Interface engineering for cement-matrix composites

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Abstract—The methods and effects of interface engineering for cement-matrix composites are reviewed. The methods include steel rebar surface treatments, admixture surface treatments and the use of admixtures. The effects relate to the improvement of the mechanical, thermal, chemical and processing behavior.

1. INTRODUCTION

Interfaces are present in a composite material and greatly affect its properties. Interface engineering refers to the design and preparation of an interface. This is widely practiced for composites with polymers, metals, ceramics and carbons as matrices, in order to improve the properties of the composites. However, partly due to the importance of low cost for practical concretes, interface engineering has received relatively little attention in relation to cement-matrix composites. Nevertheless, the need for improved concretes in today's infrastructure is recognized by countries all over the world, thus resulting in momentum in research to improve cement-matrix composites. Interface engineering is a significant aspect of recent research on the improvement of cement-matrix composites. This engineering involves surface treatments of steel reinforcing bars (rebars) and of admixtures, as well as the use of admixtures. The surface treatment of aggregates is economically not practical, due to the low cost and large volume of usage of aggregates in concretes. Interface engineering for cement-matrix composites is reviewed in this paper, with emphasis on the methods and effects of interface engineering, rather than the mechanisms, due to the infancy of the field.

2. BACKGROUND ON CEMENT-MATRIX COMPOSITES

Cement-matrix composites include concrete (containing coarse and fine aggregates), mortar (containing fine aggregate but no coarse aggregate) and cement paste (containing no aggregate, whether coarse or fine). It also includes steel reinforced concrete, i.e. concrete containing steel rebars. Other fillers, called admixtures, can be added to the mix to improve the properties of the composite. Admixtures are discontinuous, so that they can be included in the mix. They can be particles, such as silica fume (a fine particulate) and latex (a polymer in the form of a dispersion). They can be short fibers, such as polymer, steel, glass or carbon fibers. They can be liquids such as methylcellulose aqueous solution, water reducing agent, defoamer, etc. This section provides background on cement-matrix composites, with emphasis on carbon fiber cement-matrix composites for the purpose of illustration.

Carbon fiber (short) cement-matrix composites are structural materials that are gaining in importance quite rapidly due to the decrease in carbon fiber cost [1] and the increasing demand of superior structural and functional properties. These composites contain short carbon fibers, typically 5 mm in length, as the short fibers can be used as an admixture in concrete (whereas continuous fibers cannot be simply added to the concrete mix) and short fibers are less expensive than continuous fibers. However, due to the weak bond between carbon fiber and the cement matrix, continuous fibers [2–4] are much more effective than short fibers in reinforcing concrete. Surface treatment of carbon fiber (e.g. by heating [5] or by using ozone [6, 7], silane [8], SiO_2 particles [9] or hot NaOH solution [10]) is useful for improving the bond between fiber and matrix, thereby improving the properties of the composite. In the case of surface treatment by ozone or silane, the improved bond is due to the enhanced wettability by water. Admixtures such as latex [6, 11] methylcellulose [6] and silica fume [12] also help the bond.

The effect of carbon fiber addition on the properties of concrete increases with fiber volume fraction [13], unless the fiber volume fraction is so high that the air void content becomes excessively high [14]. (The air void content increases with fiber content and air voids tend to have a negative effect on many properties, such as the compressive strength.) In addition, the workability of the mix decreases with fiber content [13]. Moreover, the cost increases with fiber content. Therefore, a rather low volume fraction of fibers is desirable. A fiber content as low as 0.2 vol.% is effective [15], although fiber contents exceeding 1 vol.% are more common [16–20]. The required fiber content increases with the particle size of the aggregate, as the flexural strength decreases with increasing particle size [21].

Effective use of the carbon fibers in concrete requires dispersion of the fibers in the mix. The dispersion is enhanced by using silica fume (a fine particulate) as an admixture [14, 22–24]. A typical silica fume content is 15% by weight of cement [14]. The silica fume is typically used along with a small amount (0.4% by weight of cement) of methylcellulose to help the dispersion of the fibers and the workability of the mix [14]. Latex (typically 15–20% by weight of cement) is much less effective than silica fume in helping the fiber dispersion, but it enhances the

workability, flexural strength, flexural toughness, impact resistance, frost resistance and acid resistance [14, 25, 26]. The ease of dispersion increases with decreasing fiber length [24].

The improved structural properties rendered by carbon fiber addition pertain to the increased tensile and flexible strengths, the increased tensile ductility and flexural toughness, the enhanced impact resistance, the reduced drying shrinkage and the improved freeze–thaw durability [13–15, 17–25, 27–29, 20, 31–38]. The tensile and flexural strengths decrease with increasing specimen size, such that the size effect becomes larger as the fiber length increases [39]. The low drying shrinkage is valuable for large structures and for use in repair [40, 41] and in joining bricks in a brick structure [42, 43]. The functional properties rendered by carbon fiber addition pertain to the strain sensing ability [7, 44–58] (for smart structures), the temperature sensing ability [59–62], the damage sensing ability [44, 48, 63–65], the thermoelectric behavior [60–62], the thermal insulation ability [66–68] (to save energy for buildings), the electrical conduction ability [69–78] (to facilitate cathodic protection of embedded steel and to provide electrical grounding or connection), and the radio wave reflection/adsorption ability [79–83] (for electromagnetic interference or EMI shielding, for lateral guidance in automatic highways, and for television image transmission).

In relation to the structural properties, carbon fibers compete with glass, polymer and steel fibers [18, 27–29, 32, 36–38, 84]. Carbon fibers (isotropic pitch based) [1, 84] are advantageous in their superior ability to increase the tensile strength of concrete, even though the tensile strength, modulus and ductility of the isotropic pitch based carbon fibers are low compared to most other fibers. Carbon fibers are also advantageous in the relative inertness to chemicals [85]. PAN-based carbon fibers are also used [17, 19, 22, 33], although they are more commonly used as continuous fibers than short fibers. Carbon-coated glass fibers [86, 87] and submicron diameter carbon filaments [77–79] are even less commonly used, although the former are attractive for the low cost of glass fibers while the latter are attractive in providing high radio wave reflectivity (which results from the skin effect). C-shaped carbon fibers are more effective for strengthening than round carbon fibers [88], but their relatively large diameter makes them less attractive. Carbon fibers can be used in concrete together with steel fibers, as the addition of short carbon fibers to steel fiber reinforced mortar increases the fracture toughness of the interfacial zone between steel fiber and the cement matrix [89]. Carbon fibers can also be used in concrete together with steel rebars [90, 91], or together with carbon fiber reinforced polymer rods [92].

In relation to most functional properties, carbon fibers are exceptional compared to the other fiber types. Carbon fibers are electrically conducting, in contrast to glass and polymer fibers, which are not conducting. Steel fibers are conducting, but their typical diameter ($\geqslant 60$ μm) is much larger than the diameter of a typical carbon fiber (15 μm). The combination of electrical conductivity and small diameter makes carbon fibers superior to the other fiber types in the area of strain sensing

and electrical conduction. However, carbon fibers are inferior to steel fibers for providing thermoelectric composites, due to the high electron concentration in steel and the low hole concentration in carbon.

Although carbon fibers are thermally conducting, addition of carbon fibers to concrete lowers the thermal conductivity [66], thus allowing applications related to thermal insulation. This effect of carbon fiber addition is due to the increase in air void content. The electrical conductivity of carbon fibers is higher than that of the cement matrix by about 8 orders of magnitude, whereas the thermal conductivity of carbon fibers is higher than that of the cement matrix by only one or two orders of magnitude. As a result, the electrical conductivity is increased upon carbon fiber addition in spite of the increase in air void content, but the thermal conductivity is decreased upon fiber addition.

The use of pressure after casting [93], and extrusion [94, 95] can result in composites with superior microstructure and properties. Moreover, extrusion improves the shapability [95].

3. STEEL REBAR SURFACE TREATMENTS FOR INTERFACE ENGINEERING

The interface between steel rebar and concrete affects the bond strength between rebar and concrete, in addition to affecting the corrosion resistance of the rebar in the concrete and affecting the vibration reduction ability of the steel reinforced concrete. The shaping of the rebar (say, having surface deformations called ribs, or having a hook at an end of the rebar) enhances the mechanical interlocking between rebar and concrete, thereby improving the interface. However, this section focuses on rebar surface treatments rather than the shaping of the rebar for the purpose of interface engineering. The treatments include sand blasting, [96–98], water immersion [97], ozone treatment [96, 98] and acetone washing [96]. Some of them are useful for improving the bond strength, vibration reduction ability and corrosion resistance, as described below.

Sand blasting involves the blasting of ceramic particles (typically alumina particles of size around 250 μm) under pressure (typically around 80 psi or 0.6 MPa). It results in roughening as well as cleaning of the surface of the steel rebar. The purpose of the cleaning is to remove rust and other contaminants typically found in places on the rebar surface. The cleaning also causes the surface of the rebar to be more uniform in composition.

Water immersion means total immersion of the rebar in water at room temperature for two days. It causes the formation of a black oxide layer on the surface of the rebar. Water immersion times that are less than or greater than two days give less desirable effects on both bond strength and corrosion resistance.

Ozone treatment involves exposure of the rebar to ozone (O_3) gas (say, 0.3 vol.% in air) for 20 min at 160°C, followed by drying at 110°C in air for 50 min. This causes the formation of a dark gray oxide layer on the surface of the rebar.
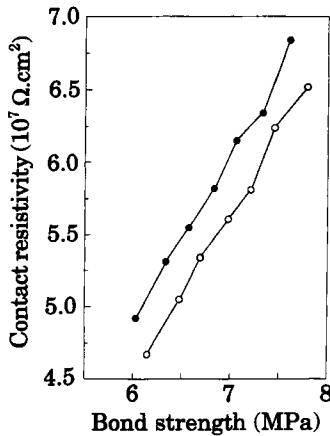
Figure 1. Variation of contact electrical resistivity with bond strength between steel rebar and concrete at 28 days of curing. Solid circles: as-received steel rebar. Open circles: acetone treated steel rebar.
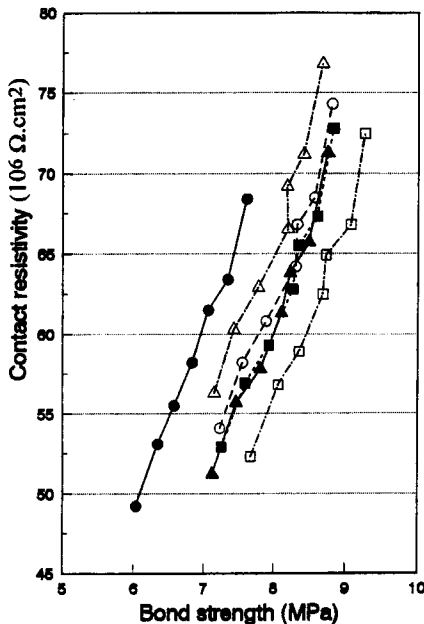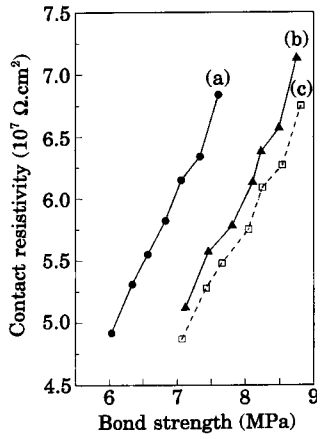

Figure 2. Variation of contact electrical resistivity with bond strength between steel rebar and concrete at 28 days of curing. Solid circles: as-received steel rebar. Solid triangles: steel rebar immersed in water for 2 days. Solid squares: steel rebar immersed in water for 5 days. Open circles: steel rebar immersed in water for 7 days. Open triangles: steel rebar immersed in water for 10 days. Open squares: O_3 treated steel rebar.

Acetone washing involves immersion of rebar in acetone for 15 min, followed by drying in air. This process removes the grease on the rebar surface.

Figures 1–3 show the correlation of the contact resistivity of the rebar–concrete interface with the shear bond strength for different surface treatments of steel rebar.

Figure 3. Variation of contact electrical resistivity with bond strength between steel rebar and concrete at 28 days of curing. (a) As-received rebar. (b) Water treated rebar. (c) Sand blasted rebar.

The contact resistivity increases almost linearly with increasing bond strength, such that the data for the different surface treatments lie on essentially parallel straight lines. Acetone treatment increases the bond strength slightly and decreases the contact resistivity slightly (Fig. 1) (as in the case of the interface between stainless steel fiber and cement paste [99]), presumably because of the degreasing action of the acetone. Water immersion for 2–5 days (Fig. 2) increases the bond strength by 14% (more than for acetone treatment) and slightly increases the contact resistivity (in contrast to the decrease in contact resistivity for acetone treatment). Increase of the water immersion time beyond 5 days causes the bond strength to decrease and the contact resistivity to increase further (Fig. 2). However, even for a water immersion time of 10 days, the bond strength is still higher than that for the as-received rebar. Thus, a water immersion time of 2 days is recommended. Figure 2 shows that ozone treatment enhances the bond strength more than any of the water treatments. The contact resistivity is also increased by the ozone treatment, but not as much as in the case of water treatment for 7 or 10 days.

It is reasonable to assume that the contact resistivity is related to the amount of oxidation product at the rebar–concrete interface, as the oxidation product is a poor electrical conductor. Hence, the differences in contact resistivity (Fig. 2) suggest that the amount of oxidation product is comparable between O_3 treatment and 2–5 day water treatments, but is larger for 7–10 day water treatments. The phase of the oxidation product differs between O_3 and water treatments, as indicated by the black color of the oxidation product of the water treatments and the dark gray color of the oxidation product of the O_3 treatment. This phase difference is believed to be partly responsible for the difference in the extent of bond strength enhancement.

The contact resistivity increases with increasing bond strength among the data for each water immersion time (Fig. 2). The origin of this dependence is associated with interfacial phase(s) of volume resistivity higher than that of concrete. The

interfacial phase enhances the bonding, unless it is excessive. It may be a metal oxide. Water treatment increases both bond strength and contact resistivity because the treatment forms a black phase that may be akin to rust on the rebar; the phase enhances the bonding but increases the contact resistivity. The longer the water immersion time, the more the black phase and the higher the contact resistivity. However, an excessive amount of the black phase (as obtained after 7 or 10 days of water immersion) weakens the bond.

At the same bond strength, the water treated rebar exhibits a lower contact resistivity than the as-received rebar (Fig. 2). As the amount of black phase increases with increasing contact resistivity, this implies that the black phase formed by the water treatment is more effective than the rust or rust-like phase(s) formed without the water treatment in enhancing the bond strength. The greater effectiveness of the former is probably partly because of the more uniform distribution of the black phase and partly because of the possible differences in phase between the black phase and the rust or rust-like phase formed without the water treatment.

Water treatment and sand blasting increase the bond strength to similar extents (Fig. 3), which are less than that provided by ozone treatment (Fig. 2). Water immersion, like ozone treatment, causes the contact resistivity to increase, but sand blasting has negligible effect on the contact resistivity. This is consistent with the presence of a black coating on the rebar after water immersion and the absence of a coating after sand blasting. Scanning electron microscopy (SEM) shows that sand blasting roughens the surface in a coarse way, whereas water treatment results in a fine surface microstructure. The uneven surface quality (due to uneven rusting) in the as-received rebar is removed after sand blasting or water treatment, as shown by visual observation. In spite of the significant roughening by sand blasting, the bond strength is similar for the sand blasted rebar and the water treated rebar. This suggests that the bond strength increase after water immersion is essentially not due to surface roughening, but is due to change in the surface functional groups (as supported by the black coating) which affect the adhesion between rebar and concrete.

The corrosion resistance of steel rebar in concrete greatly affects the durability of steel reinforced concrete. Water immersion (2 days) and sand blasting are similarly effective for treating steel rebars for the purpose of improving the corrosion resistance of the rebar in the concrete. The increase in corrosion resistance is due to the surface uniformity rendered by either treatment.

Vibration damping is valuable for structures, as it mitigates hazards (whether due to accidental loading, wind, ocean waves or earthquakes), increases the comfort of people who use the structures, and enhances the reliability and performance of structures. Both passive and active methods of damping are useful, although active methods are usually more expensive due to the devices involved. Passive damping most commonly involves the use of viscoelastic materials such as rubber, though these materials tend to suffer from their poor stiffness and high cost compared to the structural material (i.e. concrete). High stiffness is useful for vibration reduction.

These problems with stiffness and cost can be removed if the structural material itself has a high damping capacity. The use of the structural material for passive damping also lowers the cost of damping implementation. Moreover, due to the large volume of structural material in a structure, the resulting damping ability can be substantial. Therefore, the development of concrete that inherently has a high damping capacity is of interest.

The vibration reduction ability of mortar, as expressed by the loss modulus (product of loss tangent and storage modulus) under dynamic flexure (0.2–1.0 Hz), is increased by up to 91% by sand blasting the steel rebar, due to the increase in the damping capacity [98]. Surface treatment of the rebar by ozone has negligible effect on the loss modulus.

4. ADMIXTURE SURFACE TREATMENTS FOR INTERFACE ENGINEERING

Cement-based materials containing solid admixtures such as silica fume and short carbon fibers are improved by surface treatment prior to using the admixtures. Consistency (workability), static and dynamic mechanical properties, specific heat and drying shrinkage are improved [100, 101].

Mortar with high consistency, even without a water-reducing agent, is obtained by using silica fume that has been surface treated with silane, which is hydrophilic [100]. The treatment also increases the strength and modulus, both under tension and compression. In particular, the tensile strength is increased by 31% and the compressive strength is increased by 27%. Moreover, the flexural storage modulus (stiffness), loss tangent (damping capacity) and density are increased [100].

The tensile strength of cement paste is increased by 56% and the modulus and ductility are increased by 39% by using silane treated carbon fibers and silane treated silica fume, relative to the values for cement paste with as-received carbon fibers and as-received silica fume [100]. Silane treatment of fibers and silica fume contributes about equally to the strengthening effect. Silane treatment of fibers and silica fume also decreases the air void content. The effects on strengthening and air void content reduction are less when the fiber treatment involves potassium dichromate instead of silane and even less when the treatment involves ozone.

The addition of short carbon fibers to cement paste containing silica fume and methylcellulose causes the loss tangent under flexure ($\leqslant 1$ Hz) to decrease by up to 25% and the storage modulus ($\leqslant 2$ Hz) to increase by up to 67%, such that both effects increase in the following order: as-received fibers, ozone-treated fibers, dichromate-treated fibers and silane-treated fibers [100]. Silane treatment of silica fume has little effect on the loss tangent, but increases the storage modulus by up to 38% [100].

The specific heat of cement paste is increased by 12% and the thermal conductivity is decreased by 40% by using silane-treated silica fume and silane-treated carbon fibers [100]. The specific heat is increased by the carbon fiber addition, due to fiber–matrix interface slippage. The increase is also in the above order, due to the

Table 1.
Surface elemental composition (in at.%) of carbon fiber

Element	As-received	O_3 treated	Dichromate treated	Silane treated
O	13	20.2	20.6	21.8
N	—	—	—	9.3
C	85.4	79.8	79.4	54.3
Cl	—	—	—	4.0
Si	1.7	—	—	10.6

Table 2.
Surface elemental composition (in at.%) of silica fume

Element	As-received	Silane treated
O	56.7	50.0
C	10.5	19.3
Si	32.8	30.7
N	—	—

increasing contribution of the movement of the fiber–matrix covalent coupling. The specific heat is increased by the silica fume addition, due to slippage at the interface between silica fume and cement. The increase is enhanced by silane treatment of the silica fume. Silane treatment of carbon fibers decreases the thermal conductivity.

Silane treatment of carbon fibers and silica fume increases the effectiveness of these admixtures for reducing the drying shrinkage of cement paste [101].

The effects of ozone, dichronate and silane treatments on the surface elemental composition (based on ESCA) of carbon fiber are shown in Table 1 [101]. The surface carbon concentration is decreased and the surface oxygen concentration is increased by any of the three surface treatments. In the case of the O_3 and dichromate treatments, this is due to the oxidation of the fiber surface and the introduction of hydrophylic functional groups such as −OH and −COOH to the surface. The charge corrected binding energies (C_{1s} and O_{1s}) of both O_3 and dichromate treated carbon fiber surfaces confirm the existence of these functional groups. In the case of the silane treatment, the oxygen, nitrogen and silicon concentrations on the surface increase and the carbon concentration on the surface decrease after the treatment, due to the composition of the silane coating of the surface (Table 1). The charge corrected binding energies of the silane treated fiber surface confirm the presence of silane.

Table 2 shows the ESCA results of silica fume particles with and without treatment. Compared to as-received silica fume, the silane treated silica fume particle surface has more carbon, but fewer oxygen and silicon atoms. This is consistent with the fact that the surface is partly covered by the silane coating and that the silane used contains C, Si and O atoms.

The ESCA results show that both carbon fiber and silica fume particle surfaces are partly coated by or bonded to silane molecules. Due to the hydrophilic nature of silane, the treated fibers and treated silica fume are expected to be more uniformly distributed in the cement. More importantly, the formation of chemical bonds at which silane serves as bridges between the surface of fiber or silica fume and the cement matrix is expected to make the composite denser and stronger, as shown in the case of silica fume [100]. Therefore, the drying shrinkage strain is decreased by silane treatment of fibers and/or silica fume.

5. ADMIXTURES FOR INTERFACE ENGINEERING

Admixtures (liquids or solids) can be used in cement-matrix composites (whether cement paste, mortar or concrete) for interface engineering.

The use of methylcellulose (a liquid solution) and untreated silica fume (solid particles) as two admixtures in cement paste, relative to the use of untreated silica fume as the sole admixture, increases the loss tangent by up to 50% and decreases the storage modulus by up to 14% [100].

The use of silane (a liquid) and untreated silica fume as two admixtures in cement paste, relative to the use of silane treated silica fume as the sole admixture, increases the compressive modulus, but decreases the compressive ductility and damping capacity [102, 103]. It also decreases the air void content and increases the density, specific heat and thermal conductivity. The effects of the silane treatment of silica fume are due to the enhanced hydrophilicity of silica fume and the covalent coupling between silica fume particles and cement. The effects of silane and untreated silica fume as two admixtures are due to the network of covalent coupling among the silica fume particles.

Polymer admixtures (such as methylcellulose and latex) to concrete increase the bond strength between concrete and steel rebar [96]. Figure 4 shows the correlation of the contact resistivity with the bond strength for different polymer admixtures in concrete. Polymer admixtures (curves (b) and (c) of Fig. 4) are slightly less effective than ozone treatment of rebar (curve (d) of Fig. 4) for increasing the bond strength between rebar and concrete (as well as that between carbon fiber and cement paste [6]). Between the two polymer admixtures, latex (curve (c) of Fig. 4) increases the bond strength slightly more significantly than methylcellulose (curve (b) of Fig. 4), at least partly due to the large amount of latex compared to the amount of methylcellulose. The combined use of latex and ozone treatment (curve (e) of Fig. 4) gives significantly higher bond strength than ozone treatment alone (curve (d) of Fig. 4). Relative to the combination of plain concrete and untreated rebar, the combined use of latex and ozone treatment results in a 39% increase in the bond strength. Ozone treatment, latex addition and combined ozone treatment and latex addition cause similarly small increases in the contact resistivity.

The contact resistivity increase after latex addition is presumably due to the high volume resistivity of the latex present at the rebar–concrete interface. The bond
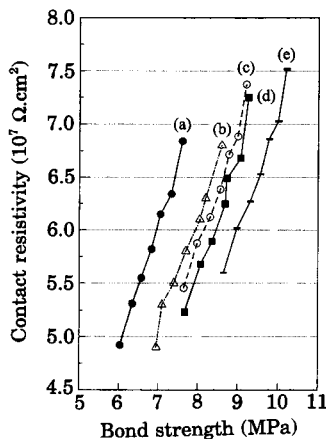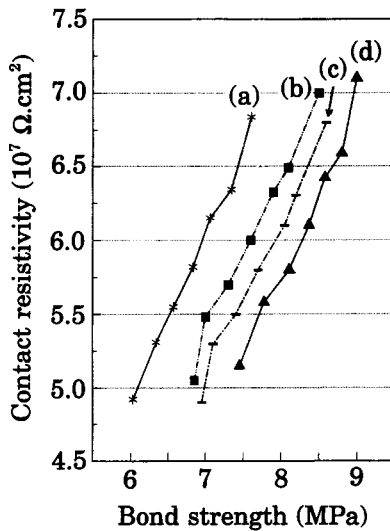
Figure 4. Variation on the contact electrical resistivity with bond strength between steel rebar and concrete at 28 days of curing. (a) Plain concrete and untreated rebar. (b) Concrete with methylcellulose addition and untreated rebar. (c) Concrete with latex addition and untreated rebar. (d) Plain concrete and ozone treated rebar. (e) Concrete with latex addition and ozone treated rebar.

strength increase after latex or methylcellulose addition is attributed to the adhesion provided by the polymer at the interface.

Silica fume as an admixture in concrete increases the bond strength between steel rebar and concrete [104–106], due to the increase in the cement matrix modulus [106], and probably partly due to the densification of the transition zone between steel and the cement paste [104, 105]. The combined use of silica fume and methylcellulose as two admixtures further enhances the bond strength between rebar and concrete [106] beyond the values attained by the use of silica fume as the sole admixture or the use of methylcellulose as the sole admixture [106], as shown in Fig. 5.

In spite of the fact that the mechanical interlocking between rebar and concrete due to the surface deformations on the rebar contributes much to the bond strength between rebar and concrete (as shown by the much higher bond strength between rebar and concrete than that between steel fiber and cement paste [107]), the ozone treatment of the rebar and the polymer admixtures to the concrete give significant increases to the bond strength between rebar and concrete. This indicates the importance of interface engineering in improving the bond between rebar and concrete. In the case of the bond between stainless steel fiber and cement paste, the polymer admixtures (latex or methylcellulose) in the cement paste cause the bond strength to increase by 90% [107]. If the surface deformations on the steel rebar were absent, the effects of ozone treatment of rebar and of polymer admixtures in concrete would have been much larger than those described here.

The beneficial effect of polymer admixtures on the bond strength between concrete and concrete has also been shown [108–110]. The presence of a polymer interlayer at the cement–aggregate interface has been shown by microscopy to be responsible for the improved adhesion between cement and aggregate [111].

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Figure 5. Variation of contact electrical resistivity with bond strength between steel rebar and concrete. (a) Plain concrete. (b) Concrete with silica fume. (c) Concrete with methylcellulose. (d) Concrete with silica fume and methylcellulose.

Admixtures (silica fume and latex) in concrete also enhance the corrosion resistance of steel rebar in the concrete [112]. However, the reason is not related to interface engineering, but is due to the decrease in the water absorptivity of concrete, and in the case of latex, also significant is the increase in the electrical resistivity of the concrete.

6. CONCLUSION

Interface engineering is effective for improving the mechanical, thermal, chemical (corrosion resistance) and processing (workability) behavior of cement-matrix composites. The techniques of interface engineering include steel rebar surface treatments, admixture surface treatments and the use of admixtures.

Acknowledgement

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