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Micromechanical studies of fresh and weathered fibre cement composites. Part 1: Dry testing

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Synopsis The durability aspects of asbestos fibre replacement programmes in cement based composites have stimulated studies concerned with micro-mechanisms of failure in fibre cement alternatives as a function of ageing. This paper deals with ageing studies related to fracture mechanisms of cement based composites reinforced with synthetic fibres and cellulose fibres. It is aimed at providing background information which could be useful for predicting the durability of the products. Conclusions drawn regarding the mechanisms of ageing have been extracted from flexural tests (tested 'dry') conducted in-situ in the scanning electron microscope. In essence, a complex fracture mechanism has been observed consisting of a combination of multiple cracking, post-cracking debonding, fibre pull-out and fibre fracture in non-aged composites. In aged composites the debonding and fibre pull-out is significantly reduced. This suggests an increase in interfacial bond with age, thereby partly explaining the measured strength and stiffness increase of the fibre/cement products with age.

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

INTRODUCTION

The importance of a clear understanding of the micromechanism of reinforcement with regard to engineering applications such as those found in the building industry has been elaborated on in a previous paper [1]. The micro-mechanisms of failure in asbestos-cement proved to be complex and the authors discuss the difficulties involved in the application of analytical models applied to the composite mechanical behaviour. Research and development programmes concerned with substitute products for asbestos-cement require a basic understanding of the mechanism of reinforcement in order to apply theoretical models to describe the composite mechanical properties. This would have an obvious, significant influence on the choice of substitute materials and reinforcing fibres particularly with respect to the economic implications and service life prediction. In order to assess the durability and predict the service life of newly developed fibre cement products, it is not only a prerequisite to describe the mechanism in a young freshly produced product but also to monitor the development of the mechanical properties during exposure to natural weathering in its particular application.

Cement-based fibre composites made with synthetic fibres such as polyvinyl alcohol (PVA type Kuralon) and cellulose fibres have revealed unconventional aspects concerning the material property behaviour particularly with regard to the development of the composite components and their interactive participation during ageing. The mechanisms describing the material property development with age are very complex and result in certain assumptions incorporated in durability studies and service life prediction.

It is the purpose of this paper to suggest possible aspects and mechanisms of ageing based on dry tested fracture studies which could be a useful guide to the models (presented in other papers [2–5]) incorporated for the service life prediction of the material. The companion paper to this one deals with 'wet' fracture studies [6]. Other workers in this field have also published much literature on the ageing properties in cement based composites [7, 10].

EXPERIMENTAL DETAILS

The mechanical property development was measured on a number of fibre cement products with varied mix formulations. It is beyond the scope of this paper to expound in great depth on the material property development of each particular product tested. It is, however,

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important to mention that an increase in strength and an associated decrease in toughness with age was measured for all products tested. The discussion will therefore be limited to two particular types of composites, these are:

Group (a)	Synthetic/cellulose fibre cement products (these products contain PVA and cellulose fibres in a ratio of 1:1 and polyethylene based fibrids; total fibre content ~8%) and,
Group (b)	Autoclaved cellulose fibre cement products (containing 8% cellulose fibres only and having a cement to silica ratio of 1:1)

The composites were manufactured using the standard Hatschek process used in the asbestos cement industry. The products manufactured in group (a) were cured under ambient conditions and those in group (b) were autoclaved using standard autoclaving procedures in the asbestos cement industry.

In-situ Scanning Electron Microscope (SEM) flexural tests and SEM fractographic studies were utilised in order to investigate and describe the varied failure mechanisms of the composite. The products were cut and polished to exact dimensional beams measuring 42 mm × 4 mm × 1 mm for all SEM studies. The development of this equipment and experimental methods used are complex and are described elsewhere [11].

RESULTS

Synthetic/cellulose fibre cement products (Group (a))

The flexural behaviour of non-aged and aged (6 years natural weathering in Switzerland) synthetic/cellulose fibre composites are significantly different and are depicted by means of the stress/deflection curves given in Figure 1.

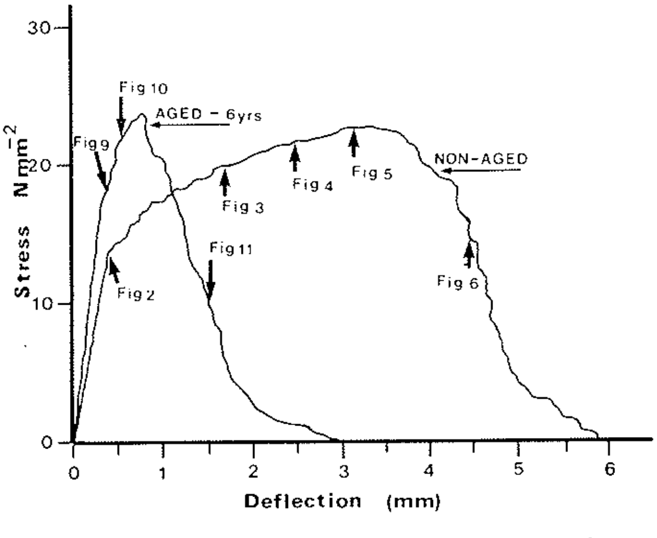


Figure 1 Typical stress/deflection curves obtained from (dry) flexural tests performed on aged and non-aged synthetic fibre products

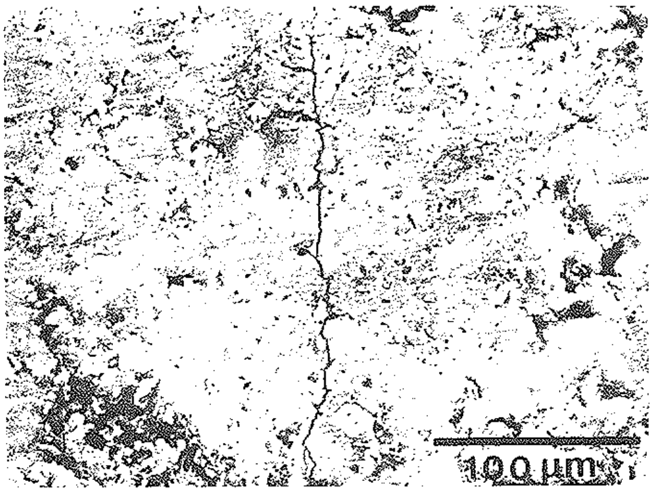


Figure 2a First crack observed at LOP (stress level ~14 N/mm^2)

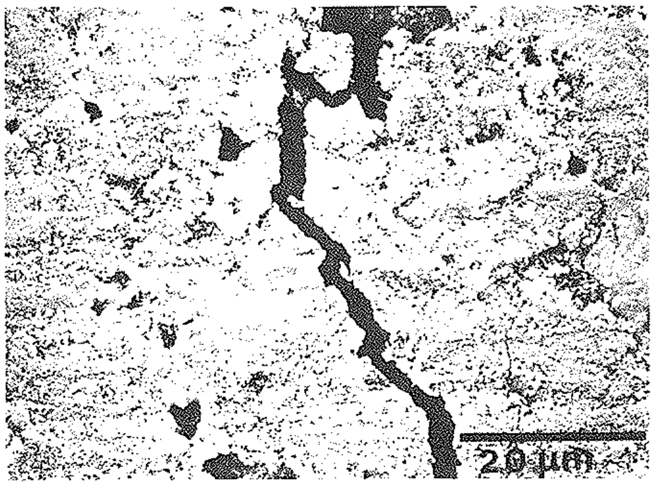


Figure 2b Enlargement of the central portion of Figure 2a crack width ~2 µm (stress level ~18 N/mm^2)

It should be noted that an increase in strength with an associated decrease in toughness with age may be seen from the curves given in Figure 1.

The limit of proportionality which is considered to be an indication of the first major crack (~2 µm wide) was easily detected in the SEM and the micrographs given in Figure 2 represent the event. It was convenient to stop the loading of the specimen at this stage and record the event given in Figure 2.

The crack detected in the tensile zone of the flexural test specimen was subsequently surveyed along its length.

At this point of loading the stress on the composite is then transferred to the fibres bridging the newly opened crack. A convenient fibre bridging the crack in the tensile zone was then chosen for study and the load on the composite was increased. With increased stress on the non-aged composite, fibre/matrix debonding occurred and fibre pull-out was easily recognised (Figure 3). It should be pointed out that the discussion in this section is limited to the synthetic fibre components of this composite only. The next section deals with the cellulose components characteristics.

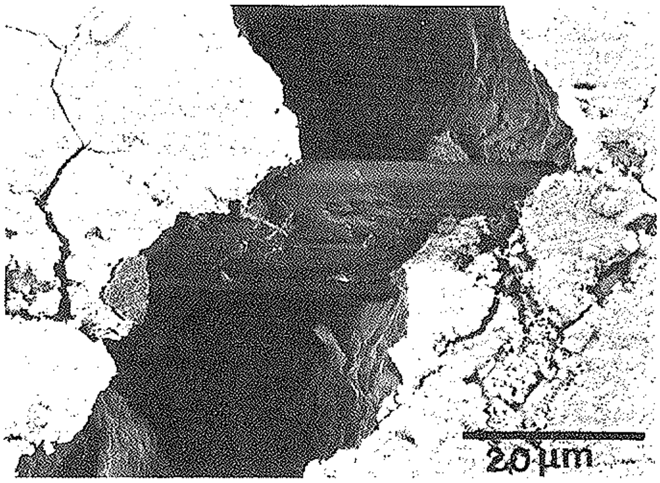


Figure 3 Fibre pull-out depicting a relatively weak interfacial bond (stress level ~20 N/mm^2)

With increased load advanced pull-out (Figure 4) and simultaneous crack opening occurred. At the same time a stress redistribution in the tensile zone of the material takes place. This results in a new formation of secondary cracking commonly called multiple cracking (Figure 5).

The events described thus far occur in non-aged synthetic/cellulose fibre composites prior to ultimate failure of the material, i.e. before maximum load is achieved. Due to the high tensile properties of the synthetic fibres used in the material, they are able to carry the transferred load with increased stress prior to failure. However, although there appears to be significant pull-out prior to ultimate failure of the composite, the synthetic fibres experience a combination of pull-out and fracture. This is depicted in Figure 6 which represents a post failure mechanism.

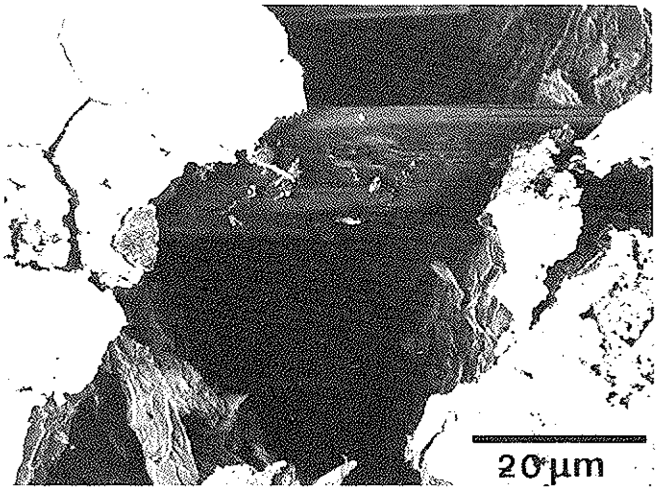


Figure 4 Advanced pull-out with simultaneous crack opening at a higher stress level (~22 N/mm^2)

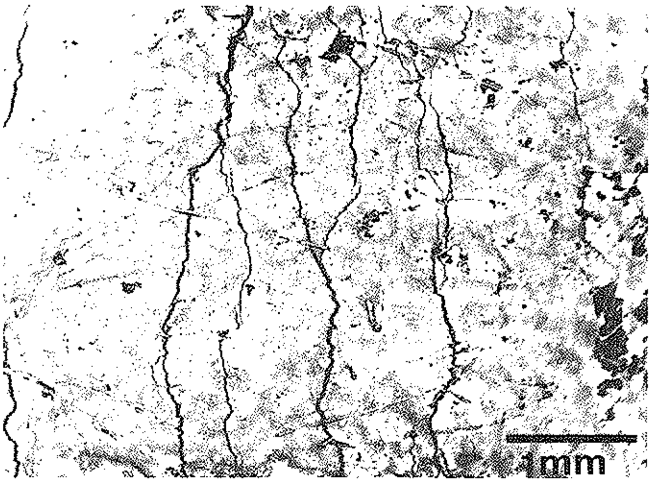


Figure 5 A typical example of multiple matrix cracking associated with fibre pull-out, debonding, crack opening and stress redistribution as secondary cracks form during the failure process

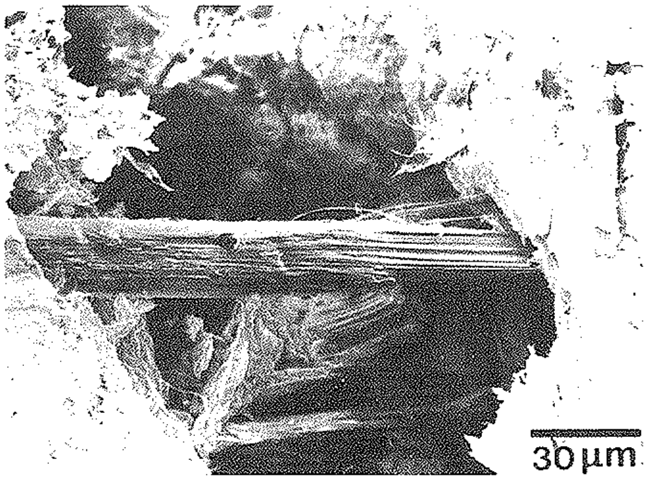


Figure 6 An example of 'post failure' fibre pull-out/fracture occurring at stress levels well below modulus of rupture (MOR) (Figure 1) i.e. after the product has essentially failed

Considering the role of synthetic fibres in the products, two possible mechanisms of failure exist in this type of composite. These are discussed in detail using a model given in the schematic representation in Figure 7.

The failure mechanism observed in non-aged synthetic/cellulose fibre composites consists of fibre pull-out combined with fibre fracture as described by variation 1 (Figure 7). Although pull-out without fibre fracture as in variation 2 was also observed, this mechanism was very rare and is considered to be the exception rather than the rule.

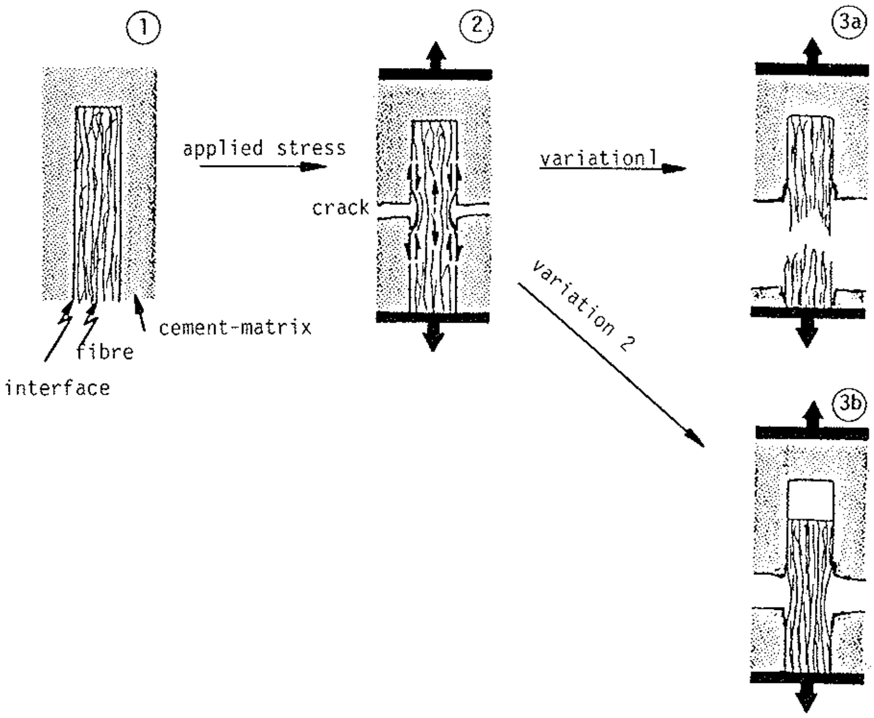


Figure 7
(1) Composite in equilibrium; (2) composite under applied stress; (3a) debonding followed by fibre fracture; (3b) debonding followed by fibre pull-out

In view of the mechanisms described for non-aged synthetic/cellulose fibre composites it would be appropriate now to consider the possible changes in fracture mechanisms which may result from ageing due to natural weathering. By comparison, aged synthetic/cellulose fibre composites display predominantly a fibre fracture mechanism with virtually no pull-out or debonding prior to fracture (Figure 8). If it is assumed that no deterioration has occurred in the synthetic fibres with age then a logical conclusion drawn from comparison of aged and non-aged products is that the interfacial bond improves with age resulting in the reduction in debonding and pull-out in aged products. This has also been reported in literature [7].

Referring back to the stress/deflection curve on an aged synthetic/cellulose fibre composite given in Figure 1, the events captured prior to ultimate failure (Figures 9 and 10) and subsequent to ultimate failure (Figure 11) indicate clearly that fibre fracture or fibrillation had occurred in aged synthetic/cellulose fibre composites.

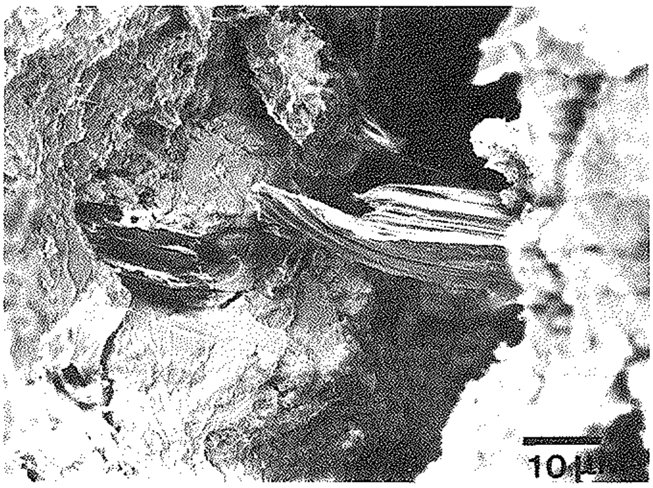


Figure 8 Fibre fracture (sheath/mechanism)

Synthetic/cellulose fibre products are not only comprised of the well defined synthetic fibres but also ill-defined fibrids. The polyethylene based fibrids (having a less ordered physical structure) behave differently. Their contribution as load bearers is not significant but they certainly supply an element of toughness or 'plastic flow' from the time the first crack has occurred to ultimate failure of the composite. Typical events occurring at stress levels between the elastic limit and MOR are given in Figures 12a and 12b. By comparison, aged synthetic fibrid composites did not display the 'rubber-like' fibrid mechanism observed in non-aged composites. There was also no evidence of 'rubber-like' remains on the post examination of the fracture surface of the aged composite. It is, therefore, logical to conclude that the interfacial bond between the fibrids and the cement improves with age and as in the case with synthetic fibres little or no debonding occurs prior to fibrid failure in aged synthetic/cellulose fibre composites.

Cellulose fibre cement composites (Group (b))

Consider the stress deflection curves given in Figure 13. By comparison, synthetic fibre composites appear to be at first glance significantly different in reinforcement characteristics. When looking at the failure mechanisms at varied stress levels on the product, however, there appears to be similarities between cellulose fibre composites and synthetic/cellulose fibre composites. Figures 14 and 15 represent the events occurring at stress levels greater than the first crack but less than MOR of the composite (Figures 13, 14 and 15). Although these events refer to a particular fibre type (eucalyptus) a

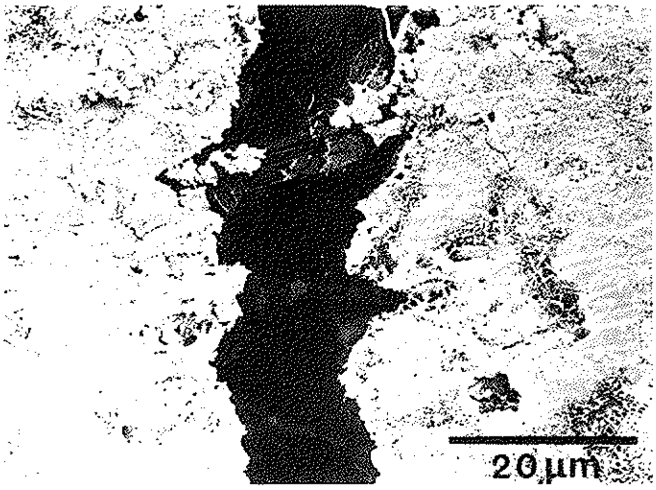


Figure 9 Complete failure of the fibre fracture occurring in an aged product prior to ultimate failure (~18 N/mm^2)

similar sequence of events has been observed for Pinus radiata fibres (Figure 16a and b) and other cellulose types. The mechanism of failure consists of a complex combination of fibre pull-out and fracture. This mechanism has also been reported elsewhere [7].

In aged cellulose fibre composites there appears to be reduced fibre pull-out and a very much more brittle type of failure seen for example in Figure 17.

Comparing aged with non-aged cellulose cement products, the complex fibre pull-out and fracture and combinations of both, occurred in aged as well as non-aged products. This would suggest that the increase in strength and associated reduction in toughness measured in cellulose fibre products with age is not as strongly dependent on the increase in interfacial bond as it is the case for synthetic fibre systems. In other words, for cellulose fibre composites the ageing process would more readily be associated with possible changes in the cellulose fibres themselves in combination with the progressive cement hardening and carbonation and increase in the interfacial bond.

Figure 10 Complete failure of the fibre (depicted in Figure 9) at a higher stress level (~22 N/mm^2)

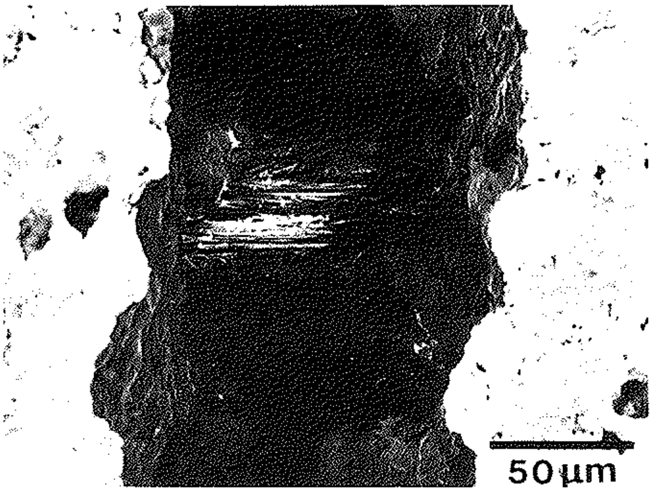


Figure 11 A typical example of fibre fibrillation taking place at stress levels much lower than MOR ('post failure frictional fibrillation')

DISCUSSION OF TEST RESULTS

The material properties of fibre cement products exposed to an external natural weathering climate are influenced significantly by factors such as relative humidity, temperature and the localised environment (for example carbon dioxide etc.) surrounding the exposed products. The products described and studied in this paper in general respond similarly to the above mentioned climatic changes; however, the sensitivity of the response is different for each material and may be related to the individual mix formulations of each product concerned. The exposure of the varied products to natural weathering tests [2–5] resulted in the following development in the material properties:

(a) A progressive increase in flexural strength and stiffness.
(b) An irreversible shrinkage and simultaneous reduction in the reversible wet/dry expansion/shrinkage properties.
(c) An increase in the degree of carbonation and density.

These three aspects are inter-related and significantly influence amongst others the fibre/matrix interfacial bond and the matrix and fibre property development. It is logical therefore to expect that the 'response' of the composite material to a flexural test would change according to its development with age.

The limit of proportionality (LOP) or 'elastic limit' of the composite may be regarded as the stress at which the first significant crack occurs with simultaneous debonding of the fibre from the interfacial zone. With increased stress subsequent fibre pull-out/fracture with

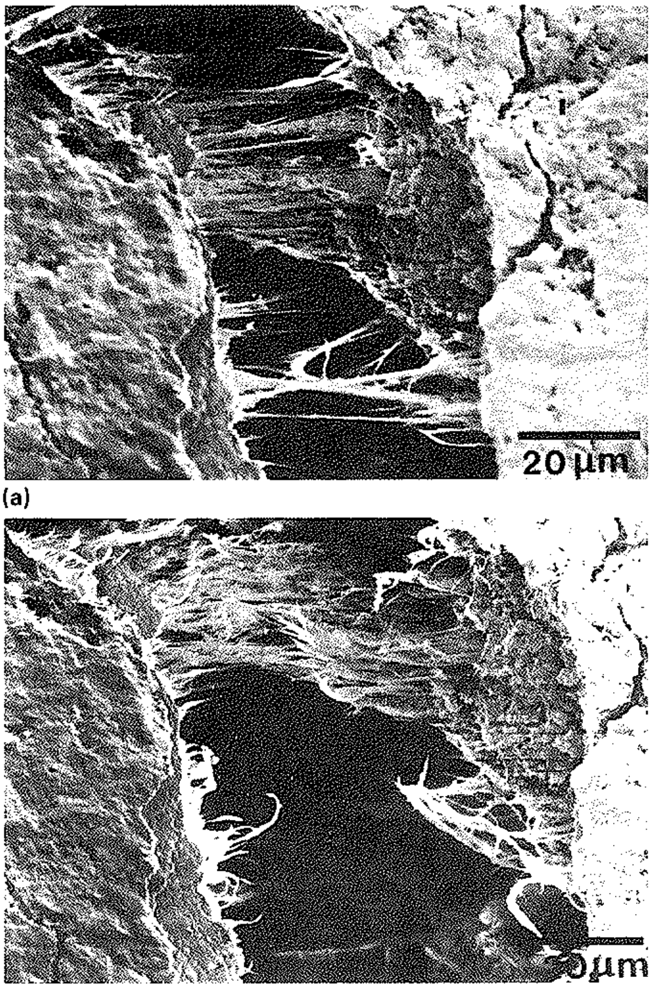


Figure 12 Fibrid fracture occurring with increasing load from micrograph 12a to 12b in non-aged synthetic fibre composites. After failure the fibrids retract or 'curl up', indicative of being stretched beyond their elastic limit

progressive debonding occurs in the tensile zone of the composite material. Because of the shift in the neutral axis from the tensile towards the compressive zone, which is the feature of a flexural type test, progressive debonding and fibre pull-out/fracture occurs in progressive stages through the thickness of the test specimen as well. This accounts partly for the so called plastic zone at stresses between LOP and MOR (modulus of rupture). In summary, the mechanisms of failure occurring at stresses greater than LOP but smaller than or equal to MOR may be considered to be a complex combination of multiple cracking, stress redistribution, fibre debonding, fibre pull-out, fibre fracture and a shift in the tensile zone towards the compressive zone through the specimen thickness.

At MOR the opening up of secondary cracks (multiple cracking) ceases and subsequent stress relaxation in the secondary cracks has been observed. (This has been reported previously [1].) The major crack which eventually leads to complete failure of the composite material propagates through the thickness of the specimen resulting in a continuation of fibre debonding, fibre pull-out and fibre fracture. Frictional shear forces are set up due to the pull-out of the fibre (fibre/matrix shearing) and fibre fracture (fibre/fibre shearing).

In essence, the mechanims of failure described in the previous paragraphs apply to both synthetic/cellulose fibre and cellulose fibre cement based composites, the only difference being that in non-aged synthetic/cellulose fibre composites the 'pseudo/ductile' zone

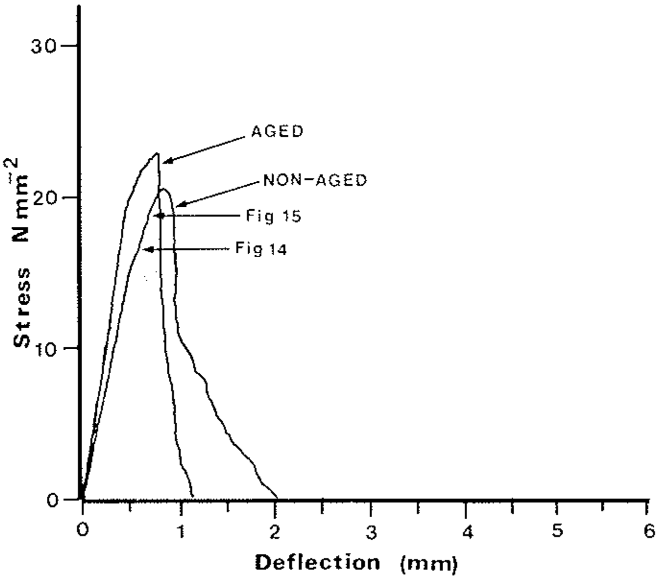


Figure 13 Stress/deflection curves obtained from dry flexural tests performed on aged and non-aged cellulose fibre-cement products (autoclaved)

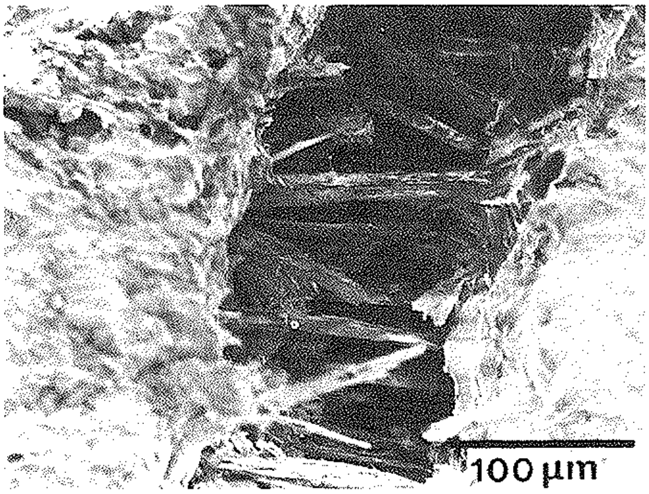


Figure 14 Cellulose fibre eucalyptus pull-out/fracture occurring in non-aged autoclaved products prior to ultimate failure (stress level of ~18 N/mm^2)

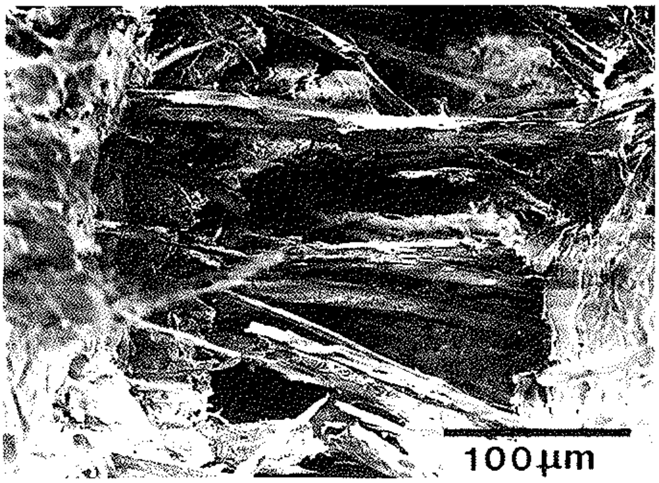


Figure 15 Advanced fibre pull-out/fracture prior to failure at a stress level of ~20 N/mm^2. This event is an advanced stage of the failure depicted in Figure 14

between LOP and MOR is more pronounced than with cellulose fibre composites. This is due to the relatively weak interfacial bond and stress/strain properties of the synthetic fibres and fibrids typical of freshly produced products. With age, however, the interfacial bond increases and this so called ductility is reduced.

This is one aspect accounting for the strength and stiffness increase with age. It should be pointed out though that carbonation of the bulk matrix is also a contributing factor to the strength development and embrittlement. This aspect has been discussed in more detail in separate publications [3–5] and extensively in the open literature [9, 10].

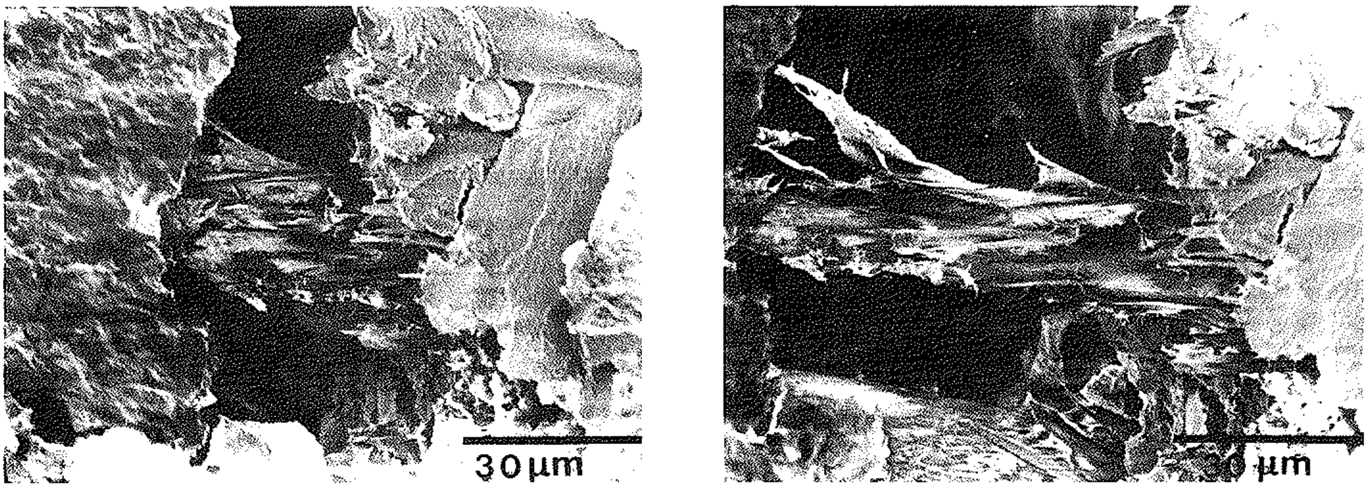
In cellulose fibre composites the cellulose fibres do not have regular dimensions and may also react chemically with the cement matrix [3, 4]. The mechanical and/or chemical bonding of these cellulose fibres to the matrix therefore is much different to those of the synthetic fibres which have regular dimensions and are virtually chemically inactive with the cement matrix. In aged cellulose fibre products, a more brittle fracture of the fibres is evident. It is suggested that this could be due to an increase in the interfacial bond and possibly a chemical or physical interaction of the cement hydration products with the cellulose fibres. The latter mechanism is complex and has been expanded upon in other publications [3–5].

CONCLUSIONS

1. The mode of failure of synthetic (Group a) and cellulose (Group b) fibre cement based composites consists of a complex combination of multiple cracking, fibre debonding, stress redistribution to secondary cracks, fibre pull-out, fibre fracture and a shift of the tensile zone towards the compressive zone through the specimen thickness.

2. It is suggested that during the ageing process in natural weathering an increase in the interfacial (fibre/matrix) bond results in reduced fibre pull-out and multiple cracking in synthetic fibre composites.

3. In cellulose cement products, a similar mechanism is evident with a possible chemical and/or physical interaction of the fibre and matrix as well. The increase in the interfacial bond with age is one aspect accounting for the composite strength increase with age.



(a) **(b)**

Figure 16a–b Similar events for pinus radiata fibres as depicted in Figures 14 and 15 for eucalyptus fibres. These events represent the failure mode of pinus radiata fibres prior to ultimate failure of the composite

Figure 17 A typical example of reduced fibre pull-out/ fracture occurring in aged cellulose fibre products

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