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Bond between reinforcing steel fibres and magnesium phosphate/calcium aluminate binders

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Abstract

The work reported provides new information on the bond between two types of steel fibres and two different rapid strengthening matrices, magnesia phosphate and accelerated calcium aluminate. Two new methods have been developed in order to investigate:

- the tensile chemical (or adhesive) bond strength between steel fibres and a cement matrix;
- the durability of the steel fibre cement matrix bond when exposed to hostile environments.

The first is a test to determine the tensile force necessary to produce fibre matrix failure. The rationale for the test was to provide a means of assessing the contribution of the chemical bond to the strength of FRC.

The second is a single-fibre pull-out test suitably fabricated to identify the bond durability after exposure of the fibre to a hostile environment.

In all tests there was the requirement of very rapid preparation as early hardening of the cement-based matrices was accompanied by rapid setting.

Keywords: Rapid strengthening cements; Steel fibre/cement bond; Tensile chemical bond strength; Bond durability

1. Introduction

The tensile chemical bond strength between a steel fibre and a cement matrix resists displacement caused by forces acting perpendicular to the interface. It can be most easily evaluated by tests that apply a tensile stress perpendicular to a relatively large interface between bulk samples of materials representing the matrix and the fibre reinforcement, using tests similar to those related to industrial adhesives [1,2]. However, specimen fabrication requires extreme care to prevent damage during handling and to ensure correct alignment. In addition, internal stresses are introduced at the interface due to restrained shrinkage of the matrix that can cause premature debonding. Further, materials used as commercially available fibre reinforcements cannot always be obtained in a bulk form from fibre manufacturers.

As a result of these limitations, very few attempts have been made to investigate the tensile bond strength of steel fibres [3,4] and these provide a qualitative rather than quantitative assessment of the contribution of the tensile bond to the strength of FRC [5]. A significant portion of studies have concentrated on the behaviour of the fibre pull-out resistance to explain the contribution of the interfacial zone in FRC. Aligned, single-fibre pull-out tests have been developed and the present understanding of these tests is quite advanced. The modelling of pull-out behaviour however is based on assumptions made from the analysis of the pull-out curves where only interfacial (frictional) shear bond stresses are considered. Little is known of the chemical bond between fibres and cement matrices and none of the recent theoretical models of pull-out appear to have taken this function into consideration [6–16].

In the present work, a test has been developed to determine the average tensile fibre/matrix chemical bond strength. The test allows direct measurements of the force to separate a bed of continuous fibres from a bulk matrix. Further, the test has the potential to provide a means of comparing various fibre surface pretreatments [17,21] and assessing the influence of hostile environments on the tensile bond strength.

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Current laboratory durability methods make use of beam specimens usually tested in flexure which contain large numbers of fibres [18–20]. These test procedures also require voluminous environmental chambers and accompanying monitoring equipment and are time consuming.

In the present work, an alternative durability method has been developed. It consists of a two-stage single-fibre pull-out test where a fibre coated with a thin layer of the cement matrix is embedded into the matrix of a dogbone pull-out arrangement after exposure in appropriate media. The method has the following advantages compared to direct composite testing: the test specimen is easier to make, inexpensive (since only a fraction of fibres is required compared to the number of fibres used in current test procedures), able to rapidly predict percentage changes in fibre/cement matrix shear bond strength and requires environmental chambers of minimal sizes.

2. Experimental procedures

2.1. Materials

Two commercially available rapid hardening cement matrices were used: a magnesia phosphate-based cement [22], namely ASR-1 supplied by FEB International plc, and an accelerated calcium aluminate cement, namely Ultracrete RSC-1 supplied by Instarmac Repair UK. Two types of melt-overflow chromium, stainless steel alloy fibres were used, offering different resistance to pull-out, one of irregular sectioned, rough surfaced kidney-shaped, 25 mm long and variable maximum cross-section dimension around 0.4 mm, and one of irregular sectioned, less rough surfaced kidney shaped, 50 mm long and variable maximum cross-section dimension around 1 mm. The data reported are for fibre surfaces in the as-received condition.

2.2. Tensile tests

The overall test arrangement is illustrated in Fig. 1. It consists of two mild steel blocks with the fibre/matrix interface assembled in between. A layer of a strong, cold cure epoxy adhesive is applied to one block surface and the required number of fibres to fill the cross-sectional area are placed adjacent to each other on the adhesive layer to form a bed. After the adhesive has cured, the block is placed on an alignment plate with a 2 mm gap from the second block. The gap edges are sealed and the matrix is poured into position to bond the fibres to the other steel block.

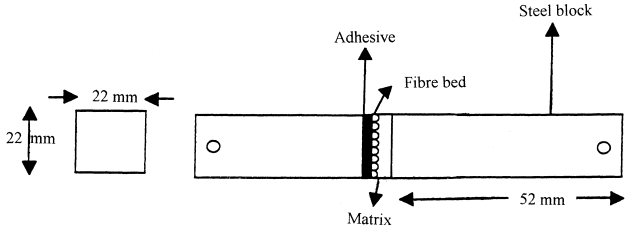

Fig. 1. Tensile debond test arrangement (not to scale).

The test specimen is loaded in tension via bars through holes in the blocks thus ensuring alignment perpendicular to the interface. Tests can be undertaken three hours after mixing. Comparison with bulk mild steel can also be made by bonding the two blocks directly with the matrix.

2.3. Durability tests

The overall test arrangement is illustrated in Fig. 2 and the method consists of the following steps:

1. A steel fibre is placed on a flat perpex plate with the curved surface facing upwards.
2. The steel fibre is coated with a thin layer (minimum dimension 0.5 mm, maximum dimension 2 mm) of the required cement material, Fig. 2(a).
3. The coated fibre is exposed to a curing environment and subsequent hostile environment (for example high temperatures, marine conditions, chemical solutions).

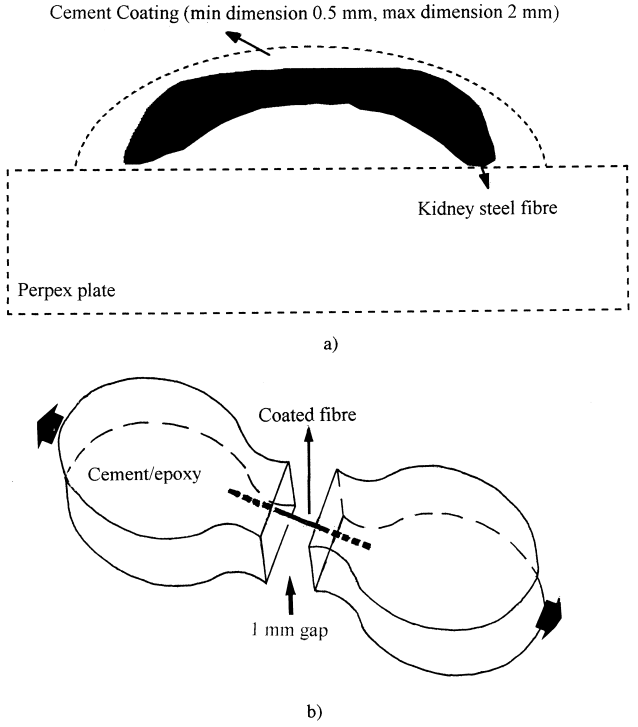

Fig. 2. Durability test arrangement: (a) coated fibre, and (b) dogbone pull-out configuration.

4. Prior to testing, the coated fibre is removed from the environment and dried if necessary using drying paper.
5. The coated fibre is embedded into a rapid strengthening cement or a rapid cold cure adhesive in a dogbone pull-out arrangement, Fig. 2(b) (the dogbone configuration was chosen because it eliminates the use of clamps).

Note that, this work involves the creation of two interfaces, one between the cement coating and the fibre and another which is related to the bond between the cement coating and the dogbone matrix (another cement or an adhesive). The bond of the latter interface is much stronger than that of the cement coating/fibre bond, as the results of the tests revealed: failure was by fibre pull-out from the coating, the coating/dogbone matrix interface was intact. Indeed, it is known that the bonding of old cement to new (or to a polymer) is much stronger than that of cement to metallic surfaces and for this reason, it is considered that, there was no effect of the coating/dogbone matrix bond to the pull-out phenomena reported in this work.

It is also necessary to mention that the interface coating/fibre resembles that in real composites, in fact it is the fibres near the surfaces of a real composite that are of most importance to the material's durability. The failure mode in a simple pull-out test does not closely resemble that in a real composite (due to the complicated forces which are involved in a real composite). However, the durability test method developed in this work provides a means of rapidly predicting the percentage changes in fibre pull-out strength (shear bond strength), that is the difference between the 3 h pull-out strength of coated fibres stored in air at laboratory conditions (controlled specimens) and those after exposure to hostile environments. It can therefore be argued that the percentage change in fibre pull-out strength will be analogous to that of the strength (in bending or tension) in a real composite, thus eliminating the need for calculations of actual composite strength values.

A number of steel fibres of kidney cross-section were first coated and kept in air in a temperature- and humidity-controlled environment for 2 h after mixing and then stored either in air or immersed in a 10% sodium sulphate solution. Prior to testing, the coated fibres were removed from the storage environment and embedded into the matrix of the dogbone arrangement, central and parallel to the mould surfaces so that the embedded coated fibre lengths in either parts of the dogbone material were 24 mm for the 50 mm long fibres and 12 mm for the 25 mm long fibres. Tests started 3 h after mixing. The durability tests were performed after exposure of the coated fibres at 3 h and at 13, 22, 29, 43 and 87 days.

All tests were undertaken using an Instron tensile-testing machine at a constant rate of crosshead movement of 0.5 mm/min recording force and extension continuously.

3. Results and discussion

3.1. Tensile tests

The chemical bond tests revealed the very poor bonding of the accelerated calcium aluminate (the tensile specimens fell apart immediately after the setting of the cement and no testing was possible) and therefore, results of the tensile chemical bond tests using the magnesia phosphate matrix were only considered and these are shown in Table 1. The chemical tensile bond strength was calculated as the nominal perpendicular stress on the curved surface of the fibre, by considering the average force to separate the bed of continuous fibres from the matrix (determined from the testing machine) and the cross-sectional area of the steel blocks ($22 \times 22\ \text{mm}^2$), and including both the tensile component and the shear component created at the interface by restrained shrinkage of the matrix [3]. The values of the ultimate force shown in Table 1 are the average of five specimens and the experimental scatter was very low since all specimens tested gave very similar results. Further, since the shape and size of the interface created closely resembles that in real composites, the results can be related to the force/displacement relationships for the subsequent development of the pull-out modelling process in more detail.

At this point it is worth mentioning that the purpose of this investigation was not to show how the tensile chemical bond strength, caused by forces acting perpendicular to the interface, can be related to the shear strength. This is rather the subject of analytical investigations. This work, provides with methods for the practical assessment of the chemical bond. However, in analytical terms, the chemical bond test results may be related to shear failure as follows: at the end of the "chemical debonding process", it can be argued that the tensile component may be considered to be zero in magnitude and that the shear component will approximately be equal to the maximum interface

Table 1
Typical tensile bond test results from the magnesia phosphate cement matrix

Material	Average ultimate force (N)	Tensile bond strength (N/mm^2)
Mild steel blocks	563	1.16
Kidney fibres	99	0.20

chemical bond stress, that is the value of 0.2 MPa from Table 1.

The results indicate that the tensile (chemical) bond at the interface between the fibres and the matrix is at least an order of magnitude less than the shear bond values reported in the literature [7,15]. The fibre/matrix tensile bond value was found to be only one-sixth of the bond strength between bulk mild steel and the matrix (this observation is used here to simply demonstrate the weak chemical bond at the fibre/matrix interface). Visual examination of the fractured surfaces revealed no matrix material on the fibre surfaces whereas in the case of the mild steel blocks there were patches of matrix adhering to the steel surface.

3.2. Durability tests

Typical pull-out test results are shown in Fig. 3 as plots of (frictional) pull-out force against displacement. The main feature is the change in the mode of fibre pull-out from the coatings from 3 h of exposure to 87 days:

3.2.1. Magnesia phosphate cement coating

(a) After maximum force is reached the fibre pulls out (or continues to pull-out) from the coating at decreasing force with an increasing amount in stick–slip behaviour after exposure at 3 h in air or in a 10% sodium sulphate solution media, Fig. 3(a), mode 1, The feature was similar for both types of kidney fibres used.

(b) At all other exposure conditions, the fibre pulls out at increasing force in stick–slip behaviour, after the bend-over point in Fig. 3, and (the fibre) breaks at maximum force, Fig. 3(b_1), mode 2. The feature was similar for both types of kidney fibres used.

3.2.2. Accelerated calcium aluminate cement coating

(c) Pull-out behaviour was similar to case (a), mode 1, for exposure at 3 h in air or in a 10% sodium sulphate

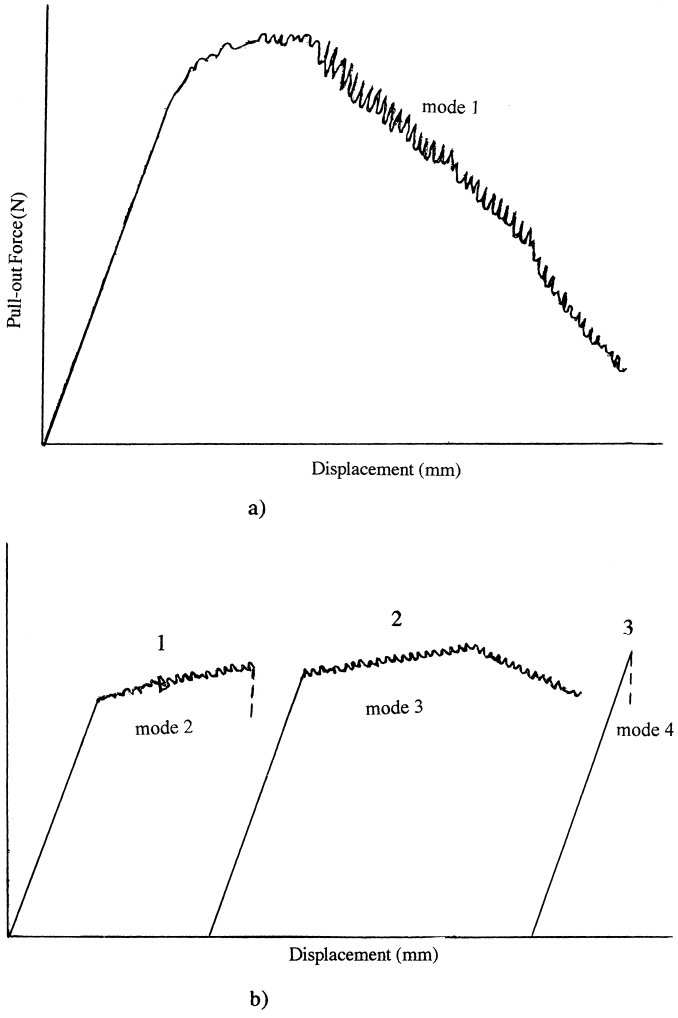

Fig. 3. Typical pull-out durability test results: (a) mode 1, and (b) modes 2, 3 and 4.

solution media but with steeper fall in the pull-out force. The feature was similar for both types of kidney fibres used.

(d) At all other exposure conditions different modes of pull-out were observed. For the 25 mm long fibres, a mixture of the following three cases:

(i) as in case (c), mode 1, but with less steeper fall in pull-out force, that is intermediate to cases (a) and (c);

(ii) as in (b), Fig. 3(b_1), mode 2;

(iii) fracture at maximum force, Fig. 3(b_3), mode 4.

For the 50 mm long fibres however, the pull-out mode was always as shown in Fig. 3(b_2), mode 3. No fibre fracture was observed compared to the 25 mm long fibres. This was attributed to the less rough surface.

Similar results in the change of fibre pull-out mode have recently been reported [18] for glass fibre-reinforced cement composites (GRC) where flexural specimens were stored in water at 60°C for up to 42 days to accelerate ageing. A specially designed SEM tensile/pull-out test arrangement together with suitably fabricated specimens was also used to demonstrate that composite failure mode changed from that of pull-out to fibre fracture, as in cases (b) and (d)(iii) above, through intermediate failures showing partial pull-out/fractures of the fibres, as in case (d) of the present work.

The data obtained in the present work clearly showed no reduction in shear bond strength with exposure time of the coated fibres. Visual examination revealed that no matrix material was retained on the fibre surfaces. Similar results have also been reported in the literature where melt extract stainless steel fibres were used to fabricate flexural specimens which were exposed to marine curing conditions for up to 1250 days [19]. For different samples at different conditions the force/deflection curves obtained were almost identical. Failure modes always occurred as a gradual pull-out of fibres at decreasing force, as in case (a) of the present work but without showing any stick-slip behaviour. Visual examination showed the absence of any corrosion in the fibres. A five-year study on the durability of FRC exposed in different environments (dry, humid and humid with cyclical wetting with a chloride solution) has recently been reported [20]. Stainless steel fibres were used to fabricate flexural specimens. Failure modes reported were similar to case (a) of the present study. The performances in terms of maximum flexural load after 5 yr were not impaired, even when influenced by highly aggressive exposure conditions.

Note that the work reported in the literature, is used here, only to demonstrate the validity and reproducibility of the durability test and not to compare between different types of fibres and cement matrices.

Overall, the data obtained from the present work were in good agreement with those reported in the literature. The amount of experimental results obtained (a large number of specimens was used) and the low levels of scatter (these are frequently high in adhesion science), typically within 15%, demonstrate the potential of the prototype tests to be used as standard industrial laboratory methods.

4. Conclusions

Two new prototype tests have been developed to provide information on the bond in steel fibre-reinforced cement composites. This paper presents the test set-up and test results.

The first test relates to the tensile chemical bond strength between steel fibres and a cement matrix in order to provide:

1. A method to evaluate the tensile fibre/matrix interfacial bond strength. The test allows direct measurements to be made of the tensile force necessary to separate a bed of continuous fibres from a cement matrix.
2. A method for comparing various fibre surface pretreatments [17,21].
3. A method for assessing the influence of hostile environments on the tensile bond strength.

The second test relates to the durability of the steel fibre/cement matrix bond when exposed to hostile environments in order to provide:

1. An alternative method to assess the durability of steel fibre-reinforced cement composites.
2. A cost-effective technique by considerably reducing the number of steel fibres and using environmental chambers of minimal sizes where the cost of accompanying monitoring equipment is also reduced.
3. An easier-to-fabricate test specimen.
4. A rapid prediction of percentage changes in fibre pull-out strength (shear bond strength).

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