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Properties of cement composites reinforced with Kevlar fibres

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The organic polyamide fibre, Kevlar, is promising as an efficient reinforcement for cementitious matrices. For cement boards, in which chopped fibres are distributed randomly in two dimensions, typical mechanical properties obtained with ~ 1.9 vol% fibre addition are as follows: ultimate tensile strength (UTS) 16 MN m^{-2}; MOR 44 MN m^{-2}; impact strength 17 kJ m^{-2}. The composite material can be produced by autoclaving if desired and at ambient temperatures they are expected to be durable in most environments. The relatively low decomposition point of Kevlar (as opposed to glass fibres or steel) is a disadvantage for its use in building components which may come into contact with high temperatures, as in a fire. It should be noted that a solvent which is used in the manufacture of the fibre and remains in the fibre in minute quantities has been found to produce cancer in rats. There is no evidence of it causing cancer in humans but the significance of this in terms of a possible health risk, if any, will need to be assessed by the appropriate medical authorities in relation to any applications.

1. Introduction

Brittle-matrix composites in general, and fibre-reinforced cement and concrete in particular, have received a great deal of attention lately. Although it has not been possible to incorporate more than say, 5 vol% fibre in practical cement composites by traditional means, useful improvements in several physical and mechanical properties have been recorded as opposed to those of the unreinforced matrix and this has formed the basis for the commercial interest shown in cement products reinforced by fibres such as glass, steel or polypropylene [1].

With all these fibres, even at small additions, the cement composite exhibits a form of ductility which results from fibre pull-outs, imparting to the material a degree of toughness it would not otherwise possess. If the fibres are strong and present in sufficient numbers to bear the load, useful gain in the tensile and bending strength is also achieved by composite action although, in some instances, the degree of retention of these improvements depends critically on the environment in which the composite is used [2].

Of all the fibres that have been tried as reinforcement for cement and concrete, carbon fibres probably best combine the advantages of high strength and stiffness, and chemical inertness. The new organic fibres based on aromatic amide polymers and marketed under various proprietary names (for example, Kevlar by Dupont) appear to possess these qualities also but the data on their performance in a cement matrix are rather scant. A programme of work aimed at examining the prospects of these fibres in cement has been continuing [3] at BRS for some time and a summary of the results obtained so far is presented in this paper.

2. Materials

Ordinary Portland Cement (OPC) passing the British Standard BS12 was used throughout in the production of composite boards. In some cases (for example, mixes which were autoclaved) 30% by weight of the cement was replaced by sand.

Kevlar 49 fibres were supplied by the manufacturer, Dupont de Nemours International SA, as continuous roving, nominally of 5067 dtex (g per 10 000 m). The roving contained about 3300 filaments, each $11.7 \pm 0.18\,\mu m$ in diameter. The properties of the yarn quoted by the manufacturer are: tensile strength $2900\,MN\,m^{-2}$, Youngs modulus $130\,GN\,m^{-2}$, elongation at break 2.6% and density $1.45\,g\,cm^{-3}$.

Very recently it has been discovered by the manufacturer that one of the constituents used in the production of Kevlar fibres is carcinogenic to rats. The manufacturer has stated* that the amount of this component remaining in the fibre is minute and that use of the fibre is not hazardous to human health if proper ventilation is provided during processing. Confirmation of health safety will be needed before the material is used in buildings.

3. Experimental

3.1. Fibre properties

The ultimate tensile strength (UTS) of the fibres was determined at room temperature (23° C) and also at several higher temperatures up to 200° C using an environmental chamber. In many civil engineering applications temperatures much higher than the ambient are sometimes encountered (for example, in fire) and if fibre reinforcements are to have structural possibilities they must withstand at least reasonable temperatures effectively.

To facilitate handling and testing of the individual Kevlar fibres they were fixed to paper mounts with an "Araldite" epoxy adhesive. The mounts were 70 mm x 13 mm with a central slot 40 mm x 4 mm. The fixing points were at each end of the slot which accurately determined the gauge length. Tensile tests were done on an Instron universal testing machine. One end of the paper mount was clamped by a steel jaw fixed to the crosshead of the machine and the other end was held in a spring steel clip suspended from the 50 g load cell. The whole of the paper mount and the clamps were enclosed within a Montford environmental chamber mounted on the crosshead. When the paper mounts were positioned in the clamps they were cut between the fibre mounting points. After the environmental chamber was sealed and before the start of the test, a period of 1 min was allowed for the temperature of the fibres and the chamber to reach equilibrium. Tensile tests were carried out with crosshead speeds in the range 0.005–$5\,cm\,min^{-1}$. Traces of load against time were obtained, from which the ultimate stress, ultimate strain and Young's modulus were calculated. In calculating the strains an allowance was made for the deflection of the load cell and clamping arrangement by calibration with a paper mount in the jaws which had not been cut. Stress relaxation in the fibre at 200° C was studied by holding the strain constant at about 1.2% for 1 h during which time the load fell to a stable value. The strain was then increased to about 1.7% and again held constant for 1 h. The strain was then reduced until the load fell to zero, subsequently the fibre was strained to failure in the usual way.

3.2. Composite fabrication

The composites containing Kevlar fibres were produced using a method similar to the spray–suction technique developed at BRE [4]. In this method an atomized slurry of cement and an air stream containing the 51 mm long fibres were directed simultaneously on to the flat surface of a mould 4 m long and 1 m wide. The spray head traversed the mould to cover it uniformly with the mixture of slurry and fibres to a depth of about 10 mm. The composite sheet was then de-watered and trowelled flat. The Kevlar fibres were cut with a chopper supplied by John Laing Res. and Dev. Ltd. This was found to be satisfactory provided the cutter was re-ground to a high precision fit before the manufacture of each board. Boards were usually kept damp under wet sacking for a period of 7 days after manufacture during which time they were cut into test coupons 150 mm long by 50 mm wide. After this curing period the coupons were randomized into four lots which were placed in the four storage conditions of air at 40% RH and 20° C, under water at 20° C and 60° C and natural weather on the exposure site at Garston. After predetermined periods of time samples were withdrawn and tested in tension, bending and for impact resistance.

One board was made from a slurry containing sand and cement in the ratio 30:70. Half of this was given a normal 28 day damp cure and the other half was autoclaved for 16 h at 180° C.

A fire test panel, 1 m square, was made from a slurry of pulverised fuel ash (pfa) and cement in the ratio 40:60. This was given a normal 28 day cure and then allowed to dry out for 6 days before being sent to the Fire Research Station for test.

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* Circular letter of 24 March 1976 signed by Prem P. Singh of Dupont De Nemours International SA 50-52 Route des Acacias, CH-1211 Geneva 24, Switzerland.

3.3. Testing the composites

Tensile tests were carried out on an Instron 100 000 N universal testing machine using strain-rate control and Instron data recording system 2415 enabling computer analysis of the load–strain data. The coupons were clamped in hydraulically operated jaws with flat serrated faces. An extensometer was fitted to the coupons which measured the strain on each side independently. The signals from this were averaged electronically and fed to the data recording system, the standard Instron chart recorder and the servo system controlling the strain rate. After the tests the data was analysed to determine the stress, strain and Young's modulus at the limit of proportionality (LOP) and the ultimate tensile stress (UTS) and the corresponding strain.

Bending tests were carried out on an Instron 50 000 N machine using a constant rate of crosshead displacement. A four-point bending jig was used, the distance between the lower outer rollers was 135 mm and the upper rollers were 45 mm apart. The load–crosshead displacement traces obtained from a chart recorder were analysed for stress, strain and Young's modulus at the LOP and for ultimate stress attained using elastic beam theory. The impact strength was determined using the Izod pendulum method, the maximum energy of the pendulum being 12.5 J. The energy absorbed in the test was normalized by dividing it by the cross-sectional area of the test piece.

Four normally cured coupons and one which had been kept in water at 60° C were subjected to cyclic loading in tension using the same equipment as the tensile tests but without the data recording system. As the peak strain was increased periodically by increments of about 1000 microstrain, the test piece was unloaded by reversing the strain rate until the load was nearly zero. This produced stress–strain traces similar to those described by Allen [5]. From these traces the residual strain and the stiffness of the material associated with each peak strain were obtained.

The response of the composite to fatigue loading in flexure was examined with the aid of a multiple-specimen loading rig developed at BRE [6] capable of testing up to 16 specimens simultaneously in four-point bending, each specimen bearing the same load. The load was made to vary sinusoidally from practically zero to a predetermined maximum at a rate of 3 cycles $\sec^{-1}$. The number of cycles undergone by each specimen when it failed was logged and a probability of failure, P, assigned to it. P was determined by $P_i = i/(n+1)$ where i is the ith specimen to fail and n is the number of specimens tested (usually 16). The number of cycles for a 50% probability of failure was obtained from the best fit straight line drawn through the points on a log cycles to failure versus probability of failure plot. This was done at a number of stress levels and the results are presented here as the usual S–N curve (stress-number of cycles) for the 50% probability of failure. The technique is described in more detail in the reference cited [6].

4. Results

4.1. Fibre properties

The stress–strain behaviour of Kevlar fibres was linear up to failure, within the range of temperature and strain rate used in the experiments, except for a small region at low stress (which is thought to be due to non-linear behaviour of the coupling to the load cell). The two stress–strain traces in Fig. 1 are typical and they form an envelope for all the other traces. The measured tensile properties showed little or no dependence on strain rate at 23° C (Fig. 2) but a tendency to increase with increasing strain rate at 200° C. The fibres tested at constant strain rate showed a fall in strength and stiffness as the temperature was increased (Fig. 3) while the failure strain remained constant.

The straining sequence in the stress relaxation experiment at 200° C is shown in the stress–strain trace of Fig. 4. Although the strain was held for 1 h in each case (i.e., at 1.2% and 1.7% respectively), the stress relaxation took place within the first 15 min. The ultimate stress and strain of the fibre were not significantly reduced by the 2 h period at 200° C.

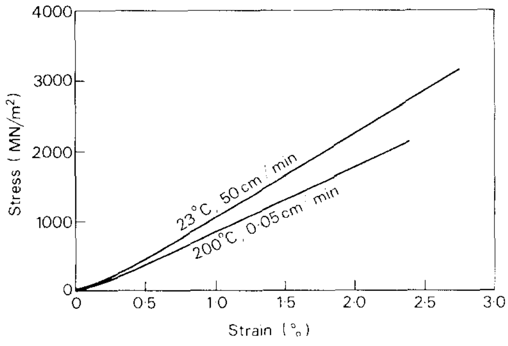
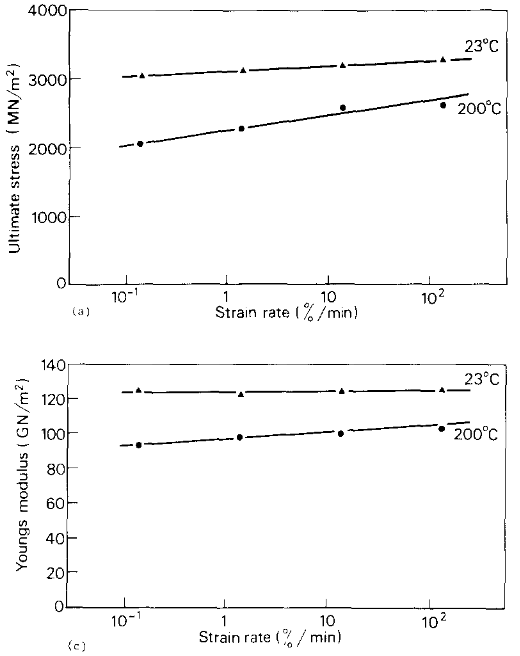


Figure 1 Stress–strain curves of Kevlar fibre at 23° C and 200° C.



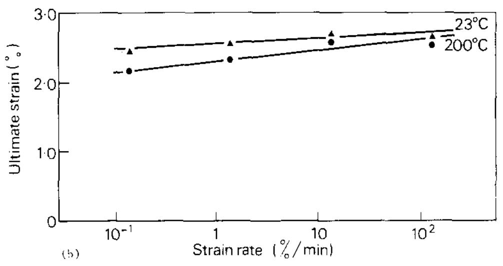


Figure 2 Variations of the tensile properties of Kevlar fibre with strain rate. (a) Ultimate stress, (b) ultimate strain and (c) Young's modulus.

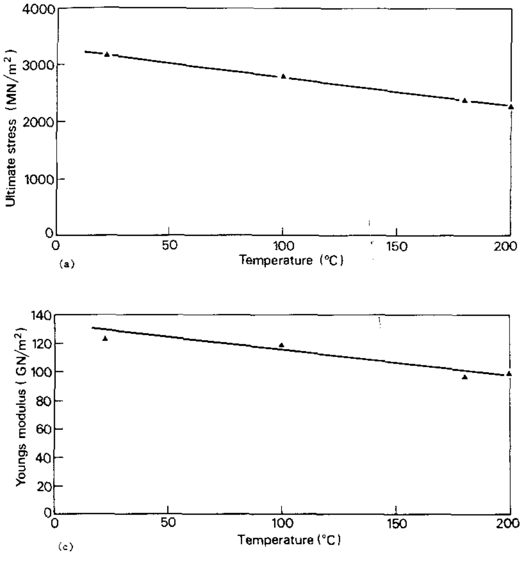
4.2. Composite properties

Some typical tensile stress–strain traces are shown in Figs. 5 and 6. These display, in common with many brittle fibre–brittle matrix systems, the now familiar form of a linear elastic region associated with the matrix followed by a region of multiple cracking and finally a linear region up to failure associated with the fibres. The analysis of this type of behaviour has been adequately dealt with by authors such as Allen [5] and Aveston, Cooper and Kelly [7].

Typical bending traces are shown in Figs. 7 and 8. The tensile, bending and impact properties obtained from normally-cured Kevlar–OPC composites containing 1.93 vol% of fibres and an autoclaved Kevlar–OPC–sand composite containing 1.78 vol% of fibres are summarized in Table I. Considerable variations were observed in the results due to non-uniform distribution of the fibres in the matrix. Six replications of each tensile and bending test were usually made and twelve for the impact tests. The standard deviation of the results was normally in the range of 10–15% of the mean for the former and 15–25% for the latter. As a consequence of this variation, differences of up to 15% between values are not statistically significant.

Typical stress–strain curves obtained during cyclic loading of Kevlar–OPC composites are depicted in Fig. 9. Cyclic loading in tension enables the stiffness (E) of the composite and any residual strain or "permanent set" (ϵ_r) to be determined as the tensile test progresses. Both these features are functions of the maximum strain reached in the test which is clearly shown in Figs. 10–13 where E and ϵ_r have been plotted against peak strain. There is no change in E and ϵ_r from

Figure 3 Variations of the tensile properties of Kevlar fibre with temperature. (a) Ultimate stress, (b) ultimate strain and (c) Young's modulus.



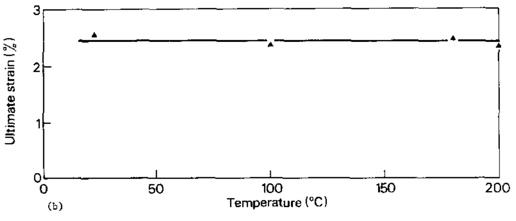


TABLE I Properties of Kevlar–cement composites

Curing conditions			Tensile properties					Bending properties				
			Ultimate		Limit of proportionality		Youngs modulus	Ultimate	Limit of proportionality		Youngs modulus	Impact strength
			Stress (MN m^{-2})	Strain (%)	Stress (MN m^{-2})	Strain (10^{-6})	(GN m^{-2})	Stress (MN m^{-2})	Stress (MN m^{-2})	Strain (10^{-6})	(GN m^{-2})	(kJ m^{-2})
Normal temperatures	Water	28 d	16.1	1.53	8.86	318	27.9	44.4	16.1	891	20.0	17.0
		180 d	15.0	1.28	9.23	252	37.1	44.4	16.3	773	21.5	14.7
		2 yrs	13.6	1.08	7.10	210	33.9	43.5	17.7	850	22.4	12.0
	Air	180 d	14.4	1.79	7.26	265	27.5	46.7	12.6	853	15.4	17.6
		2 yrs	14.8	1.69	3.82	167	24.1	45.4	9.63	587	17.5	22.0
	Weather	2 yrs	14.4	1.40	4.73	168	28.3	42.3	15.7	768	22.1	14.1
High temperatures	Water 60° C	7 d	14.7	1.24	8.87	258	34.1	39.5	13.2	713	18.8	17.1
		50 d	16.5	1.26	7.21	230	31.4	41.5	12.8	785	16.0	12.5
		180 d	12.3	1.11	6.28	185	33.9	38.2	15.9	710	22.9	11.0
	Air 150° C	7 d	13.1	1.69	7.41	348	23.0	34.4	13.7	1300	11.5	14.9
		45 d	12.1	1.91	3.68	252	16.1	37.6	15.8	964	16.6	20.1
Autoclaved 16 h 180° C			9.42	1.14	5.57	212	27.5	24.9	13.2	1290	10.6	15.9
Control-normal cure			13.4	1.41	7.65	283	27.1	36.4	12.0	883	13.7	23.0

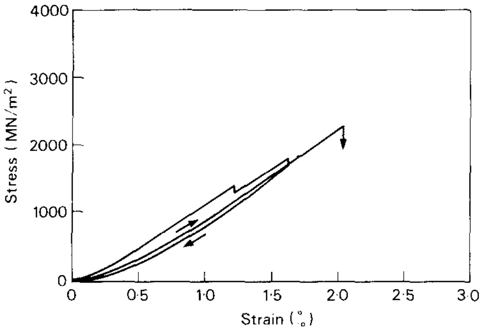


Figure 4 Stress relaxation of Kevlar fibre at 200° C. The fibre was held at 1.2% constant strain for 1 h before being strained to 1.7%.

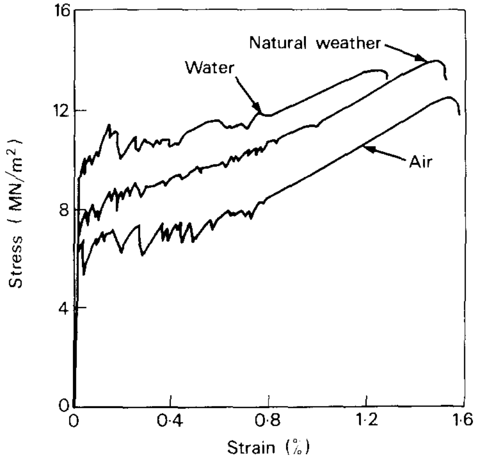


Figure 5 Tensile stress–strain traces for Kevlar–OPC composite after 2 years storage in air, water and natural weather.

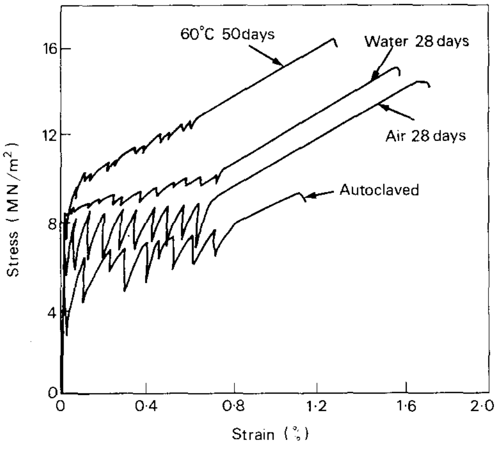


Figure 6 Tensile stress–strain traces of Kevlar–OPC composite for different curing conditions.

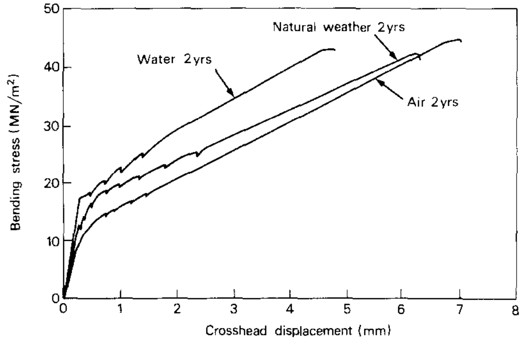


Figure 7 Bending traces of Kevlar–OPC composites after 2 years storage in air, water and natural weathering.

zero up to the elastic limit strain of about 200–300 microstrain. As the strain is increased, ϵ_r increases almost linearly while E falls rapidly at first and then more slowly to about 5% of its initial value. This type of behaviour is predicted by Allen [5] for brittle-matrix composites where the matrix is cracked.

The fatigue data were analysed in the manner described by Hibbert and Grimer [6]. Fig. 14 shows the S–N plot for 50% probability of failure (mean life) obtained with Kevlar–cement composite samples containing 2.34 vol% of fibres. The number of cycles for 10% probability of failure were generally an order of magnitude lower than the mean life, reflecting the variability found in the static bending tests. The results show the material to be quite resistant to fatigue even at stresses well above the elastic limit where the matrix is cracked. No failures were recorded for samples stressed below the elastic limit of 15 MN m^{-2} after 10^6 cycles.

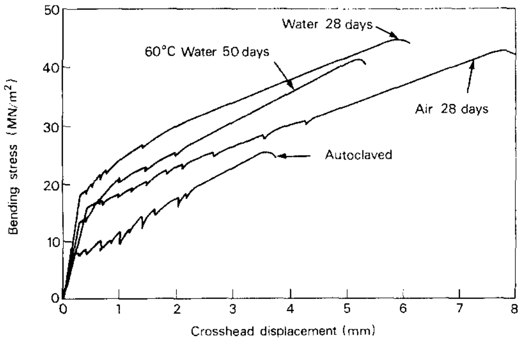


Figure 8 Bending traces of Kevlar–OPC composites for different curing conditions.

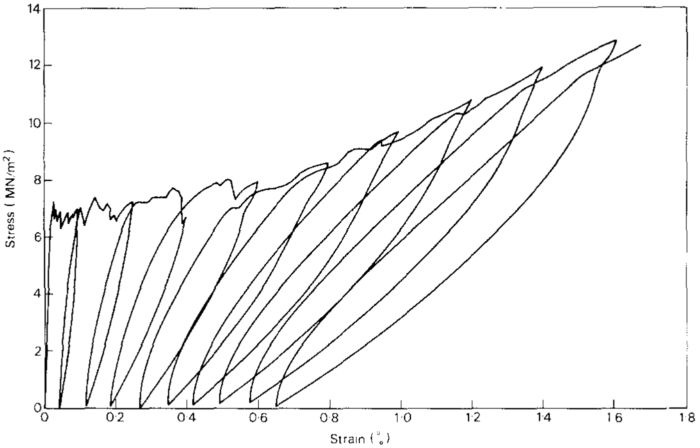


Figure 9 Cyclic loading of a Kevlar–OPC composite.

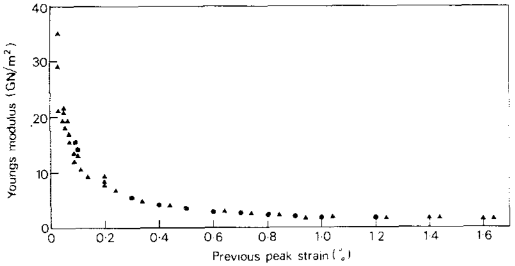


Figure 10 Stiffness versus peak strain for normally-cured Kevlar–OPC composites for 4 samples. ▲ individual results; ● coincident results.

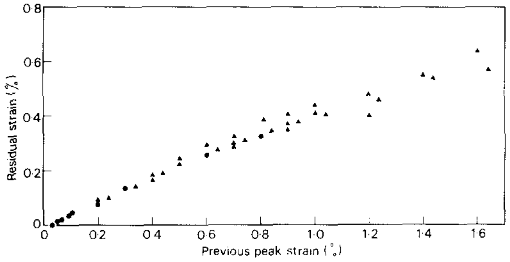


Figure 12 Residual strain versus peak strain for normally-cured Kevlar–OPC composites for 4 samples. ▲ individual results; ● coincident results.

4.3. Fire test

In the fire test one side of the panel was heated to a maximum temperature of 920° C while the other side was left open to ambient air. During the first 15 min water was driven off as steam causing delamination in patches and some surface cracking which did not penetrate through the thickness. The test was allowed to continue for 1 h when no more delamination or cracking was found to occur and the panel retained its integrity. An examination of the panel after the test showed that most of the fibres had completely charred but those nearest to the cooler surface (which had reached a maximum temperature of about 400° C) had not charred and still had sufficient strength to allow handling of the panel without disintegration. During the fire test the air in the vicinity of the panel was monitored for toxic gases but none were detected.

5. Discussion

5.1. Fibre properties

The UTS, *E* and strain to failure values of Kevlar fibres at room temperature and at higher temperatures are in accordance with the values expected from the manufacturers published data. The fibres possess high strength and stiffness which are retained at elevated temperatures. The small amount of stress relaxation observed at 200° C

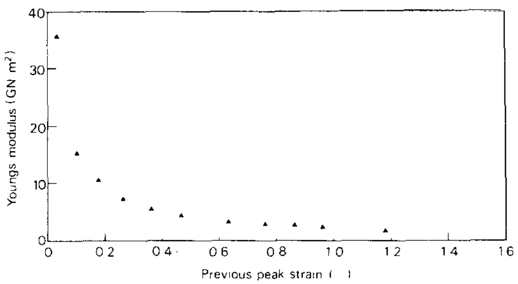


Figure 11 Stiffness versus peak strain for a Kevlar–OPC composite sample cured in water at 60° C for 60 days.

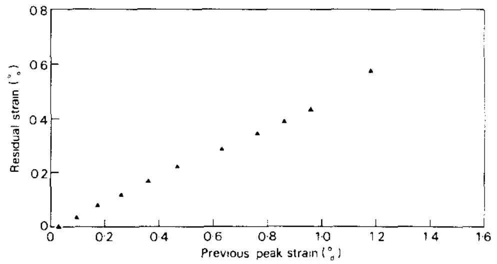


Figure 13 Residual strain versus peak strain for a Kevlar–OPC composite sample cured in water at 60° C for 60 days.

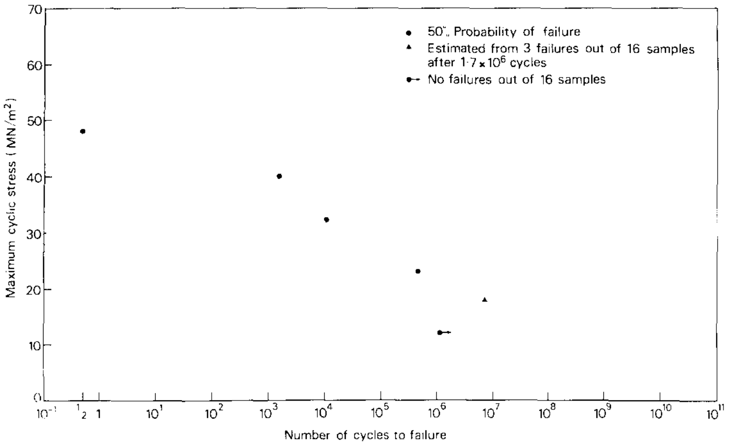


Figure 14 Fatigue life of Kevlar–OPC composites.

indicates viscoelastic behaviour that has an important bearing on the creep properties of the fibres and this is one aspect requiring further study. Bunsell [8] found a small amount of creep during the first 5 min after loading to 85% of the nominal breaking stress of the fibre at room temperature. Although no further measurable strain occurred the fibres usually broke after several hours loading. Stress-rupture (or static fatigue) of Kevlar fibre–epoxide strands has been determined at 80% and 90% of their ultimate strength by Chiao and Moore [9]. They give mean times to failure of 209 h and 1.0 h respectively. These results lead one to expect creep and static fatigue effects in Kevlar–OPC composites as they are stressed above the elastic limit when the matrix is cracked and the fibres carry most of the load.

5.2. Fabrication

The spray–suction technique was largely successful in producing Kevlar–OPC composite in sheet form but there are two areas in which improvements are desirable. A greater degree of separation of the chopped strands into filaments would be a great advantage and a more uniform distribution of the fibres both in the plane of the sheet and throughout its thickness should result in a reduced variability of properties. A smooth transfer of stress to the fibres in the multiple-cracking region could perhaps invite structural possibilities in special areas.

5.3. Composite properties

The tensile behaviour shown in Fig. 6 of the normally-cured composite may be anticipated from the mixture law and theories of multiply cracked brittle matrix–fibre systems. The UTS is about 70% of the expected value. This would indicate either poor stress transfer to the fibres or loss of fibre strength. In view of the difficulties experienced in obtaining uniform distribution of the fibres, poor stress transfer seems the more probable explanation and improved fabrication may well result in an appreciable increase in UTS.

The durability of the material has been assessed for a period up to 2 years at ambient temperatures in various environments. The ultimate strength in tension and bending is unchanged while air storage has caused an increase in strain to failure and impact strength and a decrease in the LOP stress. Water storage has had the opposite effect of decreasing strain to failure and impact strength. These changes are compatible with anticipated changes in the matrix and in increasing bond strength in water storage and a decreasing bond strength in air storage while the fibre properties remain constant. Materials stored in water at 60° C for 180 days behaved in a way very similar to that of the water-stored material at 20° C. Air storage at 150° C caused some loss of strength and an increased failure strain. This behaviour can again be attributed to changes in the matrix and bond strength.

The tensile, bending and impact strengths of the autoclaved material were 70% of the normally-cured control board. This suggests that the fibres have lost some strength during the autoclaving process.

These measured properties demonstrate that Kevlar–OPC composites can be produced with good strength and ductility and one may expect them to be retained even after long periods in adverse environments.

5.4. Cyclic tension

The analysis of the cyclic loading tests (Figs. 10–13) clearly shows the marked loss in elastic modulus and the large residual strains which occur during the multiple cracking region and beyond. The similarity in the graphs of the material normally cured, and cured at 60° C (which do not have a similar stress–strain response) would indicate that E and ϵ_r are functions of the peak strain only, a conclusion also drawn by Allen [5]. These characteristics will act as a severe constraint on the utilization of the full strength and toughness of the composite.

5.5. Fatigue

When cycled above the elastic limit the matrix is cracked and very little is known about the processes occurring during cyclic loading, either in the region of the crack or at the fibre–matrix interface. The bending deflection of the fatigue samples remained constant until just before failure suggesting that no further debonding or pull-out of the fibres occurs after the first cycle. The results show that fatigue is not a critical consideration for the use of the material even at stresses above the elastic limit.

5.6. Fire resistance

The standard specifications of different countries for fire tests stipulate that building components (for example, panels) of certain dimensions be tested. What has been done in the present study can therefore be only described as an indicative fire test. As Kevlar fibres are organic and therefore decompose at a low temperature relative to inorganic or metal fibres, such as glass or steel wires, it is to be expected that a large proportion of the fibre will actually be destroyed in a fire situation. However the fibres in the tested boards farthest away from the highest temperature did not show any significant signs of decomposition. They were obviously reasonably strong and helped the board to retain a degree of integrity after the 1 h fire test.

It is also important to note that in the fire test Kevlar–OPC board did not break up with explosive violence which is the usual feature of cement composites that are brittle. Tests on individual fibres have shown that at least up to 200° C, Kevlar remains fairly strong and its bonding to cement perhaps does not increase very significantly with increasing temperature so that the pseudo-ductility of the composite is not much affected during the initial period of the fire test. The escape of steam caused delamination in the present case but this can possibly be eradicated by better mix design and fabrication.

6. Conclusions

The mechanical properties of Kevlar–cement composites reported in this paper are sufficiently attractive to warrant further studies. The durability of these composites is likely to be very good in most environments. Even in a fire, panels made from such a composite material may not be unsatisfactory but full scale fire tests must be performed on several components before the use of Kevlar in buildings can be recommended. It should also be pointed out that before these fibres are used in composite materials or building, it should be shown beyond any reasonable doubt that this use is not hazardous to health.

Acknowledgement

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