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Microstructure of autoclaved refined wood-fibre cement mortars

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Autoclaved cement mortars reinforced with refined wood-pulp fibres have been tested in flexure and the fracture surfaces examined by scanning electron microscopy. The observations indicate that failure occurs by the dual mechanism of fibre fracture and fibre pull-out, and that the interfacial bonds between the fibres and the cement matrix are stronger than had been previously considered.

1. Introduction

The use of refined wood fibres as a source of reinforcement in autoclaved cement mortars has proved successful in both laboratory and pilot plant trials [1]. Mechanical testing of these materials demonstrated that the samples possessed sufficient flexural strength (> 20 MPa) and toughness to provide an alternative to asbestos fibre-reinforced cement sheeting.

During earlier studies of air-cured wood fibre-reinforced cement composites [2, 3] it was observed that the fracture surfaces, obtained during flexure, (three-point bend test) contained large proportions of fractured fibres. This finding contrasted with that of Andonian *et al.* [4] who proposed that autoclaved wood fibre-reinforced cement mortars (WFRC) have characteristic fracture surfaces displaying 85 to 90% fibre pull-out.

To clarify the question of whether fibres fracture or pull-out during bending to failure the fracture surfaces of a range of samples prepared from autoclaved refined WFRC were examined.

2. Materials and methods

The wood fibres used in this study were from commercial *Pinus radiata* kraft lap pulp obtained from Kinleith, New Zealand. The fibres were prepared by soaking the dry lap in water for 4 h followed by disintegration of the fibres in a Valley beater without its weight being applied. The disintegrated fibre has a freeness of 703 CSF (Pulp 1). With weights applied a second pulp was prepared with a freeness of 442 CSF (Pulp 2).

The samples of WFRC used in this investigation (containing a matrix of ordinary Portland cement and ground silica of equal weight) were prepared by a slurry/vacuum de-watering process followed by autoclaving, as reported in an earlier study [1]. Samples (of dimensions 125 mm x 40 mm) were tested in flexure (3-point bend test) at a loading rate of 0.5 mm min^{-1} (sample thickness ~ 6 mm, span = 100 mm) on an Instron testing machine (Model 1114); fracture was completed by hand. Modulus of rupture (MOR) and fracture toughness were calculated as reported in an earlier paper [1]. All samples were conditioned in a control room with relative humidity (r.h.) of $50 \pm 5\%$ and a temperature of $22 \pm 2^{\circ}$ C.

To enable close examination of individual fibres, low mass-fractions of the reinforcing fibre were used in the preparation of the composites. A scanning electron microscope (SEM) was used to examine the fracture surface of samples containing small numbers of fibres. Examination was simplified by mounting both fracture surfaces side by side on the mounting stub enabling observation of both profiles of the fracture line.

All fracture surfaces were coated with gold under vacuum and examined in a Cambridge S600 SEM at 25 KV.

3. Observations and disucssion

Representative fibres from Pulp 1 and Pulp 2 are depicted in Fig. 1a and b, respectively. The

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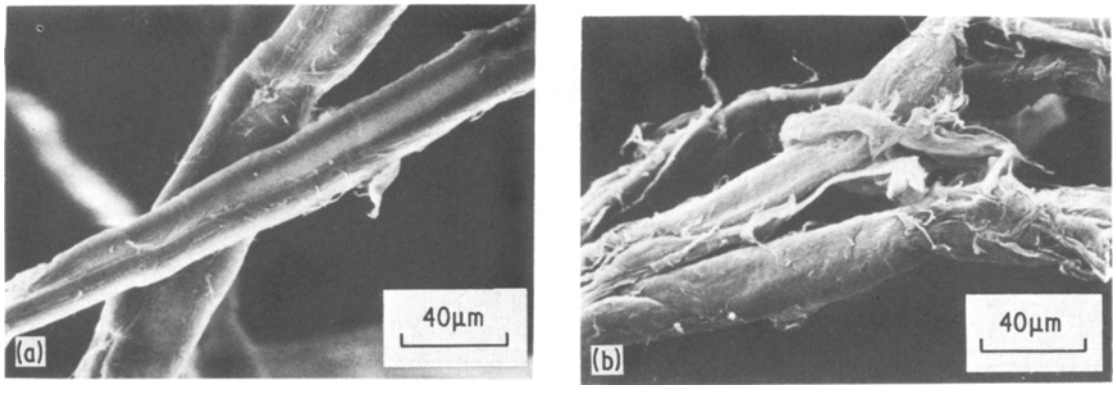

Figure 1 (a) SEM showing surface of unbeaten wood fibre; (b) surface of beaten wood fibre with fibrillation.

process of beating (or refining) wood fibres had three main effects:

(a) the fibres are shortened (often at "nodes", points of weakness in the fibre where ray cells abutted fibres in the wood structure);

(b) external fibrillation occurs, causing partial or sometimes total removal of the primary wall and causing fibrils to form on the surface of the fibre; and

(c) internal fibrillation occurs, causing the fibre to become more comformable.

It was concluded in an earlier study [1] that fibrillation of the refined fibres enabled them to form a web capable of retaining the particulate matrix whilst still maintaining sufficient drainage rate to be successfully processed on a modified Hatschek machine. It was noted in pilot plant runs that the product containing unrefined pulp (~750 CSF) was very difficult to process and gave a flexural strength less than half (~10 MPa) that obtained from a product containing pulp refined to ~500 CSF [1].

The fracture surfaces of WFRC samples containing 1 wt% of Pulp 1 and Pulp 2 are shown in Fig. 2a and b, respectively. Some fibre pull-out is evident in Fig. 2a, although fibre fracture is predominant. Fig. 2b shows short, matrix encrusted, fractured fibres. Little fibre pull-out is observed. Critical examination of the fracture line for the 1 wt% Pulp 2 by observing both fracture surfaces (Fig. 3a and b) shows that considerable damage has been rendered to the fibres. Fibre A has been stripped of its outer layer (see Fig. 3b), which is seen firmly attached to the walls of the hole in the matrix from which Fibre A was pulled out (see Fig. 3a). Fibre B (in Fig. 3b) is encrusted with matrix, and this suggests that the heterogenous matrix failed in shear or at a flaw, before sufficient stress could be exerted on the fibre to cause debonding at the interface or fibre fracture. Clearly, Fibre C was lying at an angle to the plane of the advancing crack and had been subjected to shear stresses which caused fracture between the layers of the fibre.

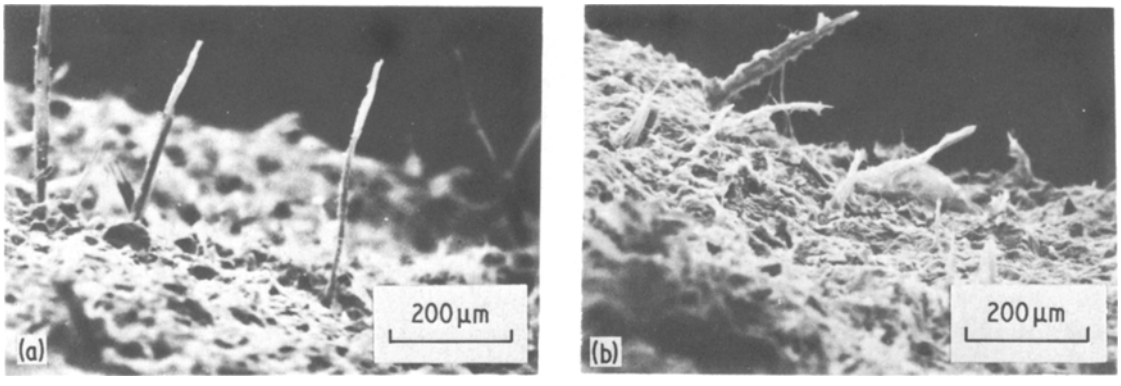

Figure 2 (a) A typical SEM of the fracture surface of a WFRC composite containing 1 wt% unbeaten fibre and (b) 1 wt% beaten fibre.

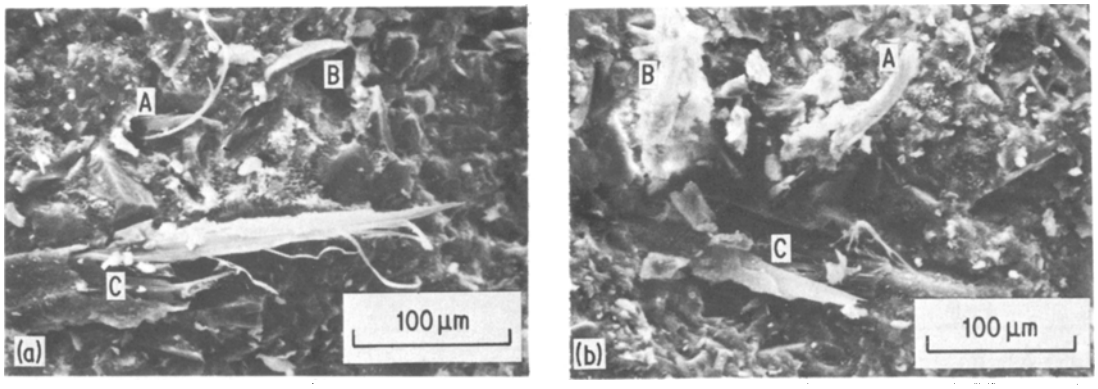

Figure 3 (a) SEM of a fracture surface of a WFRC composite containing 1 wt % of beaten fibre; (b) the matched fracture surface of (a) which shows fibre fracture has taken place.

A similar series of matched surfaces are seen in Fig. 4. Fig. 4a shows a fibre, coming out of the plane of the micrograph, which has lost its outer layer along the top half of its length and has a fractured end. This fibre is matched with an opening (Fig. 4b). Fig. 4c shows this part of Fig. 4b at higher magnification, showing that the end of the fibre contained within the hole is fractured cleanly and fibrils of the outer layer remain firmly attached to the matrix where initial debonding took place.

For the samples examined in Figs 2 to 4, no meaningful toughness values have been recorded in mechanical testing (Table I) due to the limited amount of reinforcement present in the formulations.

To draw a comparison between the earlier work of Andonian *et al.* [4] and this current study, samples containing 6 wt % fibre were prepared and examined. Andonian *et al.* [4] suggested that the fracture surfaces shown in Fig. 7 of [4] are typical of fibre pull-out yet close examination of their electron micrographs would suggest that fibre fracture is prevalent. The load–deflection curve shown in their Fig. 3a of [4] would suggest that the materials are failing in a brittle manner and little fibre pull-out is taking place. This tends to contradict their hypothesis that 85 to 90% pull-out is the source of fracture toughness.

Samples prepared by Andonian *et al.* [4] were dried at 116° before testing. It is our experience that over-dried samples show high strength and low toughness and a considerable degree of fibre fracture [1]. Work is in progress to explain this behaviour in terms of the moisture content of the samples [5].

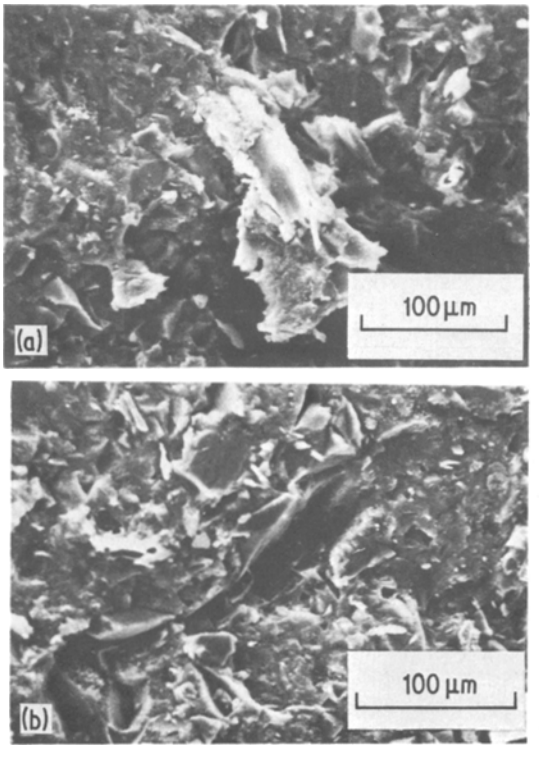

Figure 4 (a) SEM of fracture surface of a WFRC composite containing 1 wt % of beaten fibre; (b) the matched fracture surface of (a); (c) high-magnification SEM of (b).

TABLE I Mechanical properties of WFRC composites

Fibre % (wt %)	Freeness (CSF)	Cure	MOR (MPa)	Fracture toughness (kJ m^{-2})	Fig. number
1	703	Autoclaved (318 kPa, 8 h)	13.3	0.10	2a
1	442	Autoclaved (318 kPa, 8 h)	14.7	0.10	2b, 3, 4
6	442	Autoclaved (318 kPa, 8 h)	22.3	1.41	5, 8, 9
6	442	Air-cured (5 days)	11.5	2.13	6

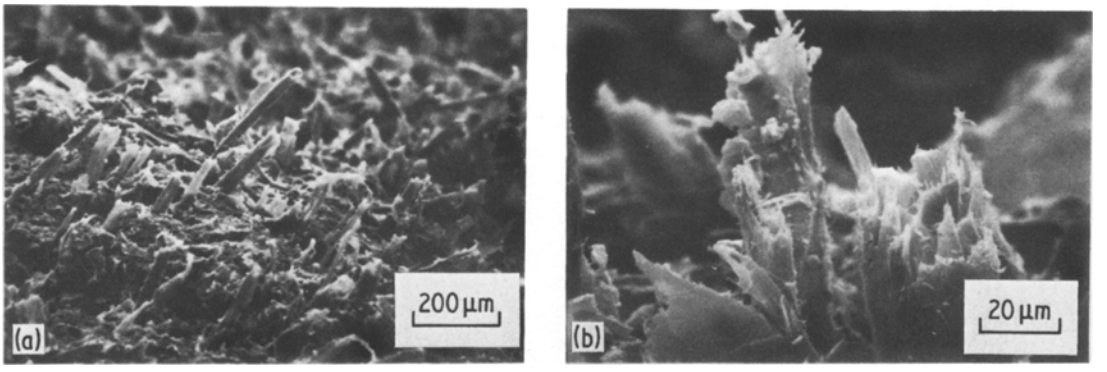

Figure 5 (a) SEM of a fracture surface of a WFRC composite containing 6 wt % of beaten fibre; (b) high-magnification SEM of (a) showing fractured fibres.

Fig. 5a in the present study, which shows the fracture surface of a WFRC composite containing 6 wt % fibre, is very similar to Fig. 7b in the work of Andonian *et al.* [4] and might suggest that higher magnifications should have been looked at in their examination. In their study, Fig. 4a of [4], which is at a higher magnification, shows considerable fibre fracture in the background.

A higher magnification view of the 6 wt % fibre sample used in this study is seen in Fig. 5b and is typical of the fibre fracture observed at the fracture surface. The fractured fibre ends are seen to be devoid of attached matrix. To confirm that fibres were not damaged before or during the fabrication of the composites, samples were prepared and air-cured for 5 days instead of autoclaving. This resulted in a weak matrix and a composite with a flexural strength of only 11.5 MPa compared with 22.3 MPa for the autoclaved sample of the same formulation. The fracture surface of the uncured sample showed almost total fibre pull-out (see Fig. 6) and few fractured fibres could be found. This negates the possibility of fibre damage during fabrication accounting for the multitude of fractured fibres observed here.

The shape of the load–deflection curves for typical WFRC composites are shown in Fig. 7. The composite containing 1 wt % fibre had a curve indicative of an almost brittle material with a flexural strength similar to the matrix (approximately 15 MPa); little fracture toughness was observed. When the fibre content was increased to 6 wt % the shape of the curve indicated a considerable increase in post-cracking ductility or toughness. Samples are of different thickness when the fibre content is increased from 1 wt % to 6 wt % fibre, and this has an effect on the maximum load recorded.

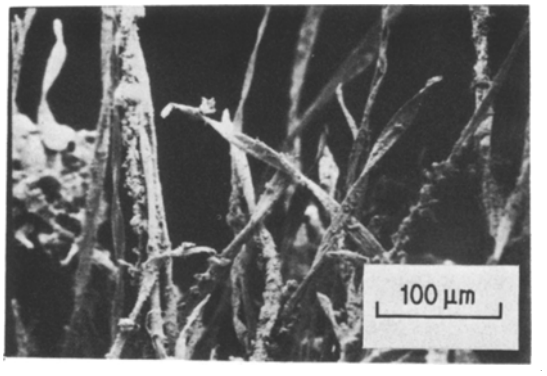

Figure 6 SEM of a fracture surface of an uncured WFRC composite showing fibre pull-out.

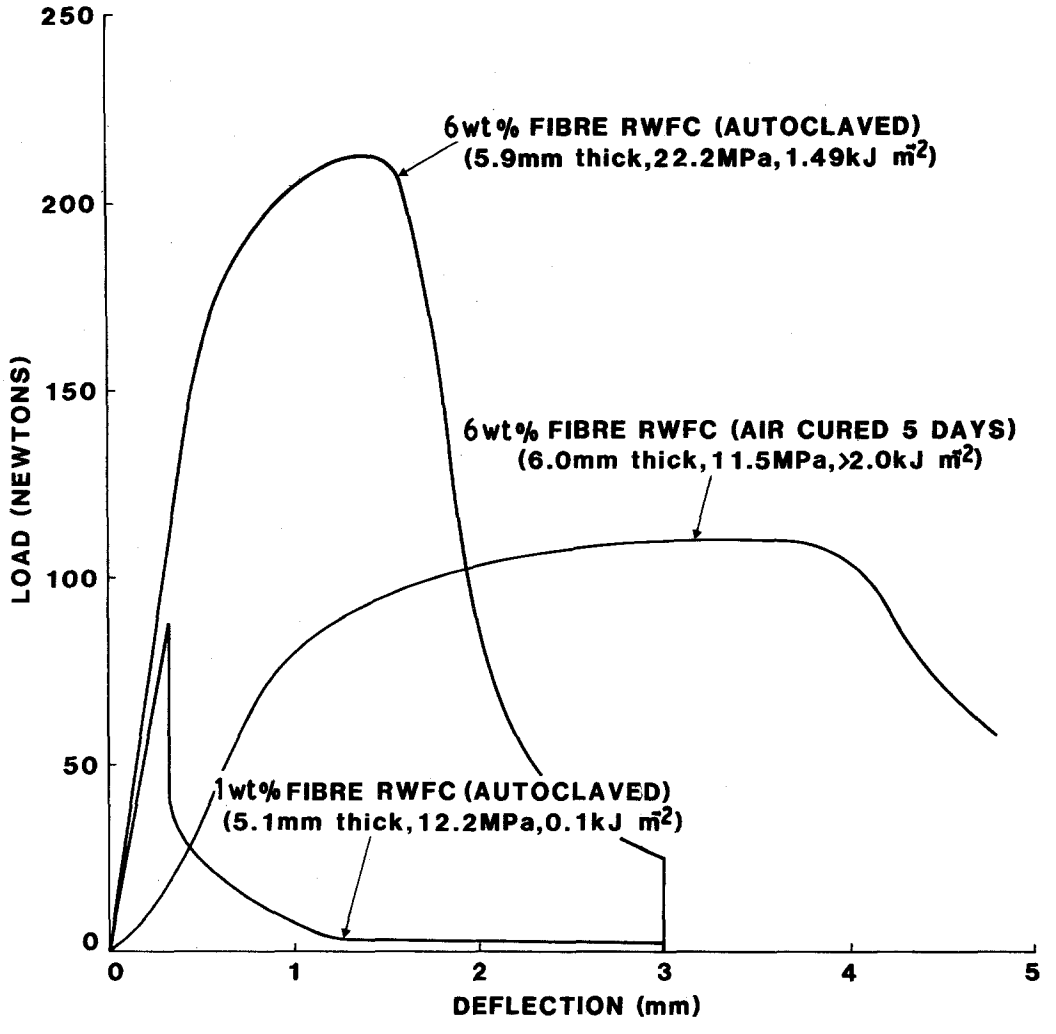

Figure 7 Typical load–deflection curves for WFRC composites.

In the case of the uncured sample (air-cured for 5 days), containing 6 wt % fibre, a low strength was observed but also considerable toughness due to fibre pull-out.

Figure 8 SEM of a fibre removed from the surface of the WFRC composite showing damage to outer layer of the wood fibre.

The bond between the fibre and matrix has been considered to be relatively weak [2, 4, 6], but is shown in this study to be substantial. The bond is sufficiently strong to enable wood fibres with a tensile strength of approximately 500 MPa [7] to be stressed to fracture (see Figs 3, 4c and 5b). The nature of this bond is further demonstrated in Figs 8 and 9 which show the outer layer of the fibre being stripped away when the fibre lies in the plane of the crack, instead of debonding at the interface. The nature of the bond between fibre and matrix is currently under investigation and may be dependent on the presence of hydroxyl bridging and/or hydrogen bonding [5].

4. Conclusions

Examination of the fracture surfaces of autoclaved, refined WFRC shows that the reinforcing fibres

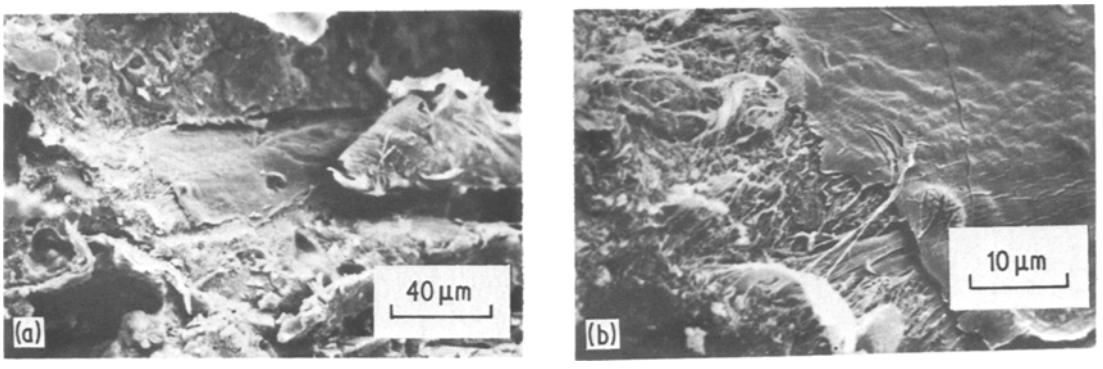

Figure 9 (a) SEM of fibre lying in plane of fracture surface, showing detached outer layer firmly bonded to matrix; (b) high-magnification SEM of (a) showing fibrils attached to cement matrix.

can be stressed to failure. The main mechanism taking place when a composite is tested in flexure is not fibre pull-out, as proposed in earlier work [4], but a dual mechanism of fibre fracture and fibre pull-out, with the former predominating.

The large number of fibres observed to fail between the internal layers of the fibre structure (as seen in the earlier work of Davies [8]), instead of at the interface between the wood fibre and the cement matrix, would suggest that the interfacial bond between the two components of the composite is relatively strong.

It is believed that the mechanism of fracture is dependent upon the moisture content of the sample and work is currently in progress to demonstrate this hypothesis [5].

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