JOURNAL OF MATERIALS SCIENCE 21 (1986) 289–296

The glass fibre/cement bond

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The multiple fibre pull-out test developed at the Building Research Establishment for determining the bond strength between glass fibre strands and cement is described. Data are presented on bond strength as a function of time and storage condition for Cem-FIL 2 fibre and compared with data for Cem-FIL fibre. The results are discussed.

1. Introduction

In recent years there has been considerable progress in understanding the factors that determine the strength properties and durability of glass fibre reinforced cement (grc). The factors include the strength of the fibre and of the matrix, and their interaction, both chemical and mechanical. While a considerable amount of data has been published on the change with time of the strength of the glass fibre strands in a cement environment [1–3] only limited data are available on the properties of the interfacial region that affect the transfer of stress between fibre and matrix. In particular, there is a dearth of information on the strength of the bond between the fibre strand and the matrix, and the effect of prolonged storage on the bond strength. This arises at least in part because of problems in obtaining a reliable measurement of bond strength.

Bond strength is most often determined using a pull-out test [4] in which the load to debond and pull-out a fibre from a button of the matrix is measured. This apparently simple test presents considerable difficulties even when performed on single rods or filaments, both because of the high variability of the results, and because of problems in interpretation. Lawrence [5] and later Bartos [6] have shown how, in principle, the pull-out data can be interpreted in terms of the interfacial bond and the frictional bond opposing pull-out after the interfacial bond has failed. In practice, however, the method has been applied only to air-cured samples of a high water/cement ratio, that is where the fibre strand strength is high and the bond strength low. Uncertainty also arises from the multi-filament nature of the fibre strand used in commercial grc composites.

Previous work [7] has suggested that it is the frictional bond rather than the interfacial bond that is important in brittle matrix/fibre composites, and Laws [8] has shown recently, how this relates to the average bond strength obtained from a pull-out test.

In this paper a multiple fibre pull-out test is described which was developed to reduce the variability of the results, and at the same time to simulate more accurately conditions in an actual composite. Data are presented on the average bond strength for alkali resistant glass in OPC (ordinary Portland cement) as a function of time and storage condition.

For most of the work, Cem-FIL 2* fibre has been used. Cem-FIL 2 fibre is a second generation alkali resistant fibre; its composition is similar to that of the earlier Cem-FIL fibre but its alkali resistance has been improved by surface treatment. For comparison, some limited tests have been carried out using Cem-FIL fibre, and these results also are presented.

2. The multiple fibre pull-out test

In the test, 16 parallel fibre strands mounted in two rows of eight, are pulled out simultaneously. The test specimens are cut from two strips (each providing five specimens) produced using the mould (Fig. 1) shown in section in Fig. 2.

The mould was constructed mainly of "Tufnol" and consists of three bars 6.5 mm × 17.5 mm × 483 mm, which form the "sides" and central separator of the assembled double mould (see Fig. 1). These are separated at each end by circular cylindrical rollers (E, Fig. 2) over which the ends of the fibre strands are located. The "base plate" (Fig. 2) consists of sections (types A and B) shaped to locate the brass guide plates C, through which the strands are threaded. The sections are held in the mould by long brass bolts D which are fitted from one of the sides of the mould, through the base plate section, etc, to the other side. The brass fibre guide plates contain 2 rows of eight holes 1 mm in diameter. The separation between rows is ~1.5 mm, and the separation within rows is ~3 mm. They are fitted in pairs as shown and perform the double function of holding the fibres in position during specimen fabrication and of transferring the load to the finished specimens during the test. The guide plate adjacent to the pull-out section of the finished specimen acts also as an artificial crack, and to encourage matrix fracture and pull-out to occur at a well-defined point, the holes in the guide plate are hemispherically bevelled on the side facing the partner guide plate. The mould has the advantage over earlier

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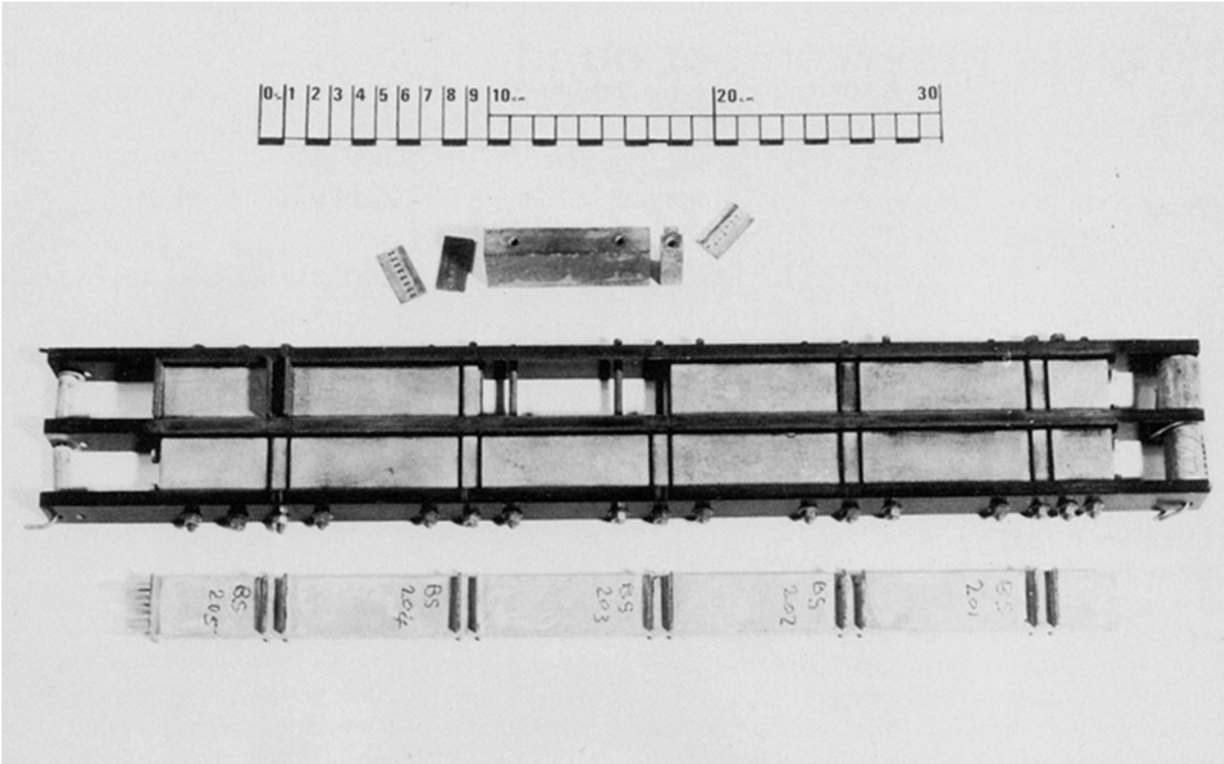

Figure 1 The mould, partly assembled, and a strip specimen.

designs in that, being fully demountable, it can be removed with minimum damage to the strip specimens. To further facilitate this release, the assembled mould and guide plates are coated with a PTFE release agent before the fibres are threaded.

Sixteen fibre strands are threaded by hand through the guides in the assembled moulds and are taped over the rollers E at one end. Small weights (steel nuts) are tied to their free ends to hold them taut while the matrix is poured. The cement paste is hand mixed and, to ensure even filling of the mould and penetration of the paste between the strands, the mould is vibrated on a vibrating table while the paste is being poured.

The specimens are allowed to set and are stored as strips. Each strip is subsequently cut into five specimens leaving a length of several centimetres below the lower brass guide to anchor the fibres at this end. The pull-out length is the length allowed above the top brass guide. Specimens are mounted in the double jig shown in Fig. 3, which is fitted via locating "pins" to the tensile testing machine. The "average bond strength" is obtained from the maximum load achieved as the fibre strands are withdrawn from the top (pull-out) length.

3. Materials and test conditions

The fibre was in the form of strands consisting of approximately 200 individual filaments, each 12.5 μm diameter. Unless otherwise stated, the pull-out samples consisted of 16 fibre strands. The cement was ordinary Portland cement (OPC) at a water/cement ratio of 0.3.

The specimens were prepared under ambient laboratory conditions and then transferred to conditions of 90% r.h. and 20° C and allowed to set. Most were demoulded approximately 24 h after manufacture, and after excess cement had been removed, were stored at 90% r.h. and 20° C. After 7 days (from manufacture), the specimens were transferred to one of three storage conditions, i.e. air at 40% r.h. and 20° C; water at 20° C; or water at 50° C. The strips were cut into individual specimens approximately 24 h before testing. The pull-out lengths varied from the minimum length practicable (about 1.5 mm) to a maximum of about 10 mm. A portion of the material abutting the top (pull-out) section was retained for porosity determinations.

Pull-out tests were performed using an Instron tensile testing machine at a crosshead speed of 1 mm

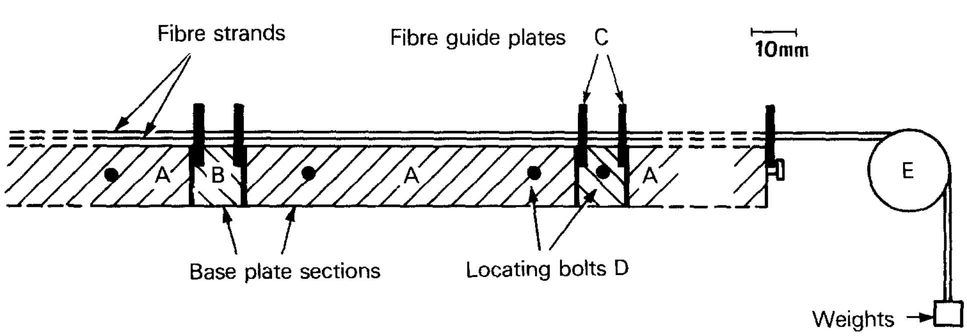

Figure 2 The mould (section view) showing details of the base plate, fibre guides, etc.

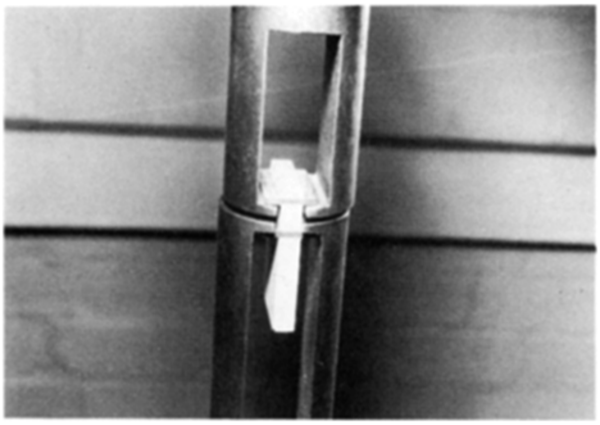

Figure 3 Specimen mounted in the double jig, ready to test.

min^{-1}, under constant temperature and humidity conditions (19° C, 40% r.h.). Water stored samples were tested wet. Pull-out lengths (i.e. "fibre embedded lengths") were obtained directly using a calibrated eyepiece in a low power stereoscopic microscope. Measurements were made from the point of emergence of the strands from the cement (i.e. from the top of the "dome" produced by the holes in the brass guide plates). Where strands had been partly broken during pull-out, measurements were made to the end of the intact filaments. Normally the average of six strands per specimen was taken.

Porosity was determined from the water uptake following drying at 100° C, evacuation and saturation with degassed water; the volume being measured by the upthrust on the saturated material suspended in distilled water.

4. Results

Load/displacement curves during "pull-out" generally were of the three types illustrated in Fig. 4. At sufficiently short embedment ("pull-out") lengths, the load rose to a maximum (curves C and D) and then fell slowly as the fibre strands pulled out. In some cases the slope of the curve beyond the maximum was initially fairly constant, and then decreased as the load fell to zero. At longer embedment lengths, where there is substantial fibre breakage, curves of the type A resulted. Embedment lengths in between the "pull-out" and fibre breakage curves resulted in curves such as curve B illustrated.

The maximum load achieved during a typical test using Cem-FIL 2 fibre is plotted against the pull-out length (i.e. "embedded fibre length") in Fig. 5a. In all the tests the scatter is considerable, but there is nevertheless an indication of an initial "linear" increase of

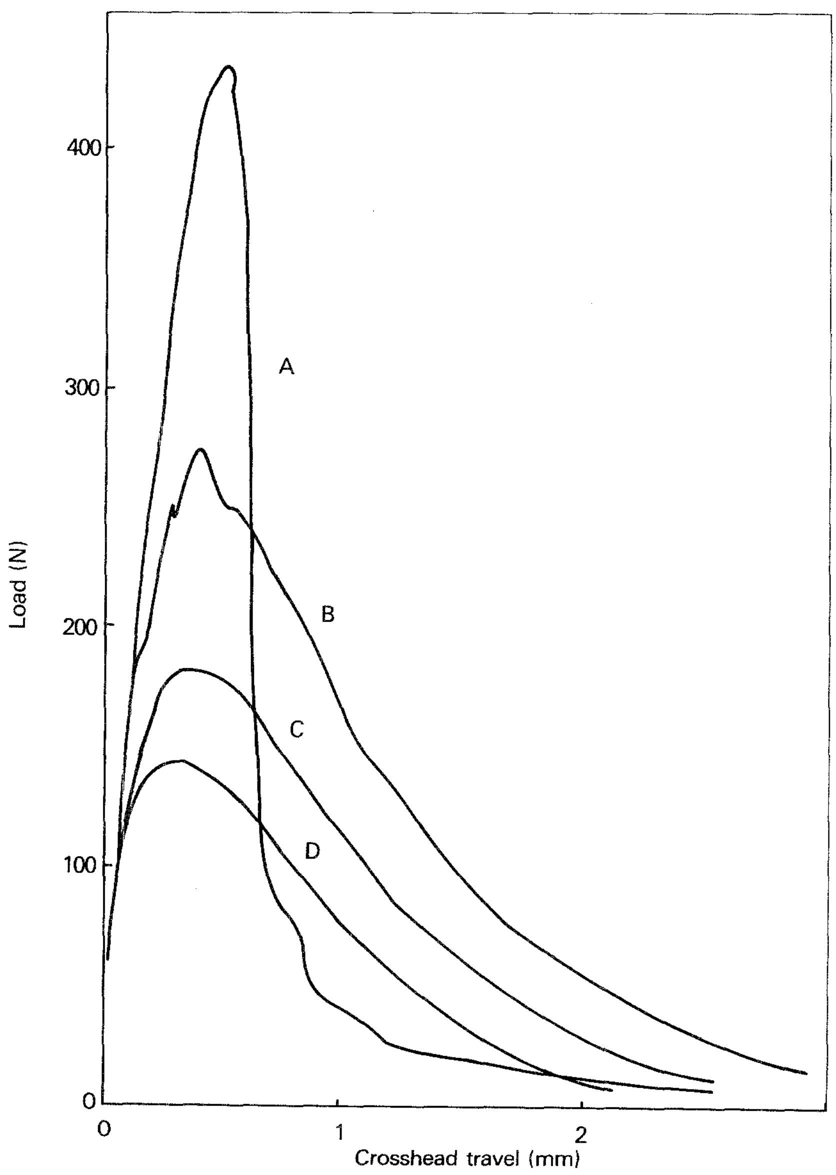

Figure 4 Load/displacement curves during "pull-out". Embedment lengths were: A, 10.3 mm; B, 5.4 mm; C, 4.5 mm; and D, 3.5 mm. Cem-FIL 2 fibre, 28 days air storage at 20° C and 40% r.h.

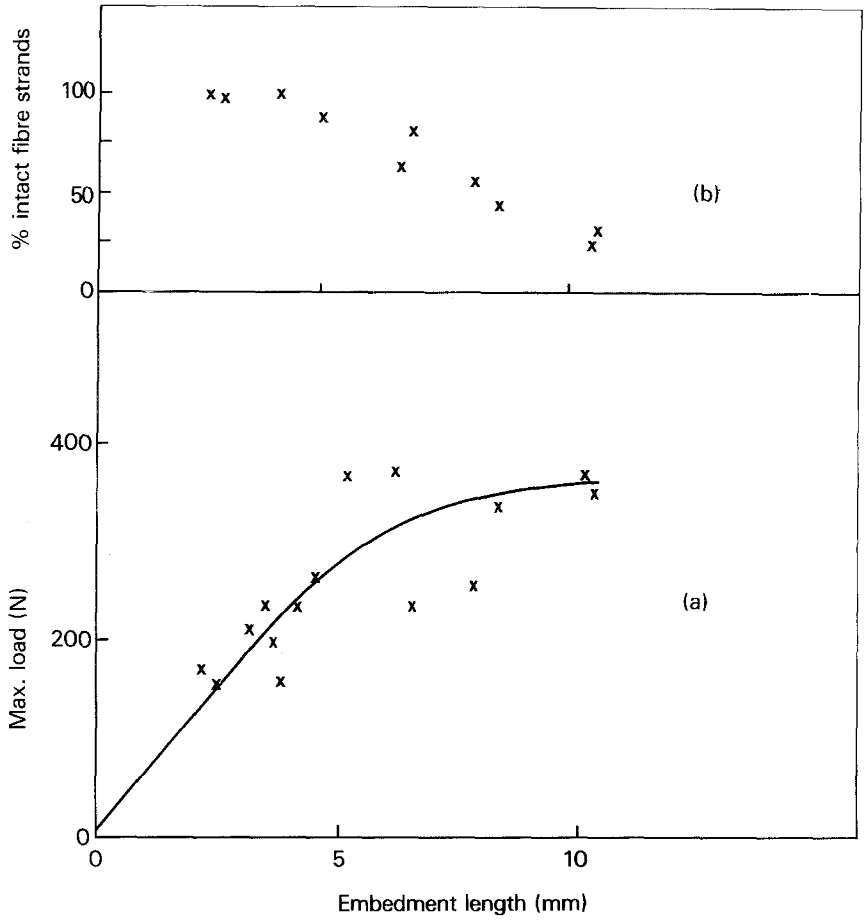

Figure 5 (a) Maximum load achieved during test, and (b) proportion of intact fibre strands remaining after test, as a function of embedment length. Cem-FIL 2 fibre, 7 days initial cure in air at 20° C and 90% r.h.

load with embedment length, followed by a region over which an increasing number of fibres break (Fig. 5b) and the pull-out load approaches a constant value (the load to break all the fibres). At sufficiently short embedment lengths most of the fibre strands remain intact and the average bond strength, τ, is simply given by

$$\tau = \frac{P}{npl}$$

where P is the maximum load achieved, n is the number of fibre strands, p is the "fibre" perimeter at the interface, and l is the embedment length. In this work, however, the product of perimeter and bond strength ($p\tau$), that is the pull-out force per fibre strand per unit length, is calculated rather than τ alone, because of the uncertainty of the value of p that applied to the multifilament strands of variable shape in a porous matrix. $p\tau$ is, in effect, the slope of the initial part of the load (per fibre strand)/embedment length curve.

During the 7 day initial cure period of the Cem-FIL 2 fibre samples at 90% r.h. and 20° C, the value of $p\tau$ increased from 1.4 N mm^{-1} at 1 day to 4 N mm^{-1} after 7 days. On transfer to water storage at both 20 and 50° C, $p\tau$ decreased. The effect of storage time and condition is shown in Figs. 6 and 7. For water storage at 20° C, $p\tau$ increased with storage time. Storage in air at 40% r.h. and 20° C led to lower values of $p\tau$, and storage time had little effect (Fig. 6). At 50° C, $p\tau$ appeared to reach a maximum value and then decrease slowly (Fig. 7). Prolonged water storage at 50° C of Cem-FIL 2 fibre samples led to lower values of $p\tau$ than water storage at 20° C.

Tests using Cem-FIL fibre comprised initial cure and storage in water at 50° C only, for a maximum of 28 days. Nevertheless where there is a comparison, values of $p\tau$ for Cem-FIL 2 fibre are generally higher than those for Cem-FIL fibre (Fig. 7). Unfortunately the extent of fibre breakage made it impossible to obtain values of $p\tau$ for the Cem-FIL fibre specimens in water at 50° C at storage times beyond 28 days and it is not known therefore whether the trend of increasing bond strength with time continues.

At long embedment lengths most fibres break, and the maximum load achieved approaches the failure load of the array of fibre strands. The maximum load achieved at approx 10 mm embedded length is plotted in Fig. 8 as a function of time of storage in water at 50° C for both Cem-FIL fibre and Cem-FIL 2 fibre and in water at 20° C for Cem-FIL 2 fibre only. Provided that all the strands break, the strand strength can be calculated. However, in some cases, particularly in the early stages of storage, some did not break (Fig. 5b), and the average maximum stress on the fibre strands during "pull-out" at 10 mm embedment length then underestimates the strength of the fibre strand. Nevertheless the results suggest that the strand strength of the Cem-FIL 2 fibre stored in water at 20° C did not change much over the period of the test. Storage in water at 50° C resulted in decreased strength for both Cem-FIL fibre and Cem-FIL 2 fibre, the latter consistently showing the higher strength.

The porosity (Fig. 9) of the samples stored in air at 40% r.h. and in water at 20° C fell continuously over the test period (720 days). In water at 50° C (Fig. 10) the porosity dropped sharply and at the end of the test

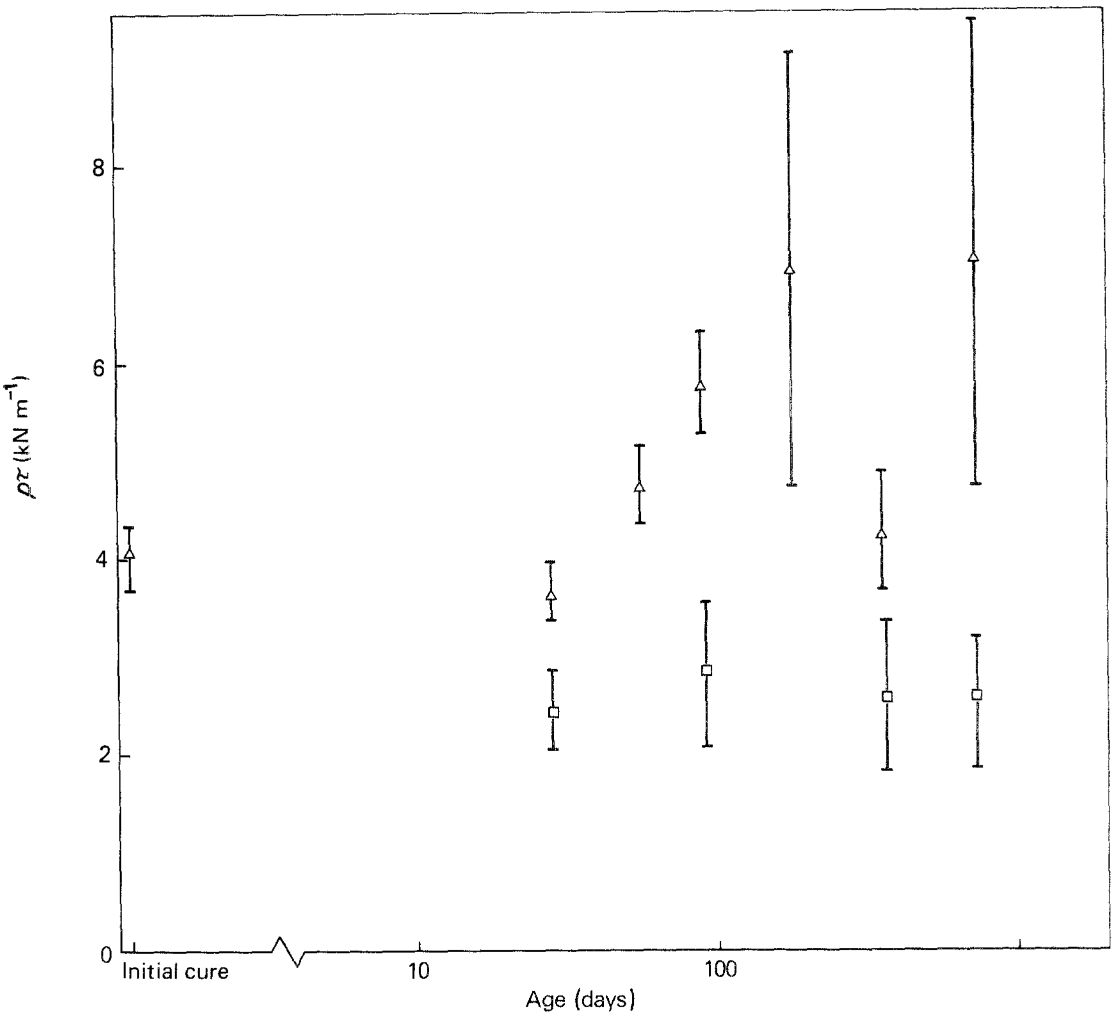

Figure 6 Variation of bond strength with time of storage at 20° C. Cem-FIL 2 fibre. Δ, cured in water; □, cured in air at 40% r.h. p is the perimeter of the fibre strand. The 90% confidence limits are shown.

period (90 days) had apparently reached a constant value which was lower than that resulting from an equal time in either of the other storage conditions. On continuous storage in air or water at 20° C, however, the porosity continued to decrease and after 720 days was well below the "constant" value attained after 90 days' storage in water at 50° C.

5. Discussion

The load/displacement curves during pull-out where there was no fibre breakage, showed the expected rise to a maximum followed by what appeared to be a linear descending portion as the fibre strands were withdrawn from the matrix. The slope of this descending region, however, is higher than the value of $p\tau$

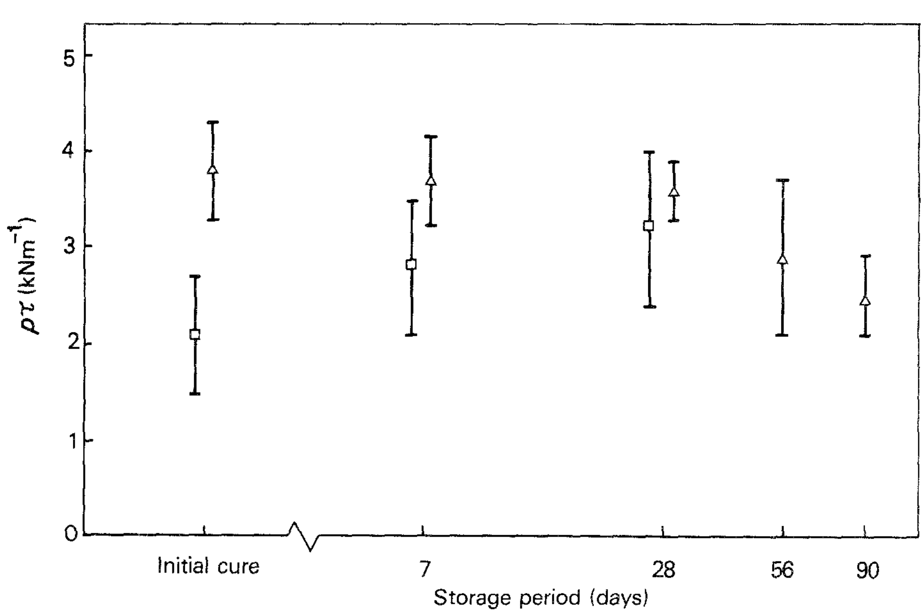

Figure 7 Variation of bond strength with time of storage in water at 50° C. Δ, Cem-FIL 2 fibre; □, Cem-FIL fibre. The 90% confidence limits are shown.

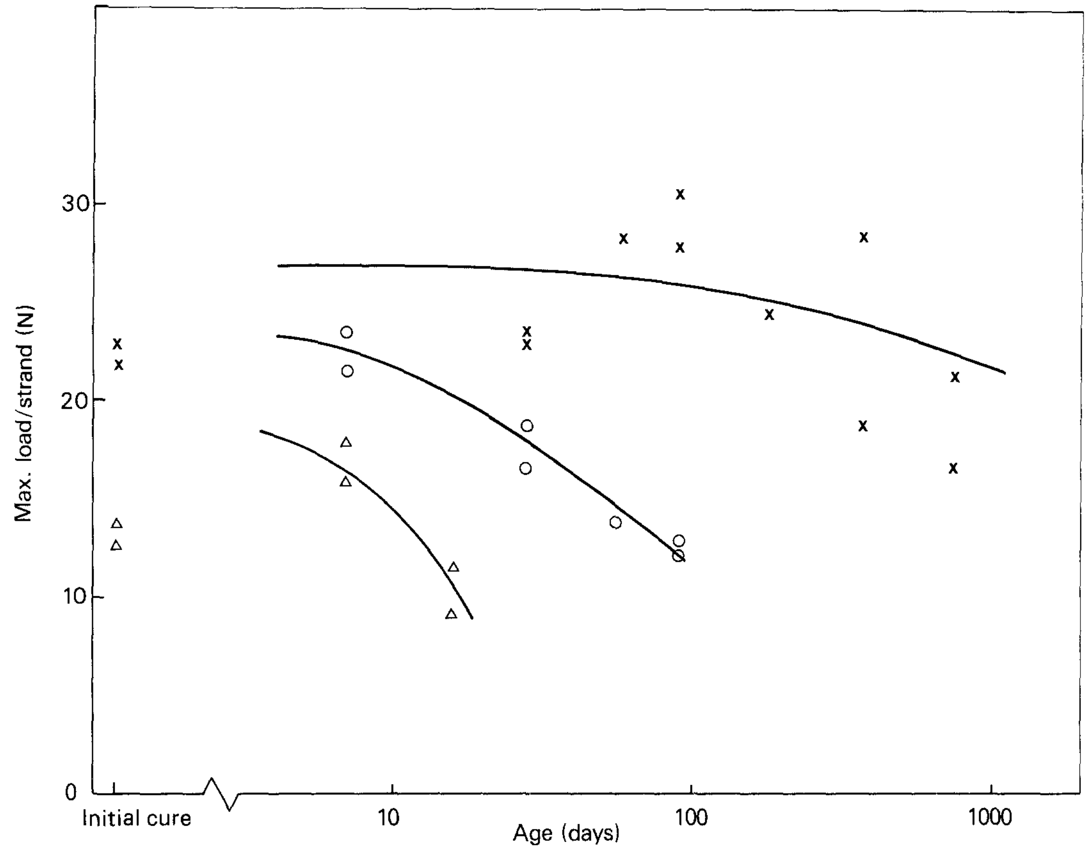

Figure 8 The maximum load achieved at long (~ 10 mm) embedment lengths, as a function of storage time and condition. ×, Cem-FIL 2 fibre, water 20° C; o, Cem-FIL 2 fibre, water 50° C; Δ, Cem-FIL fibre, water 50° C.

calculated from the maximum load, suggesting that the embedment length is shorter than that calculated from the crosshead travel (to zero load). This would be the expected behaviour of an assembly of fibre strands of equal $p\tau$ but unequal lengths; and the total crosshead travel would be a measure of the length of the longest fibre strand.

However, the average fibre length measured using a microscope was not consistently lower than that obtained above, and it seems probable that the shape

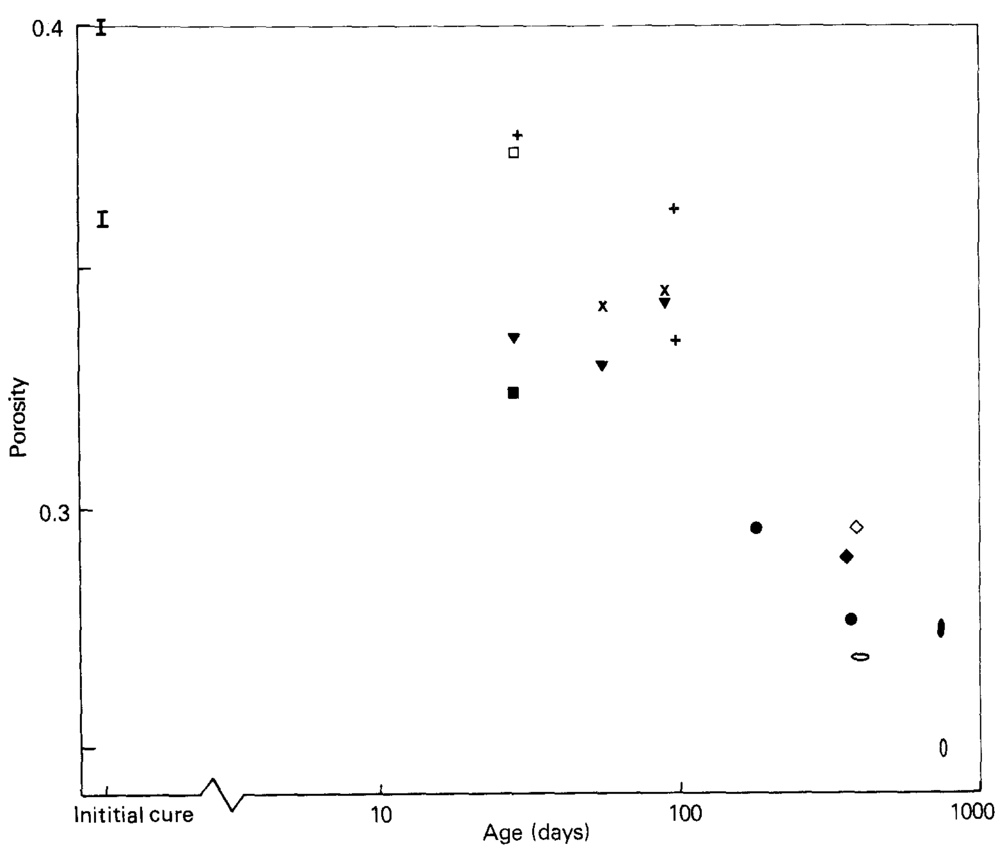

Figure 9 Variation of porosity with storage time at 20° C, Cem-FIL 2 fibre. × and hatched symbols refer to water storage; + and unhatched symbols refer to storage in air at 40% r.h. The different symbols refer to different batches.

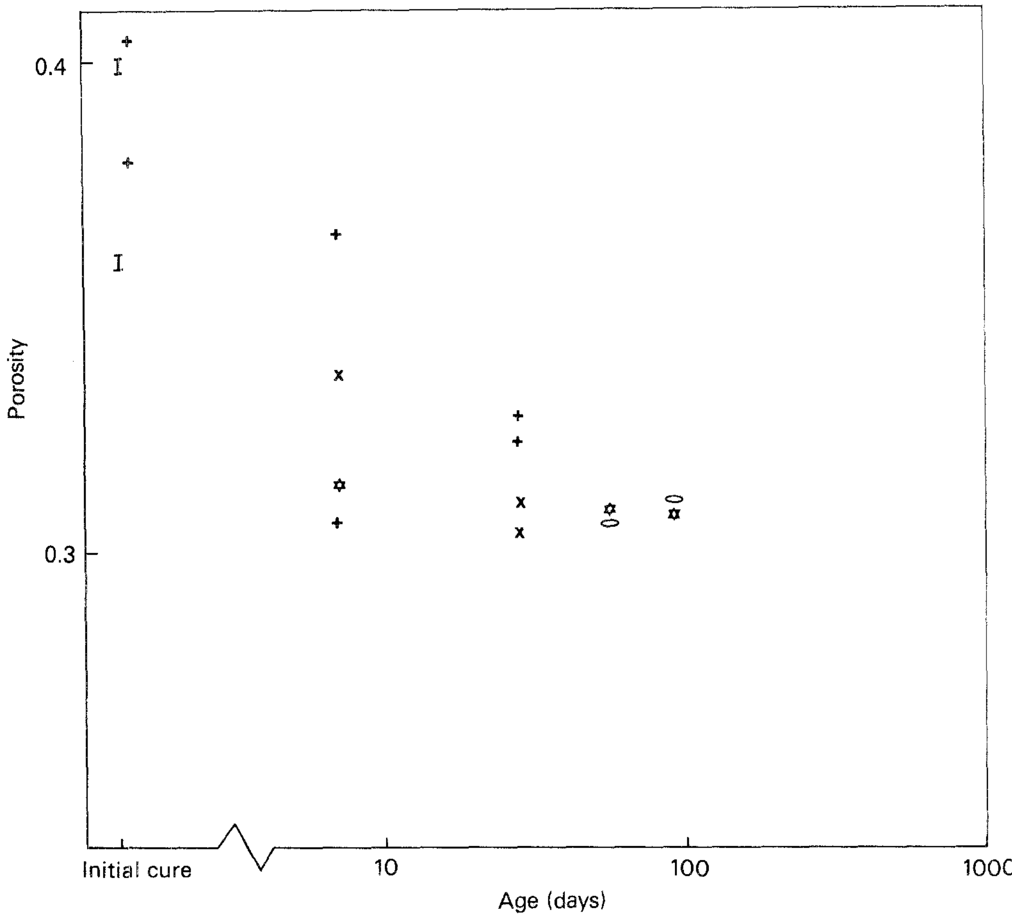

Figure 10 Variation of porosity with storage time in water at 50° C. +, Cem-FIL fibre; other symbols, Cem-FIL 2 fibre. The different symbols refer to different batches.

of the pull-out curve is determined by other factors, for example by matrix crumbling leading to (frictional) bond which decreases as the fibre strands are withdrawn.

The bond strength results present some problems of interpretation. Previous work [9] showed that, after 28 days, the strength of the glass fibre/Portland cement bond increased with time of storage in air (100% r.h.) and in water at 18° C. They used single uncoated rods, 1 mm diameter, of several different glasses including an alkali resistant glass. The bond strength (τ) values they obtained were high, and reached as much as $20\,\mathrm{N\,mm^{-2}}$. On the other hand, Oakley and Proctor [10] calculated bond strengths from crack spacings for grc sheets made by the spray-suction method and stored in air. The values they deduced were of the order of $1\,\mathrm{N\,mm^{-2}}$ or less, and decreased with time. The glass fibre was Cem-FIL alkali resistant glass in the form of multi-filament strands. Oakley and Proctor used a value of 2.8 mm for the perimeter of the strand and the $p\tau$ values deduced (and also one value obtained directly by pull-out) are approximately $3\,\mathrm{N\,mm^{-1}}$ and of the same magnitude as the values reported here.

The results for storage in water at 20° C show an increasing trend of $p\tau$ with time, and this accords with current understanding of the microstructural changes that take place at the fibre/matrix interface as the cement matrix hydrates [11]. The initial storage period leads to a rapid bond development (Fig. 8). In the early stages of storage the matrix is highly porous, but with time of storage in water the cement hydrates and the porosity decreases. In the immediate vicinity of the fibre strand the porosity is somewhat higher than in the bulk of the cement because the accumulation of water from the slurry around and within the fibre strand results in a higher water/cement ratio in this region. Initially then the contact area between fibre and matrix is small, and the strength of the matrix in the interfacial region is low, resulting in a low bond strength. As the cement hydrates, the contact area increases, the strength of the interfacial region increases and the bond strength increases. Additionally hydration products have been seen to penetrate into the strand in Cem-FIL fibre composites [12], further increasing the effective contact area.

Our results show that the porosity of the specimens decreased with time. This happened even for air-storage; but the Cem-FIL 2 fibre strand/cement bond strength did not change much. For 50° C water storage the porosity fell quickly to reach an apparently constant value presumably when the hydration was complete. However, after first increasing, the bond strength decreased and this is not consistent with the increased contact area that is expected as the porosity falls. Furthermore the values of $p\tau$ obtained at all times of storage at 50° C were below that at the end of the initial cure period. At the longer storage times it was more difficult to ensure that the fibre strands pulled out completely and fibre breakage leads to a reduced estimate of the true bond strength. At the shorter storage times, however, fibre breakage is less and the problem of explaining the lower bond strength remains. The results for Cem-FIL fibre strands on the other hand, showed an increase in bond strength with time of storage in water at 50° C although fibre breakage was more of a problem in this case.

The reason for the apparently lower bond strength

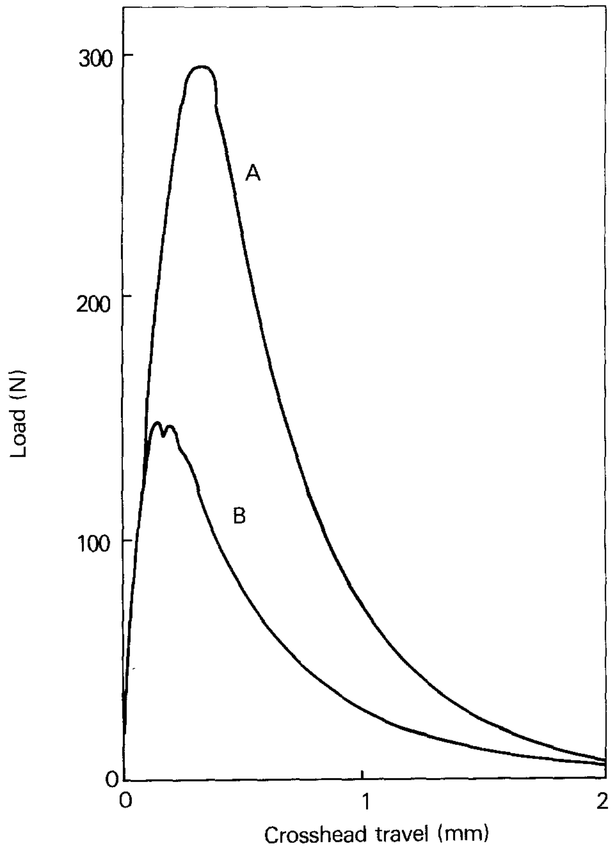

Figure 11 Load/displacement curves during pull-out. 3.5 mm embedment length. Curve A refers to 90 day water storage at 20° C; curve B refers to 90 days water storage at 50° C. Cem-FIL 2 fibre.

for Cem-FIL 2 fibre specimens stored in water at 50° C compared with those stored in water at 20° C must also be the subject of some speculation. It might at least in part be the result of an increasing number of partial failures of the fibre strands with increasing storage time at 50° C. The microstructure of the cement stored in water at 50° C might well be very different from that stored at 20° C and there might also be an effect of temperature on the contribution of the fibres to the shear properties of the interfacial region (if, for example, they are coated).

The lower strand/cement bond strength found for Cem-FIL fibre compared with Cem-FIL 2 fibre has also to be explained. Further information is needed too, on the microstructural changes that take place in Cem-FIL 2 fibre composites as they age, and whether for example, hydration products are deposited within the fibre strand and cement the filaments together to form a rod-like reinforcing element as happens with Cem-FIL fibre composites.

Evidence to date suggests that the fibre strand remains loose. If this is so the effect of bending stresses on the reinforcing element at composite failure will be reduced, and the composite strength might be calculated from the load/embedment length curve [13]. There is still some uncertainty about the value of the factor to be applied to allow for the orientation of the fibres, but relative predictions might be made.

Fibre pull-out is an important factor contributing to the impact strength of fibrous composites [14] and the pull-out curves are useful also in indicating differences. For example, Fig. 11 indicates the difference in energy required to withdraw fibres of length approx. 3.5 mm, from the matrix stored for 90 days in water at 20 and 50° C.

6. Conclusion

The multiple fibre pull-out test described is capable of yielding data on the strength of the bond between fibre strand and cement, and at sufficiently long embedment lengths, the strength of the fibre strands. In this latter regard it is analogous to the strand-in-cement test [2], but it has the potential added advantage that the effective strength of the short strand of the length used in the composite could be measured.

Further work is needed to see how well the composite strength and energy to break can be predicted using pull-out data.

The results described here show that the bond strength between Cem-FIL 2 fibre strands and cement increases with storage time, for water storage at 20° C Where there is a comparison, both the bond strength and the strand strength are higher for Cem-FIL 2 fibre than for Cem-FIL fibre.

Acknowledgement

The work described has been carried out as part of the research programme of the Building Research Establishment of the Department of the Environment and this paper is published by permission of the Director.

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Received 6 February and accepted 13 March 1985