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$CaCO_3$ whisker modified Engineered Cementitious Composite with local ingredients

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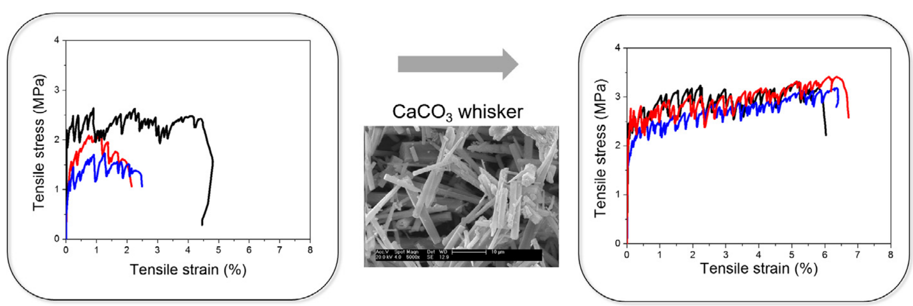
HIGHLIGHTS

- Incorporating $CaCO_3$ whisker in optimal content of 0.5% can increase the tensile strength of ECC by 53.5%.
- Incorporating $CaCO_3$ whisker can improve the tensile strain capacity of ECC significantly.
- Incorporating $CaCO_3$ whisker also improve the robustness of ECCs' mechanical properties significantly.
- When $CaCO_3$ whisker was added, the frictional bond strength between fiber and matrix was significantly affected by whisker.

GRAPHICAL ABSTRACT



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ABSTRACT

This paper aims at developing $CaCO_3$ whisker modified ECC with local ingredients in China. $CaCO_3$ whisker is a kind of micro scale fibrous material (inorganic single crystal) with a diameter of 0.5–2 µm and an aspect ratio of 20–60. It has a high tensile strength of 3–6 GPa and high elastic modulus of 410–710 GPa. Therefore, $CaCO_3$ whisker can potentially reinforce the ECC materials at microscopic level. In this study, cube compressive and uniaxial tensile tests were conducted to investigate the influence of $CaCO_3$ whisker on ECC's mechanical properties. The experimental results indicated that incorporating $CaCO_3$ whisker can improve compressive strength and tensile strain-hardening behavior (especially tensile strain capacity). When $CaCO_3$ whisker was added at the optimal content of 0.5% by volume of total ECC mixture, the compressive strength of composite increased from 23 MPa to 30 MPa, and the ultimate tensile strength and tensile strain capacity increased by 53% and 114%, respectively. Addition of $CaCO_3$ whisker can also enhance the robustness of ECC mixtures. The coefficient of variation of mechanical properties was found to be reduced by 90% compared to that of the ECC without $CaCO_3$ whisker. In addition, when $CaCO_3$ whisker is added, fly ash content has negligible influence on ultimate tensile strength of the composite, which suggests that the frictional bond strength of fiber/matrix interface is significantly affected by $CaCO_3$ whisker.

1. Introduction

Engineered Cementitious Composite (ECC), a special kind of High Performance Fiber Reinforced Cementitious Composites (HPFRCC), was developed by Li and coworkers in the 1990s [1]. It

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exhibits an excellent tensile strain hardening behavior with high tensile ductility in the range of 2–5% (200–500 times that of normal concrete or FRC) [2,3]. A typical uniaxial tensile stress-strain-crack width curve of ECC is shown in Fig. 1 [4]. As can be seen in this figure, after first cracking, the crack width stabilizes at about 60 μm while the number of cracks continues to increase with load, which is the source of the excellent tensile ductility of ECC material [4–6]. In addition, the multiple and tight cracks is another advantage of ECC material. Unlike normal concrete and FRC materials, the tight crack width is an intrinsic property of ECC, independent of structural size, steel reinforcement, or the load applied to a structure built with ECC. Owing to the excellent ductility, ECC has been applied in field applications successfully, including repair of concrete structures such as dam and irrigation channels [6], coupling beams in high rise buildings [7] and bridge deck link slab [8].

On the other hand, due to the high cost of imported PVA fiber, re-development of ECC with local ingredients (especially PVA fiber) is necessary for the broader adoption of ECC materials. A series of ECCs with Chinese local ingredients were developed by Zhang and Qian [9], Qian and Zhang [10], Pan et al. [11] and Ma et al. [12]. Zhang and Qian analyzed the feasibility of developing ECC with local ingredients (PVA fibers) through four-point bending test. Pan et al. developed ECCs with a combination of domestic and imported PVA fibers. Ma et al. developed ECCs with two domestic PVA fibers (WW PVA fiber and BHL PVA fiber). Although the ECCs in above literatures have an acceptable tensile ductility, their strengths are relatively lower. The ECC in [11] with 0.6% domestic PVA fiber and 1% imported PVA fiber has an ultimate tensile strength of 3.5 MPa. The ECC in [12] with 2% WW PVA fiber only has a compressive strength of 23 MPa and ultimate tensile strength of about 2.2 MPa, which greatly restricted their adoption. Therefore, further modification of these ECCs is necessary.

$CaCO_3$ whisker is a kind of micro scale fibrous material (inorganic single crystal) with a diameter of 0.5–2 μm and an aspect ratio of 20–60. It has a high tensile strength of 3–6 GPa and high elastic modulus of 410–710 GPa. Some previous investigations have attempted the use of $CaCO_3$ whisker to improve mechanical properties of cementitious material. According to the results of Cao et al. [13], incorporating $CaCO_3$ whisker can effectively postpone the onset of microscopic damage due to delayed initiation and propagation of micro-cracks. In other words, the $CaCO_3$ whisker can reinforce the composite material at microscopic level. Although incorporating $CaCO_3$ whisker can increase total porosity of cement mortars, it also refines the pore distribution in cement mortar [14].

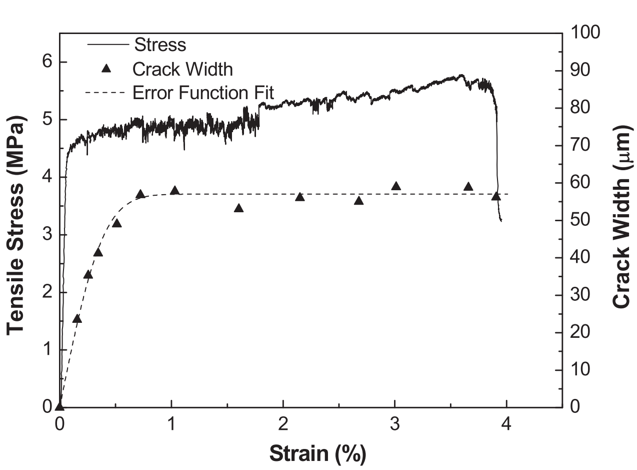

Fig. 1. A typical uniaxial tensile stress-strain-crack width curve of ECC [4].

Cao et al. [15] studied hybrid fibers effect, including steel fiber, PVA fiber and $CaCO_3$ whisker, on mechanical properties of cementitious composite. Their results indicated a slight improvement in compressive strength, and a significant improvement in flexural strength and flexural toughness, with a combination of 1.25% steel fiber, 0.55% PVA fiber and 2% $CaCO_3$ whisker. The results of Li et al. [16] indicated that the cementitious composite containing 10% $CaCO_3$ whisker and 0.3% carbon fiber has a higher compressive and flexural strength than that of the composite with only 10% $CaCO_3$ whisker or 0.3% carbon fiber. Cai and Pan [17] attempted to utilize $CaCO_3$ whisker to improve ECC material and simultaneously reduce the cost. Their results indicated that the ECC with $CaCO_3$ whisker for partial replacement of the PVA fiber shows a favorable tensile strain hardening behavior.

In this study the $CaCO_3$ whisker was used in combination with domestic PVA fiber to address some of the issues facing local ECC development in the literature, such as limited strength, limited ductility and/or poor uniformity. The optimal content of $CaCO_3$ whisker was determined through evaluating compressive strength and uniaxial tensile behavior of ECCs. And the coefficient of variation (CV) was used to evaluate the influence of $CaCO_3$ whisker on ECC mixtures' robustness. Besides, the influences of $CaCO_3$ whisker on ECC's microstructure and fiber/matrix interface properties were investigated by SEM and single fiber pullout test. The combined effect of $CaCO_3$ whisker and fly ash on ECC's mechanical properties was also investigated in this study.

2. Experimental programs

2.1. Raw materials and mix proportions

In this study, raw materials include cement, fly ash, silica sand, $CaCO_3$ whisker and PVA fiber. Table 1 lists the chemical compositions of Portland cement and fly ash. The fine silica sand has a size distribution of 106–212 μm and a mean size of 150 μm. The $CaCO_3$ whisker has a length of 20–30 μm and a diameter of 0.5–2 μm. The physical properties and chemical compositions of $CaCO_3$ whisker provided by manufacturer are listed in Table 2. The microscopic morphology of $CaCO_3$ whisker is shown in Fig. 2. The WW PVA fiber was used in this study, and its physical and mechanical properties are listed in Table 3.

The mix proportions of ECCs in this study are listed in Table 4, where D4-0 is the control mix without $CaCO_3$ whisker. The influence of $CaCO_3$ whisker content on ECC's mechanical properties was investigated based on this mix proportion. Mixtures with 0.5%, 1% and 2% $CaCO_3$ whisker by volume form the first test series to screen for the optimal $CaCO_3$ whisker content. Once the optimal $CaCO_3$ whisker content was determined (0.5%), the combined effect of fly ash and $CaCO_3$ whisker was also investigated by varying the content of fly ash (FA/C = 3.0, 2.2 and 1.2). The ECC mixtures were labeled as D3.0-0.5, D2.2-0.5 and D1.2-0.5, which form the second test series.

2.2. Specimen preparation and tests

All ECC mixtures were mixed by a planetary mixer with 10 L capacity. Firstly, all solid ingredients, including cement, silica sand and fly ash, were mixed for 3 min. Secondly, water and high range water reducer were added and mixed for another 5 min. When the fresh mixture reached a uniform state, $CaCO_3$ whisker and PVA fiber were added slowly and mixed for 10 more minutes until the fibers were distributed evenly. The specimens were demolded after 24 h, and then cured in sealed condition at 90 ± 5% RH and temperature of 20 ± 2 °C until 28 days. For each test and each mixture, three specimens were prepared.

Mechanical tests on ECCs include compressive test and uniaxial tensile test. The compressive test was conducted on a hydraulic pressure testing machine with a load capacity of 2000 KN. The compression cube specimens have an edge dimension of 70.7 mm. The uniaxial tensile test was conducted on dog-bone specimen using a 20 KN SANS test machine. Two LVDTs were fixed on either sides of the specimen to measure the deformation. The test was conducted under a displacement control of 0.5 mm/min as recommended by the Japan Society of Civil Engineers (JSCE) [18].

In order to investigate the influence of $CaCO_3$ whisker on ECC's microstructure and fiber/matrix interface properties, the SEM and single fiber pullout test were conducted. The single fiber pullout test is shown in Fig. 3. The test configuration, data interpretation and calculation procedure of the interfacial parameters follow those of Redon et al. [19]. To ensure that the PVA fiber is aligned with the load direction, a cross regulator was set as pedestal. In addition, in order to ensure the fiber can be pulled out, the fiber embedded length was set as below 1 mm. The test was conducted under a displacement control of 0.4 mm/min.

Table 1
Chemical compositions of cement and fly ash (%).

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	P_2O_5	Na_2O	K_2O	TiO_2	MgO
Cement	21.26	7.67	2.88	57.82	4.04	5.26	0	0.78	0.21	–
Fly ash	52.25	27.42	4.84	7.22	1.83	0.89	0.4	1.32	1.10	2.57

Table 2
Physical properties and chemical compositions of $CaCO_3$ whisker.

Physical properties		Chemical compositions (%)			
Density	2.8 g/cm^3	CaO	54.93	SO_3	0.31
Tensile strength	3–6 GPa	SiO_2	0.29	MgO	2.14
Elastic modulus	410–710 GPa	Al_2O_3	0.11		
Length	20–30 μm	Fe_2O_3	0.07		
Diameter	0.5–2 μm	CO_2	42.07		

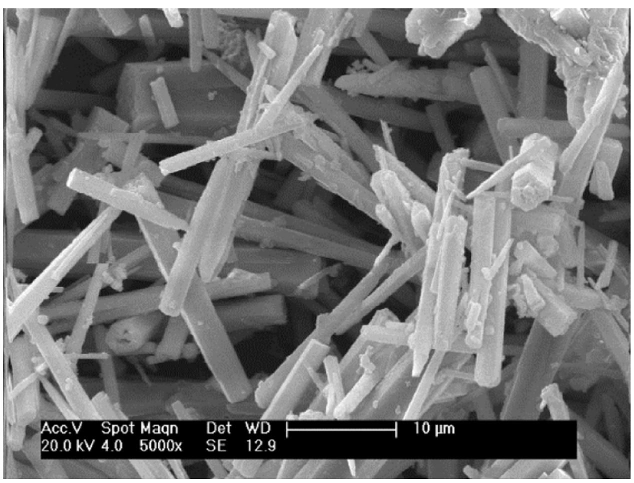

Fig. 2. The microscopic morphology of $CaCO_3$ whisker.

3. Results and discussions

3.1. Compressive strength

The compressive strengths of ECCs with different $CaCO_3$ whisker content are shown in Fig. 4. As can be seen, when 0.5% volume fraction of $CaCO_3$ whisker was added, ECC has the largest compressive strength of 30 MPa. It increases by 30% compared to that of D4-0 (without $CaCO_3$ whisker). This could be a result of the filler effect of $CaCO_3$ whisker in composite, which increases the compactness of matrix [13]. In addition, due to the small diameter and high aspect ratio of whisker, it can bridge flaws at microscopic level, which may delay micro-cracks from developing into macro-cracks, as shown in Fig. 5. However, when the $CaCO_3$ whisker content increases to 1% and 2%, a significant decrease in compressive strength can be observed comparing to D4-0.5. Firstly, the very low activity of $CaCO_3$ whisker causes the relative weaker strength of matrix when excessive $CaCO_3$ whiskers were added. Secondly, the poor dispersion and agglomeration of $CaCO_3$ whiskers when excessive content was added may result in new defects in composite and therefore reduces the compressive strength.

3.2. Tensile strain hardening behavior

The uniaxial tensile results of ECCs with different $CaCO_3$ whisker content are shown in Table 5 and Fig. 6. As can be seen from Table 5, the first cracking strength of ECC D4-0.5 is slightly larger than that of D4-0. However, subsequent increase of $CaCO_3$ whisker content leads to reduced first cracking strength, which is even smaller than that of D4-0. The first cracking strength of ECC is mainly governed by matrix strength. As mentioned in the previous section, the matrix compressive strength reached highest at whisker content of 0.5%, and reduced gradually with increasing whisker content. The results from matrix compressive strength are consistent with that from first cracking strength.

Table 3
Physical and mechanical properties of WW PVA fiber.

Diameter	Length	Density	Elongation	Elastic Modulus	Tenacity
35 μm	12 mm	1.3 g/cm^3	7.3%	31.3 GPa	1287 MPa

Table 4
Mix proportions of ECCs.

Mix No.	Cement	Fly ash	Sand	Water	HRWRA	PVA fiber (by volume)	$CaCO_3$ whisker (by volume)
D4-0	1	4.0	1.8	1.5	0.02	2%	0%
D4-0.5	1	4.0	1.8	1.5	0.02	2%	0.5%
D4-1	1	4.0	1.8	1.5	0.02	2%	1%
D4-2	1	4.0	1.8	1.5	0.02	2%	2%
D3-0.5	1	3.0	1.5	1.2	0.02	2%	0.5%
D2.2–0.5	1	2.2	1.2	0.96	0.02	2%	0.5%
D1.2–0.5	1	1.2	0.8	0.66	0.02	2%	0.5%

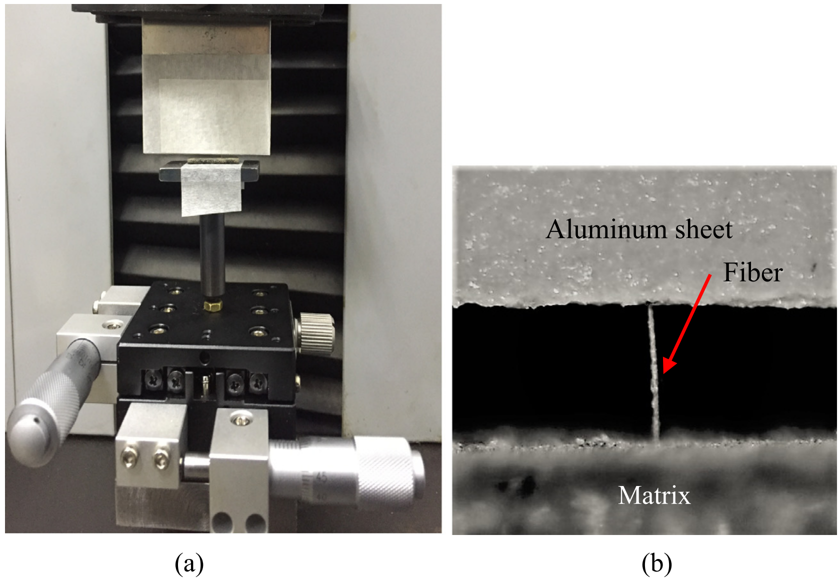

Fig. 3. Single fiber pullout test: (a) test setup; (b) close-up view.

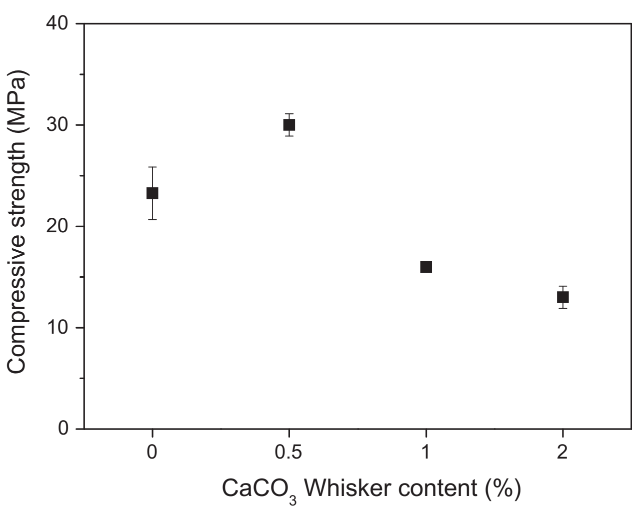

Fig. 4. Compressive strength of ECCs with different $CaCO_3$ whisker contents.

The ultimate tensile strength presents a similar trend with the first cracking strength, i.e. initial increase followed by subsequent decrease, as shown in Table 5. With $CaCO_3$ whisker content at 0.5%, the ultimate tensile strength of ECC reaches 3.3 MPa, increasing by 53.5% compared to that of D4-0. The ultimate tensile strength is governed by the fiber bridging capacity σ_0 of the weakest cross-section in ECC specimen. The fiber bridging capacity σ_0 can be calculated in Eq. (1), which ignores fiber rupture, slip-hardening, and snubbing effect for simplicity [20].

$$\sigma_0 = \frac{4V_f\tau_0}{L_f d_f}\left(\frac{L_f}{2}\right)^2 \cdot \eta_B \tag{1}$$

where V_f is the volume content of fiber; τ_0 is the frictional bond strength of interface between fiber and matrix; L_f and d_f is the length and diameter of fiber, respectively; η_B is defined as the efficiency of fiber bridging [21], and the value of η_B is $2/\pi$ and 1/2 in the case of 2D and 3D distribution of fiber, respectively [22].

As can be seen in Eq. (1), the fiber bridging capacity σ_0 is governed by the frictional bond strength τ_0 for a given fiber type and volume fraction. According to the results of single fiber pullout test (Fig. 7), the frictional bond strength τ_0 is highest for the composite with 0.5% $CaCO_3$ whisker, which agrees well with the trend of ultimate tensile strength of ECCs with different $CaCO_3$ whisker content.

The frictional bond strength is directly related to roughness and compactness of fiber/matrix interface [23]. Incorporating a proper amount (0.5% by volume) of $CaCO_3$ whisker can increase the roughness of interface and frictional bond. This will subsequently increase fiber bridging capacity and ultimate tensile strength. Sim-

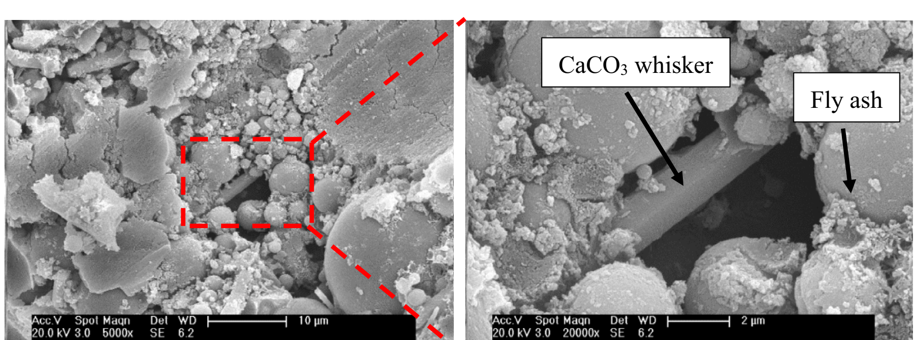

Fig. 5. $CaCO_3$ whisker bridging the micro flaw in composite.

Table 5
Tensile test results of ECCs with different $CaCO_3$ whisker contents.

Mix No.	First cracking strength (MPa)	Ultimate tensile strength (MPa)	Tensile strain capacity (%)	Number of crack
D4-0	1.64 ± 0.42	2.15 ± 0.37	2.95 ± 1.06	31 ± 11
D4-0.5	2.43 ± 0.27	3.29 ± 0.10	6.30 ± 0.30	67 ± 9
D4-1	1.41 ± 0.06	2.34 ± 0.15	6.76 ± 0.51	64 ± 7
D4-2	1.32 ± 0.10	2.15 ± 0.05	6.77 ± 0.11	63 ± 5

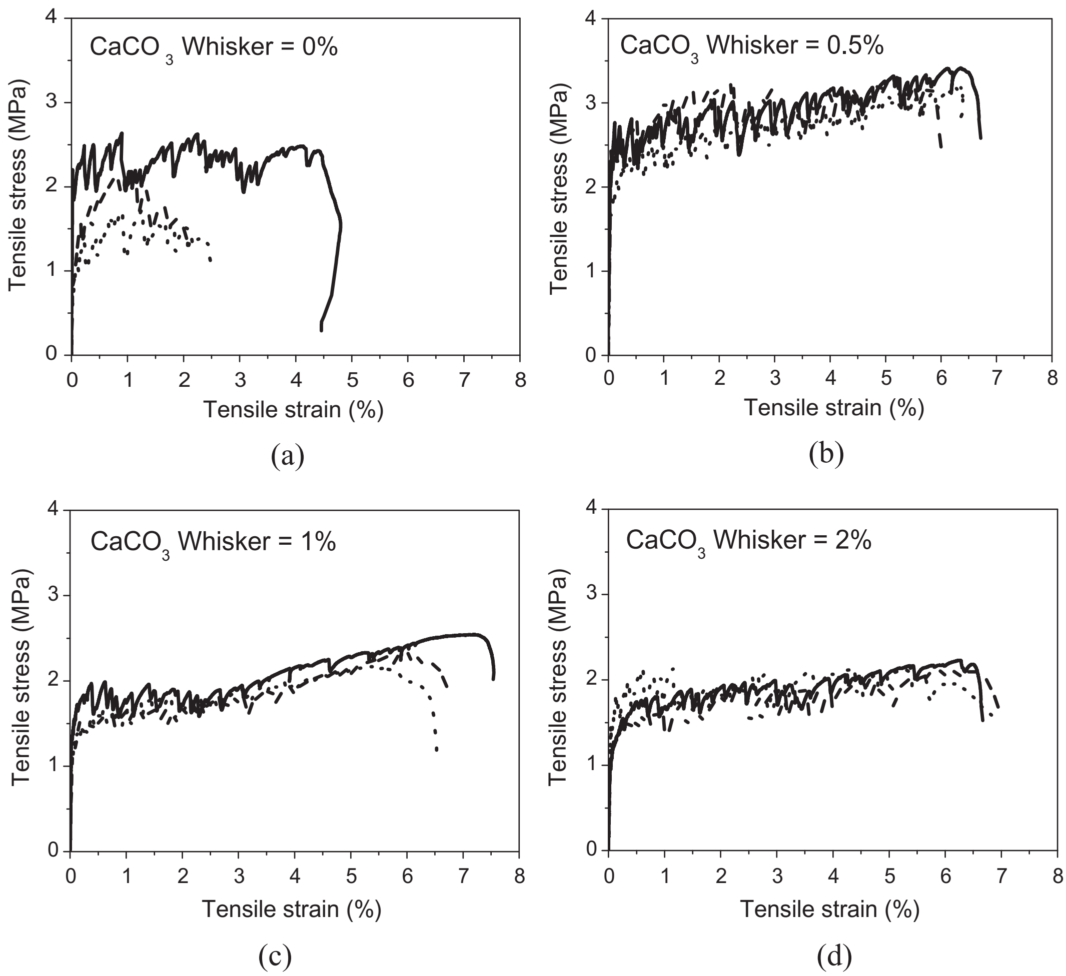

Fig. 6. Typical tensile curves of ECCs with different $CaCO_3$ whisker contents: (a) 0%; (b) 0.5%; (c) 1% and (d) 2%.

ilarly, the filler effect of whisker [13] can also increase the density/-compactness of interface, which results in increased frictional bond strength and eventually ultimate tensile strength. However, excessive whiskers tend to agglomerate, which increases pore in the ITZ instead of making it more compact. Therefore, the ultimate tensile strength reduces with increasing whisker content after 0.5%.

As can be seen in Fig. 6, incorporating $CaCO_3$ whisker improves the tensile strain capacity of ECCs significantly. When 0.5% $CaCO_3$ whisker is incorporated, the tensile strain capacity increases by 114% compared to that of D4-0 (from 2.95% to 6.30%). With further increase in $CaCO_3$ whisker content, the tensile strain capacity increases slightly and stabilizes at around 6.7%.

As is typical of single fiber pull-out studies, the data show high variability (Fig. 7). Even so, the averaged chemical bond G_d of fiber/matrix interface shows a significantly reduction when $CaCO_3$ whisker is added. The $CaCO_3$ whiskers attached to the surface of PVA fiber can potentially reduce the contact area of PVA fiber

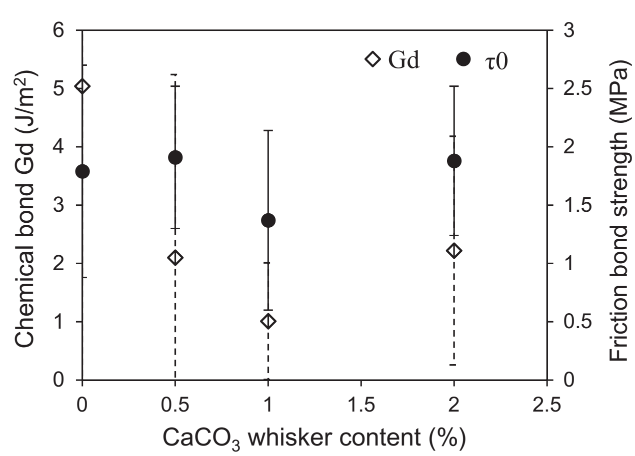

Fig. 7. Chemical bond G_d and frictional bond strength τ_0 at different $CaCO_3$ whisker contents.

and cementitious matrix (as shown in Fig. 8), and subsequently reduce the metal cation Al^{3+} and Ca^{2+} concentration on the interface which governs the chemical bond G_d [5,23]. Fig. 7 shows a slight increase in friction bond for the case with 0.5% $CaCO_3$ whisker, but the error bar is too large for this to be conclusive.

According to the ECC design theory, the energy criterion, as shown in Eq. (2) [5], should be satisfied for tensile strain hardening behavior.

$$J_{tip} \leqslant \sigma_0\delta_0 - \int_0^{\delta_0} \sigma(\delta)d\delta \equiv J_b' \quad (2)$$

where J_{tip} is the crack tip toughness of matrix, J_b' is the complementary energy, σ_0 is the maximum fiber-bridging strength, and the corresponding crack opening is δ_0. In the presence of a smaller chemical bond G_d, interface de-bonding can initiate at a lower tensile load leading to a higher complementary energy J_b' [24]. Thus, it enlarges the margin for energy criterion, and enhances the tensile strain capacity. In addition, due to acicular morphology of $CaCO_3$ whisker (Fig. 2), it can increase the roughness of interface (Fig. 5) and ultimately the frictional bond strength as they are closely related [23]. This can further enlarge the margin of energy criterion and improve the tensile strain capacity.

3.3. Variability of material properties

In order to investigate the influence of $CaCO_3$ whisker on robustness of ECC mixture, a coefficient of variation (CV) of energy dissipation capacity under tension is defined:

$$CV = \frac{\sqrt{\frac{\sum_{i=1}^{n}(S_i-\bar{S})^2}{n-1}}}{\bar{S}} \times 100\% \quad (3)$$

where S_i is the integral area of one tensile stress-strain curve with abscissa, as shown in Fig. 9, which represents the energy dissipation before failure; $\bar{S}$ is the average value of one batch of ECC mixtures; n is the specimen number of each batch, which is three in this study.

The CV of energy dissipation capacity of different ECC mixtures are shown in Fig. 10. The ECCs incorporating $CaCO_3$ whisker have much smaller variability than the ECC without whisker. The CVs of D4-0.5, D4-1 and D4-2 reduced by 90%, 79% and 97% compared to that of D4-0. It indicates that incorporating $CaCO_3$ whisker can reduce the variability of ECC significantly, which is beneficial to the mass production and application of ECC materials. As mentioned above, incorporating $CaCO_3$ whisker in ECC reduces chemical bond and increases frictional bond strength, which results in the increasing of complementary energy J_b' and fiber bridging capacity σ_0. Therefore, the strain hardening criteria, i.e. strength criterion and energy criterion, can be met with a wider margin. This is likely a reason for the reduction of the variability of the tensile behavior of ECC mixtures incorporating $CaCO_3$ whisker [25].

Besides the CV of energy dissipation capacity, the variability of compressive strength and first cracking strength were also investigated in this study. Fig. 11 shows the CVs of compressive strength and first cracking strength with different $CaCO_3$ whisker contents. The CV of first cracking strength reduces significantly when adding 0.5% $CaCO_3$ whisker. However, it increases gradually with higher content of $CaCO_3$ whisker. The CV of compressive strength shows the same tendency. It is very likely due to the agglomeration of $CaCO_3$ whisker in composite when excessive content was added. It also results in the reduction of compressive strength and first cracking strength when excessive $CaCO_3$ whisker was added, as mentioned previously.

3.4. Combined effect of $CaCO_3$ whisker and fly ash

Based on previous studies on the influence of $CaCO_3$ whisker content on mechanical properties (compression and tension) of ECC with local ingredients, it appears that incorporating $CaCO_3$ whisker can improve the tensile strain hardening behavior, espe-

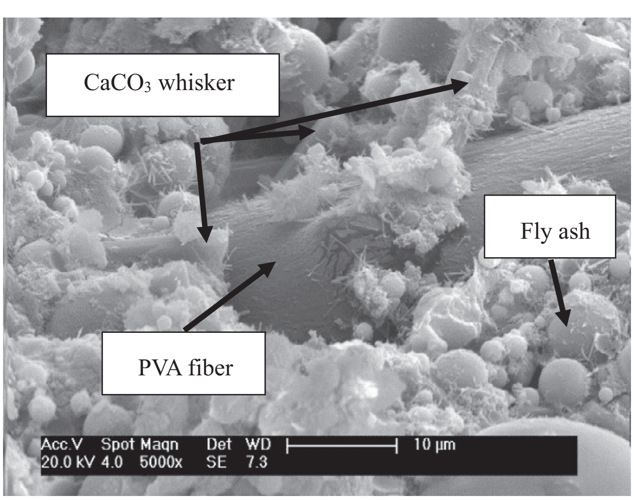

Fig. 8. Microstructure of PVA fiber/matrix interface with $CaCO_3$ whisker.

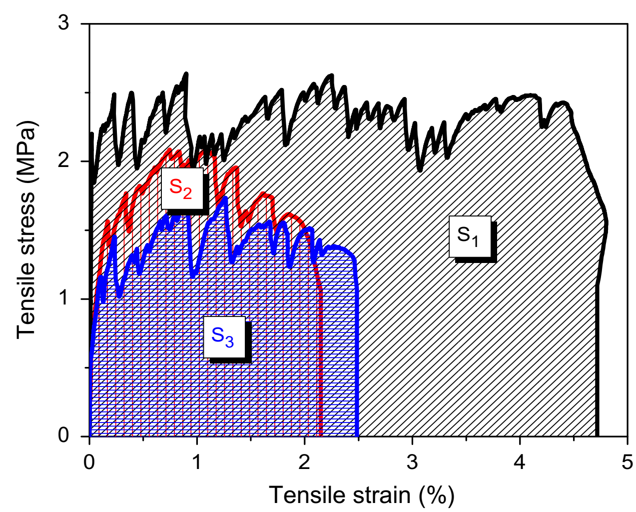

Fig. 9. The schematic diagram of integral area S_i.

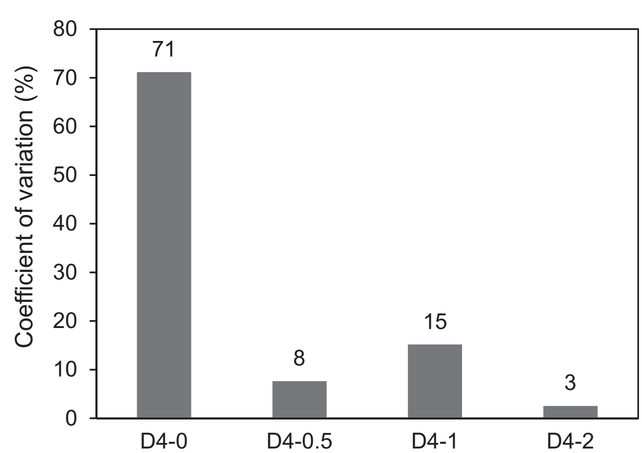

Fig. 10. Coefficient of variation of energy dissipation capacity of different ECCs.

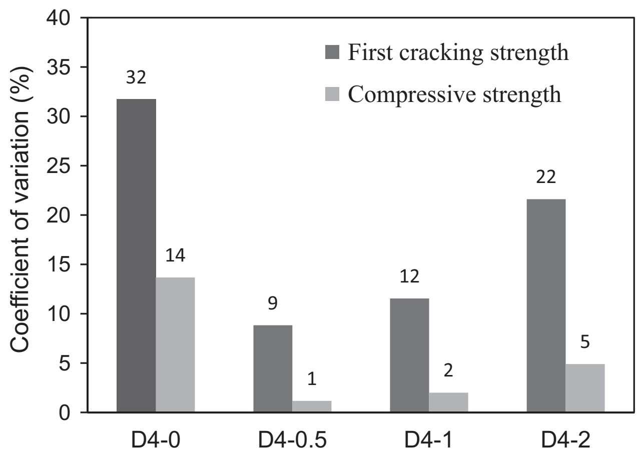

Fig. 11. Coefficients of variation of first cracking strength and compressive strength.

cially tensile strain capacity. The optimal content of $CaCO_3$ whisker is 0.5% by total mixture volume. In this section, with $CaCO_3$ whisker content kept constant at 0.5%, the influence of fly ash content on ECCs' tensile property will be discussed.

As can be seen in Fig. 12, the tensile strain capacity of ECCs increases gradually with fly ash content increasing. With fly ash increasing (cement decreasing) the metal cations of Al^{3+} and Ca^{2+} on the interface between fiber and matrix decrease [5,23,26], which results in the decreasing of chemical bond G_d and further increases the complementary energy J_b'. Therefore the tensile strain capacity increases with increased fly ash content.

On the other hand, the fly ash content has almost no influence on ultimate tensile strength. The average ultimate tensile strength of D1.2-0.5 is 3.33 MPa, and that of D4-0.5 is 3.26 MPa. The ultimate tensile strength of ECC is governed by the frictional bond strength which is in turn determined by the roughness and compactness of interface between fiber and matrix. Therefore, the results in this study indicate that the interface roughness and compactness were significantly affected by $CaCO_3$ whisker, and fly ash content has minimal influence on frictional bond strength.

4. Conclusions

This paper focuses on using $CaCO_3$ whisker to modify ECC with local ingredients and PVA fiber in China. The compressive strength and uniaxial tensile behavior of ECCs were evaluated, and the optimal content of $CaCO_3$ whisker was determined. The combined effect of $CaCO_3$ whisker and fly ash was also investigated. The specific conclusions can be drawn as follows:

(1) Incorporating optimal content of $CaCO_3$ whisker can improve the tensile strain capacity of ECC significantly plausibly due to the reduction of chemical bond of fiber/matrix interface. In addition, acicular morphology of $CaCO_3$ whisker can increase the roughness of interface and therefore the frictional bond strength. This can further enlarge the margin of strain hardening criteria and improve the tensile strain capacity.

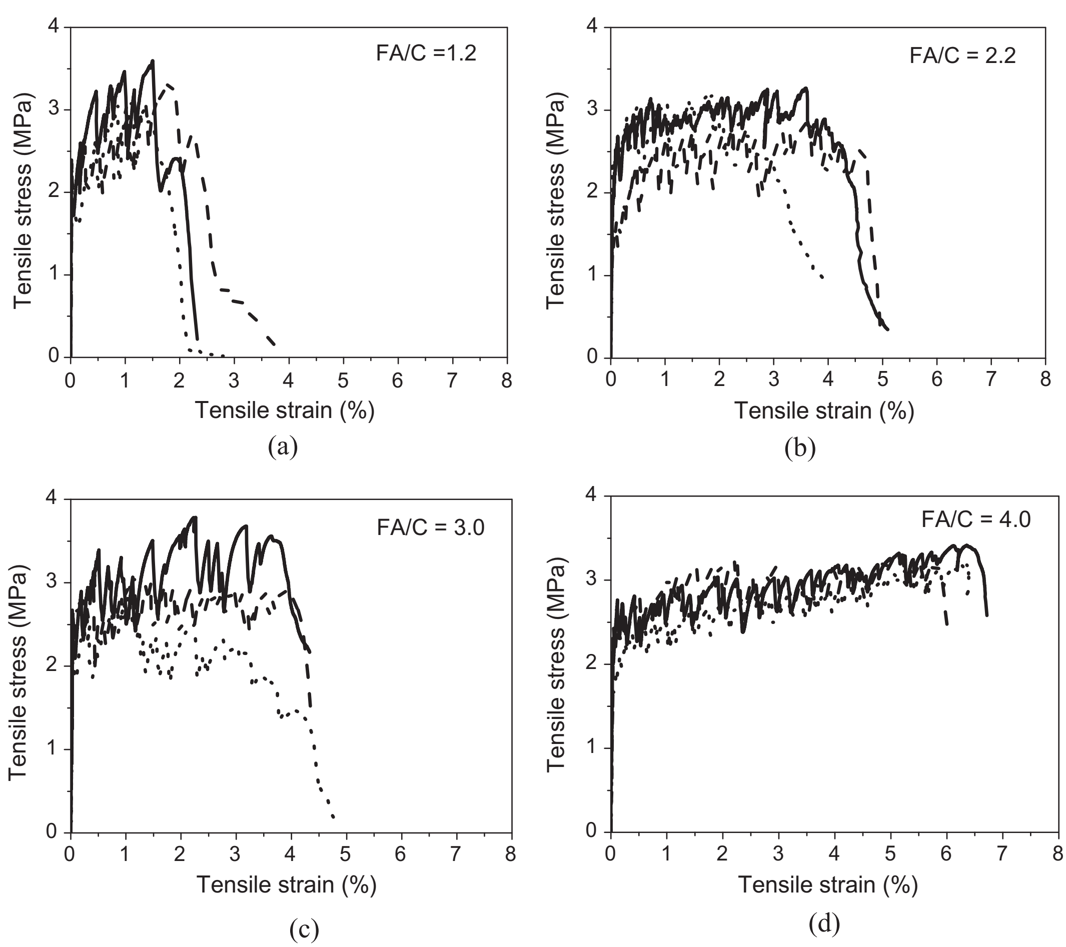

Fig. 12. Typical tensile curves of ECCs with different fly ash content: (a) FA/C = 1.2; (b) FA/C = 2.2; (c) FA/C = 3.0; (d) FA/C = 4.0.

(2) The optimal content of $CaCO_3$ whisker is 0.5% by volume of total ECC mixture. With this content, the ECC D4-0.5 shows a compressive strength of 30 MPa, ultimate tensile strength of 3.3 MPa and tensile strain capacity of 6.3%.

(3) Incorporating $CaCO_3$ whisker also improve the robustness of ECCs' mechanical properties significantly. When optimal $CaCO_3$ whisker was used, the coefficients of variation of ECC's properties reduce drastically compared to that of the ECC without $CaCO_3$ whisker (D4-0). This is likely due to a wider margin in complementary energy for satisfying the strain hardening criteria.

(4) When $CaCO_3$ whisker was added, the frictional bond strength between fiber and matrix was significantly affected by whisker. Therefore, the ultimate tensile strength of ECC shows insignificant change for different fly ash content.

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