

Micromechanical studies of fresh and weathered fibre cement composites. Part 2: Wet testing

R. B. Tait* and S. A. S. Akerst†

Synopsis This paper presents a study involving in situ SEM fracture studies of fibre cement composite materials tested in a wet environment. An in situ three point bend loading device coupled with 'wet cell' techniques have facilitated the simultaneous loading and observation of micro fracture processes in wet synthetic and cellulose fibre cement composite materials. The micromechanistic fracture behaviour is similar to earlier dry testing in that failure is a complex combination of microcracking, stress redistribution, fibre debonding, fibre pull-out and fibre failure. The onset of the first microcracking stage of this process has been identified as occurring at the limit of proportionality and which develops progressively to the ultimate strength, as the tensile zone moves through the thickness of the composite. During ageing due to natural weathering it is believed that there is an increase in interfacial fibre bond which increases the propensity for fibre failure as opposed to fibre pull-out, as observed, and leads to greater strengths in aged products. The extra effect of localised moisture on performance is substantial additional localised microcracking when wet, as well as a small drop in strength. This may be associated with enhanced stress redistribution and more widespread microcrack initiation under wet conditions.

Keywords Fibre cement composites, cellulose fibres, fracture behaviour, cracking (fracturing), wet testing, moisture content, synthetic fibres, ageing, scanning electron microscopy, composite materials.

INTRODUCTION

One of the major considerations about the serviceability of fibre cement products is their long term service life and durability as a function of moisture, temperature and age. For realistic modelling of such long term behaviour, a micromechanistic understanding of the mechanism of reinforcement under load is required. A recent research programme [1] has directed its efforts towards developing such an understanding of the micro-mechanics of composite failure under load, making use of in situ fracture studies inside the specimen chamber of a scanning electron microscope. The mechanism of strength development and the composite failure has been studied in this way for both freshly prepared material as well as for fibre cement composites subjected to natural weathering and is described elsewhere [1]. Reference has also been made to publications on ageing mechanisms in cement based composite materials.

This previous paper [1] necessarily undertook to examine both fresh and aged specimens prepared conventionally for the SEM, i.e. they were totally and rigorously dried and coated with a carbon and gold palladium layer to facilitate electron imaging in the high vacuum conditions (10^{-7} torr) of the SEM using secondary electron emission. Such drying could be said to inhibit the specimens' fracture behaviour and hence, to complete this in situ SEM fracture study, corresponding 'wet cell' tests needed to be undertaken. This paper describes the details and results of a series of 'wet cell' in situ SEM tests, parallel to those described earlier [1], where the effect of ageing on micromechanic performance was examined. Similarities and differences in micromechanical behaviour under load are highlighted in an effort to add to the interpretation of durability effects, and the consequent service life prediction capability for the composite material.

* Department of Metallurgy and Materials Engineering, University of the Witwatersrand, Johannesburg, R.S.A.

† Ametex AG, Research and Development, Niederurnen, Switzerland.

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EXPERIMENTAL DETAILS

Material

As in the companion paper [1], the composite materials considered here include (a) synthetic polyvinyl alcohol (PVA) fibre and cellulose fibre cement products and (b) autoclaved cellulose fibre cement products. Specimens

were cut to size (typically $42 \times 4 \times 1.7$ mm), polished to 1200 grit finish and soaked for between 24 and 30 hours prior to testing. A final polish was undertaken immediately before SEM observation to remove any newly formed hydration and carbonation products. The specimens were uncoated and inserted into the test rig (and SEM) in a dripping wet condition.

In situ SEM wet cell test system

The in situ SEM loading system used in these tests was based on an earlier in situ double torsion loading system which facilitates crack growth studies in brittle materials [2–6]. In the present case, the three point bend loading rig comprised a small DC electric motor and gearbox driving pulley wheels which retracted two threaded clevis pin loading arms. The specimen was thus loaded in three point bending with its tensile face uppermost and nominally continually in focus and thus available for SEM observation. The central loading point was seated on the spherical load button of a small commercial load cell so that load/time and load/displacement traces could be obtained. The whole system was mounted on the goniometer unit of the SEM so that cracks could be followed, and lead throughs for the electric DC supply for the motor and load cell were achieved using vacuum tight electrical seals which facilitated remote external control and data monitoring. Straining rate was controllable (typically 0.01 to 0.5 mm/sec) and the whole system calibrated.

Of more particular interest in the present application was the so called 'wet cell' facility which is based on a similar development by Diamond, Mindess and Lovell [7]. As this system is described elsewhere [7–9], only salient features will be mentioned here. Instead of using secondary electron emission for observation, backscattered electrons are used (using a Robinson detector) in the SEM (an ISI SX30). This enables much higher contrast and reflectivity from materials of differing atomic number to be imaged, as well as, in particular, virtually eliminating charging artefacts. This detection mode is used in conjunction with a CFAS (charge free acquisition system) unit which enables uncoated wet specimens of nonconducting material to be examined without the normal charging effects.

The existing CFAS unit was coupled to a water bubbler intake system [7] so that moist air was sucked into the chamber of the SEM, separated from the column and at much lower vacuum, which facilitated the examination of wet specimens. The chamber vacuum was independently controllable [at typically 0.2–0.3 torr] which meant that wet composite specimens inserted into the SEM dripping wet could be examined for up to an hour without exhibiting significant drying cracking or shrinkage cracking. Unfortunately the poorer vacuum and moisture in the chamber lead to reduced resolution at high magnification and this is the price paid in such studies using this 'wet cell' technique. Nonetheless, at reasonable magnifications (1000 to 2000 times) resolution was still sufficient to add to the understanding of microfracture of these materials in a wet environment.

Beam damage appeared to be very much less under wet backscatter operation as well, enabling accelerating voltages of 15 kV, as opposed to 2.5 to 5 kV [1], to be used.

As soon as the specimens had been inserted and the appropriate vacuum achieved, the emission system was activated and the CFAS bubbler device stabilised. This whole operation took approximately 3–4 minutes after which time loading of the sample could proceed – either in a continuous or interrupted manner. Typically, the approach was to load the samples to their elastic limit, i.e. to the LOP (limit of proportionality) and arrest loading just as 'non linearity', corresponding to first microcracking, was observed. Close examination of the surface then frequently revealed a microcrack of the order of 1 to 2 microns in width which could then be focused on and observed and further studied while additional loading and cracking took place. In this way the composite crack behaviour prior to ultimate load could be studied in a wet condition.

TEST RESULTS

The response of the composite products to ageing obviously depends on the curing and ageing conditions such as relative humidity, temperature and localised environment, as mentioned elsewhere [1–11]. The development of material strength and toughness, as determined from these present wet cell in situ tests, is, as may be expected, very similar to the interpretation from the dry tests. (Toughness in this context refers generally to the area under the load deflection curve.) The only significant difference is that under wet testing conditions the composites appear to exhibit slightly lower strengths but greater toughnesses compared to dry tests. The basic similarities for both wet and dry tests are that as a function of ageing and weathering there is a progressive increase in flexural strength and stiffness, with an associated increase in degree of carbonation.

With the in situ SEM testing facility it is possible to relate and evaluate the fracture mechanism as observed in the SEM to the position on the load-deflection (or load-time) trace in both a dry condition [1] and wet condition, and these latter aspects are discussed below.

Synthetic fibre cement products

Aged specimens (subjected to six years of natural weathering in Switzerland) exhibited typically less ductility than non-aged specimens, when tested wet (Figure 1). This characteristic is analogous to dry test behaviour [1], where there is significantly less 'ductility' in aged compared to non-aged specimens. It is always difficult in such tests to characterise so called 'typical' behaviour unless one draws up a composite curve from a series of numerous but otherwise nominally identical tests. The present work reflects the results of some twenty eight in situ SEM bend tests as well as incorporating experience from hundreds of other parallel full scale bend specimen tests. The 'typical' results illustrated in Figure 1 refer to a PVA fibre composite material tested in a wet condition.

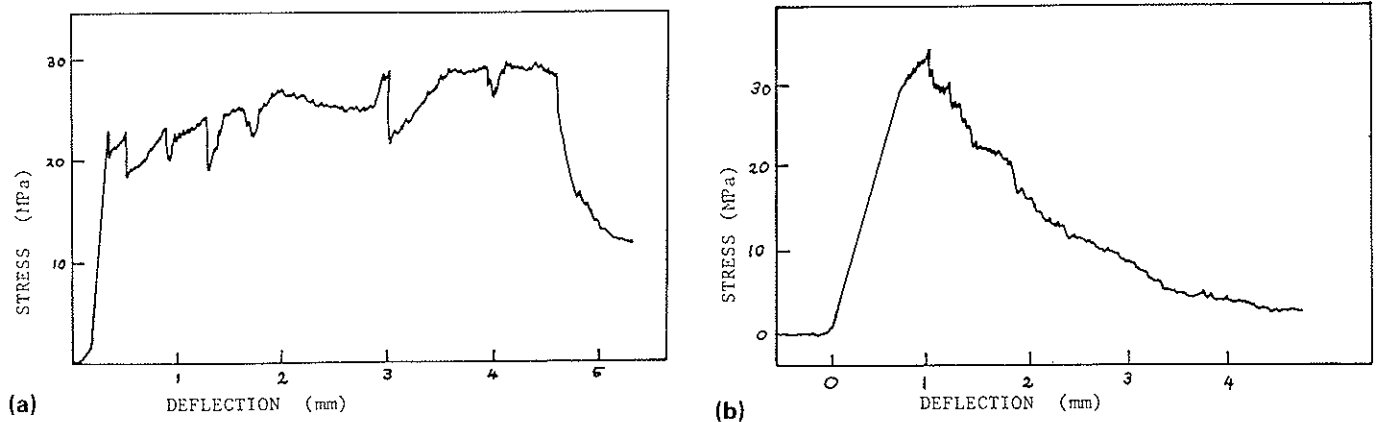


Figure 1 Load deflection curves for (a) non-aged and (b) aged in situ SEM flexural tests of PVA fibre composites tested in a wet condition

An interesting feature of the Robinson/CFAS system is that because of the atomic weight difference of the fibres and matrix and of the way the backscattered electrons are scattered and detected there is more contrast between fibres and matrix than is the case for secondary electron detection. This aspect is shown clearly in Figure 2 and could well prove to be a useful analytical tool if quantitative evaluations of fibre orientation were required by making use of linked quantitative image analysis facilities.

Characteristic features of all the tests were that the first significant microcrack could be determined from the load deflection tests as it was virtually co-incident with the limit of proportionality (LOP). After a further small load increment, this crack extended and frequently exhibited fibre pull-out, typical of fresh specimen failure. Load is transferred to the fibres and on further straining the fibre then increases its load capability until either fibre failure or fibre pull-out occurs. In wet non-aged specimens fibre pull-out tended to predominate.

The single most notable feature of wet in situ SEM fracture testing compared to dry testing was that there was a significant increase in both micro- and macro-cracking under wet conditions. This was especially the case for fresh as opposed to aged samples as can be seen for example in Figure 2 for non-aged PVA fibre composite. Figure 2 in fact shows ten major macrocracks (and this sample actually had a total of thirteen). The phenomenon appears to be quite definitely moisture related and associated with load redistribution. In several cases, the first microcrack was observed when it was (a) the only microcrack, typically 1 micron wide and (b) at the LOP point. On further loading, however, this first crack tended to open only slightly more in such a way that the stresses were transferred to the fibres and the stress redistributed. Consequently another region of high local stress was developed and when it exceeded local strength, a parallel microcrack (or several microcracks) opened up. Some of these ultimately became macrocracks with further loading, Figure 2. Indeed the specimens were strained to the limit of deflection (some 7mm in this test rig) and no single macrocrack failure could be induced.

The interpretation given to these results is that the interfacial bond presumably increases with ageing and, assuming there is no significant deterioration in strength

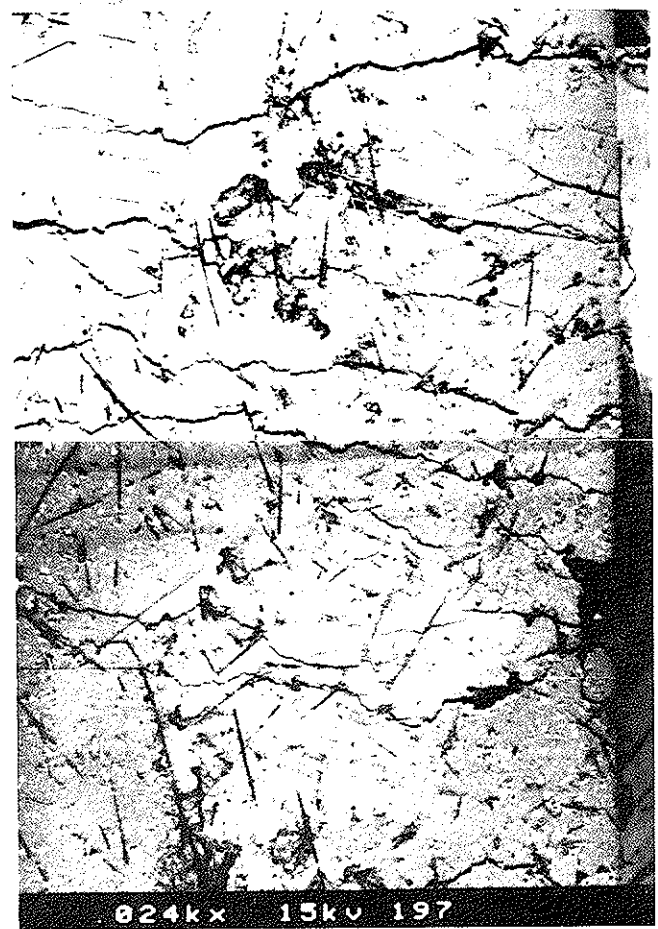


Figure 2 Low magnification wet cell micrograph of non-aged PVA fibre cement composite after loading, illustrating the contrast effect between fibres and matrix with backscatter detection. Note that extensive multiple microcracking is also illustrated for such wet tests

of the fibres, a consequent strength increase with ageing occurs, as observed. This is also consistent with the decreased post LOP toughness with ageing in that fibres in aged composites are more prone to fracture than pull-out [1], thus pull-out lengths are less and toughness is decreased. The scenario when the composite is tested under wet conditions is less obvious but the mechanism that is proposed is that under such (wet) conditions and as a result of small microcracks developing and locally relieving stresses there is increased redistribution of stresses. With a moist environment such redistributed stresses initiate and open up other microcracks at the 'next weakest link'. This is aided by the reduced matrix strength when wet and possibly a water assisted stress corrosion cracking mechanism [8, 10]. This would account for the observed increase in microcracking when wet (Figure 2) as well as the increased toughness (more microcracks, hence more fibre pull-out) in non-aged wet specimens.

Cracking features similar to dry tests

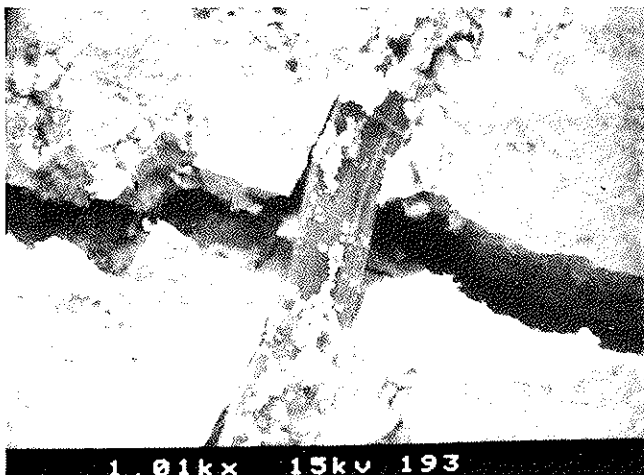
Except for the phenomenon of multiple cracking in wet testing, several of the features observed in dry tests were also observed in wet tests. In particular, despite the more multiple cracking, an increased propensity for fibre pull-out between LOP and ultimate stress in non-aged specimens, tending to more fibre failure in aged specimens, was observed. Evidence of the debonding model suggested in the companion paper on dry testing [1] has also been observed in these wet tested specimens. In Figure 3, fibre debonding, fibre pull-out and shear effects on the fibre during loading of the composite was clearly apparent.

Fibre failure (or fibrillation) in aged PVA containing composite is illustrated in Figure 4, although this occurred very close to the ultimate stress limit and not simply just after LOP as was the position for the cases mentioned earlier.

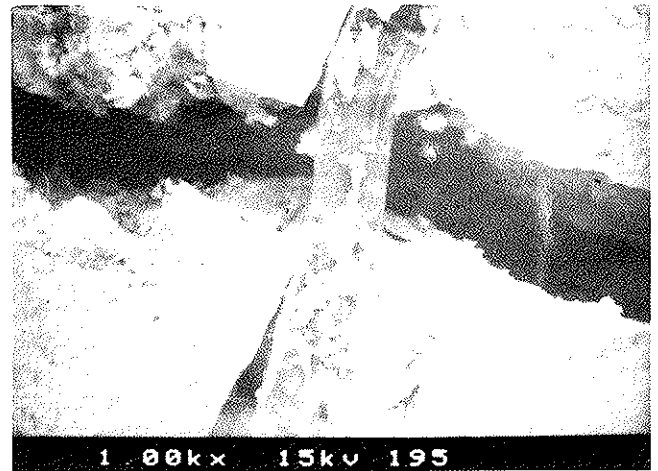
Cellulose fibre cement composite

Load deflection traces of in situ SEM bend tests of cellulose fibre reinforced cement composite tested in a dry and wet condition are shown in Figure 5 (a) and (b) respectively. It is apparent for these cellulose fibre cement composites that there is not the significant post LOP toughness difference between aged and non-aged material as was the case for the synthetic fibre materials. This would suggest that the increase in interfacial bond with ageing is less important than in the case for synthetic fibre materials (which led to greater strength and reduced toughness) but rather that composite failure is controlled by fracture of the fibre itself and that fibre pull-out is less significant. This behaviour is consistent with the single, as opposed to multiple, microcracking nature of the bending beams for cellulose fibre cement composites. It should, however, be remembered that the ageing of the matrix itself also contributes to changes in the mechanical properties, and these changes would be different for autoclaved and non-autoclaved products. Therefore, the mechanism proposed above is one aspect explaining the strength and toughness development.

Under wet test conditions for cellulose composites there does appear to be an increase in post LOP wet toughness compared to dry tests (Figure 5). There is not, however, very much difference between aged and non-aged specimens from a toughness viewpoint although the wet strength is usually lower than the dry strength by up to 20%. This is again consistent with the lower fibre strengths and less pull-out toughness contribution. In the wet tests, however, the increased post LOP toughness is considered to arise from changes in the properties of the cellulose fibre itself which can absorb water. Such water absorbed cellulose fibres presumably have possibly either (a) poorer fibre cement bond hence assisting fibre pull-out (and increasing toughness), or (b) the ability to sustain greater elongation to failure and hence provide greater post LOP toughness. These observations are somewhat preliminary,



(a)



(b)

Figure 3 Fibre pull-out in fresh PVA composite after various load increments. Shear debonding of the fibre is also illustrated

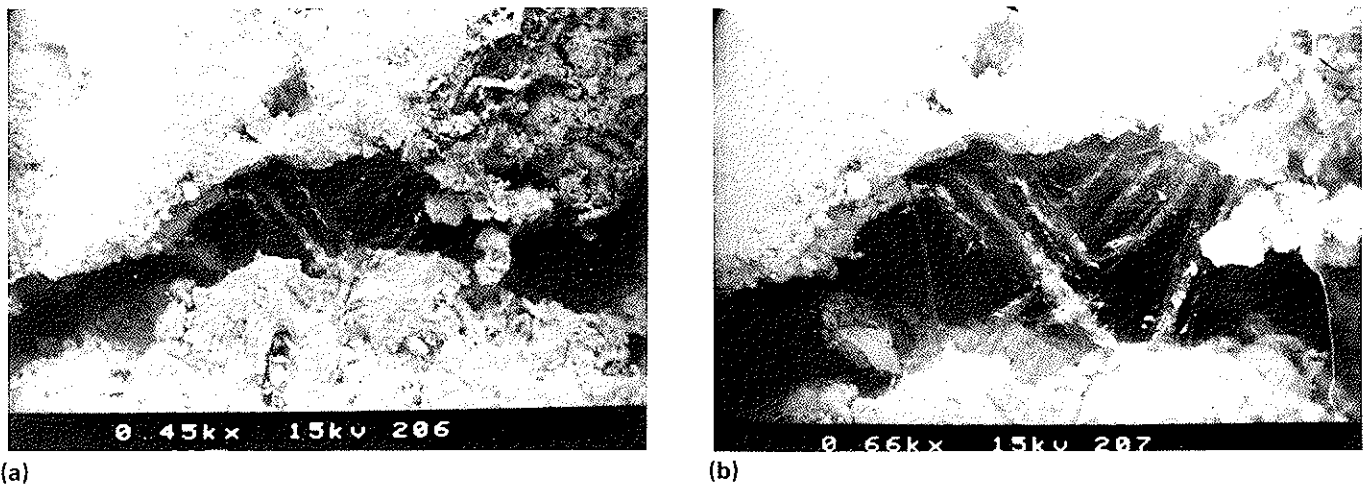


Figure 4 Fibre failure in aged PVA composite (a) before and (b) after an increment of loading

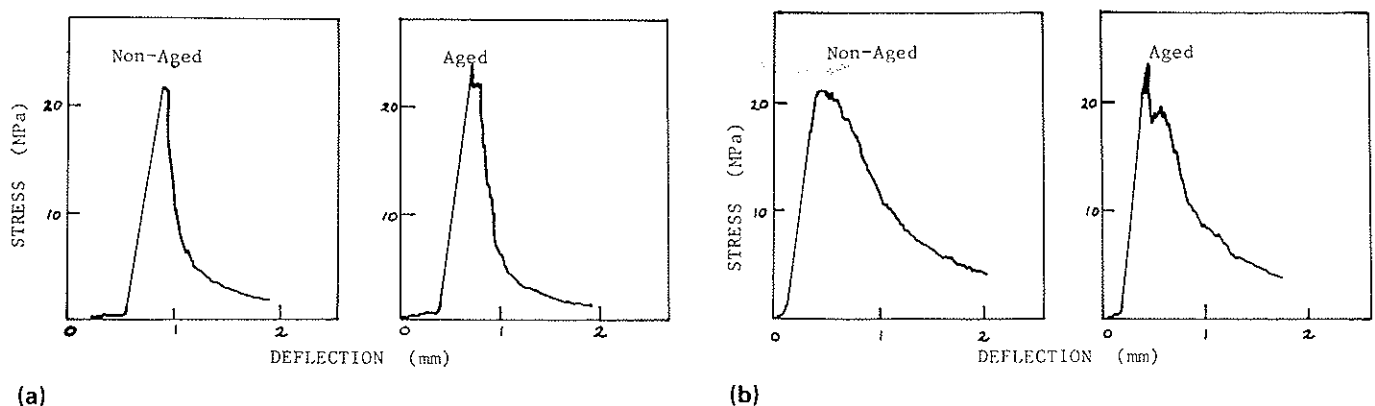


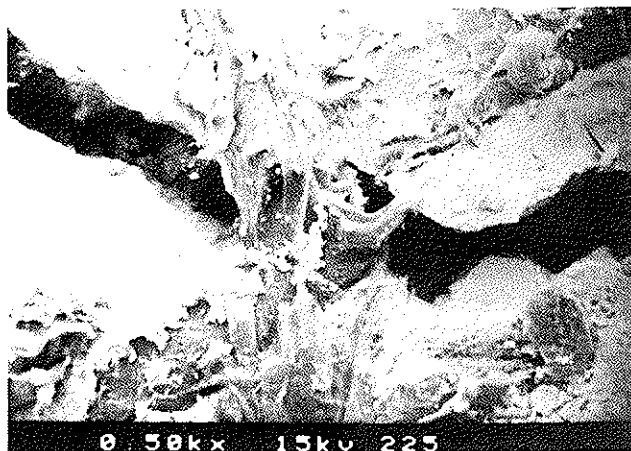
Figure 5 Typical load deflection traces for non-aged and aged specimens respectively of autoclaved cellulose fibre reinforced cement composite tested in situ in the SEM under (a) dry and (b) wet conditions. Although there is no longer the significant difference between non-aged and aged (as was the case for the synthetic fibre composites), the wet tested specimens (b) show higher toughness values compared to dry tests (a)

however, and need further observation for consolidation. This aspect has also been observed by other researchers. Micrographs illustrating progressive crack opening in non-aged wet cellulose fibre cement composite, after two load increments, are shown in Figure 6. Surface fibre failure and some subsurface fibre pull-out (of relatively short fibres) of cellulose are also illustrated in this figure.

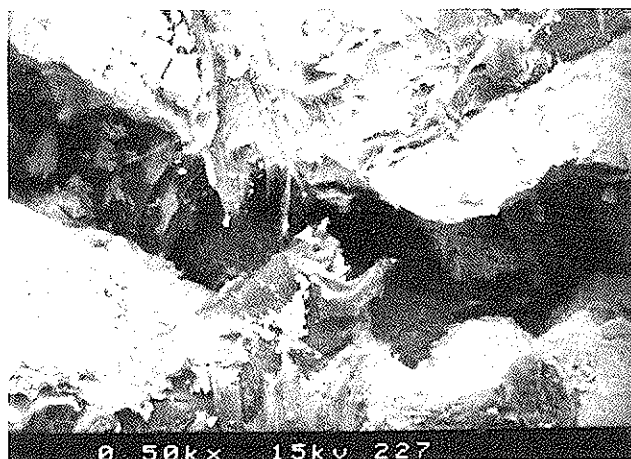
DISCUSSION OF TEST RESULTS

This paper has undertaken to compare the micro-mechanisms of failure of fibre cement composite products using in situ SEM fracture techniques in dry and wet testing conditions as a means of contributing to the understanding of the effect of durability and ageing on such composites. In general, the response of the materials to testing under wet conditions has effectively been similar to dry tests [1] with a few notable additions to be discussed below. From both this work and previous dry studies [1], the transition from pure elastic behaviour (limit of proportionality) to non-linear so called 'ductile' behaviour can be associated with the development of

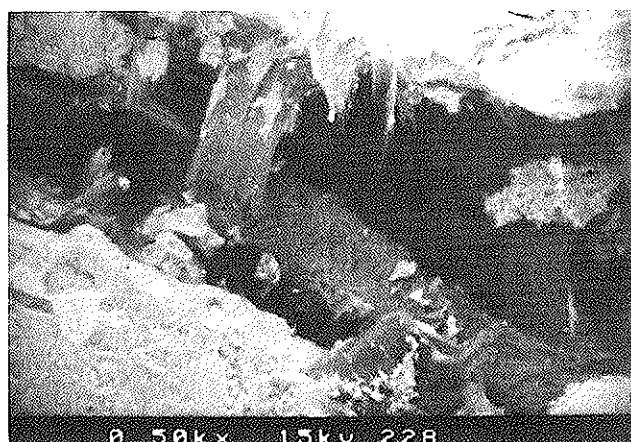
microcracking of the composite. At LOP such deviation has been identified with the first crack in the matrix. Subsequent loading can lead to partial fibre debonding and either fibre pull-out or fibre failure, or a combination of the two [1], depending on bonding conditions, fibre type and degree of ageing. Between LOP and the peak of the load deflection curve (i.e. the materials modulus of rupture, MOR), there tends to be significant stress redistribution as such microcracks bridged by fibres open up. This stress redistribution in turn leads to the development of more microcracking along the concept that 'the next weakest link' of the material fails locally. This localised microcracking from redistributed stresses appears to be enhanced by the presence of water as evidenced by the greater number of micro- and macro-cracks (Figure 2) as well as the greater 'ductile' appearance of the load deflection curves. The cement matrix is acknowledged to contain significant flaws which can readily act as microcrack initiation sites. With the low intrinsic toughness of this matrix material and its increased propensity to cracking when wet [8, 10], it is



(a)



(b)



(c)

Figure 6a–c Surface cellulose fibre failure and subsurface fibre pull-out in non-aged autoclaved cellulose composite material after two load increments, tested wet

consistent that under conditions of high localised tensile stresses in the tensile stress regime of the beam, multiple microcracking occurs.

As the cracks propagate and as other microcracks develop so the cracks widen and fibres either pull out or

fail in some manner [1], depending on local bonding conditions. The tensile stress zone and hence the microcracking region gradually shifts through the depth of the composite as bending increases and as the neutral axis moves closer to the (originally) compressive face. Both these aspects would contribute to the apparent plasticity or ductility of the composite when viewed as a continuum. Beyond MOR some stress relaxation occurs, and certain macrocracks tend to dominate and control behaviour of the composite. The toughness as is observed would be associated with fibre friction and fibre sheen during pull-out as well as non-linear yielding of the fibres themselves.

The effect of ageing on this scenario that is proposed, is that through increased hydration, the fibre to matrix bond improves and the fibre is thus less likely to pull-out. The observed effects in aged specimens of increased strength, reduced toughness, more susceptibility to fibre failure (as opposed to fibre pull-out), lower strain to failure and lower ductility are all consistent with this model.

Under wet conditions, the predominant observations of greater toughness and generally slightly reduced strength for both aged and non-aged specimens of synthetic fibre composites are consistent with the increased observed microcracking and enhanced stress redistribution model. This may be enhanced by possible stress corrosion cracking mechanisms [10] but at this stage, this contention is simply conjecture. For cellulose fibre cement products there is less difference between aged and non-aged behaviour than for synthetic fibre products, but there is again an increase in toughness when wet and a slight decrease in strength. In this case, this behaviour may be due to the cellulose fibres absorbing moisture and thus having lower E-modulus, but increased strain to failure, with lower bonding strength and hence greater post LOP toughness. These latter aspects need confirmation, however, as the role of the matrix should not be neglected in the ageing behaviour of both products.

CONCLUSIONS

1. The micromechanistic fracture behaviour of PVA and cellulose fibre cement composites under wet conditions (as examined using a wet cell in situ SEM system comprising a Robinson backscatter detector, charge free acquisition system (CFAS), variable moisture and pressure control unit and electric motor loading and load measuring unit) was basically similar to dry tests.

2. Failure comprised a complex combination of microcracking, stress-redistribution, fibre debonding and pull-out as well as fibre failure. The limit of linearity or proportionality (i.e. LOP) in load deflection traces is consistent with the development of the first matrix microcrack, typically of approximately 1 micron width. There is a load increase between LOP and the ultimate strength or (MOR) which is associated with the complex microcracking, stress redistribution and fibre pull-out and failure just mentioned.

3. With ageing there appears to be an increase in interfacial fibre bond which leads to a greater propensity to fibre failure rather than fibre pull-out and thus higher strengths and lower toughnesses.

4. In situ SEM tests under wet conditions exhibit similar behaviour to dry tests with the additional effect that there appears to be more microcracking in wet conditions during stress redistribution and an associated toughness increase and slight strength decrease.

5. Cellulose fibre cement products, while exhibiting less difference between aged and non-aged fracture behaviour whether tested wet or dry, still exhibit a significant toughness increase when wet. In the case of these cellulose fibres this may be partially due to moisture absorption by the cellulose fibres themselves.

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