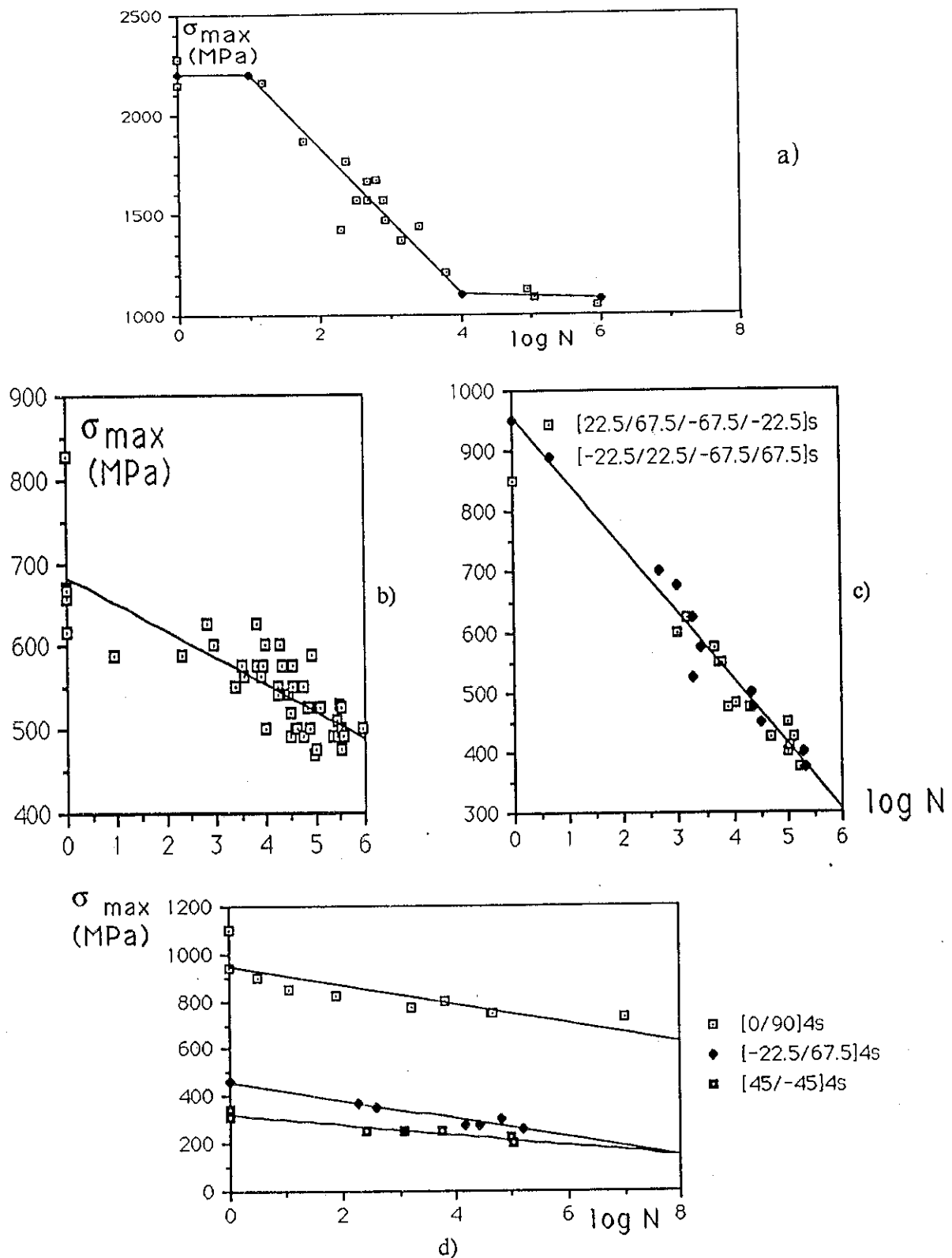


Fig 4 Fatigue data for different laminates : a) unidirectional b) quasi-isotropic in-axis c) quasi-isotropic off-axis d) cross-ply, angle-ply in-axis and off-axis.

3. Results

Static load : As can be easily seen from fig. 1 there are two different laminate behaviour : one where matrix failure is predominant, characterized by great deformation and the other where the failure mechanism is mainly fibre dependent and characterized by small deformation. To the first type belong angle-ply in-axis and off-axis, transverse [90] and peek matrix alone. For angle-ply in-axis and off-axis the stress-strain curve is strongly nonlinear with the presence of a break point (B.P.) after which the slope markedly decreases. This corresponds to sharp change in the transverse and shear modulus [8] and at the microscope level to a first spread of matrix cracking, followed by delamination, as shown in fig. 2. On the other hand the fibre dependent mechanism is characterized by little or no matrix cracking, as depicted in fig. 3.

Fatigue data are represented in the σ_{\max} -log N plane. As can be noted in fig. 4 the greatest scattering is present in quasi-isotropic laminates. Thickness and ply order play a limited role in explaining scattering [9], which can be interpreted by scattering in the elastic modulus. Fig. 4c, that refers to two different quasi-isotropic laminates loaded off-axis, shows a Woehler curve which is partly higher, for $1 < N < 10^3$, and partly lower, for $N > 10^3$ than the in-axis case of fig. 4b. Furthermore scattering is limited and strength seems to be independent on the stacking sequence. Deterioration during fatigue is negligible for almost all the laminates and is often concentrated in the first cycle. Extended matrix cracking and layer debonding can be observed only in angle-ply laminates (in-axis and off-axis) during the first cycle, but their increase during fatigue is small. For quasi-isotropic laminates damage is spread only near the fracture region and is concentrated in the 45° and 90° layers. Continuous recording and processing of the load cell and extensometer signals leads to the calculation of the elastic mean secant modulus and the hysteresis loop area. This second quantity, that can be correlated with the imaginary part of the complex modulus, is expected to be more sensitive to damage than the real part. On the other hand, it is more subjected to external noises then resulting less reliable. Some example of the real modulus and cycle area variation during fatigue life is shown in fig. 5, for various laminates. The most interesting thing in fig.5 is represented by the two curves related to $[-22.5/22.5/-67.5/67.5]_2s$ and $[22.5/67.5/-67.5/-22.5]_2s$ sequences, which are very different with respect to damage accumulation. With the specification that they are really representative of the two laminates behaviour, if we refer to the

Woehler curve of fig. 4c we do not find any difference between them in fatigue strength. It seems from these results that not necessarily higher damage amounts correspond to shorter lives.

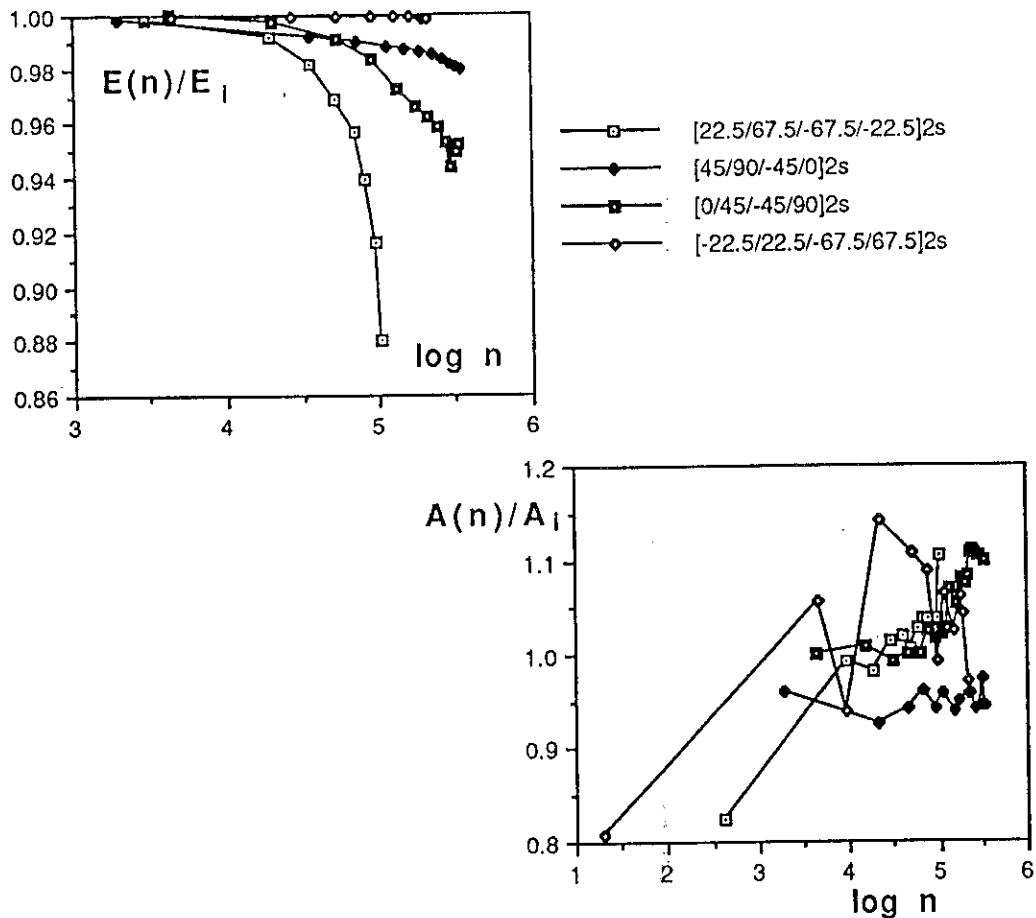


Fig. 5 Variation of the real modulus and of the cycle area during the specimen life.

4. Microstructure

At the optical microscope, fibre distribution along a normal section appear dishomogeneous as in fig. 6. When analysed at the the image processor, local fibre density shows noticeable scattering along the thickness, with lower values at the interface between two layers. We think that local dishomogeneity should play a significant role in determining rise and propagation of fracture in materials like reinforced peek, characterized by prevalent fragile behaviour, but at present we have no specific experience on this field.

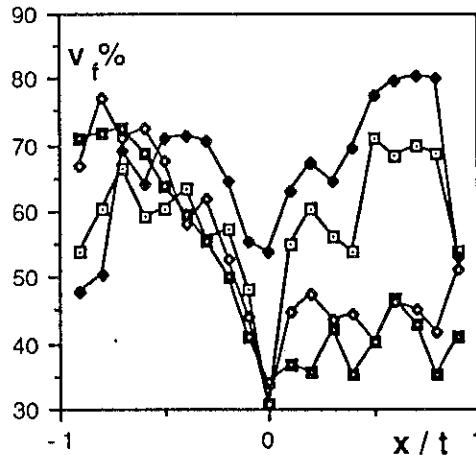
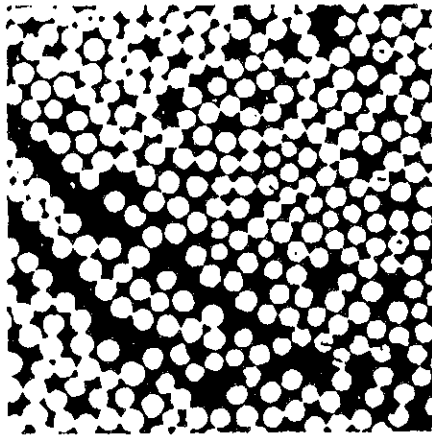


Fig. 6 Fibre distribution in a 0° layer. The four curves of the diagram refer to different locations in the same section; x is the transverse coordinate starting from the interface and t is the layer thickness.

5. Failure criteria

The structural designer needs to be able to predict fatigue life of multiangle laminates starting from data obtained on the unidirectional one. But it is well known that when a layer is inserted into a laminate its properties vary. The problem can be solved by considering tests on cross-ply and angle-ply laminates as able to furnish supplementary data. Two strength criteria have been used: M.S.C. (maximum stress) and H.C. (Hashin), together with a bilinear material behaviour of the type described in [8]. All considerations were performed on the simplified Woehler curves of fig. 4. Stresses were calculated by means of the elementary elastic layer theory [10]. Initial elastic constants and static strength values were assumed to be: $E_x=134$ MPa (fibre direction); $E_y=8.9$ MPa (transverse); $E_s=7$ MPa (shear); $\nu_x=0.24$; $X=2200$ MPa (fibre direction); $Y=88$ MPa (transverse); $S=68$ MPa (shear), all determined by specific experiments except E_s and S that were taken by the supplier data sheet. When a critical situation is reached in a layer, the corresponding elastic constants are reduced. Possible critical situations are: B.P. due to prevalent shear stress; failure for the same reason; transverse failure due to prevalent transverse stress; fibre breakage due to longitudinal stress. The corresponding elastic constant values become: $E_y = 0$, $E_s = 0.26$ GPa; $E_y = E_s = 0$; $E_y = 0$; $E_x = 0$. Ultimate failure is assumed to happen when for all the layers $E_y = E_s = 0$ [11].

Cross-ply, angle-ply in-axis and off-axis :

Microscopic observation does not reveal any preliminar matrix cracking in the 90° ply except for static or nearly static load. When it happens, transverse cracking occurs just before final failure. From the Woehler curve of fig. 4d, if compared with the curve of fig. 4a through the M.S.C., it is possible to calculate column X and Y of Table 2. The hypothesis that matrix failure in the 90° ply and fibre failure in the 0° ply occur at the same time is made.

N	X	X'	Y	S	S'
1	1838	2200	121	167	161
10	1735	2200	115	154	149
10 ⁴	1445	1100	95	120	113
10 ⁶	1379	1080	91	92	89

Table 2. Stress values are in MPa. X' is drawn from unidirectional tests. X and Y are drawn from the cross-ply, S and S' from the angle-ply (in-axis and off-axis) laminates with the H.C. and M.S.C. respectively.

Angle-ply in-axis and off-axis static tests of the type of fig. 2 can be well interpreted with the assumption that after the break-point (B.P.) elastic constants E_y and E_s change their initial values to 0 and 0.26 GPa. Fatigue loads always exceed the B.P. so the above values were taken. Starting again from the Woehler curves of fig. 4d through M.S.C. and H.C. we obtain S' and S of Table 2.

In-axis and off-axis quasi-isotropic :

For in-axis case the longitudinal stress corresponding to B.P. in the $\pm 45^\circ$ plies is 388 MPa. It then follows that in these plies E_y and E_s assume the reduced values. By the application of the M.S.C. or H.C. (there is no difference in this case) to the 0° ply and comparing with X and X' of Table 2 we obtain data of Table 3.

N	σ_{exp}	σ	σ'
1	683	643	770
10	651	607	770
10 ⁴	554	506	385
10 ⁶	489	483	378

Table 3. σ and σ' are calculated from X and X' of Table 2. All stress values are in MPa.

In off-axis laminates the B.P. is reached in all plies for a laminate longitudinal stress of 552 MPa. Then for $1 < N < 10^3$ the reduced values for E_y and E_s must be assumed, while for $N > 10^3$ the original elastic constants remain unchanged. Application of the H.C. and M.S.C. results in values of columns 3 to 6 of Table 4.

N	σ_{exp}	σ	σ'	σ''	σ'''
1	956	765	915	765	915
10	848	722	915	722	915
10^4	522	568	469	707	538
10^6	305	502	432	674	528

Table 4. σ and σ' are calculated with the H.C. using X and X' of Table 2 respectively.; σ'' and σ''' with the M.S.C. Stresses are in MPa.

The Woehler curves corresponding to Tables 3 and 4 are represented in fig 7.

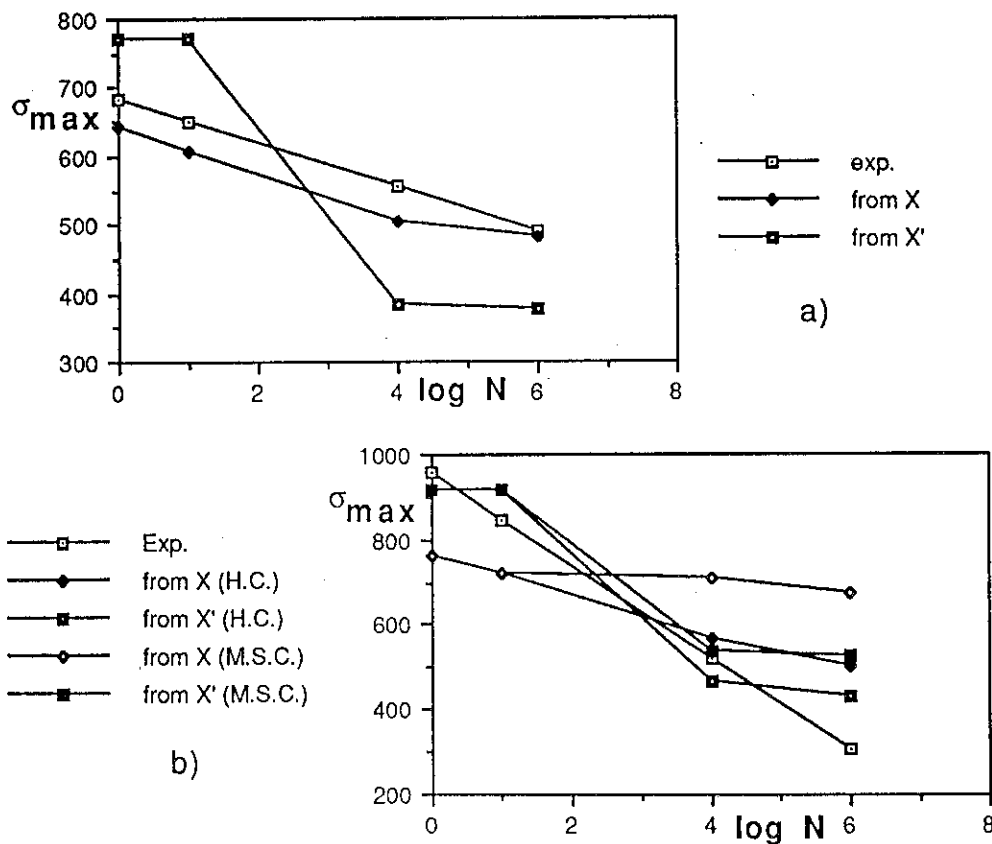


Fig. 7 Prediction of fatigue curves : a) quasi-isotropic in-axis laminate b) off-axis. Stress values are in MPa.

6. Conclusion

The analysis of several static and fatigue data of long graphite fibre-peek laminates can be summarised as follows :

- 1) Fatigue data in the Woehler plane are normally not greatly dispersed. Except for the quasi-isotropic case, where different laminate sequences and test conditions were considered, data are well condensed near a simple curve.
- 2) The real elastic modulus loss is limited in almost all the laminates treated. In any case, loss is small if compared with similar epoxy based laminates found in bibliography. The hysteresis loop area variation is greater, but at present relevant scatter makes this parameter unreliable.
- 3) The original static and fatigue strength data achieved on unidirectional laminate can result useless in predicting the laminate behaviour. Better results are obtained by using angle-ply data for shear strength and cross-ply for transverse and longitudinal strength. This last quantity, however, leads to contradictory results between in-axis and off-axis quasi-isotropic laminates, as shown by fig. 7.
- 3) Strength criteria like the maximum stress criterion and the Hashin criterion, together with assumed bilinear material behaviour, elastic constants degradation and a systematic optical observation method of matrix cracking and delamination, can be used in predicting Woehler curves with reasonable engineering approximation.

References

- [1] Brewster, I. and Cattanach, J.B. : *Engineering with long fibre thermoplastic composites*, SAMPE Conf. Proceedings - Engineering with Composites, London, 1983
- [2] Bishop, S.M. : *The mechanical performance and impact behaviour of carbon-fibre reinforced peek*, Proc. of the 3rd Int. Conf. on Composite Structures (1985) 295-318
- [3] Belbin, G.R. : *Thermoplastic structural composites a challenging opportunity*, Materials and Design Vol.6 no. 1 Feb./March 1985 9-17
- [4] Poursatip, A., Ashby, M.F. and Beaumont, P.W.R. : *The fatigue damage mechanism of a carbon fibre composite laminate, 1 - Development of the model*, Composite Science and Technology 25 (1986) 193-218
- [5] Kim, R.Y. and Aoki, R.M. : *Transverse cracking and delamination in composite materials*, Fibre Science and Technology 18 (1983) 203-216
- [6] Burk, R.C. : *Standard failure criteria needed for advanced composites*, Aeronautics and Astronautics June 1983 58-62
- [7] Hashin, Z. : *Failure criteria for unidirectional fiber composites*, Journal of Applied Mechanics Vol. 47 June 1980 329-334
- [8] McLaughlin, P.V.Jr and Ingle, A.A. : *Analysis of damage propagation and ultimate failure in laminated composite flywheels*, SAE paper SAE/P-85/164 1985

- [9] Picasso, B. and Priolo, P. : *Influence of the stacking sequence and load direction on the fatigue behaviour of quasi-isotropic graphite-peek laminates*, Proc. 5th Int. Conf. on Composite Structures, July 1989 671-677
- [10] Tsai, S.W. and Hahn, H.T. : *Introduction to composite materials*, Technomic Publ. Co., 1980
- [11] Sun, C.T. and Zhou, S.G. : *Failure of quasi-isotropic composite laminates with free-edges*, Journal of Reinforced Plastics and Composites Vol. 7 - Nov. 1988 515-557