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Fatigue evaluation and dynamic mechanical thermal analysis of sisal fibre – thermosetting resin composites

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Abstract: Sisal composites were manufactured in a hot press from asreceived and 0.06M NaOH treated sisal fibres with polyester and epoxy resin matrices. Tensile tests were conducted on the composites to establish loading levels for fatigue testing. A fatigue evaluation of the sisal fibrethermosetting resin composites was undertaken at loading levels of 75%, 60%, 50% and 35% of static tensile strength and at an R ratio of 0.1. S-N curves for the composites are presented for untreated and 0.06M NaOH treated sisal fibres. Epoxy matrix composites have a longer fatigue life than polyester matrix composites. The effect of chemical treatment on fatigue life is significantly positive for polyester matrix composites but has much less influence on the fatigue life of epoxy matrix composites. Dynamic Mechanical Thermal Analysis (DMTA) was conducted on samples from failed epoxy resin fatigue specimens and the influence of fatigue history on the tan δ peak temperatures and T_g of the composites is examined. Significant shifts in T_g are observed following fatigue testing. The fatigue performance of natural fibre composites suggests that they are suitable for

use in dynamically loaded structures and may be used as a substitute for Glass Fibre Reinforced Plastic composites in fatigue.

Key words: Fibres, interfacial strength, sisal, DMTA, fatigue

1. INTRODUCTION

The subject of fatigue failure in composites was given serious thought from the earliest days of development. Although it was initially thought that composites did not suffer fatigue damage at stresses well below their static strength, normal and shear strains at the fibre/resin interface could cause deterioration leading to fatigue failure [1].

Composite materials made of unidirectionally oriented natural fibre bundles in a resin matrix are anisotropic and the fibre bundles have random cross sections (figure 1). However, at a macroscopic level they can be regarded as being homogeneous. Fatigue failure in conventional composites does not occur in a localized fashion but throughout the volume of the material. Damage in such composites takes the form of fibre breakage, matrix cracking, debonding, transverse ply cracking, and delamination, depending on the method used in manufacturing the composite [2]. These processes occur sometimes interactively or independently. Testing conditions and material variables may sometimes influence the predominance of one mechanism over another. One aim of the work presented here is to evaluate fatigue damage mechanisms for natural fibre composites.

Composite materials have greater variability in fatigue response as compared to metals. Carrying out more replicate tests at fewer stress levels has been found to be more satisfactory since it provides statistical information at each stress level and also provides probability/stress/life curves in addition to median-life or mean-life curves [1]. Most of the fatigue studies on composites have concentrated on synthetic fibre/resin composites and fatigue studies on natural fibre composites are relatively new. Due to the current strong interest in natural fibre composites, a study of their fatigue properties is timely. The static properties of natural fibre composites are often variable so fatigue lives are also likely to be highly variable.

Gassan [3] conducted a study on the fatigue behaviour of composites made of flax and jute yarns and epoxy, polyester and polypropylene resin matrices under load controlled mode with step-wise loading increments. A comparison between unidirectional fibre reinforced composites and woven fibre reinforced composite found that the former were less sensitive to fatigue induced damage. A study by Thwe and Liao [4] on the fatigue of bamboo fibre reinforced polypropylene composites (BFRP) and bamboo-glass fibre reinforced polypropylene hybrid composites (BGRP) showed that the latter had a better fatigue resistance than the former at all load levels. The composites were loaded cyclically at 35, 50, 65, and 80% of their ultimate tensile stress. S-N curves were used to compare the fatigue resistance of the two composites.

The use of sisal as a reinforcement in polymer matrices has been studied over the past few decades by several researchers. Investigations on the mechanical, rheological, electrical and viscoelastic properties of short fibre reinforced LDPE composites as a

function of processing method, fibre content, fibre length and fibre orientation were conducted by Joseph et al [5-8]. Bisanda and Ansell [9] reported on the effect of silane and alkali treatment on mechanical and physical properties of sisal-epoxy composites.

The objective of this research is to evaluate the effect of fibre surface treatment on the fatigue life of sisal fibre/synthetic resin composites. The composites evaluated were made up of sisal fibres in as received and chemically treated states. Sisal fibres were treated using a 0.06M NaOH solution regarded as an optimum concentration by Mwaikambo and Ansell [10]. Prior to evaluation of fatigue properties, the shear strength of the fibre to resin interface was studied using the droplet pull out test [11] to confirm the positive effect of chemical treatment.

A second aim is to evaluate changes in the dynamic response of natural fibre composites following fatigue loading by Dynamic Mechanical Thermal Analysis (DMTA). Changes in storage modulus and loss tangent are expected to reflect the state of fatigue damage.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

The grade CRYSTIC 2-406PA polyester resin and Strand M catalyst were supplied by Scott Bader Company, UK. Epoxy resin Araldite LY5052 and Araldite hardener H5052 were supplied by Aeropia Limited of the UK. NaOH solution was supplied as a general laboratory reagent.

Sisal fibre obtained from Tanzania was used to manufacture unidirectional composites using the two resins. The fibres were used in as received and treated states with 0.06M solution of NaOH being used for surface treatment.

2.2 Fabrication of Composites

The composites were prepared by moulding in a hot press. Each composite specimen contained 12 g of sisal fibres that were divided into three layers of four grams each prior to introduction into the mould. This ensured proper wetting of the fibres when the resin was poured onto the individual layers in sequence. Preparation of the polyester composites involved mould curing at a pressure of 60 bars and temperature of 50° C for 20 minutes followed by post curing overnight at 80° C. Epoxy resin composites were prepared by mould curing at a pressure of 60 bars and temperature of 80° C for 20 minutes. Epoxy matrix samples were cured at $23\pm1^{\circ}$ C for 23 hours followed by curing at 100° C for 4 hrs. The average fibre volume fraction for all composites was determined using the equation

$$v_f = 1 - \frac{\left(m_c - m_f\right)}{\rho_m V_c}$$

where v_f is the fibre volume fraction, m_c is the mass of the composite, m_f is the mass of fibres, ρ_h is the density of the matrix and V_c is the volume of the composite. The average volume fractions for untreated and treated fibre polyester resin composites were 68.2% and 64.4% respectively with standard deviations of 3.2% and 1.9% respectively. Average volume fractions for the untreated and treated fibre epoxy resin composites

were 71.5% and 68.5% respectively with standard deviations of 2.5% and 1.5% respectively.

2.3 Mechanical Properties

2.3.1. Static Tests

To establish the stress levels for fatigue testing, the static tensile strengths of the different composites were measured using EN ISO 527-5:1997 [12]. The dimensions of the tensile test specimens are shown in Figure 2.

The tensile strengths obtained were used as a basis for the determination of the maximum loads used at each load level for the four types of composites tested in fatigue. A minimum of four samples was tested for each composite formulation. To obtain the elastic modulus of the composites, an extensometer with a gauge length of 25mm was attached to the specimen as shown in Figure 3.

2.3.2. Fatigue Tests

The fatigue tests were performed according to BS ISO 13003:2003 [13] with some modification of specimen thickness and width because of the coarse nature of sisal fibre bundles ($100 - 200\mu m$). The tests were at an R ratio of 0.1 at four different stress levels. These were 75%, 60%, 50% and 35% of the tensile strength of the composites. Four specimens were tested at each stress level. Specimens were manufactured in a mould that produced the exact size required for the test and continuous fibre bundles were retained within the specimen with no cut edges. This technique markedly reduces

variability in fatigue life. Testing was performed under load control at constant stress (load) amplitude in the tension-tension mode.

A loading rate (or rate of stress application, RSA) of 200 MPa/s was used during the test. The selection of a constant rate of stress application (RSA) prevents hysteretic heating in the specimen, accelerates testing and eliminates rate effects. The machine used for fatigue testing was a 200KN capacity Denison–Mayes test machine. Testing was continued until failure of the composite was observed.

The regression lines for the S-Log N plots were obtained by plotting the Log N values as the dependent variable on the vertical axis and the maximum stresses as independent variable on the horizontal axis. The regression values were plotted with the experimental data on the conventional S-Log N system of axes. This approach was used because the number of cycles is the dependent variable in the analysis of fatigue data.

2.4. Dynamic Mechanical Thermal Analysis

Dynamic mechanical thermal analysis of the composites that had been subjected to fatigue testing was conducted using a TRITEC 2000 DMTA and results were compared with unfatigued specimens. The samples were obtained from regions of the composites that had not suffered visible damage after fatigue failure. Testing was conducted in single cantilever mode at a frequency of 1 Hz and temperature ramp rate of 2° C/min from -50°C to a maximum temperature of 180° C. The minimum temperature was chosen to be well below the glass transition temperature, T_g, of the resin matrix whilst

the maximum temperature was selected due to the deterioration of sisal fibres at higher temperatures. Figure 4 shows the tangent construction method used for determination of glass transition temperature from the storage modulus versus temperature characteristic. The tan δ peak temperature is also indicated.

The sample shown is an unfatigued sisal epoxy composite presented by way of example.

3. **RESULTS**

3.1 Static Tests

Table 1 presents data for the tensile strength of the untreated and treated sisal fibre composites. Treatment of the fibres using a 0.06M NaOH solution has increased the mean tensile strength of the polyester resin composites from 222.6 to 286.0 MPa, an increase of 28.5%. In contrast, the mean tensile strength for the epoxy resin composites has only increased from 329.8 to 335.4 MPa, an increase of 1.7%. These results show that treating the sisal fibres using 0.06M NaOH solution improves the tensile strength of the polyester resin composites but shows no significant improvement in the strength of the epoxy resin composites. These results were confirmed by performing a Student's t-test on the means of the tensile strengths of the chemically treated and untreated composites.

The high strength of the sisal fiber bundle/epoxy matrix composites offers strong potential for applications in engineering and construction.

3.2 Fatigue testing of polyester matrix sisal composites

Based on the static test results, maximum stress levels were applied to specimens in the range from 78 to 167 MPa for the untreated fibre composites corresponding to 35% to 75% of the static strength, and 100 to 215 MPa for the treated sisal fibre composites corresponding to 35% and 75% of the static strength (see Table 2) at an R ratio of 0.1.

Figure 5 presents S- Log N fatigue data for sisal fibre composites in a polyester resin matrix for both untreated and 0.06M NaOH treated sisal fibres at 75%, 60%, 50% and 35% of the static failure stress. The results show the scatter in the number of cycles to failure for up to four specimens that were tested at each stress level. The static strengths of the composites have been plotted at Log N = -0.3 (half a load cycle). The composites treated using 0.06M NaOH show an improvement in the load carrying capacity in fatigue. The fatigue lives of the NaOH treated composites are shorter at high loading levels as compared to those with untreated sisal fibres as shown in Table 2. As the loading level decreases, the two graphs merge, suggesting that at lower stress levels, the need for treating the fibres becomes unnecessary. Nevertheless, during normal operation, some components may only be subjected to low cycle fatigue and static loads. Hence, the use of treated fibre composites would be more beneficial due to the increase in load carrying capacity.

Figures 6 (a) and (b) show the type of failure observed for the untreated and treated sisal fibre polyester resin composites. The untreated fibre composites, figure 7(a), suffered severe damage to the fibres, with splitting and dislodging of resin being the most prominent failure mode. The damaged untreated fibre composite had a brushier

appearance than that of the damaged treated fibre composite. The treated fibre composites, figure 6(b), suffered splits and delamination coupled with explosive tensile failure in some areas, similar to that shown by the untreated fibre composites. The treatment of fibres creates better bonding between resin and fibre.

The brushy nature of failure in these composites prevented the evaluation of fatigued polyester resin matrix composites using the DMTA.

3.3 Fatigue testing of epoxy matrix sisal composites

In Table 3, the fatigue stress levels for the untreated and treated composites are close because their static strengths are similar. The stress levels for the untreated composites range from 115 to 247 MPa, corresponding to 35% to 75% of static strength, while the values for the treated composites range from 117 to 252 MPa, corresponding to 35% to 75% of static strength. This represents an increase in applied stress of only 1.7% and 2% for the lower and upper stress levels respectively. This is further reflected in the average cycles to failure, which do not show substantial differences between the two composites at the four stress levels selected.

Figure 7 presents S-Log N fatigue data for untreated and 0.06M NaOH treated sisal fibre composites. The static strengths of the composites have been plotted at Log N = -0.3 (half a load cycle). The treated fibre composites have a slightly higher load bearing capacity than the untreated ones, but this does not have a noticeable impact on the fatigue lives of the two types of composites. As reported by Towo and Ansell [14], micro-bond tests on chemically treated and untreated sisal fibre/epoxy resin composites

demonstrated that a strong bond was formed between the epoxy resin and untreated sisal fibres to the extent that fibres failed rather than the resin droplet shearing from the fibre bundle surface.

Figures 8 (a) and (b) show untreated and treated sisal fibre epoxy resin composites loaded at the 75% stress level following fatigue failure. The composites suffered stepped longitudinal fractures coupled with delamination and tensile failure. The nature of failure observed here is quite different to that of polyester resin composites that suffered more dramatic failures (Figure 6 (a) and (b)). This can be attributed to the stronger bond that is formed between the epoxy resin and sisal fibres, resulting in a more brittle, less fibrous failure mode.

Epoxy matrix composites behave predictably in fatigue and trends in behaviour are similar to those for GFRP and CFRP. Natural fibres are a viable alternative for synthetic fibres in fatigue applications.

3.4 Dynamic Mechanical Thermal Analysis results

Figures 9 and 10 present the storage modulus data for the untreated and 0.06M NaOH treated sisal fibre/epoxy resin composites at different load levels.

The storage modulus plots for the fatigued specimens show a shift to the left of the unfatigued specimen for the untreated sisal fibre epoxy matrix composites suggesting that there is a decrease in the glass transition temperature, T_g , of the untreated fibre composite following fatigue.

From Table 4, it can be seen that the glass transition temperature for the fatigued untreated fibre composite ranges from 75 to 82.5 $^{\circ}$ C while that for the fatigued treated fibre composites ranges from 106.25 to 115 $^{\circ}$ C.

The glass transition temperatures for the untreated and treated unfatigued fibre epoxy resin composites are 116.25 and 117.5 °C respectively, which represents no significant change. From the T_g values of the two composites, those for the fatigued untreated composites show a shift of between 11.54 and 41.25 °C from that of the un-fatigued composite. The T_g for the fatigued treated fibre composites shows a shift of between 2.5 to 11.25 °C, which is not as significant as that for the untreated fibre composites.

It can therefore be assumed that fatigue has modified the resin to fibre bundle interface to such and extent that the apparent glass transition temperature is shifted downwards.

Figures 11 and 12 present the tan δversus temperature data for untreated and 0.06M NaOH treated sisal fibre epoxy resin specimens obtained from composites that had failed due to fatigue loading at 75%, 60%, 50% and 35% loading levels. Figure 11 shows a shift of the tan δpeaks to the left in the untreated sisal fibre-epoxy resin composite. This is not present in Figure 10 where no shift in the tan δpeaks is evident for treated fibre composites.

The tan δ peak temperatures for the fatigued untreated fibre specimens are all lower than those for the un-fatigued specimens. This is likely to be due to damage at the fibre to

matrix interface. The internal friction is manifested at a lower temperature as a result of this damage. Untreated sisal fibres are reported to bond weakly with thermosetting resins, which will result in debonding between fibre and matrix, as the material is fatigued. Conversely, Figure 12 for treated fibre composites shows no significant change in tan &peak temperatures. This shows that treated fibres create composites that are resistant to structural changes when fatigued, regardless of the load levels. The DMTA results for tan & been able to show that though the fatigue lives of the untreated and treated composites do not show significant differences, the untreated fibre composites experience considerably more interfacial damage.

As seen in Table 5, the tan δ peak temperatures for the untreated sisal fibre composites range from 112.2 to 118.7 °C for the four stress levels, while for the treated fibre composites the tan δ peak temperature ranges from 128.8 to 135.6 °C for the same stress levels. The un-fatigued untreated and treated composites have similar tan δ peak temperature values of 134.1 and 133.9 °C respectively. This represents a decrease in tan δ peak temperatures for the untreated fibre composites of between 15.4 and 21.9 °C, while the treated fibre composites remained almost unchanged. There is evidently some change that has occurred in the state of the untreated composites that has resulted in a decrease in the tan δ peak temperature values that are closely related to the glass transition temperature or the composites. A likely explanation is the gradual weakening of the bond between the fibre and resin, as the material is fatigued. The better bonding expected in treated fibre composites is reflected by the small variation in peak tan δ temperatures of the treated fibre composites at all stress levels.

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4. CONCLUSIONS

- The results show that treatment of sisal fibres with 0.06M NaOH solution improves the tensile strength of sisal fibre polyester matrix composites but does not necessarily influence the fatigue lives especially at lower stress levels.
- Severe damage to the fibres, with splitting and loss of resin was the most prominent failure mode experienced by the polyester resin composites. It was not possible to subject these composites to DMTA tests.
- Sisal fibre epoxy matrix composites manufactured with treated fibres did not show a marked improvement in fatigue life over those with untreated fibres. This can be attributed to the strong bond formed between the epoxy resin and untreated sisal fibres.
- Sisal fibre epoxy matrix specimens failed in a brittle mode with some delamination failure and stepped longitudinal splitting.

DMTA analysis has shows that the treatment of epoxy resin composites with 0.06M NaOH solution does lower the glass transition temperature, T_g , of the composites. The glass transition temperatures of the untreated fibre composites, which suffered fatigue failure, decreased by an amount between 11.54 and 41.25 $^{\circ}$ C compared to the un-fatigued composites.

- DMTA analysis has shown that the tan δpeak temperatures of the untreated epoxy resin composites decreases by between 15.4 and 21.9°C following fatigue loading, while the treated fibre composites do not experience significant reduction in tan δpeak temperature values following fatigue.
- Changes in the T_g and tan δpeak temperature for untreated sisal epoxy composites following fatigue damage is not apparent from the S-Log N curves
- The behaviour of sisal fibre composites is similar to that of conventional synthetic fibre composites and static and fatigue strengths are suitably high for many commercial applications.

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Figure 1 Cross section of sisal fibres

Figure 2 Tensile test specimen

Figure 3 Tensile test of composites showing extensometer attached to the specimen

Figure 4 Determination of glass transition temperature, Tg, and tan δ peak temperatures from the storage modulus and tan δ plots for an unfatigued untreated sisal fibre epoxy resin composite.

Figure 5 Stress-life data for polyester resin composites at maximum stresses of 75%, 60%, 50% and 35% of failure stress at R=0.1. Arrow indicates run-outs.

Figure 6 Photographs of (a) ntreated fibre polyester resins composite at 50% loading level after 228,040 cycles, and (b) treated fibre polyester resin composite at 50% loading level after 45,038 cycles.

Figure 7 Stress-life data for epoxy resin composites at 75%, 60%, 50% and 35% of failure stress at R=0.1.

Figure 8 Photographs of (a) untreated fibre epoxy resin composite at 75% loading level failed after 873 cycles, and (b) treated fibre polyester resin composite at 75% loading level failed after 1917cycles

Figure 9 Storage modulus data for untreated sisal fibre-epoxy resin composites fatigued to failure at 75%, 60%, 50% and 35% of the mean static strength, including an un-fatigued specimen.

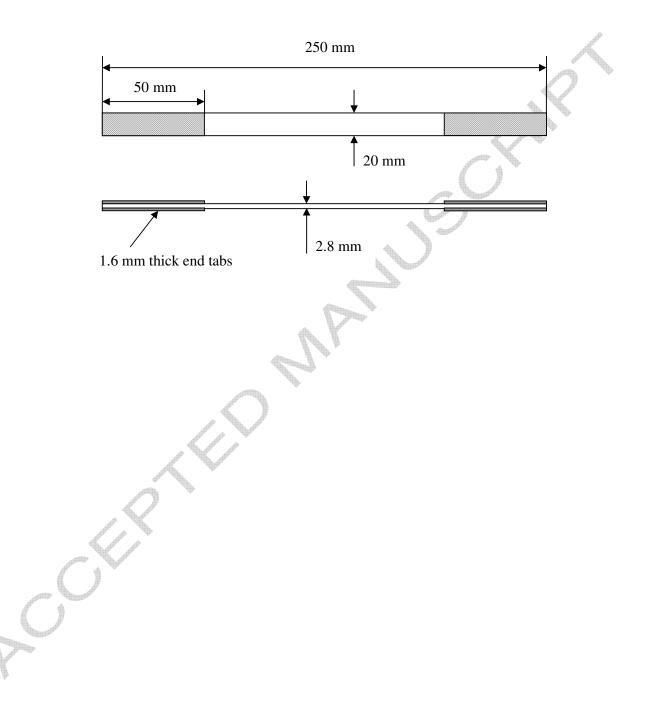
Figure 10 Storage modulus data for treated sisal fibre-epoxy resin composites fatigued to failure at 75%, 60%, 50% and 35% of the mean static strength, including an un-fatigued specimen.

Figure 11 Tan δdata for untreated sisal fibre epoxy resin composites at 75%, 60%,
50% and 35% of failure stress and un-fatigued.

Figure 12 Tan δ data for treated sisal fibre epoxy resin composites at 75%, 60%, 50% and 35% of failure stress and un-fatigued.

C)X

Figure 2





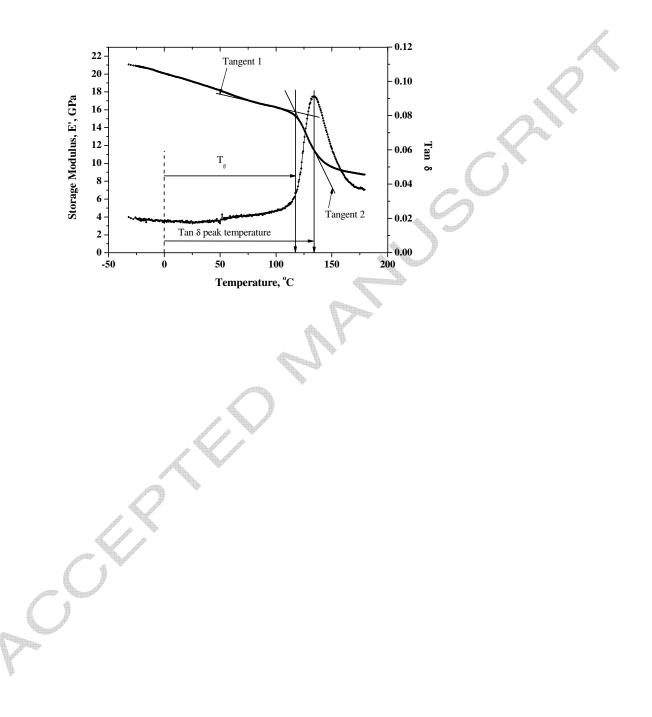
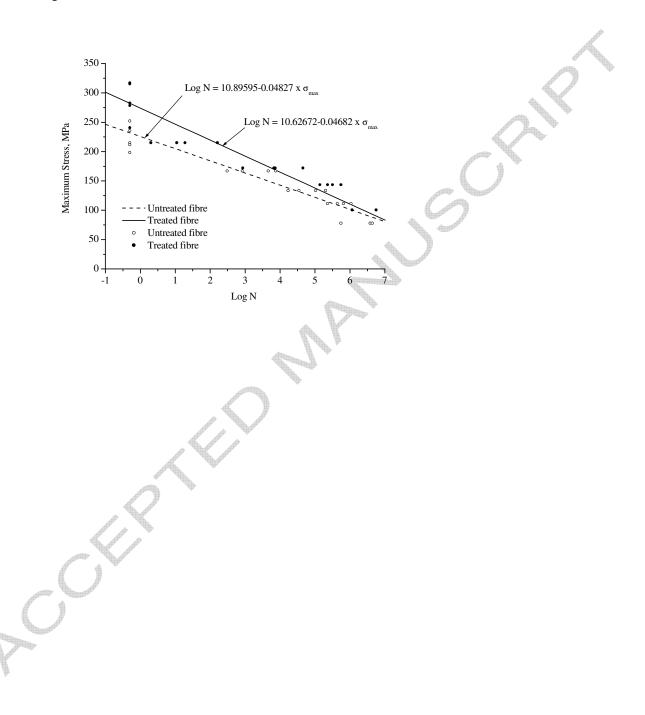
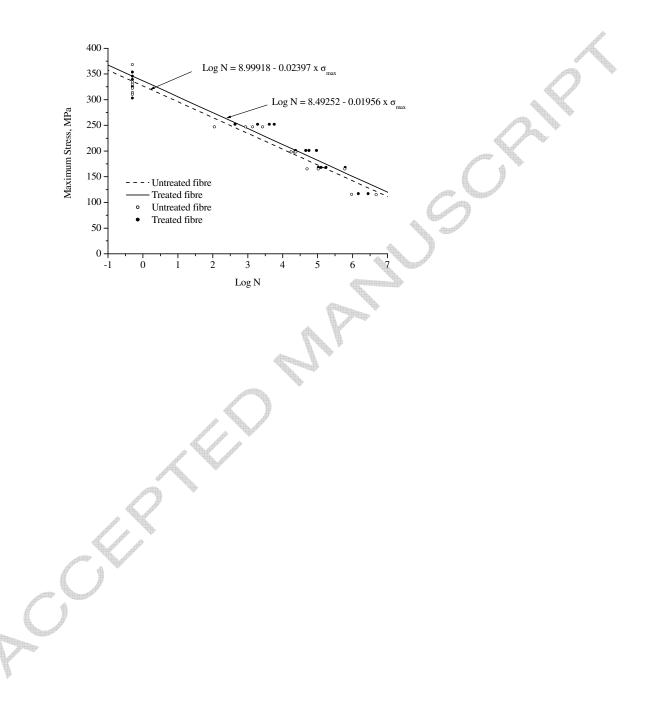


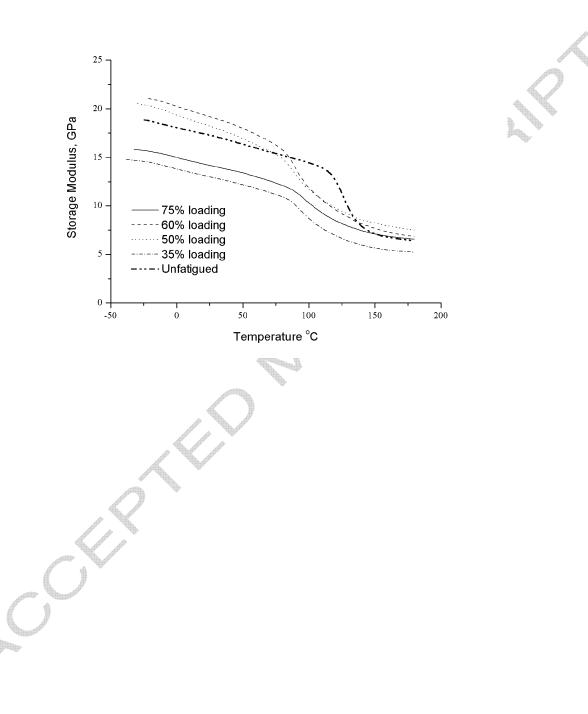
Figure 5



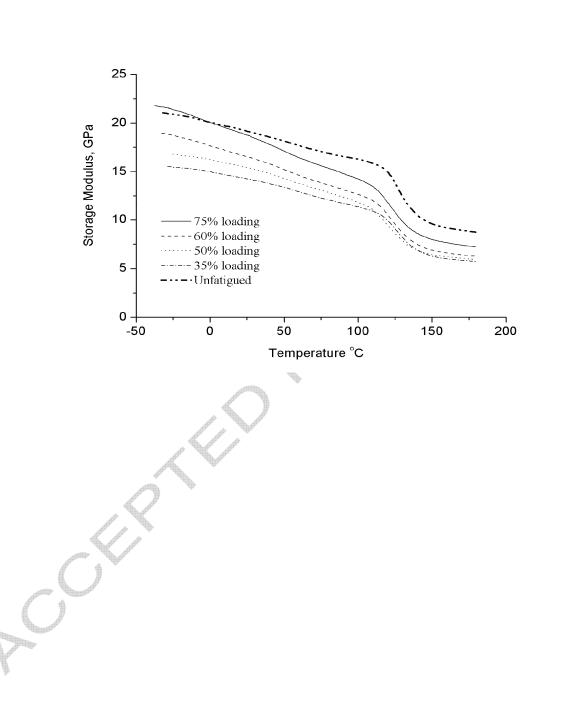




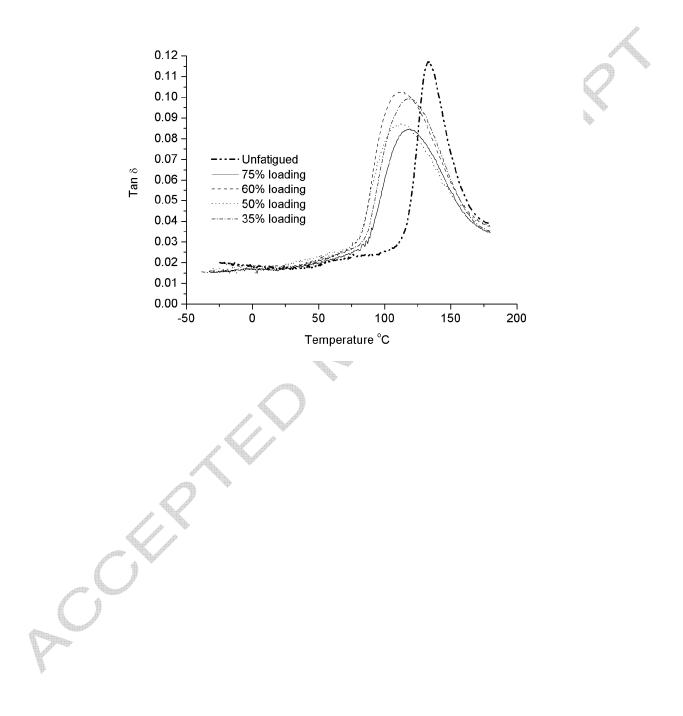




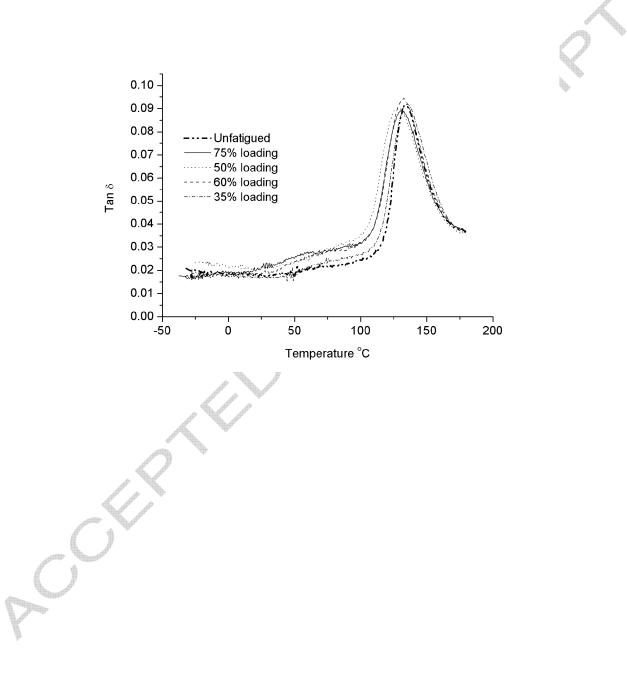












Type of Composite	Mean Strength (MPa)	Standard Deviation (MPa)	Maximum Strength (MPa)	Minimum Strength (MPa)	Median Strength (MPa)
Untreated fibre/polyester	222.6	21.2	252.1	198.5	214.8
Treated fibre/polyester	286.0	31.3	316.9	240.8	282.8
Untreated fibre/epoxy	329.8	20.9	368.1	310.3	326
Treated fibre/epoxy	335.4	18.4	353.6	303.1	342.4

 Table 1
 Static test results for the untreated and 0.06M NaOH treated sisal fibre

composites

Color Color

Fibre Type	Stress level (%)	G _{Max} (MPa)	Frequency (Hz)	Average Cycles to failure, N
	75	167	2.7	3,312
Untreated	60	133	3.3	89,187
Sisal Fibres	50	111	4	599,378
	35	78	5.7	2,184,288
0.06M	75	215	2.1	48
NaOH	60	172	2.6	14,953
Treated	50	143	3.1	310,082
Sisal Fibres	35	100	4.4	1,697,120

Table 2Fatigue data for polyester resin composites at different stress levels.Average lives are for 4 specimens.

Contraction of the second seco

28

Fibre Type	Stress levels (%)	G _{Iax} (MPa)	Frequency (Hz)	Average Cycles to failure, N
Untreated	75	247	1.8	1,636
Sisal Fibres	60	198	2.2	20,059
	50	165	2.7	252,738
	35	115	3.9	4,755,712*
0.06M	75	252	1.8	3,052
NaOH Treated	60	201	2.2	54,764
Treated Sisal Fibres	50	168	2.7	255,778
	35	117	3.9	1,466,752*

Table 3Fatigue data for epoxy resin composites at different stress levels.Average lives are for 4 specimens. (* Only one specimen was tested at

35%).

Contraction of the second seco

Loading level (%)	Glass Transition Temperature, T _g °C		
	Untreated sisal fibre	Treated sisal fibre	
75	80	110	
60	75	112.5	
50	76.25	106.25	
35	82.5	115	
Unfatigued	116.25	117.5	

Table 4Glass transition temperatures for untreated and 0.06M NaOH treated
sisal fibre/epoxy resin composites at different load levels. (Note: Resin
manufacturer quotes the Tg onset for the epoxy resin to be in the range
118-124°C and Tg to be in the range 120-130°C [15])

Color Contractions

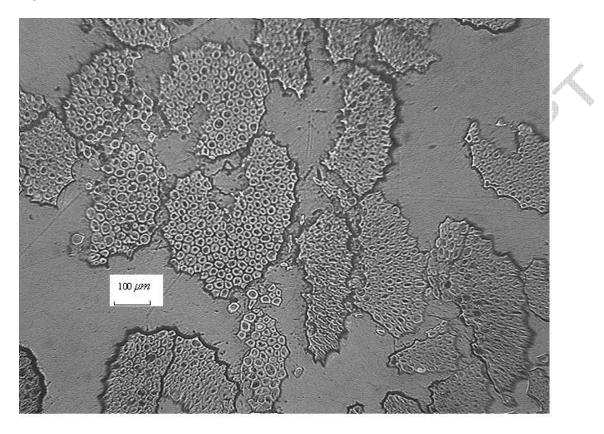
Loading level (%)	Tan δ peak tem	perature °C	Peak tan Svalue	
	Untreated sisal fibre	Treated sisal fibre	Untreated sisal fibre	Treated sisal fibre
75	118.7	131.4	0.08461	0.08916
60	114.7	132.5	0.10257	0.09458
50	112.2	128.8	0.08723	0.09057
35	118.3	135.6	0.09938	0.09184
Un-fatigued	134.1	133.9	0.11739	0.09132

 Table 5
 Tan δdata for untreated and 0.06M NaOH treated sisal fibre/epoxy resin

 composites at different load levels

Contraction of the second seco

Figure 1



Contraction of the second seco

Figure 3





Figure 6a



Figure 8a

