

# The effects of alkali treatment on the interfacial adhesion of bamboo fibres

K J Wong<sup>1\*</sup>, B F Yousif<sup>2</sup>, and K O Low<sup>3</sup>

<sup>1</sup>Centre for Composites, Faculty of Mechanical Engineering, Universiti Teknologi Malaysia, Johor, Malaysia

<sup>2</sup>Faculty of Engineering and Surveying, University of Southern Queensland, Toowoomba, Queensland, Australia

<sup>3</sup>Faculty of Engineering and Technology, Multimedia University, Melaka, Malaysia

*The manuscript was received on 5 October 2009 and was accepted after revision for publication on 13 April 2010.*

DOI: 10.1243/14644207JMDA304

**Abstract:** In the present work, the potential of using bamboo fibres as reinforcement for polyester composites was evaluated. Two types of bamboo fibres were used: untreated and treated with different NaOH concentrations (1, 3, and 5 wt%). Mechanical properties of both treated and untreated fibres were investigated. In addition, single-fibre pull-out tests were performed to study the interfacial shear strength of the fibres with the polyester matrix at different embedment length of fibres. Scanning electron microscopy was used to study the surface morphology of the fibres before and after the tests. Results revealed that an untreated fibre has the best strength and stiffness but lowest strain at break. An increase in alkali concentration reduces the strain at failure and ductility of bamboo fibres. However, the strength and stiffness of the fibres were increased. In addition, the interfacial shear strength was improved with longer embedment length and higher NaOH concentration.

**Keywords:** bamboo fibres, polyester, alkali treatment, interfacial shear strength

## 1 INTRODUCTION

In recent years, natural fibres have received more attention as reinforcements for polymeric composites. This is because natural fibres have the advantages of low density, low cost, renewability, recyclability, and biodegradability [1]. Reinforcing polymers with natural fibres produces composites with low weight and cost, as well as being environmentally friendly [2]. However, natural fibres are hydrophilic, in which they exhibit a high level of moisture absorption and poor wettability [3, 4]. Incompatibility of hydrophilic fibre and hydrophobic matrix results in poor adhesion at the fibre–matrix interface, leading to debonding of the fibre under a certain loading [3, 5–7]. This prevents the applied load from being transferred effectively from matrix to the fibre. Hence, it is important to study the

interfacial adhesion and surface modification of such fibres to improve the polymeric composite properties.

Researches have been carried out to improve the interfacial adhesion of natural fibres through alkali treatment. Studies have been carried out on short henequén fibre-reinforced polyethylene (PE) matrix, where the fibres were treated by using 2 per cent of alkali treatment [7, 8]. Interfacial shear strength (IFSS) was characterized by using two methods, single-fibre pull-out test (SFPT) and single-fibre fragmentation test (SFFT). SFPT is commonly employed to evaluate the bonding quality at the fibre–matrix interface due to its simplicity and versatility [9, 10]. This method can be used for any fibre–matrix combination as long as the interfacial adhesion is not too strong and the fibre is sufficiently thick [11, 12]. Results showed that for treated fibre PE composites, the IFSS was improved for both SFPT and SFFT.

Van de Weyenberg *et al.* [13] treated flax fibres with NaOH at concentrations of 1 per cent, 2 per cent, and 3 per cent. The strength and modulus of treated flax/epoxy composites in both longitudinal and transverse directions were improved. For longitudinal strength, the highest improvement was

\*Corresponding author: Centre for Composites, Faculty of Mechanical Engineering, Universiti Teknologi Malaysia, UTM Skudai, Johor 81310, Malaysia.  
email: [jye1189@yahoo.com](mailto:jye1189@yahoo.com)

achieved at 3 per cent NaOH concentration. As for longitudinal modulus, transverse strength, and transverse modulus, fibres treated by 1 per cent NaOH solution promoted the greatest positive effect.

Aziz and Ansell [14] reinforced polyester resin matrices with long and short hemp and kenaf fibres. Those fibres were treated by a 6 per cent NaOH solution. The results showed that both flexural modulus and strength were improved for all types of composites compared with untreated ones. However, the work of fracture deteriorated except for both long and short kenaf polyester composites. Similar results were observed when cashew nut shell liquid was used as the matrix [15], except for long kenaf fibre, where the work of fracture of the treated one was lower. In addition, Mohanty *et al.* [3] treated sisal fibres with 5 per cent NaOH solution and found that there was a marginal increase in tensile, flexural, and impact strengths in treated sisal–polypropylene (PP) composites.

Besides, Mishra *et al.* [16] treated sisal fibres with 5 per cent and 10 per cent NaOH solution and found that all tensile, flexural, and Izod impact strengths of the sisal/glass hybrid polyester composites were improved compared with the untreated one. However, 5 per cent NaOH was found to promote better mechanical properties than 10 per cent NaOH.

The aim of this article is to present how surface modification of fibres using NaOH treatment affects the interfacial adhesion of bamboo fibres with polyester. Single-fibre tensile tests (SFTTs) were first carried out to determine the mechanical properties of untreated and treated bamboo fibres. Then, micro-mechanical analyses were carried out using SFPT. Effects of treatment and length of fibre were studied. Scanning electron micrographs were observed on various samples.

## 2 EXPERIMENTAL

### 2.1 Materials

The resin used in this study was unsaturated polyester (Reservol P 9509) supplied by Jiashan Anserly Glass Fibre Company Ltd, Malaysia. Bamboo fibres were obtained from a household shop at the state of Sarawak, Malaysia. Figure 1 shows a bundle of bamboo fibres used in this study. All fibres were obtained from the outer layer of the bamboo stem. Only fibres from the same bunch were used to ensure the consistency. The bamboo fibres were carefully extracted from the bundles manually. Assuming that these fibres are of uniform circular shape, the diameters were measured using NJF-1 optical microscope. Five measurements were taken on each fibre to ensure that the assumption of uniform cross-sectional area is valid.



Fig. 1 Bamboo fibres used in this study

### 2.2 Alkali treatment

Selected bamboo fibres were cut into an average length of 70 mm and immersed in NaOH aqueous solution for 24 h at room temperature. From the literature [3, 7, 8, 13, 14, 16], the concentrations of the NaOH were varied from 1 to 10 wt%. According to reference [16], high concentration of NaOH worsened the mechanical properties of the fibres. Therefore, in the current work, three different NaOH concentrations were prepared as 1, 3, and 5 wt%. After treatment, the fibres were washed with water and then dried for 24 h under the sun.

### 2.3 Fabrication of samples

To fabricate the SFTT specimens, both ends of the selected fibres (0, 1, 3, and 5 wt% of NaOH treatment) were adhered to Okada adhesive tape with tabs of size approximately  $30 \times 30 \text{ mm}^2$  to maximize the area of contact between the tabs and the clamping jigs. Before folding the adhesive tapes to form the tabs, the fibres were further glued with adhesive liquid to enhance the adhesion of fibres with the adhesive tape surface to prevent slipping of fibres during the tests. All the specimens were fixed to a gage length of 10 mm. Gage length was set to be short because longer fibres tend to have a higher possibility of flaws and hence will affect the consistency of results. Figure 2 shows the schematic diagram of an SFTT specimen. The specimen was then clamped tightly on 100Q Standalone Universal Test System for testing. The crosshead speed was set to be 1 mm/min. Tests were performed on fibres with 0, 1, 3, and 5 wt% of NaOH treatment. Five test replicates were performed for each percentage of treatment, and the average values were determined.

For SFPT specimens, a metal mould with dimensions of  $50 \times 10 \times 10 \text{ mm}^3$  as shown in Fig. 3 was used. Both ends of the fibre were mounted onto the

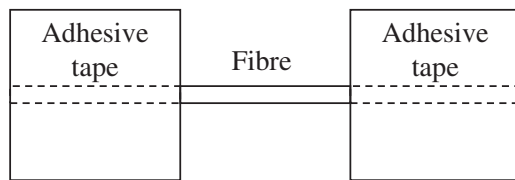


Fig. 2 Schematic diagram of an SFPT specimen

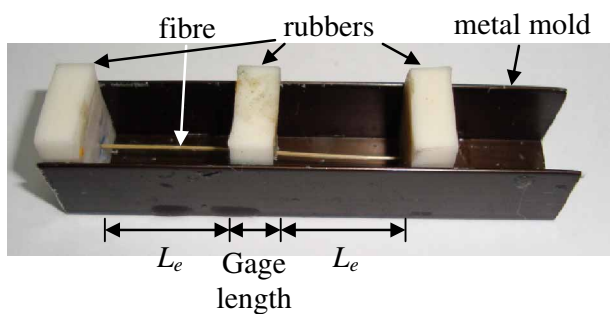


Fig. 3 Mould for an SFPT specimen

mid-plane of the pieces of rubbers. Two rubbers were placed at both ends of the mould to hold the ends of the fibre and also to prevent the resin from leaking during curing process. Another rubber was placed in the middle of the mould, where the distance between the rubbers determines the fibre embedment length and the thickness of the middle rubber determines the gage length. Before proceeding to the fabrication of the specimens, the inner wall of the mould was coated with a thin layer of wax as a release agent. Subsequently, the polyester mixed with 2 per cent of hardener was stirred gently and poured into the mould. The samples were left to be cured at room temperature for 24 h. The specimens were fabricated with different fibre embedment lengths,  $L_e$ , of 1, 3, and 5 mm for both untreated and treated fibres. Figure 4 shows the schematic diagram of an SFPT specimen with embedment length of 5 mm. All specimens were tested using 100Q Standalone Universal Test System, at the crosshead speed of 1 mm/min. At least five tests were repeated to obtain the average.

To obtain the interaction surface of the resin, a nail was pointed to the centre of the resin (which is also the fibre embedment region) and knocked by a hammer carefully to break the specimen. The broken resin is then ready to be studied under microscope.

## 2.4 Morphology study

The morphology of the fibre and matrix surfaces was studied under scanning electron microscope (JEOL-JSM 840, Japan). All specimens were gold sputtered before the analysis.

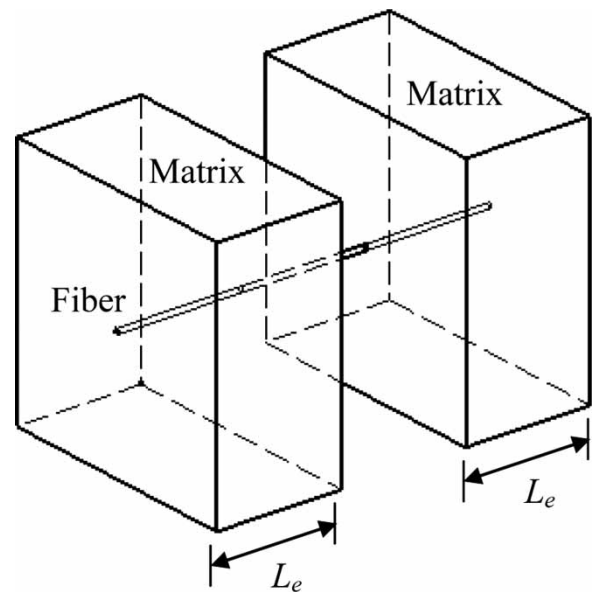


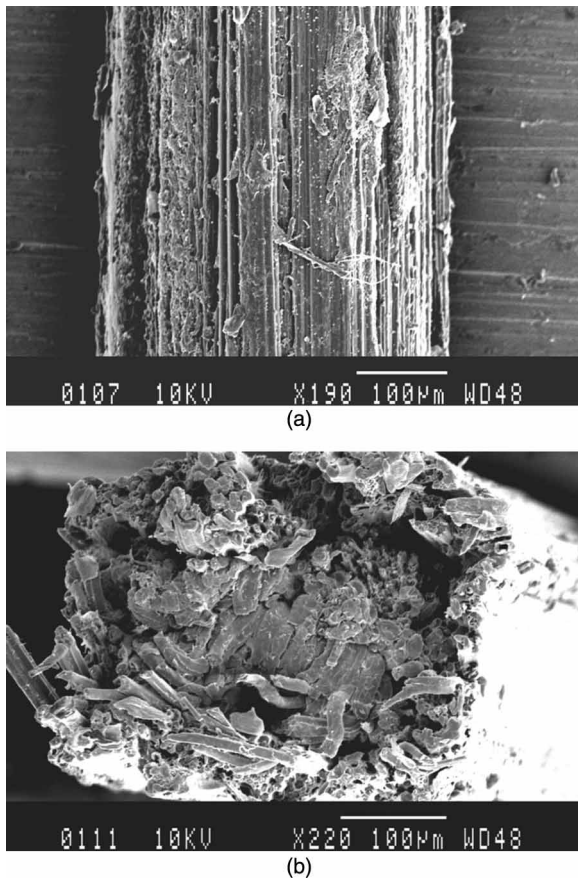
Fig. 4 Schematic diagram of an SFPT specimen

## 3 RESULTS AND DISCUSSION

### 3.1 Surface topography of fibres

Figure 5(a) shows the lateral view of an untreated bamboo fibre. There are considerable hemicelluloses, lignin, waxes, oils, and surface impurities on the fibre surface. Bamboo is a natural lingo-cellulose composite that contains cellulose, hemicelluloses, and lignin [17]. Cellulose is not observed on the fibre surface as it is contained inside the lignin matrix. Figure 5(b) shows the transverse view of an untreated bamboo fibre, where cellulose micro-fibrils could be observed. This indicates that the fibre is actually composed of many ultimate fibres. In other words, every single fibre is a fibre bundle by itself. This is one of the characteristics of lingo-cellulose-based natural fibres [17]. Waxes and oils are substances on the fibre surface that protect the fibre [14, 18] and are generally observed on natural fibres, such as hemp [14, 18] and jute [19]. Certain amount of gaps illustrates that the fibre has a certain degree of porosity, which is also reported in reference [17].

Figures 6(a) to (c) show the micrographs of bamboo fibres treated with 1, 3, and 5 wt% of NaOH, respectively. Hemicelluloses, lignin, waxes, oils, and surface impurities were removed from the fibre surface. It has also been reported elsewhere [7, 8, 14] that the above-mentioned substances on the fibre surface are soluble in NaOH aqueous solution. Besides, it can be observed that different percentages of NaOH caused different levels of treatment on the fibre surface. For 1 wt% NaOH-treated fibre, some fibrous-like impurities can still be observed. When the fibre was treated with 3 wt% NaOH, some holes are observed, indicating that the

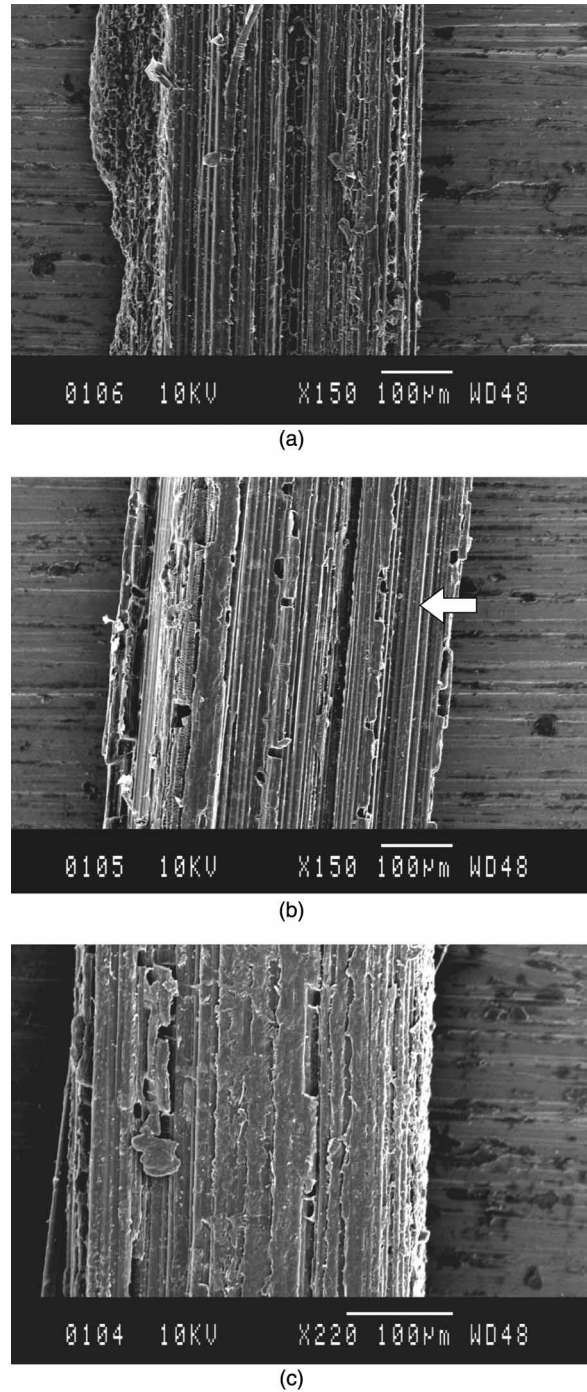


**Fig. 5** (a) Lateral and (b) transverse views of an untreated bamboo fibre

caustic soda has removed larger amount of the soluble substances from the respective layers. Moreover, individual ultimate fibres start to appear, as labelled by the arrow shown in Fig. 6(b). As for fibre treated with 5 wt% of NaOH, the surface topography is very much different, where all the above-mentioned substances on untreated fibre surface are hardly observed. In addition, individual ultimate fibres are more visible. Alkali treatment is believed to have cleared the micro-pores in fibres and roughened the fibre surface [20]. Aziz and Ansell reported that alkalization cleans and makes the fibre surface look smooth, but the fibre surface is actually roughened [14]. Roughening of fibre surface through alkalization was also reported for hemp, sisal, and jute fibres [7, 18, 19]. Higher NaOH concentration could have cleaned the fibre even more thoroughly.

### 3.2 Tensile properties

Table 1 displays the mechanical properties of unsaturated polyester as well as untreated and treated bamboo fibres. Comparing the density,  $\rho$ , among different types of bamboo fibres, it can be seen that the untreated fibre has the highest density, followed by 1, 3, and lastly 5 wt% of NaOH treatment. Compared with other types of natural fibres such as flax, kenaf, hemp,



**Fig. 6** Scanning electron micrographs of treated fibres with (a) 1, (b) 3, and (c) 5 wt% of NaOH treatment

jute, sisal, ramie, and cotton ( $\approx 1.5 \text{ g/cm}^3$ ) [1, 21], the density of untreated bamboo fibre is lower, which implies higher porosity. This could be further verified from Fig. 5(b), where gaps are observed. Reduction in the density of treated fibres could be due to removal of hemicelluloses, lignin, waxes, oils, and surface impurities from the fibre surface through alkali treatment, as mentioned earlier. Consequently, the primary cell wall of the fibre is damaged, leading to reduction in the total weight of the fibre core, and hence lowering

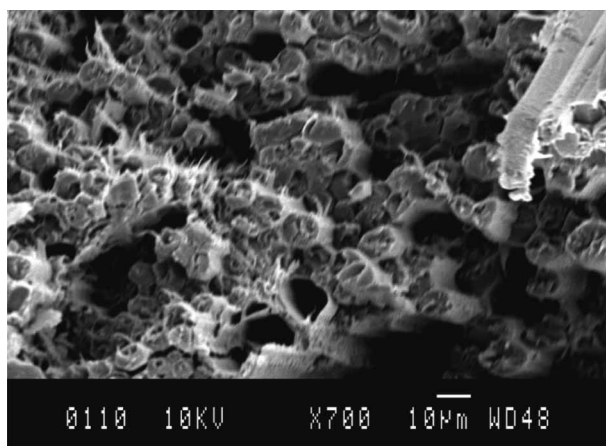
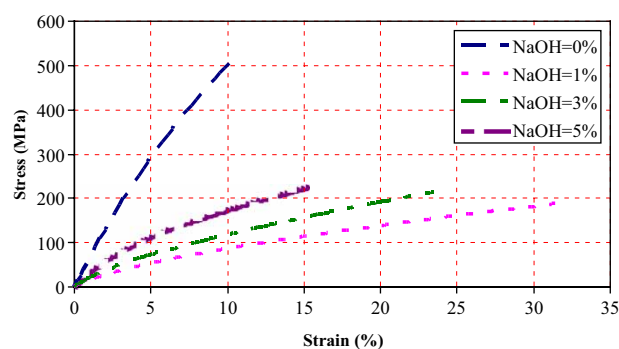


**Table 1** Physical and mechanical properties of polyester [24] and bamboo fibres

Materials	Physical properties			Mechanical properties		
	Diameter range $d_f$ ( $\mu\text{m}$ )	Density $\rho$ ( $\text{g}/\text{cm}^3$ )	Yield strength $\sigma_Y$ (MPa)	Ultimate tensile strength $\sigma_{UT}$ (MPa)	Strain at break $\varepsilon^*$ (%)	Young's modulus $E$ (GPa)
Polyester	–	1.2–1.5	–	40–90	2	2.0–4.5
Fibre 0%	300–380	$1.15 \pm 0.29$	$123 \pm 8$	$518 \pm 51$	$10.4 \pm 0.2$	$5.96 \pm 0.43$
Fibre 1%		$0.95 \pm 0.07$	$45 \pm 6$	$187 \pm 40$	$31.6 \pm 0.5$	$1.31 \pm 0.18$
Fibre 3%		$0.83 \pm 0.18$	$60 \pm 22$	$213 \pm 43$	$23.4 \pm 0.4$	$1.51 \pm 0.54$
Fibre 5%		$0.78 \pm 0.16$	$83 \pm 13$	$222 \pm 47$	$15.2 \pm 0.5$	$2.01 \pm 0.32$

the density. Moreover, reduction in density could also be due to an increment in the porosity of the fibre. Munawar *et al.* have reported that treated fibre has more separated fibre bundles, which is an indication of a porous bundle structure [22]. Mwaikambo and Ansell also mentioned that more cellulose is found to be dissolved at severe alkalization [18]. As shown in Fig. 7, individual ultimate fibres are obvious, with certain amount of internal separation from fibre bundle and relatively more gaps. Results show that higher concentration of the NaOH aqueous solution leads to lower density, indicating higher alkalization effect. Similar observation is also reported in reference [18], in which high NaOH concentration causes the ultimate fibres to be unable to bond together strongly. Negative change in bulk density was also reported in reference [23]. Besides, removal of the soluble substances does not cause significant reduction in fibre diameter, and therefore the diameter range remains the same for all types of fibres. This could be verified through Figs 5 and 6. This indicates that 5 wt% of NaOH solution is still mild enough to dissolve all lignin and pectin, which are the natural cementing substances of natural fibres [18]. Otherwise, separation of fibre bundle could occur and the average diameter could deviate much. However, it should be noted that the diameter of untreated fibres is, on an average, at the upper boundary whereas 5 wt% treated fibres is at the lower boundary of the range.

Stress–strain curves of SFTT for bamboo fibres with different NaOH concentrations are shown in Fig. 8. It can be observed that all bamboo fibres exhibit linear behaviour at initial stage for a relatively small amount of elongation (approximately 1 per cent of strain). Beyond that, all bamboo fibres in different concentrations show non-linear behaviour. The non-linear region shows the ductility of the respective fibre. Untreated fibre is found to have higher yield strength, ultimate tensile strength, and stiffness than treated fibres. This can be explained as follows: for treated fibres, the roughness of the fibre surface is increased, hence creating more peaks and valleys on the fibre surfaces. Decrease in the level of smoothness for treated fibres causes high concentration at certain areas, thereby weakening the fibre. This causes the fibre to yield and break at lower strength. Besides,

**Fig. 7** Transverse view of a 3 wt% treated bamboo fibre**Fig. 8** Stress–strain curves of SFTT for bamboo fibres

the outer layer of the bamboo is one of the regions with broad lamellae, which have lower lignin content [17]. Moreover, removal of lignin could weaken the fibre strength as lignin is the binder of cellulose fibres. As reported, high lignin amount in bamboo fibres exhibits high tensile properties along the fibre direction [17]. Furthermore, as mentioned earlier, porosity of treated fibres is higher, which indicates lower cellulose content and thus worsens the fibre strength. This could be particularly true for natural fibres with low micro-fibril angle (angle between the fibre axis and fibril) [18]. Indeed, bamboo fibre is reported to have low micro-fibril angle [19]. However, treatment

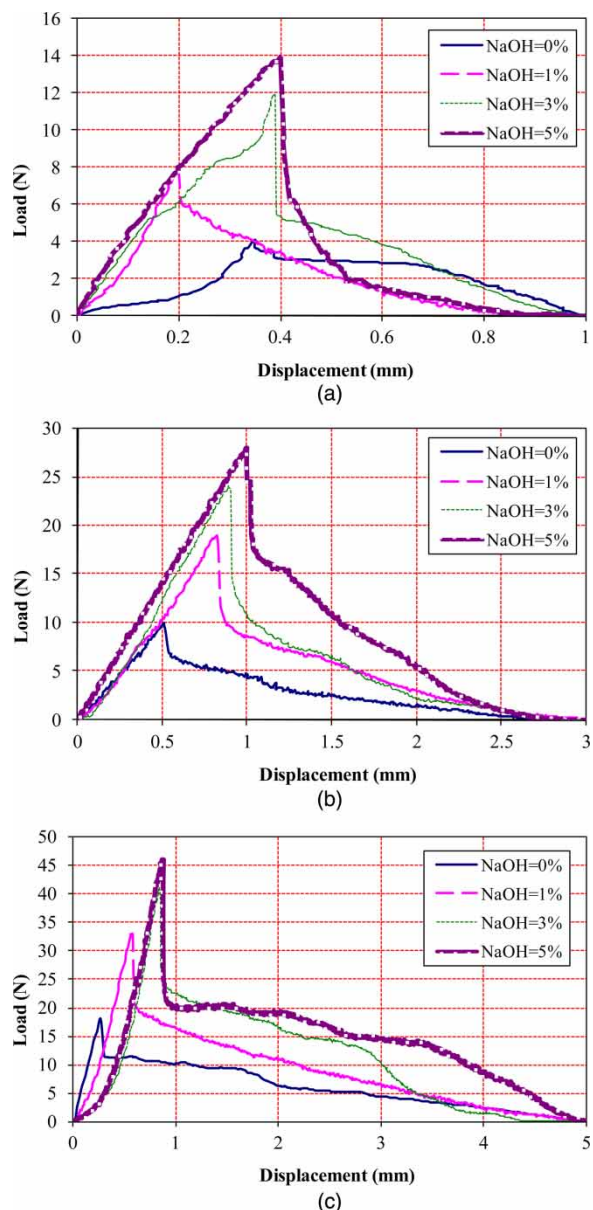
increases the strain at break. This is because higher porosity leads to lower true volume. This increases the actual fibre aspect ratio (length-to-diameter ratio). As the diameter is reduced, the resistance to elongation due to neighbouring ultimate fibre is decreased, so that the fibre could elongate more before ultimate failure. This also indicates a reduction in the rigidity and an improvement in the ductility of treated fibres. Without further measurement, it could also be predicted that the Poisson's ratio is decreased because axial strain is improved. It also needs to be mentioned that reduction in yield strength and increment in strain lead to decrement in the stiffness of treated fibres.

However, among the treated fibres, it is found that the trend of the mechanical properties is in the reverse order of the NaOH concentrations. One weight per cent of NaOH treatment has the highest strain at break and ductility, with the lowest yield strength, ultimate tensile strength, and stiffness. It could be due to the change in micro-fibril angle. As reported by McLaughlin and Tait [25], increment in micro-fibril angle increases the strain. In addition, Mwaikambo and Ansell [18] also reported that strength and stiffness of fibres are dependent on micro-fibril angle, and natural fibres with low micro-fibril angle exhibit high strength [17]. Hence, it is predicted that micro-fibril angle decreases as caustic soda concentration increases. This explains why the strain at break reduces whereas the strength and stiffness increase. However, further work needs to be carried out to have a better view on this factor.

Comparing all the different types of fibres with unsaturated polyester, it is clear that all types of fibres are better than polyester in strength, stiffness, ductility, and compressibility. Hence, bamboo fibres have a potential as a constituent for reinforcement. With regards to the percentage of treatment, this will depend on the applications of the composite.

### 3.3 Interfacial properties

Figures 9(a) to (c) show load–displacement curves obtained from SFPT of bamboo fibres at different fibre embedment lengths. From the figures, it can be observed that upon loading, the force increases until a peak load is attained. The increment is almost at the same rate except for embedment length of 1 mm. This could be because the stress transfer is not fully developed before pull-out is initiated, as short embedment length exhibits weaker bonding. Peak load is also known as debonding load,  $F_{deb}$ , where total debonding of the fibre from the matrix occurs. Before attaining the peak load, the fibre is actually not pulled out yet and is assumed to be still bonded with the matrix. The applied force is directly carried by the matrix and transferred from the matrix to the fibre through fibre–matrix interface. The amount of load carried by



**Fig. 9** Load–displacement curves of a single bamboo fibre pull-out test with (a) 1, (b) 3, and (c) 5 mm of embedment lengths

each of them depends on their mechanical property (Young's modulus), geometrical property (area of each material), and interfacial property (bonding quality). Besides the axial force carried by both materials, there is frictional shear force acting in the opposite direction of applied force across fibre–matrix interface. Based on static equilibrium theory, the net amount of the axial and shear forces is always equal and opposite to the applied force.

At the instant where the load reaches maximum, the load drops drastically. This is due to less interpenetration between the matrix and the fibre surface, indicating a release of stored energy. The stored energy can be calculated by measuring the area under the force–displacement curve for the region before debonding

is initiated. Debonding could be initiated by the axial force loading, which results in a Poisson's contraction of the fibre in the radial direction. Consequently, this reduces the normal radial stress on the fibre, which is due to the shrinkage of matrix [8]. The shrinkage of matrix upon hardening of unsaturated polyester is relatively significant compared to other polymers such as epoxy [26]. Reduction in the normal radial stress decreases the frictional component of the interfacial strength. This could initiate the interfacial failure and lead to subsequent fibre pull-out. Broutman [27] studied the single-filament fibre pull-out test and obtained a similar load-displacement curve. Similar observation was reported by Morlin and Czigány [28] on the study of three different natural fibres (flax, hemp, and sisal) reinforced with three different thermoplastic matrices (PP, Mater-Bi, and Purasorb PL).

After this stage, the mechanism changes from static to kinetic motion, where the fibre slides against the resin with any further displacement imposed until the fibre is totally pulled out. This is a similar mechanism as in friction and hence the interfacial failure is basically considered as a frictional-type failure. The load decreases almost linearly at the kinetic region, which is expected due to the decrement of cross-sectional area of contact between fibre and matrix as the fibre was pulled out progressively at a constant crosshead speed. The respective load at any instant represents the force needed to overcome the shear force. Generally, higher alkali concentration gives higher load at the kinetic region except for embedment length of 1 mm. This could be because the embedment length is so short that, upon debonding, it is very difficult to control the steady pull-out of the fibre. At the point where the fibre is totally pulled out, the load becomes zero. Meo *et al.* [29] found a similar trend of frictional pull-out of the fibre. Such behaviour represents a constant shear distribution defined by the classical Coulomb friction law, where, upon debonding, the shear stress is maximum and always constant. Slight fluctuation can be seen, which is due to irregularity of the contact surfaces.

The average values of the IFSS at different embedment lengths and percentages of NaOH treatment are shown in Figs 10 and 11. The interfacial shear strength,  $\tau_{\text{IFSS}}$ , is determined by using equation (1) by Kelly and Tyson [30]. In this equation, the shear stress across fibre-matrix interface is assumed to be constant

$$\tau_{\text{IFSS}} = \frac{F_{\text{deb}}}{d_f \pi L_e} \quad (1)$$

where  $\tau_{\text{IFSS}}$  is the interfacial shear strength,  $F_{\text{deb}}$  the debonding load,  $d_f$  the fibre diameter, and  $L_e$  the fibre embedment length.

Figure 10 compares the IFSS when the fibre embedment length increases for fibres with different percentages of treatment. For all cases, the trend

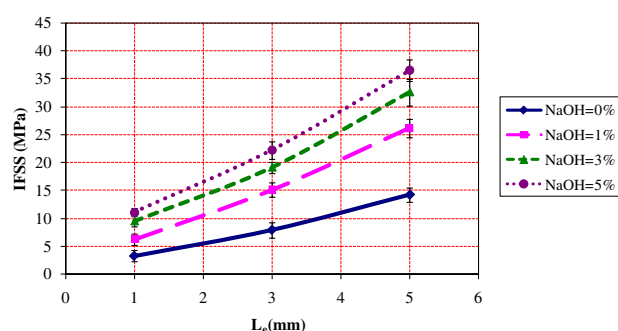


Fig. 10 The effect of fibre embedment length to IFSS for untreated and treated fibres

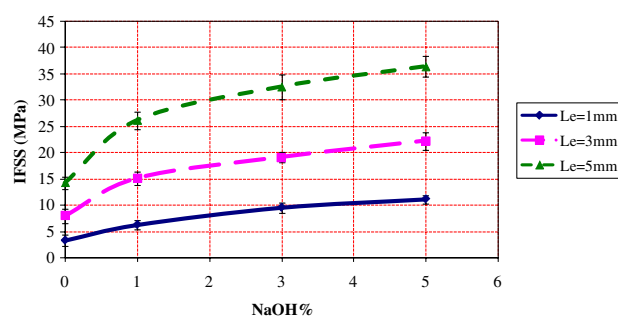


Fig. 11 The effect of alkali treatment to IFSS at different fibre embedment length

shows an improvement in IFSS when the embedment length is increased. With embedment length of 1 mm as reference, the IFSS is at least doubled when the embedment length is 3 mm. With embedment length of 5 mm, the percentage improvement is more than 230 per cent as compared to 1 mm embedment length. This indicates that the peak load dominates over the fibre embedment length, as IFSS is proportional to the peak load and inversely proportional to the embedment length. This could be due to longer embedment length, leading to larger area for bonding; thus, upon solidification of the matrix, the total residual compressive stress due to the temperature change during resin curing is larger. It is therefore also known as thermal residual stress [26]. The largest residual compressive stress is believed to exist when the matrix is totally hardened because in solids molecules are arranged in a proper order with a shorter distance among them, thereby having a stronger bonding compared to liquids.

Hence, it is predicted that with further increment in the embedment length, the IFSS will be further improved. However, Favre and Perrin [31] reported that there is a critical embedment length for effective stress transfer across the fibre-matrix interface, where the IFSS is optimum at that particular embedment length. Beyond that, the IFSS will not be further increased with any further increment in fibre length. Further work is needed to determine this. In the works

of Favre *et al.*, the critical embedment lengths of carbon fibre reinforced in various thermoset polymers including epoxy, polyester, and polyimide matrix were not determined as well.

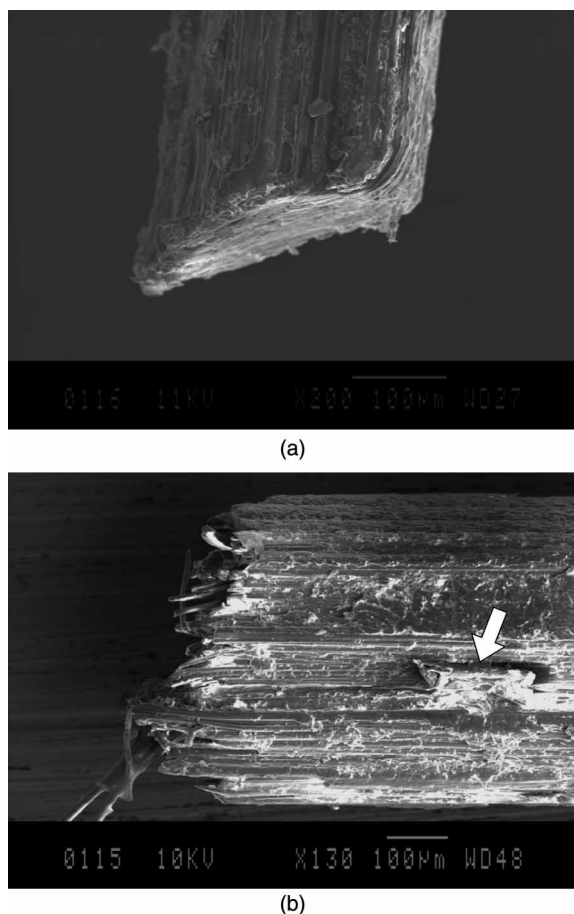
Figure 11 shows the effect of percentage of alkali treatment to IFSS. It is clear that with alkali treatment, the IFSS increases. At a particular fibre embedment length, untreated fibres are always having the lowest IFSS, followed by 1, 3, and finally 5 wt% of alkali treatment. Improvement in IFSS is believed to be due to surface roughening through treatment. This increases the total fibre surface area, thus giving larger contact area between the fibre and the matrix [3, 7]. As a result, this promotes better fibre–matrix mechanical locking [8, 19] and improves the interfacial adhesion. Also, higher porosity of treated fibres allows the matrix to impregnate the cavities and induces better bonding [17]. In addition, treatment increases the hydroxyl groups and thus improves the wetting ability of the fibre [32].

However, Fig. 11 also shows that when the percentage of alkali treatment is increased, the improvement is less significant. This can be observed from the figure such that the slopes of all curves are in decreasing manner. Hence, it is expected that with further increment in NaOH percentage, the percentage of increment will be even less significant. After a certain percentage of alkali treatment, the improvement will be saturated. This will be the optimum percentage of treatment by using NaOH.

### 3.4 Morphology study after fibre pull-out

Interfacial characteristics of bamboo fibres with polyester matrix are further explained through scanning electron microscopy shown in Figs 12 and 13. Figure 12 shows the debonded surfaces of untreated and 3 wt% NaOH-treated fibres, together with their fibre ends, whereas Fig. 13 demonstrates the corresponding debonded surfaces of the matrix. From Fig. 12(a), it can be observed that the surface of untreated fibre is clean. This is an indication of poor interfacial bonding, which is due to incompatibility (poor surface wetting) of hydrophilic fibre and hydrophobic matrix [3, 7, 14]. Weak bonding implies composite failures by delamination, which is an evidence of shear failure [16]. Same figure illustrates that the fibre end is almost intact, indicating very minimal energy dissipation at fibre end.

For the treated fibre, some matrix patches are found to be attached to the fibre surfaces, which are indicated by arrows shown in Fig. 12(b). Such observation is an indication of better interfacial adhesion. As described, this is due to improved roughness, wettability, and impregnation of matrix. Besides, certain amount of fibre end damage is observed, which is indicated by fibrillar splitting. This could be because of higher

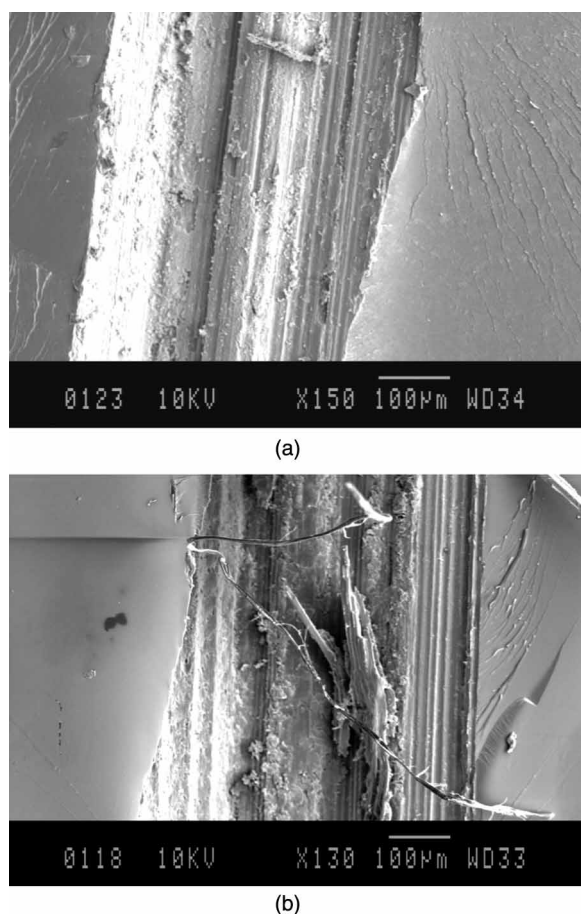


**Fig. 12** Debonded fibre surfaces and ends of (a) untreated and (b) 3 wt% NaOH-treated fibres

interfacial shear strength, as higher load is needed to initiate the debonding. Fibrillar splitting implies energy dissipation at fibre end.

For matrix surfaces, which are the counter-surfaces of the respective fibres, it is shown that for untreated fibre, the matrix surface is relatively clean (i.e. not much fibre at the surface is attached to the matrix). This further verifies poor interfacial adhesion, where the fibre surface is sliding along the surface upon fibre pull-out, without strong mechanical locking between the fibre and the matrix. This explains low IFSS of untreated fibres, as shown in Fig. 11. For 3 wt% NaOH-treated fibre, it is clearly observed that there is more fibre surface attached to the matrix surface. This indicates better interfacial adhesion, where there is better interpenetration at the interface of the fibre–matrix. Interfacial shear strength may exceed the inter-bonding strength in the fibre itself at certain portions, and hence upon debonding, the surface is detached from the fibre. Mohanty *et al.* [3] reported that this is due to the chemical modification by alkali treatment that the hydroxyl groups of the fibre surface are transformed, and the polymer chains can hence be covalently attached to the fibre surface.





**Fig. 13** Debonded matrix surfaces from (a) untreated and (b) 3 wt% NaOH-treated fibres

#### 4 CONCLUSIONS

From this study, several points can be concluded as follows:

1. Alkali treatment increases the strain at break and ductility compared to untreated ones. However, density, yield strength, ultimate tensile strength, and Young's modulus are lowered.
2. Higher NaOH concentration improves the yield strength, ultimate tensile strength, and Young's modulus of the fibre, but lowers the density and strain at break.
3. The IFSS improves with increment in fibre embedment length (i.e. higher IFSS is found at 5 mm of embedment length compared to 1 and 3 mm).

The IFSS increases with the increase of alkali concentration. However, as the concentration of NaOH increases, the percentage of improvement decreases.

© Authors 2010

#### REFERENCES

- 1 **Wambua, P., Ivens, J., and Verpoest, I.** Natural fibres: can they replace glass in fibre reinforced plastics? *Compos. Sci. Technol.*, 2003, **63**(9), 1259–1264.
- 2 **Herrera-Franco, P. J. and Valadez-González, A.** Mechanical properties of continuous natural fibre-reinforced polymer composites. *Compos. A, Appl. Sci. Manuf.*, 2004, **35**(3), 339–345.
- 3 **Mohanty, S., Nayak, S. K., Verma, S. K., and Tripathy, S. S.** Effect of MAPP as coupling agent on the performance of sisal-PP composites. *J. Reinf. Plast. Compos.*, 2004, **23**, 2047–2063.
- 4 **Doan, T.-T.-L., Gao, S.-L., and Mäder, E.** Jute/polypropylene composites I. Effect of matrix modification. *Compos. Sci. Technol.*, 2006, **66**(7–8), 952–963.
- 5 **Doan, T.-T.-L. and Mäder, E.** Performance of jute fibre reinforced polypropylene. In Proceedings of the Seventh International AVK-TV Conference, Baden-Baden, 28–29 September 2004.
- 6 **Lucka, M., Bledzki, A. K., and Michalski, J.** Influence of the hydrophobisation of Flax fibres on the water sensitivity, biological resistance and electrical properties of Flax-polypropylene composites. In Proceedings of the 5th Global Wood and Natural Fibre Composites Symposium, Kassel, Germany, 27–28 April 2004, pp. A15.1–A15.7.
- 7 **Herrera-Franco, P. J. and Valadez-Gonzalez, A.** A study of the mechanical properties of short natural-fibre reinforced composites. *Compos. B, Eng.*, 2005, **36**(8), 597–608.
- 8 **Valadez-Gonzalez, A., Cervantes-Uc, J. M., Olayo, R., and Herrera-Franco, P. J.** Effect of fiber surface treatment on the fiber-matrix bond strength of natural fiber reinforced composites. *Compos. B, Eng.*, 1999, **30**(3), 309–320.
- 9 **Fu, S. Y., Yue, C. Y., Hu, X., and Mai, Y. W.** Analyses of the micromechanics of stress transfer in single- and multi-fiber pull-out tests. *Compos. Sci. Technol.*, 2000, **60**(4), 569–579.
- 10 **Bannister, D. J., Andrews, M. C., Cervenka, A. J., and Young, R. J.** Analysis of the single-fibre pull-out test by means of Raman spectroscopy. Part II: micromechanics of deformation for an aramid/epoxy system. *Compos. Sci. Technol.*, 1995, **53**(4), 411–421.
- 11 **Herrera-Franco, P. F. and Drzal, L. T.** Comparison of methods for the measurement of fibre/matrix adhesion in composites. *Composites*, 1991, **23**(1), 2–27.
- 12 **Gu, W.** *Interfacial adhesion evaluation of uniaxial fiber-reinforced-polymer composites by vibration damping of cantilever beam.* PhD Thesis, Virginia Polytechnic Institute and State University, 1997.
- 13 **Van de Weyenberg, I., Ivens, J., De Coster, A., Kino, B., Baetens, E., and Verpoest, I.** Influence of processing and chemical treatment of flax fibres on their composites. *Compos. Sci. Technol.*, 2003, **63**(9), 1241–1246.
- 14 **Aziz, S. H. and Ansell, M. P.** The effect of alkalization and fibre alignment on the mechanical and thermal properties of kenaf and hemp bast fibre composites. Part 1: polyester resin matrix. *Compos. Sci. Technol.*, 2004, **64**(9), 1219–1230.

- 15 Aziz, S. H. and Ansell, M. P. The effect of alkalization and fibre alignment on the mechanical and thermal properties of kenaf and hemp bast fibre composites. Part 2: cashew nut shell liquid matrix. *Compos. Sci. Technol.*, 2004, **64**(9), 1231–1238.
- 16 Mishra, S., Mohanty, A. K., Drzal, L. T., Misra, M., Parija, S., Nayak, S. K., and Tripathy, S. S. Studies on mechanical performance of biofibre/glass reinforced polyester hybrid composites. *Compos. Sci. Technol.*, 2003, **63**(10), 1377–1385.
- 17 Jain, S., Kumar, R., and Jindal, U. C. Mechanical behaviour of bamboo and bamboo composite. *J. Mater. Sci.*, 1992, **27**(17), 4598–4604.
- 18 Mwaikambo, L. Y. and Ansell, M. P. Mechanical properties of alkali treated plant fibres and their potential as reinforcement materials. I. Hemp fibres. *J. Mater. Sci.*, 2006, **41**(8), 2483–2496.
- 19 Eichhorn, S. J., Baillie, C. A., Zafeiropoulos, N., Mwaikambo, L. Y., Ansell, M. P., Dufresne, A., Entwistle, K. M., Herrera-Franco, P. J., Escamilla, G. C., Groom, L., Hughes, M., Hill, C., Rials, T. G., and Wild, P. M. Review: current international research into cellulosic fibres and composites. *J. Mater. Sci.*, 2001, **36**(9), 2107–2131.
- 20 Vasconcelos, P. V., Lino, F. J., Magalhães, A., and Neto, R. J. L. Impact fracture study of epoxy-based composites with aluminium particles and milled fibres. *J. Mater. Process. Technol.*, 2005, **170**(1–2), 277–283.
- 21 Agarwal, B. D., Broutman, L. T., and Chandrashekhara, K. *Analysis and performance of fiber composites*, 3rd edition, 2006 (John Wiley and Sons, Inc., New York, USA).
- 22 Munawar, S. S., Umemura, K., Tanaka, F., and Kawai, S. Effects of alkali, mild steam, and chitosan treatments on the properties of pineapple, ramie, and sansevieria fiber bundles. *J. Wood Sci.*, 2008, **54**(1), 28–35.
- 23 Mwaikambo, L. *Plant-based resources for sustainable composites*. PhD Thesis, Department of Engineering and Applied Science, University of Bath, 2002.
- 24 Hull, D. and Clyne, T. W. *An introduction to composite materials*, 2nd edition, 1996 (Cambridge University Press, Cambridge, UK).
- 25 McLaughlin, E. C. and Tait, R. A. Fracture mechanism of plant fibres. *J. Mater. Sci.*, 1980, **15**(1), 89–95.
- 26 Feih, S., Wonsyld, K., Minzari, D., Westermann, P., and Lilholt, H. Testing procedure for the single fiber fragmentation test, Risø National Laboratory, December 2004, 30 p.
- 27 Broutman, L. J. Measurement of the fiber–polymer matrix interfacial strength. In *Interfaces in composite*, 1969, vol. 27, ASTM STP 452 (ASTM, Philadelphia, Pennsylvania, USA).
- 28 Morlin, B. and Czigány, T. Investigation of the surface adhesion of natural fibre reinforced polymer composites with acoustic emission technique. In Proceeding of the Eighth polymers for Advanced Technologies International Symposium, Budapest, Hungary, 13–16 September 2005, pp. 1–3.
- 29 Meo, M., Achard, F., and Grassi, M. Finite element modelling of bridging micro-mechanics in through-thickness reinforced composite laminates. *Compos. Struct.*, 2005, **71**(3–4), 383–387.
- 30 Kelly, A. and Tyson, W. R. Tensile properties of fibre-reinforced metals: copper/tungsten and copper molybdenum. *J. Mech. Phys. Solids*, 1965, **13**, 329.
- 31 Favre, J. P. and Perrin, J. Carbon fiber adhesion to organic matrices. *J. Mater. Sci.*, 1972, **7**, 1113.
- 32 Felix, J. M. and Gatenholm, P. Effect of transcrystalline morphology on interfacial adhesion in cellulose/polypropylene composites. *J. Mater. Sci.*, 1994, **29**(11), 3043–3049.