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The Effect of Processing on the Bond and Interfaces in Steel Fiber Reinforced Cement Composites

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Abstract

The object of the present work was to study the influences that processing may have on the pull-out resistance of a steel fiber from a normal cementitious matrix, consisting of portland cement binder cast as paste or mortars with varying sand content. In order to determine the mechanisms by which the processing affects the pull-out resistance, the nature of the interfacial transition zone (ITZ) was evaluated simultaneously by microhardness testing and characterization of its microstructure by SEM. It was found that processing of the matrix and modifications in its composition by changing the sand content had a considerable influence on the steel fiber–matrix average bond, which was increased by a factor of two. The influences are due to changes in the interfacial microstructure and the content of sand. In pastes, the interfacial microstructure seems to be the dominant mechanism controlling bond, and processing which leads to densening results in higher average bond. The influences in mortars are more complex; average bond is less sensitive to processing and is more influenced by the sand content. Copyright © 1996 Published by Elsevier Science Limited.

INTRODUCTION

The bond across the interface in fiber reinforced cementitious composites is a significant parameter in controlling the overall performance of the composite.[1,2] It is usually determined by the testing of special pull-out configurations[3] in which load–displacement curves are obtained and are interpreted in terms of an average pull-out resistance (sometimes referred to as average bond strength) and adhesional and frictional bond strength. The modeling and testing of pull-out is quite advanced.[4] In view of the significance of bond, attempts have been made to improve it, either by modification of the fiber shape and surface or by matrix modification.[3] The latter means might be quite useful, and increases in bond by a factor of two have been reported.[5–7] Most of these improvements were achieved by modification of the matrix composition with special additives such as silica fume and polymers. It was suggested that the effect of such treatments is due not only to modifications of the interface itself, but to changes of the whole area of the interfacial transition zone (ITZ). This zone, which forms in most portland cement composites, extends from the interface into the matrix to a distance of up to $\approx 50\ \mu m$, is characterized by a more porous and heterogeneous microstructure than the bulk,[8] and is usually perceived as the weak link in the fiber–matrix interaction.[9]

The formation of the ITZ is associated with inefficient packing of the cement grains in the fresh mix at the inclusion surface, due to the 'wall effect' and local bleeding.[8,9] Much attention has been given to elimination of these influences by modification of the binder composition. However, the ITZ might also be affected by the method of processing of the composite in the fresh state, in which the shearing stresses applied during mixing can change the bleeding tendencies and modify the packing of particles near the surface of the inclusion, whether fiber or aggregate. The issue of the influences of processing on bond has not received adequate attention. It can be important for optimizing the bond for a given composition, and it might also have an influence on the pull-out test results themselves. Such tests are done with special specimens whose preparation involves quite different procedures than those applied in the production of the composite itself. Resolving the influence of processing on the pull-out resistance is essential in the evaluation of the significance of the test results and their applicability to the composite.

The object of the present work was to study the influences that processing may have on the pull-out resistance of a steel fiber from a normal cementitious matrix, consisting of portland cement binder cast as paste or mortars with varying sand contents. To determine the mechanisms by which the processing affects the pull-out resistance, the nature of the ITZ was evaluated simultaneously by microhardness testing and characterization of its microstructure by SEM.

EXPERIMENTAL

In the present study the processing was obtained by rotation of the fresh matrix against a stationary steel fiber using the test set-up shown schematically in Fig. 1. First, the matrix was prepared by mixing the solid ingredients (cement and sand) with water, and casting and compacting the specimen in the mold. The mold was sealed on both sides, placed in the processing apparatus and rotated at a speed of 50 revolutions per min for periods of up to 40 min. The specimen was demolded after 24 h and placed in 20°C water for curing. At the age of 14 days it was tested for pull-out and was then prepared for SEM observation and microhardness evaluation.

The specimen consisted of a matrix with a square cross-section of 15 mm and a length of 30 mm, with the fiber placed along its centroidal axis. At the middle of the mold, perpendicular to the fiber, a Teflon separator

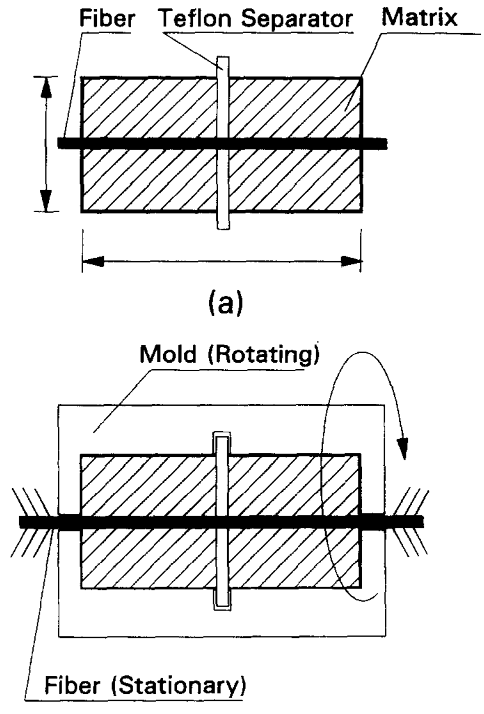


Fig. 1. Schematic description of the pull-out specimen (a) and the processing involved in its production (b).

was placed, so that after demolding, the specimen consisted of two 15 mm long blocks, bridged by the fiber. Pull-out tests were carried out in the rig shown schematically in Fig. 2 at a rate of 0.024 mm/s. Load vs load displacement curves were recorded.

For the microhardness testing the specimen was cut with a diamond saw on a plane perpendicular to the surface, and was then polished using the following procedure: 5 min on #600 paper and 10 min on #1200 paper. The microhardness test was carried with a Leitz mini-load apparatus using a load of 0.05 N (5 gmf). Microhardness values were determined as a function of the distance from the fiber interface. Considerations regarding the microhardness

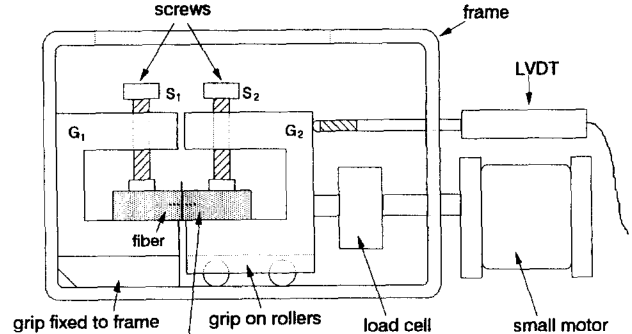


Fig. 2. Schematic description of the pull-out test.

testing of cementitious materials and the interpretation of results are provided elsewhere.[10] Microhardness profiles were the average of tests carried out on all four sides of the fiber: below, above and at the two sides. In specimens where bleeding cavities underneath the fiber were seen (i.e. specimens which were not subjected to rotational processing) the microhardness values were the average of determinations on three sides only: left and right at mid-height, and above the fiber.

The microstructure was determined by back-scattered electron (BSE) imaging of impregnated and polished specimens, using a Hitachi S-2300 SEM. The specimens were vacuum impregnated with ultra low viscosity epoxy.

The specimens were all made with a 0.40 w/c matrix with varying sand content: no sand (paste), and mortars with cement:sand ratios of 0.5 and 1. The portland cement was CSA type 10 (equivalent to ASTM type I).

RESULTS

The average pull-out stress values were determined by calculating the maximum load and dividing it by the embedded surface area of the fiber. This value will be referred to as average bond strength. The term processing time will be used to describe the duration of rotation of the fresh mix in the apparatus shown in Fig. 1.

Average bond strength

The effects of rotation and composition of the matrix on the average interfacial bond strength are presented in Table 1. It can be seen that the trends for the paste and the mortars are different. In the paste, bond strength increases monotonically with an increase in the processing time, while in the mortars bond is essentially the same for the unprocessed and 20 min processed systems. There is also a tendency for an increase in the bond values with an increase in the sand content of the mix (Fig. 3).

Microhardness

The effects of rotation and mix composition of the microhardness profiles are presented in Figs 4–6. As in the case of bond, there is a difference between the paste and the mortars. In the former, an increase in processing duration is accompanied by an increase in microhardness values in the ITZ, and by an increasingly ascending shape of the curve as the actual interface is approached. These characteristics do not occur in the mortars: the processing time hardly affects the curve and the tendency for an ascending curve towards the actual interface is very mild (Fig. 6) or even absent (Fig. 5). It is interesting to note that in the bulk paste (i.e. $\approx 100\ \mu m$ away from the interface) the microhardness values in each system are essentially constant, with the microhardness values being higher in the paste. The addition of sand (1:0.5 cement:sand ratio) results in considerable reduction in the microhardness values in the bulk (compare Figs 4 and 5), but a further addition of sand (1:1 cement:sand ratio) led to an increase in the microhardness values in the bulk (compare Figs 5 and 6), to a level which is only slightly lower than that in the paste (compare Figs 6 and 4).

Microstructure

Typical micrographs at low magnification are shown in Figs 7 and 8 for the paste and the mortar of 1:0.5 cement:sand ratio. It can be observed that for both unprocessed paste and unprocessed mortar the microstructure around the fiber is highly non-uniform, with large bleeding voids underneath the fiber. The specimens after 20 min of processing did not exhibit such bleeding. In the mortar specimens (Fig. 8(b)) it can be seen that the rotation also

Table 1. Effect of processing on the average bond strength in paste and mortars

Processing time (min)	*Average bond strength (MPa)*		
	Paste	*Mortar with cement:sand ratio of: 1:0.5*	*1:1*
0	0.69	1.22	1.58
20	0.98	1.34	1.55
40	1.31	—	—

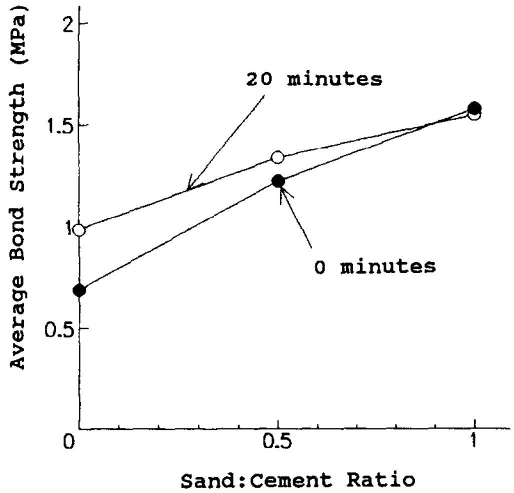


Fig. 3. Effect of the sand content in the mix on the average bond strength.

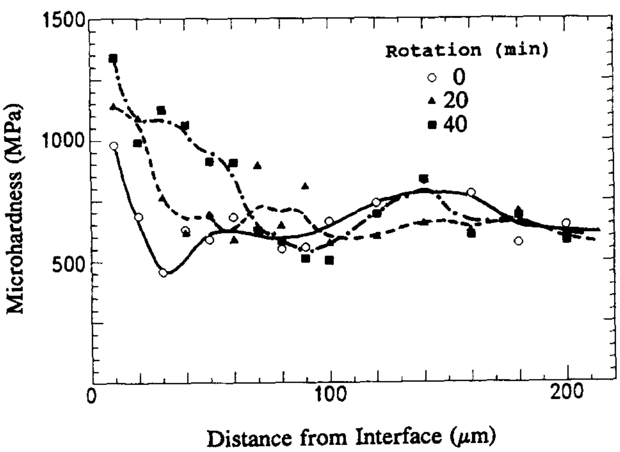


Fig. 4. Microhardness profiles in the ITZ in pastes subjected to different processing times.

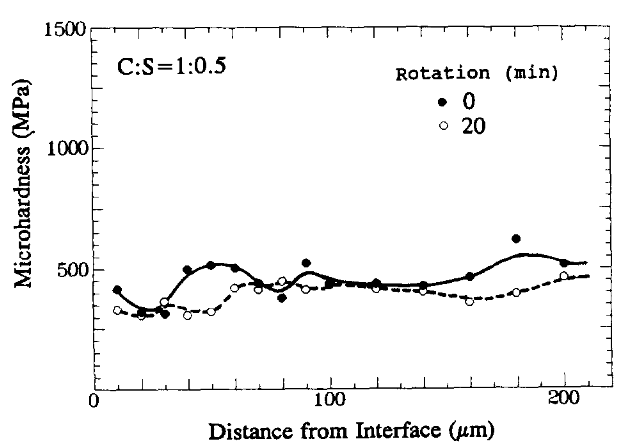


Fig. 5. Microhardness profiles in the ITZ in 1:0.5 cement:sand ratio mortars subjected to different processing times.

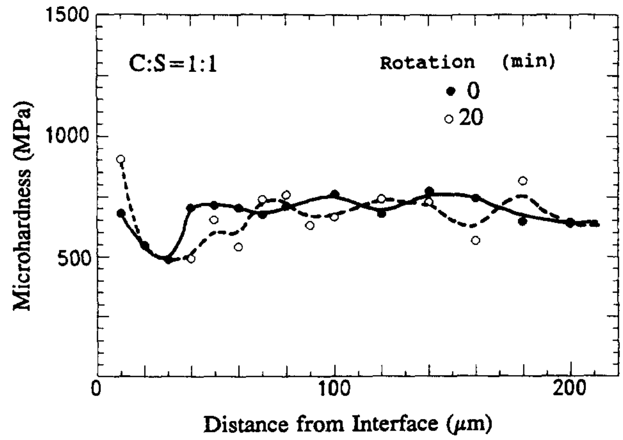


Fig. 6. Microhardness profiles in the ITZ in 1:1 cement:sand ratio mortars subjected to different processing times.

resulted in a more uniform dispersion of the sand particles in the vicinity of the fiber, whereas in the specimen that was not processed there was a wide rim around the fiber where there were fewer sand particles. In cases of pro-

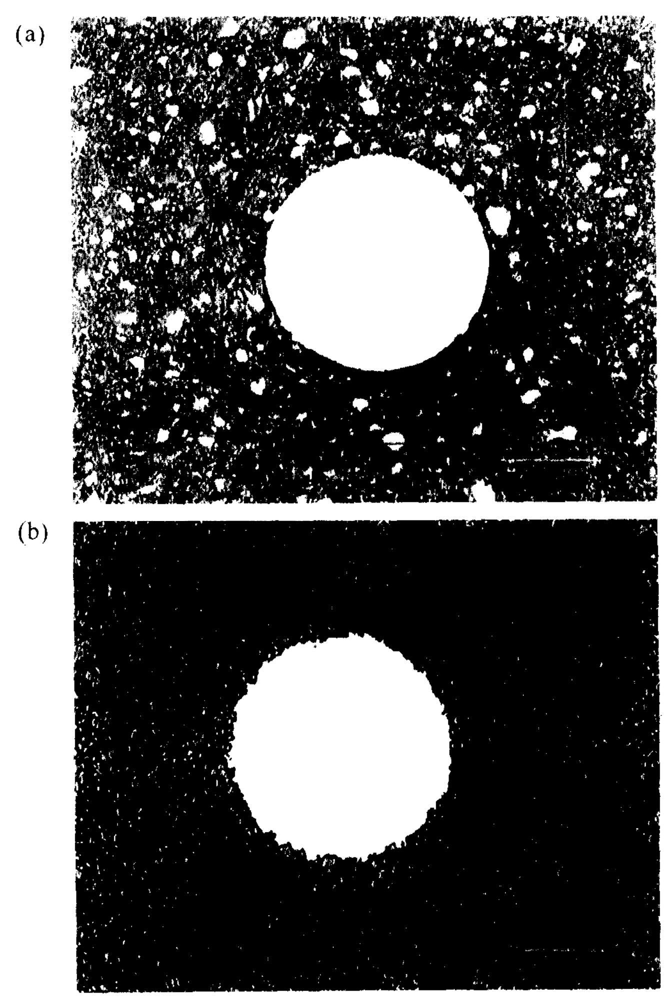


Fig. 7. BSE images of steel fiber–cement paste specimens (a) unprocessed, (b) 20 min processing.

cessed and unprocessed mortar specimens there was evidence that some of the sand grains formed direct contact with the fiber (e.g. the sand particle to the left of the fiber in Fig. 8(a)).

To better quantify the microstructure, back-scattered images were analyzed to resolve the porosity and its distribution. The pores considered by this technique are the larger ones, approximately 0.5 μm and above. Typical images of the pores are shown in Figs 9–12 for the paste and 1:0.5 cement:sand mortars with and without processing. These images can best be described by subdividing the paste volume surrounding the fiber into three zones: a rim of 1–20 μm, in which large pores and sometimes continuous porosity are observed, then a transition zone characterized by dispersed pores with porosity greater than that of the bulk, and the bulk itself. In the case of the unprocessed paste (Fig. 9(a)) it can be seen that the rim underneath the fiber is relatively wide and continuous, which is clearly the effect of bleeding. It becomes narrower and the pores are discontinuous at the sides and upper part of the fiber. The transition zone beyond this rim seems to have a larger porosity than in the bulk. Processing for 20 min resulted in elimination of this rim, above and below the fiber (Fig. 10). It also led to a considerable reduction in the porosity in the transition zone, although such a transition zone with porosity higher than the bulk can still be observed.

(a)
(b)

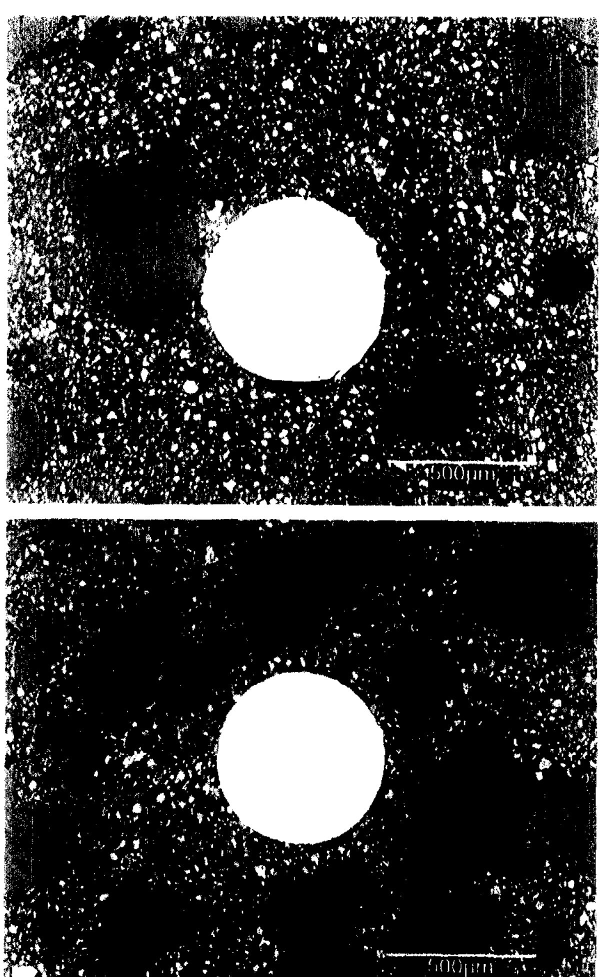


Fig. 8. BSE images of steel fiber–mortar (1:0.5 cement:sand ratio) specimens (a) unprocessed, (b) 20 min processing.

In the case of mortars, the porosity distribution in the unprocessed specimen seems to be quite similar to its unprocessed paste counterpart, i.e. a continuous porous rim underneath the fiber which becomes smaller and discontinuous above it (Fig. 11). A different trend is observed for the mortar processed for 20 min (Fig. 12). Its interfacial porosity distribution is more uniform, but a rim containing pores adjacent to the interface is present, and although not as porous as in the unprocessed specimen, it was not eliminated as in the processed paste. It is interesting to note that in the processed mortar (Fig. 12) the transition zone is often confined between the fiber and sand grains, and on both sides it seems to be bordered by a porous rim, one on the fiber side and the other on the sand grain side.

DISCUSSION

Paste

The results for the paste seem to be consistent. Increasing the processing time results in a denser microstructure and elimination of the porous rim, which can account for the enhanced mechanical performance: higher microhardness values in the ITZ (Fig. 4) and higher bond strength values (Table 1). Attention should be drawn to the microhardness profile in the ITZ in the processed specimens: although the porosity in the ITZ seems to be higher than in the bulk (Fig. 8) the microhardness profile in the ITZ ascends towards the actual interface, without any trough such as the one occurring in the unprocessed paste. This was highlighted in a previous publication.[10] It was suggested that such a trend is the result of the presence of a rigid, well bonded inclusion, which restrains the stress field developed under the indenter, even if it is somewhat distant from the inclusion, leading to an indentation which is smaller than would be expected from the nature of the paste only. In the present results it can be seen that a continuous increase in microhardness towards the inclusion, without any trough, occurred in the processed paste in which the high porosity interfacial rim was eliminated. This apparently ensured adequate bond across the interface, which is essential if the rigid inclusion is to have any influence on the indenter stress field.

When considering the pore images it is of interest to address the nature of the porous rim and its location (below, above and to the sides of the fiber). In the unprocessed specimen, the large rim below is largely due to bleeding. Above the fiber, bleeding is less likely to occur, and the presence of a discontinuous porosity rim might be due to the 'wall effect' which prevents the cement grains from packing efficiently at the fiber surface. The elimination of this rim with processing may be due to the shear applied to the cement grains in the fresh mix, forcing them to pack efficiently in the vicinity of the surface.

Mortars

The results for the mortars seem not to have this consistency: an increase in processing time did not result in enhanced fiber–matrix bond or higher microhardness profiles, although it eliminated the formation of a large porosity rim underneath the fibers. Also, the trends in the microstructural characteristics as determined from the porosity images are somewhat complex. Processing resulted in elimination of the bleeding cavity rim underneath the fiber, but a rim of pores (though discontinuous and not as porous) could still be observed above and at the

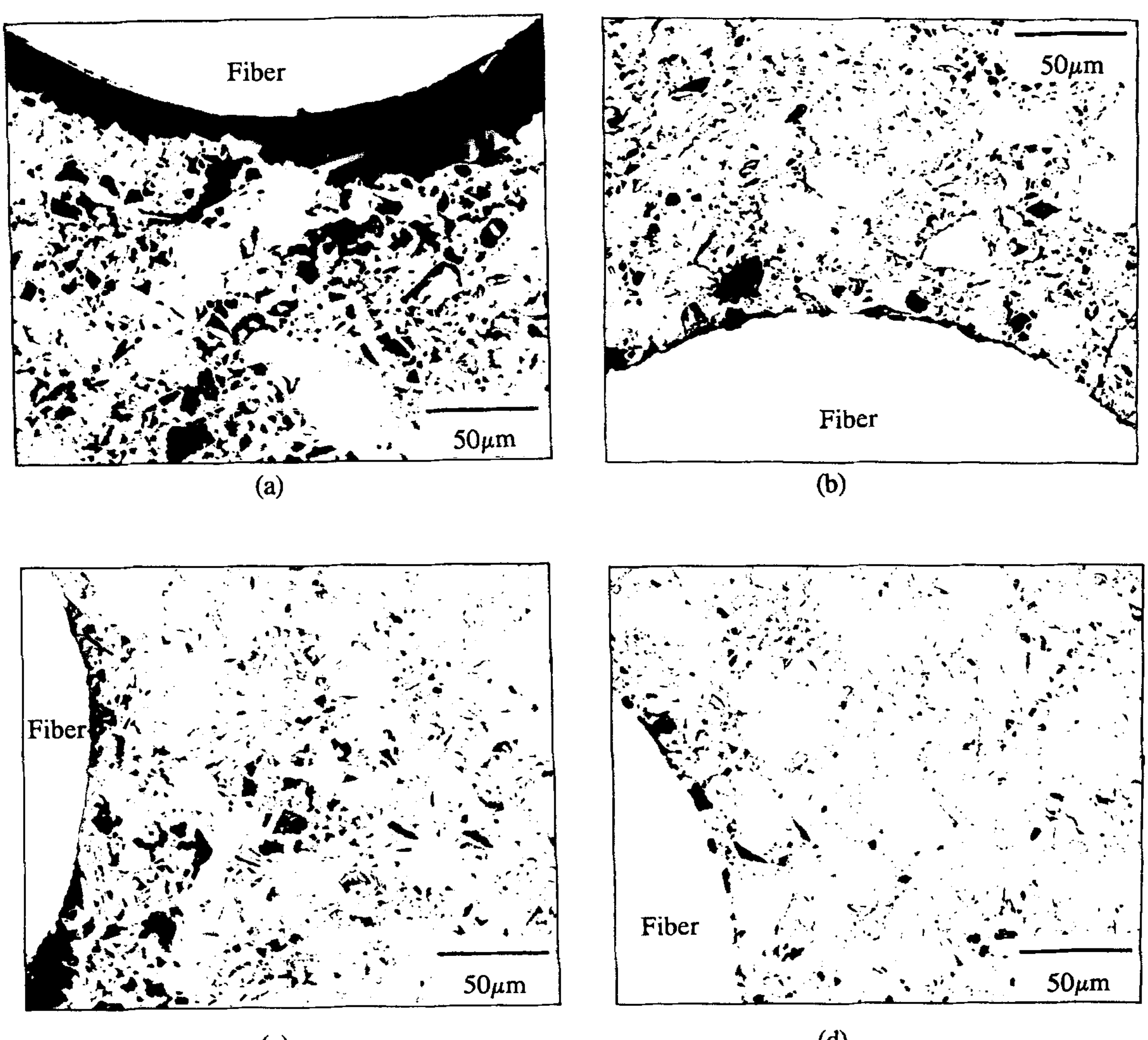
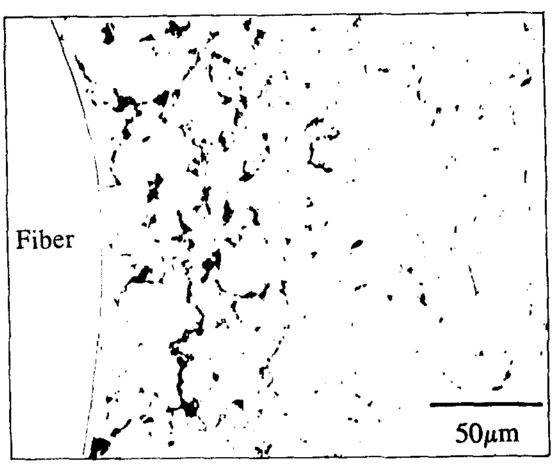
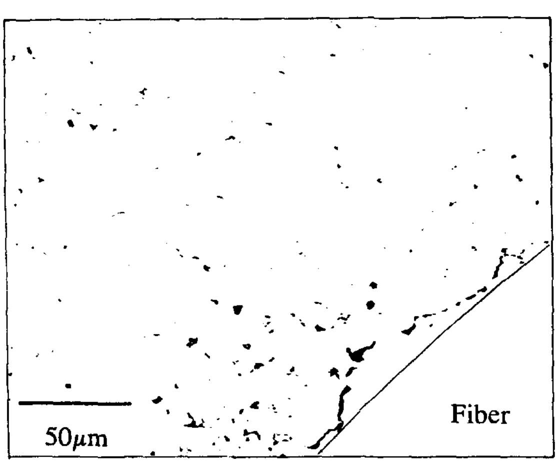


Fig. 9. Porosity images of fiber–cement paste specimens, unprocessed: (a) below the fiber, (b) above the fiber, (c) right side and (d) left side.

sides of the fibers, backed by a somewhat porous transition zone. These characteristics are similar to those observed around the unprocessed mortar specimen. This may account for the similarity in bond strength and microhardness profiles in the unprocessed and processed mortars though it should be kept in mind that in the unprocessed specimens microhardness was determined only at the sides and above the fiber, where there was no large bleeding cavity. This is different than in the paste specimens where processing resulted in general densification, which might be explained by the observation that in the mortars, superimposed on the effect processing is expected to have on the paste matrix microstructure, is the rearrangement of the sand grains, which approach the fiber during processing (compare Figs 8(a) and 8(b)). It is possible that due to this rearrangement of the sand grains, a confined area of $\approx 100\ \mu m$ is formed between the fiber surface and the neighboring grains, which is within the zone where high porosity ITZ is formed. This influence may counteract the densening effect that would be expected due to longer processing. Diamond *et al.*[11] suggested that in concrete systems most of the paste is confined in narrow spaces between the aggregates, and the microstructure of most of the paste in concrete is thus expected to be the same as that characteristic of the ITZ.



(a)



(b)

Fig. 10. Porosity images of fiber–cement paste specimens, 20 min processing.

The presence of rims of porosity even in the processed mix may imply poor bond at the actual interface, which limits the influence of the inclusion on the indenter, and therefore the ascending microhardness profile towards the fiber surface observed in the pastes (Fig. 4) was not seen here to the same extent (Figs 5 and 6).

The discussion above can explain the observations that for the same mortar composition the processing has no effect on the fiber–matrix average bond strength. As explained above this is the result of limitations in the mortar to improve their microstructure during processing. Yet, when comparing the different compositions it is seen that an increase in sand content results in improved average bond strength (Fig. 3). A possible explanation for this trend is that this pull-out resistance reflects a different mechanism, which is influenced by fiber–sand interlocking, as seen, for example, in Fig. 11. The likelihood for such interlocking to occur is greater when the sand content is higher. Thus, the overall pull-out resistance should be evaluated in terms of the influences of the microstructure of the hydrated material, which is dominant in paste, and sand–fiber interlocking which becomes of greater significance in mortars. Processing of mortars has a complex influence on both characteristics; on the one hand it leads to greater proximity of sand particles to the fiber surface, but on the other hand the paste microstructure is less favorable as more of it is in the region of the ITZ.

CONCLUSIONS

Processing of the cementitious matrix and modifications in its composition by changing the sand content in mortars can have a considerable influence on the steel fiber–matrix average bond, which can be increased by a factor of two. The influences are due to changes in the interfacial microstructure and the content of sand. In pastes, the interfacial microstructure seems to be the dominant mechanism controlling bond, and processing which leads to densening results in higher average bond. The influences in mortars are more complex; average bond is less sensitive to processing and is more influenced by the sand content.

These conclusions are of significance for both developing methods of improving bond, and for evaluating the significance of pull-out tests. In the latter case, the method by which the composite for testing is prepared (i.e. processed) may have a significant influence on the pull-out resistance, in particular when a paste matrix is involved. When considering methods to improve pull-out resistance by matrix modification, the parameters to be considered are not just w/c ratio; sand content and processing may exert considerable influences.

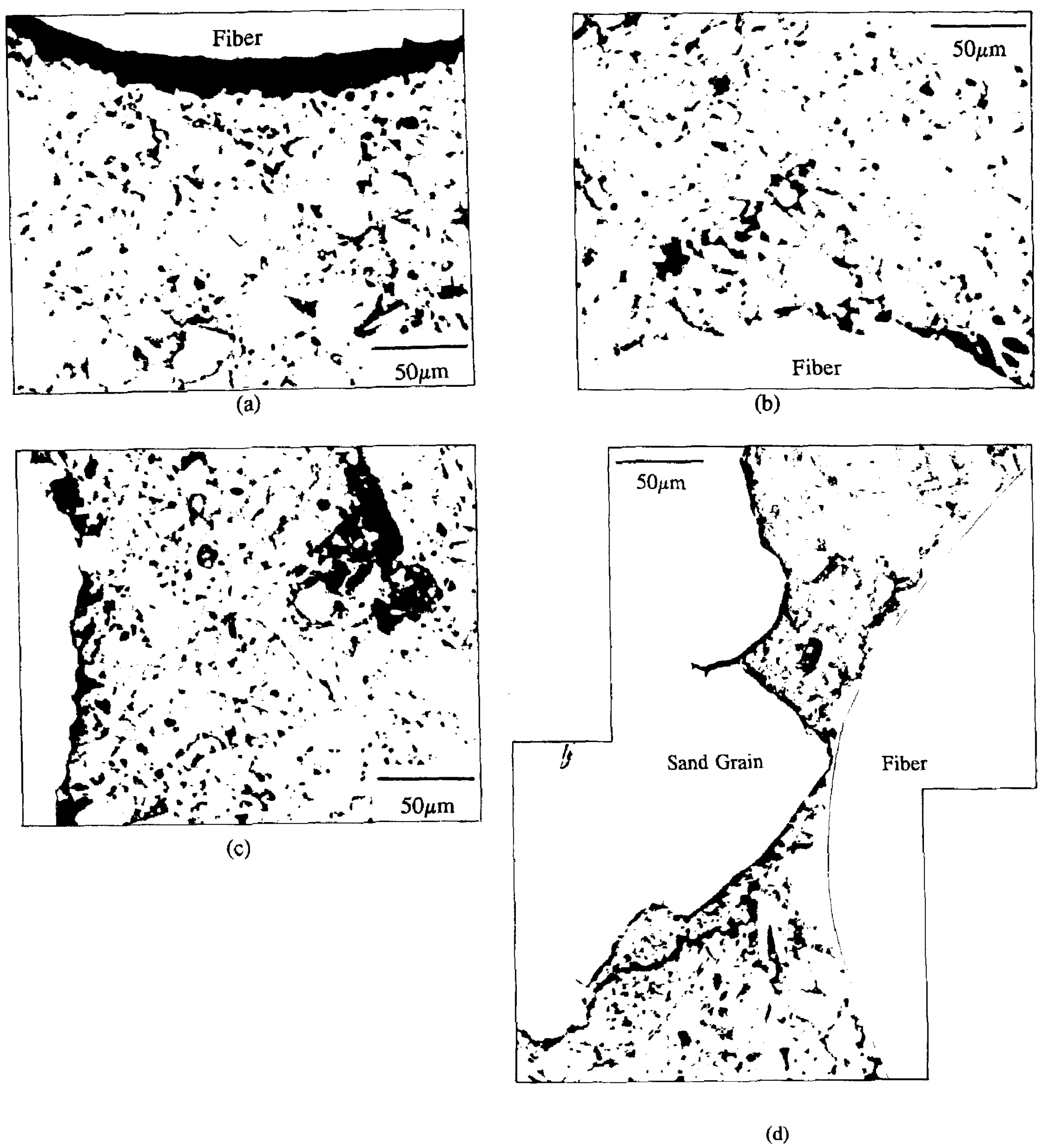


Fig. 11. Porosity images of fiber–mortar (1:0.5 cement:sand ratio), unprocessed: (a) below the fiber, (b) above the fiber, (c) right side and (d) left side at fiber–sand grain intersection.

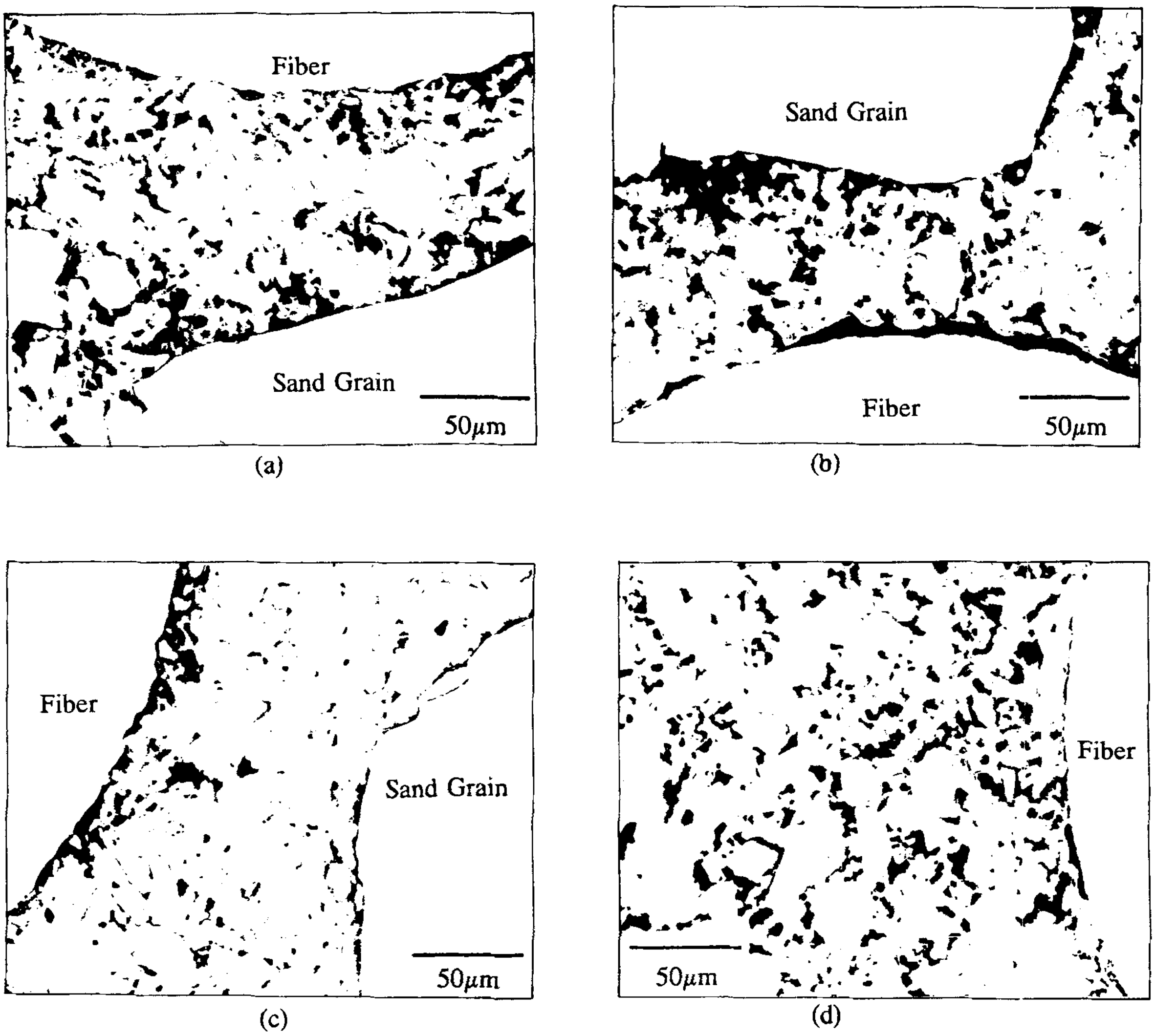


Fig. 12. Porosity images of fiber–mortar (1:0.5 cement:sand ratio), processed: (a) below the fiber, (b) above the fiber, (c) right side and (d) left side.

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