Chapter 2

Structure of fibre reinforced cementitious materials

The properties of fibre reinforced cementitious materials are dependent on the structure of the composite. Therefore, in order to analyse these composites, and to predict their performance in various loading conditions, their internal structure must be characterized. The three components that must be considered are:

1 The structure of the bulk cementitious matrix.
2 The shape and distribution of the fibres.
3 The structure of the fibre–matrix interface.

2.1 Matrix

The bulk cementitious matrix is not significantly different from that in other cementitious materials, and it can be divided into two types depending on the particulate filler (aggregate) which it contains: paste/mortar (cement/sand–water mix) and concrete (cement–sand–coarse aggregate–water mix) [1–3].

Fibre reinforced cement pastes or mortars are usually applied in thin sheet components, such as cellulose and glass fibre reinforced cements, which are used mainly for cladding. In these applications the fibres act as the primary reinforcement and their content is usually in the range of 5–15% by volume. Special production methods need to be applied for the manufacturing of such composites.

In fibre reinforced concretes, the fibre volume is much lower (<2% by volume) and the fibres act as secondary reinforcement, mainly for the purpose of crack control. The production of such reinforced concretes is carried out by conventional means. Higher contents of fibres can be incorporated by relatively simple mixing technologies, but using advanced matrix formulations which are based on sophisticated control of the rheology and microstructure of the mix. Such formulations combine dispersants and fillers (e.g. DSP, RPC and DUCTAL® [4–6]). The dense microstructure in these composites, as well as their improved rheology can enable the incorporation and uniform dispersion of 2–6% by volume of short fibres, which can provide effective reinforcement.

2.2 Fibres

A wide range of fibres of different mechanical, physical and chemical properties have been considered and used for reinforcement of cementitious matrices, as outlined in Chapter 1. The fibre-reinforcing array can assume various geometries and in characterizing its nature two levels of geometrical description must be considered: (i) the shapes of the individual fibres and (ii) their dispersion in the cementitious matrices (Figure 2.1) [7].

The individual fibres may be subdivided into two groups: discrete monofilaments separated one from the other (e.g. steel – Figure 2.2) and fibre assemblies, usually made up of bundles of filaments, each with a diameter of 10μm or less. The bundled structure is typical of many of the man-made fibres, whether inorganic (e.g. glass – Figure 2.3(a) and (b)) [8] or organic (e.g. carbon, kevlar), and it also shows up in some natural fibres (e.g. asbestos). The bundled fibres frequently maintain their bundled nature in the composite itself (Figure 2.3(c)), and do not disperse into the individual filaments. The monofilament fibres which are used for cement reinforcement rarely assume the ideal cylindrical shape, but are deformed into various configurations (Figure 2.2), to improve the fibre–matrix interaction

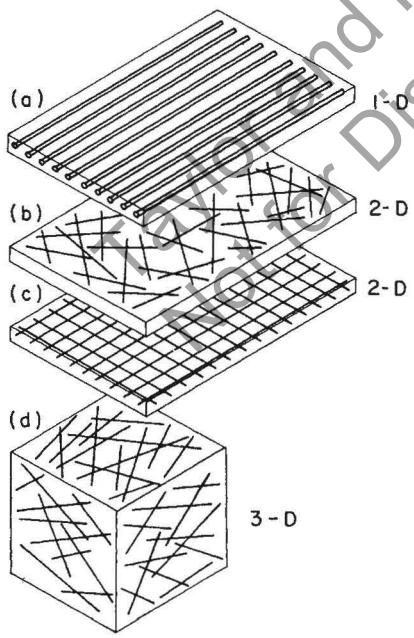


Figure 2.1 Classification of fibre arrangements in one, two and three dimensions and as continuous (a,c) or discrete, short fibres (b,d) (after Allen [7]). (a) 1D arrangement; (b,c) 2D arrangement; (d) 3D arrangement.

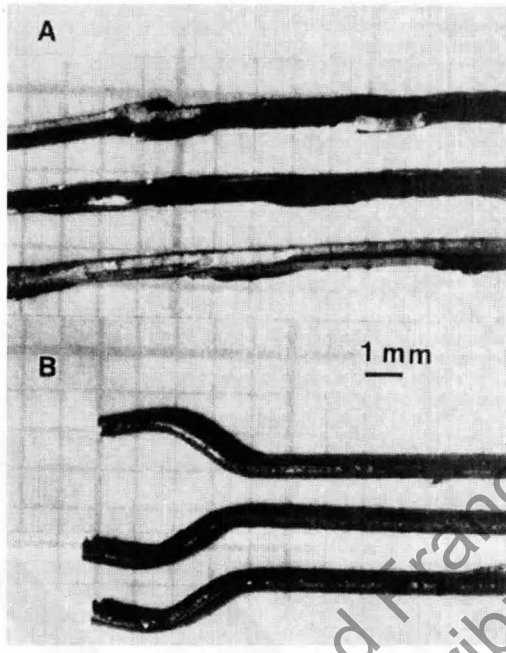


Figure 2.2 Various shapes of steel fibres (a) deformed; (b) hooked.

through mechanical anchoring. A range of complex geometries, ranging from twisted polygonal cross sections to ring type fibres have been evaluated, to provide effective anchoring, while maintaining adequate workability (e.g. [9–11]).

There are two distinctly different types of fibre-reinforcing arrays: (i) *continuous reinforcement* in the form of long fibres which are incorporated in the matrix by techniques such as filament winding or by the lay-up of layers of fibre mats; and (ii) *discrete short fibres*, usually less than 50 mm long, which are incorporated in the matrix by methods such as spraying and mixing. The reinforcing array can be further classified according to the dispersion of the fibres in the matrix, as 1D, 2D or 3D (Figure 2.1).

In the continuous form, the fibres can be aligned in a preferred orientation, which is controlled by the production process (orientation of winding, or lay-up direction of the mat) and the structure of the mat. This type of fibre reinforcement bears some resemblance to ferrocement applications; it is less common in FRC composites which are usually reinforced by discrete, short fibres, but has recently been the focus of intense development efforts (see Chapter 13 for details). In the case of dispersed fibres the dispersion in the matrix is more uniform, and the short fibres tend to assume a more random orientation. However, even in these systems the fibre distribution is rarely completely uniform, and their orientation is not

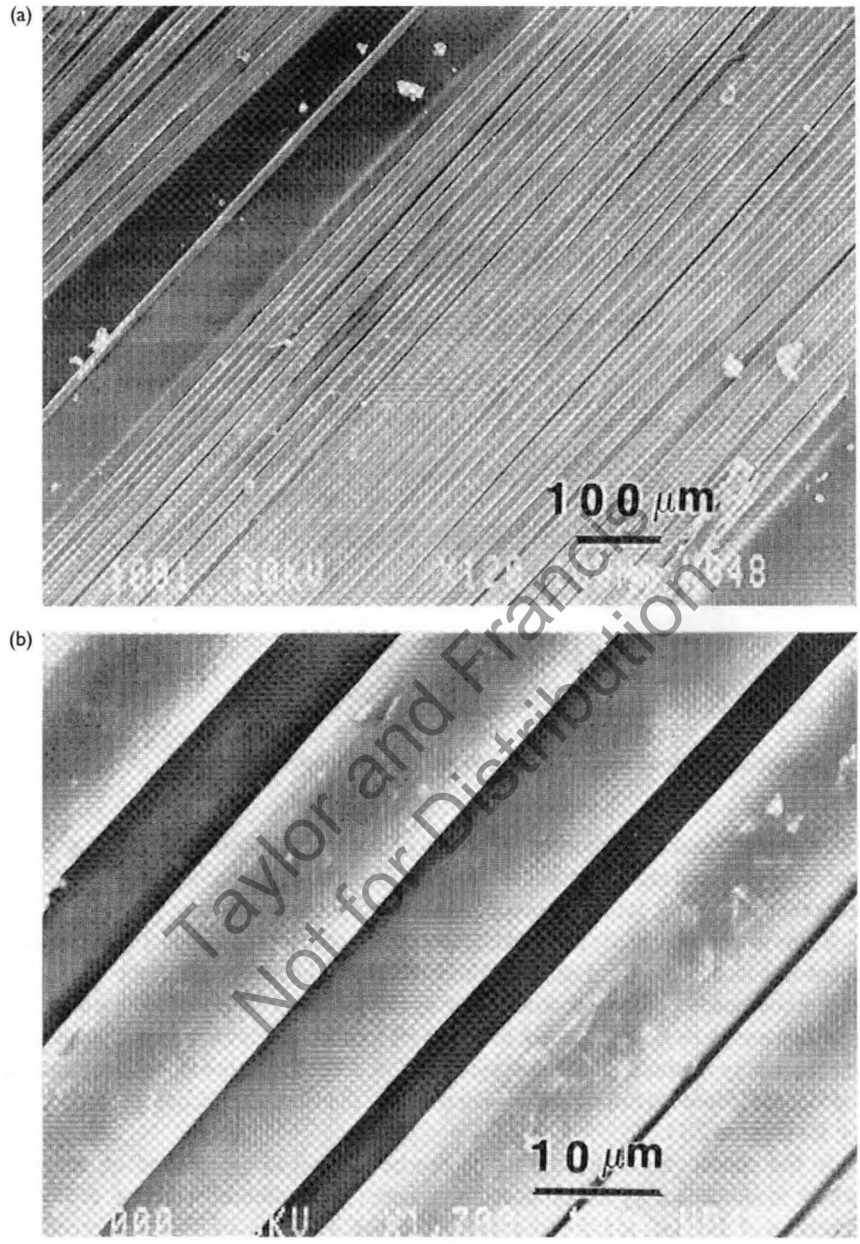


Figure 2.3 The bundled structure of glass filaments (after Bentur [8]). (a) Strands, each composed of 204 individual filaments grouped together. (b) Higher magnification of (a), showing the individual filaments in a strand. (c) The structure of the glass fibres in the cement composite, showing the bundled nature of the strand which does not disperse into the individual filaments.

(c)

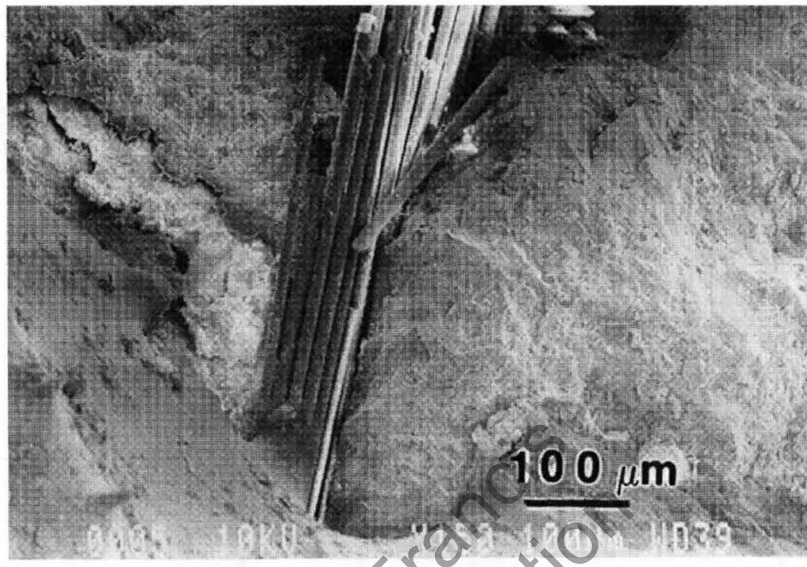


Figure 2.3 Continued.

ideally random. If the ratio of the fibre length to the thickness of the composite is sufficiently large, the fibres will assume a 2D distribution (Figure 2.1(b)), which is usually the case in thin components or thin cast overlays. A preferred 2D distribution can also be promoted in thick components due to vibration. This will give rise to anisotropic behaviour.

The uniformity of volume distribution of the fibres is very sensitive to the mixing and consolidation process, and in practice a uniform distribution is rarely achieved (Figure 2.4). The analytical treatment of fibre distribution can be based on various stereological models [12–15].

A geometrical parameter which is of significance in controlling the performance of the composite is the distance (spacing) between the fibres. Assuming a uniform fibre distribution, and using various statistical concepts, the average fibre–fibre spacing has been calculated, and several expressions have been derived. For cylindrical fibres, some of these equations take the form [16,17]:

$$S = \frac{K \cdot d}{V_f^{1/2}} \tag{2.1}$$

where S is the fibre spacing; K, a constant; d, the fibre diameter; V_f, the fibre volume content; and K varies in the range of 0.8–1.12 depending on the orientation (1D, 2D or 3D) and the assumptions made in the calculation.

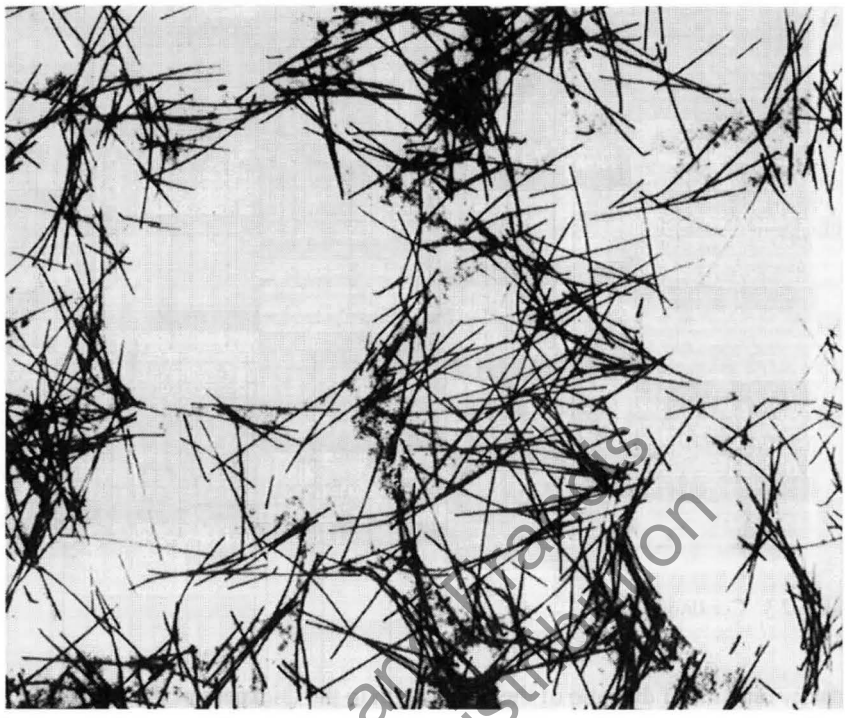


Figure 2.4 Distribution of steel fibres in concrete as observed by X-ray, showing non-uniform distribution (after Stroeven and Shah [12]).

To illustrate the relationship between fibre diameter and fibre spacing, Figure 2.5 [18] is a nomograph which yields either the fibre count (number of fibres per unit volume of FRC), or the surface area of fibres per unit volume of FRC, for unit length of fibres. If the specified volume of fibres is entered along the abscissa, then the number of fibres (or surface area) per unit volume may be found on the ordinate for a given fibre diameter.

Another way of quantifying the geometry of fibres is by using the *denier* unit common in the textile industry. A denier is the weight in grams of a 9000 m long staple. The relationship between fibre diameter and denier is shown in Figure 2.6 [19]. The fibre count and the surface area of fibres per unit volume of FRC can be expressed as functions of the weight, volume, specific gravity, denier, length and diameter as shown in the equations of Table 2.1 [19].

2.3 The structure of the fibre–matrix interface

Cementitious composites are characterized by an *interfacial transition zone* (ITZ) in the vicinity of the reinforcing inclusion, in which the microstructure of the

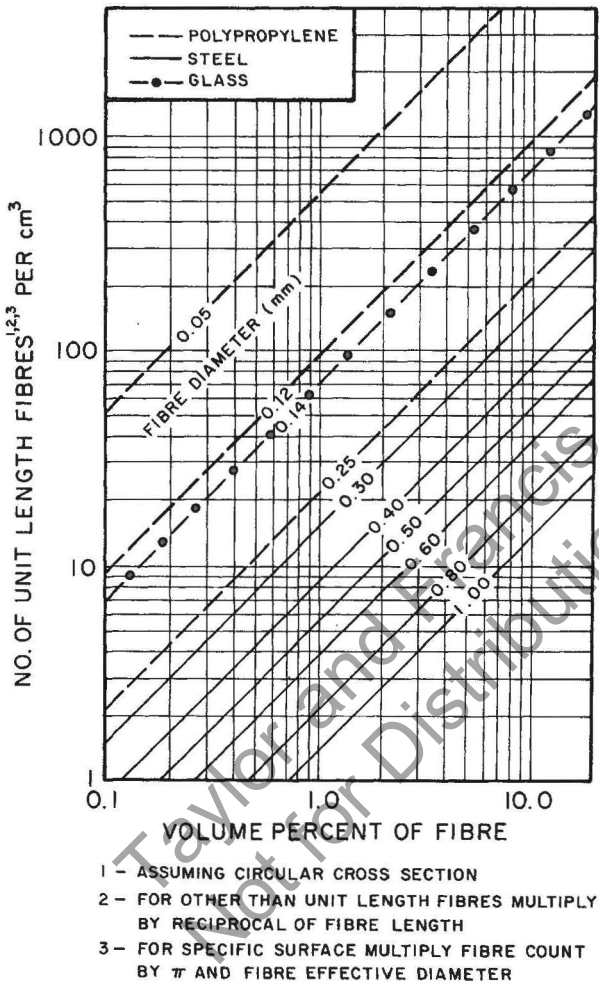


Figure 2.5 Number of fibres per unit volume, or surface area of fibres per unit volume, as a function of the volume per cent of fibres and the fibre geometry [18].

paste matrix is considerably different from that of the bulk paste, away from the interface. The nature and size of this transition zone depends on the type of fibre and the production technology; in some instances it can change considerably with time. These characteristics of the fibre–matrix interface exert several effects which should be taken into consideration, especially with respect to the fibre–matrix bond, and the debonding process across the interface (see Chapters 3 and 4).

The special microstructure of the transition zone in cementitious composites is closely related to the particulate nature of the matrix. The matrix consists of

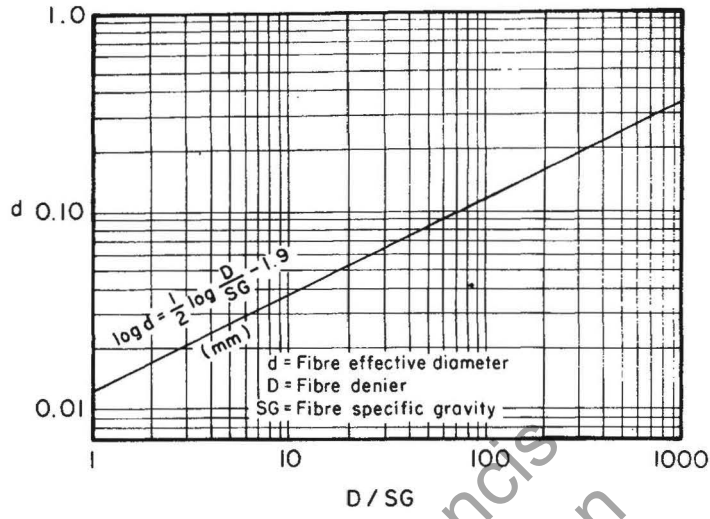


Figure 2.6 Fibre diameter vs. denier relationship [19].

Table 2.1 Fibre count (FC) and surface area (SS) of fibres per unit volume (cm^3) of FRC [19]

FC = 0.077	$WT/(\ell)(d)^2(SG)$
FC = 0.077	$(V)/(\ell)(d)^2$
FC = 3.112	$WT(10)^5/(\ell)(D)$
FC = 3.112	$V(10)^5/(\ell)(D)$
SS = 0.244	$(WT)/(d)(SG)$
SS = 0.244	$(V)/(d)$
SS = 48.81	$WT/(D)^{1/2}(SG)^{1/2}$
SS = 48.81	$V/(D)^{1/2}(SG)^{1/2}$

Note
WT = weight; V = volume; SG = specific gravity; D = fibre denier; d = fibre diameter; ℓ = fibre length.

discrete cement particles ranging in diameter from ~1 to ~100 μm (average size of ~ 10 μm) in the fresh mix, which on hydration react to form mainly colloidal CSH particles and larger crystals of CH. The particulate nature of the fresh mix exerts an important influence on the transition zone, since it leads to the formation of water-filled spaces around the fibres due to two related effects:

1 bleeding and entrapment of water around the reinforcing inclusion and
2 inefficient packing of the ~ 10 μm cement grains in the 20–40 μm zone around the fibre surface.

Thus, the matrix in the vicinity of the fibre is much more porous than the bulk paste matrix, and this is reflected in the development of the microstructure as hydration advances: the initially water-filled transition zone does not develop the dense microstructure typical of the bulk matrix, and it contains a considerable volume of CH crystals, which tend to deposit in large cavities.

When considering the development of the microstructure in the transition zone, a distinction should be made between discrete monofilament fibres separated one from the other (e.g. steel), and bundled filaments (e.g. glass). With monofilament fibres, the entire surface of the fibre can be in direct contact with the matrix; with bundled filaments only the external filaments tend to have direct access to the matrix.

2.3.1 *Monofilament fibres*

The microstructure of the transition zone around monofilament fibres has been studied primarily in steel fibre reinforced cement pastes [20–25]. It was observed that the transition zone in the mature composite is rich in CH (usually in direct contact with the fibre surface), and is also quite porous, making it different from the microstructure of the bulk paste. These characteristics are probably the result of the nature of the fresh mix, as discussed above. The CH layer can be as thin as 1 μm (duplex film), or it can be much more massive, several μm across [23]. The porous nature of the transition zone is the result of pores formed between the CSH and the ettringite in a zone which backs up the CH layer. A schematic description of the transition zone showing the different layers (duplex film, CH layer, porous layer consisting of CSH and some ettringite) is presented in Figure 2.7(a), along with some micrographs which demonstrate the microstructure of each of the layers (Figure 2.7(b)–(d)). The formation of a CH rich zone at the fibre surface is probably the result of its precipitation from the solution in the space around the fibre, with the fibre surface being a nucleation site. The CH layer adjacent to the fibre surface is not necessarily continuous and it contains some pockets of very porous, needle-like material (Figure 2.7(c)) consisting also of CSH and some ettringite. The thin duplex film can usually be observed in the vicinity of the porous zone (Figure 2.7(d)) but not around the massive CH.

The microstructure in Figure 2.7 clearly indicates that the weak link between the fibre and the matrix is not necessarily at the actual fibre–matrix interface; it can also be in the porous layer, which extends to a distance of $\sim$ 10–40 μm from the interface, between the massive CH layer and the dense bulk paste matrix. This is consistent with characteristics of the mechanical properties of the transition zone determined by microhardness testing [26–28], showing lower values in the paste matrix in the immediate vicinity of the inclusion (aggregate, fibre) than in the bulk paste away from the inclusion surface, Figure 2.8. This is reflected in observations reported in [29] showing that during pull-out of a fibre high shear displacements occurred in an interfacial zone which appeared to be 40–70 μm wide.

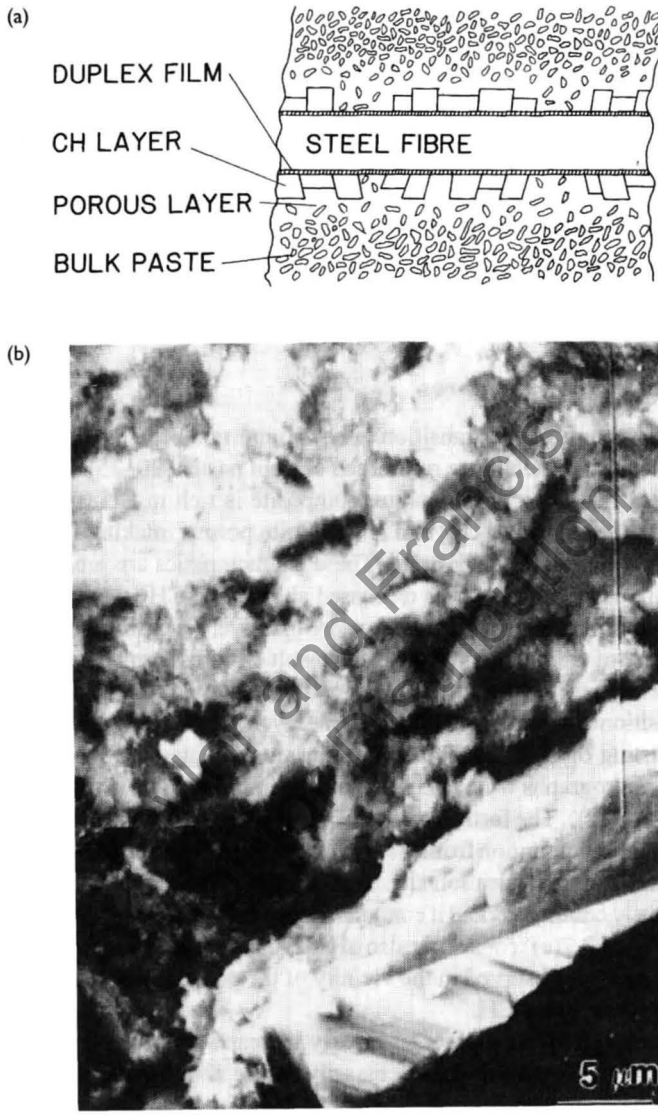
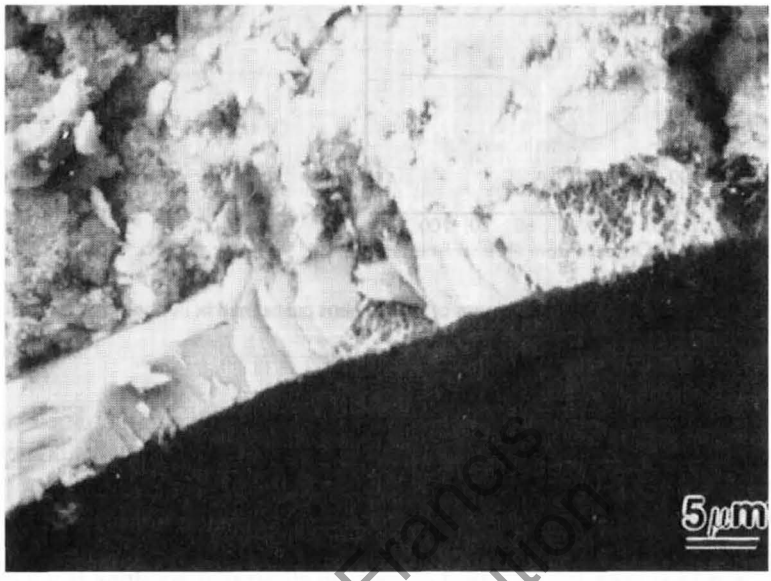


Figure 2.7 The transition zone in steel fibre reinforced cement (after Bentur *et al.* [23]). (a) schematic description; (b) SEM micrograph showing the CH layer, the porous layer and the bulk paste matrix. (c) SEM micrograph showing discontinuities in the CH layer and (d) SEM micrograph showing the duplex film backed up by porous material.

(c)



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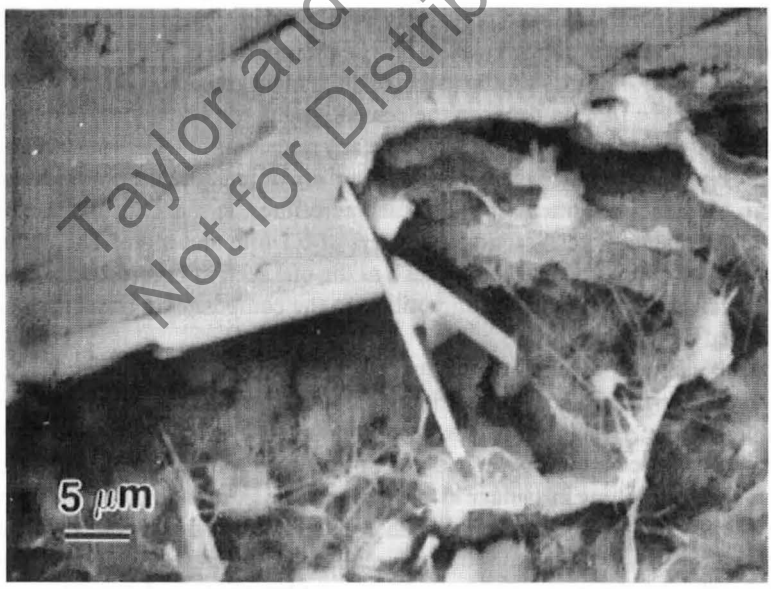

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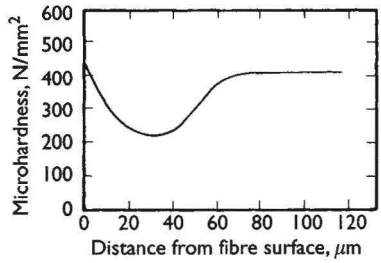

Figure 2.8 The microhardness of the cement paste matrix in contact with a steel fibre (after Wei *et al.* [26]).

It should be noted that the interfacial zone is sensitive to the processing and to the nature of the matrix. Intensive processing, which involves higher shear stresses in the fresh mix will result in a denser and smaller transition zone [30]. In the case in which the matrix is made of a well-graded mix, with fine fillers of the size of cement grains and smaller, and the fibre cross section is sufficiently small, the transition zone can be almost completely eliminated, resulting in a high bond matrix [31]. This kind of a microstructure is more likely to occur in systems such as RPC and DSP discussed previously [4–6], and in systems where the fibres are particularly small in diameter, a few tens of microns or less. In this range, the size of the fibre cross section is similar to that of the cement grains and fillers, and efficient packing of the fibre in between the cement grains can take place, resulting in an extremely dense microstructure, without any transition zone, as seen in Figure 2.9. Fibres in this size range, which is characteristic of many of the polymer and glass fibre filaments, are often referred to as microfibres, to make the distinction from macrofibres with cross-section diameters of 0.1 mm and more. The potential of getting the dense microstructure, such as the one seen in Figure 2.9, is dependent on efficient dispersion of the microfibres in the composite, to break their original bundled morphology.

Processing of softer fibres by special means, such as extrusion, can result in marked interfacial changes which are associated with abrasion of the fibre and its fibrillation, resulting in enhanced bonding [32]. Interfacial microstructural changes can occur during the pull-out of fibres induced during the loading of the composite, resulting in damage to the fibre or to the surrounding matrix, depending, to a large extent, on their relative stiffness [33]. These characteristics will be given special attention in Chapter 3. Interface tailoring is thus becoming an important tool in the development of high performance fibre reinforced cements (e.g. [34]).

2.3.2 *Bundled fibres*

In fibres consisting of bundled filaments, which do not disperse into the individual filaments during the production of the composite, the reinforcing unit is not a single

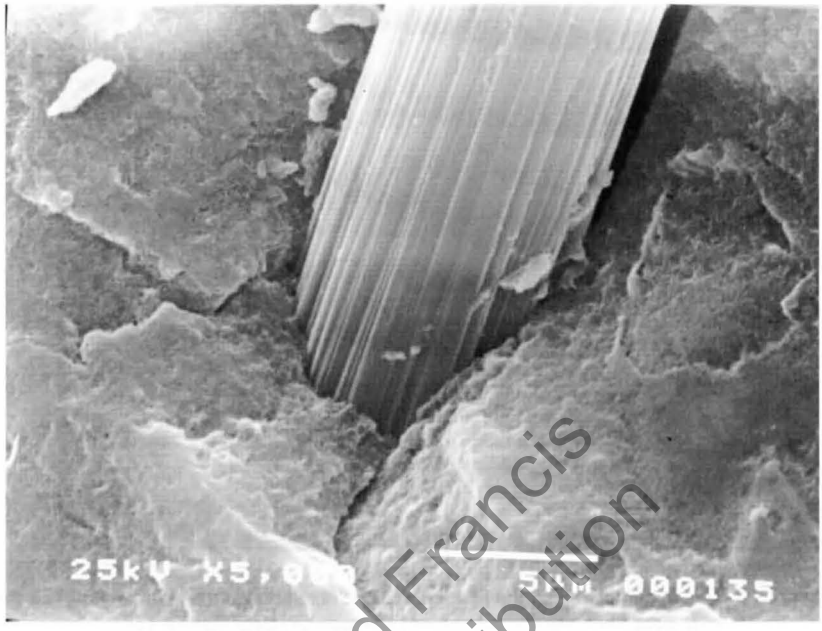

Figure 2.9 A dense interfacial microstructure formed around a microfibre (carbon) which was well dispersed as monofilament in the cement matrix (after Katz and Bentur [31]).

filament surrounded by a matrix, but rather a bundle of filaments [11,35–37] as shown in Figure 2.3(c) for glass fibres. The filaments in the fibre bundles of this kind are quite small, with diameters of $\sim$ 10 μm or less. The size of the spaces between the filaments does not exceed several μm, and as a consequence it is difficult for the larger cement grains to penetrate within these spaces. This is particularly the case with glass fibres, which have much less affinity for the cement slurry than does asbestos. The resulting microstructure after several weeks of hydration is characterized by vacant spaces between the filaments in the strand or limited localized formation of hydration products in some zones between the filaments (Figure 2.10). As a result, the reinforcing bundle remains as a flexible unit even after 28 days of curing, with each filament having a considerable freedom of movement relative to the others. Some stress transfer into the inner filaments may occur through frictional effects, aided by the point contacts formed by the hydration products and the sizing applied during the production of the glass fibre strands. In such a bundle, the bonding is not uniform, and the external filaments are more tightly bonded to the matrix.

The spaces between the filaments can be gradually filled with hydration products if the composite is kept in a moist environment. This process involves nucleation and growth stages, and the filament surfaces can serve as nucleation sites. Mills

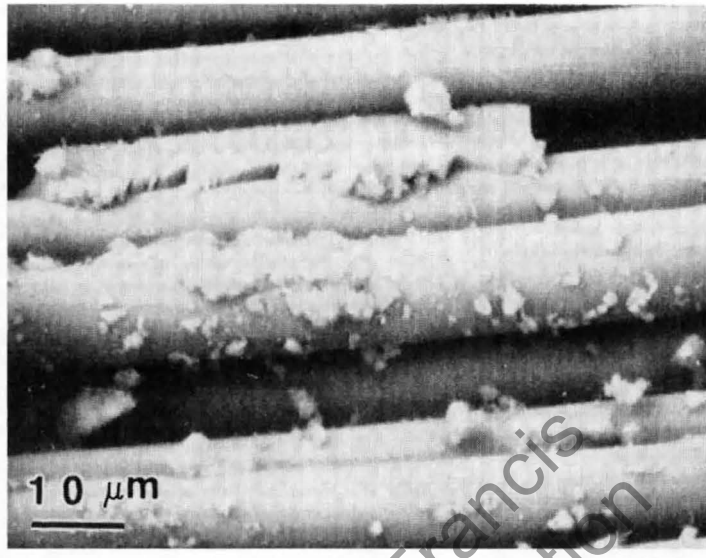

Figure 2.10 The spaces between the filaments in the reinforcing strand in a young (28 days old) glass fibre reinforced cement composite (after Bentur [37]).

[38] has demonstrated the affinity of an alkali-resistant glass fibre (AR) for nucleation and growth of CH crystals on its surface, when it was in contact with a Portland cement pore solution. This affinity is evident in the aged composite, prepared with high zirconia AR glass, where massive deposits of CH crystals were observed between the filaments [35,37], cementing the whole strand into a rigid reinforcing unit (Figure 2.11(a)). The nature of the deposited products can change, depending on the surface of the fibre. In newer generations of AR glass fibres (Cem FIL-2), in which the surface was treated by special coating [39], the hydration products deposited tend to be more porous, presumably CSH, rather than the massive crystalline CH (Figure 2.11(b)). Also, the rate of deposition is much slower [40]. This is a demonstration of the effect that the fibre surface may have on the microstructure developed in its vicinity.

The absence of CH-rich zones in the vicinity of the fibres was reported by Akers and Garrett [41] for asbestos–cement composites and by Bentur and Akers [42] for cellulose FRC composites produced by the Hatscheck process. This may be the result of the affinity of these fibres for the cement particles, and the processing treatment which involves dewatering, both of which lead to a system with very little bleeding, and probably reduce the extent of formation of water-filled spaces around the fibres in the fresh mix. This is reflected in the nature of the fibre–matrix bond failure; in asbestos composites, the cement matrix was sometimes seen to be sticking to the asbestos fibre bundle. This suggests that a strong interface was

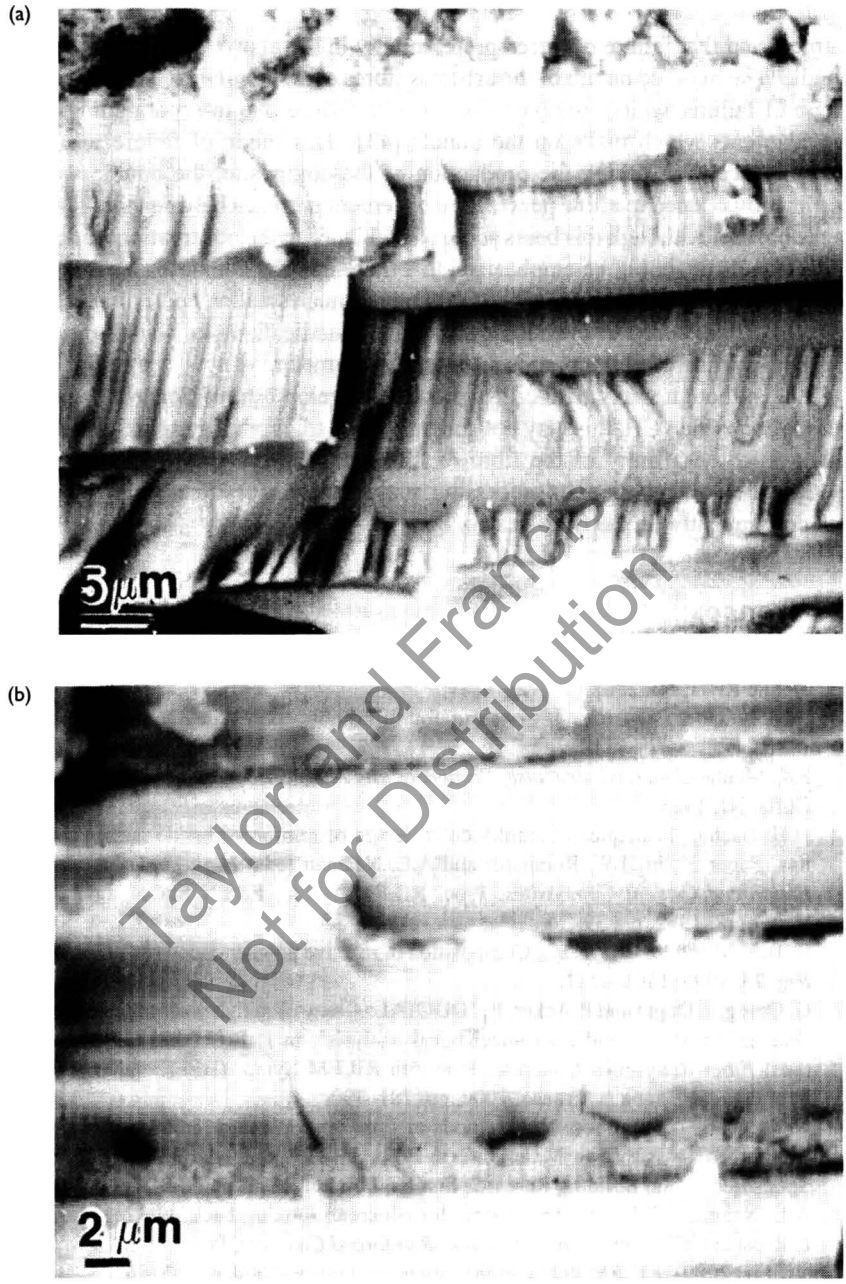

Figure 2.11 The spaces between the filaments in an aged glass fibre reinforced cement, showing them to be filled with massive CH crystals in the case of CemFIL-1 fibres (a) and more porous material in CemFIL-2 fibres; (b) (after Bentur [37]).

formed, and that failure occurred preferentially in the matrix away from the fibre bundle. The bundled nature of the asbestos fibres occasionally gave rise to another mode of failure, which involved fibre bundle failure due to separation between the filaments which make up the bundle [43]. This mode of failure was more likely to occur if, during the production of the composite, the bundle was not sufficiently opened to allow penetration of cement particles between the filaments in the bundle. Although this bears some resemblance to the observations with glass fibre strands, it should be emphasized that there is a considerable size difference between the two systems: the asbestos bundle is much smaller, consisting of fibrils of ~ 0.1 μm diameter or even less, with a fibre bundle diameter being ~ 5 μm; in the glass system each filament is $\sim$ 10 μm in diameter.

Thus, although many of the FRC systems develop a transition zone which is porous and rich in CH, this may not generally hold true for all systems. Substantial changes in the affinity of the fibre for the matrix, combined with rheological modification of the mix or its processing, may have a major effect on the interface, and consequently on the fibre–matrix bond.

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