Strain Resilient Cementitious Composites of Unclassified Calcareous Fly Ash and PP Fibers: Performance by also Considering Durability Effects

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Abstract. Strain Resilient Cementitious Composites (SRCC) is a class of ECCs, following the design philosophy in terms of grain fineness of mixing materials and the high dosage of plastic fibers, but differentiating in the tensile response: after first cracking they develop a wide parabolic response sustaining adequate tensile strength up to high levels of strain. The material's deformation is concentrated near a limited number of cracks emanating from the anchorage conditions of the embedded fibers. In the case of SRCC made by the less studied calcareous fly ash (of low pozzolanic ingredients and high lime) and polypropylene (PP) hydrophobic fibers, early studies have shown that strong chemical bond links are developed in the fiber-binder interface as the material hardens with time. This time-dependent effect alters the mechanical behavior of fibers from partial pullout at early age to elongation at advance time; the latter mechanism promotes the occurrence of early crack stabilization with the development of few cracks which sustain the tensile resistance due to the sufficient anchorage length of the fibers. This paper presents experimental results for the mechanical characterization of four SRCCs with parameters the Greek calcareous fly ash and the PP fiber contents at advanced age. In total 120 specimens were tested in compression (cubes) and tension (3-point bending prisms and 4-point bending beams for definition of material's tensile strength/fracture energy and deformation capacity respectively) by also considering assessment of materials' durability in regards to the impact of hearing and freeze-thaw cycling.

Keywords: SRCC \cdot Mechanical response \cdot Temperature \cdot Freeze-thaw \cdot Testing age

1 Introduction

The incorporation of engineered cementitious composites (ECC, Li et al. 1993) in reinforced concrete structures attaches multiple benefits namely (i) resistance to environmental/corrosive attack due to the restrain for cracking initiation and growth (Flores Medina et al. 2014, Banthia and Gupta 2006), (ii) reduction of the extensive

amount of transverse reinforcement in strategic regions of the structure since the ECCs are able to carry tensile stresses at high strains comparable to those undertaken by the reinforcement (Zhang et al. 2015) or even (iii) increase of the strain development capacity of the reinforcement anchorages (Tastani et al. 2016) by favorably affecting the descending branch of the bond-slip law. ECC materials incorporated polypropylene fibers (Zhang et al. 2015, Tastani et al. 2016) show a stress – strain response where up to first cracking the material behaves elastically, then exhibits mild hardening up to peak followed by mild softening, both in a wide range of resilient deformation up to 15% loss of strength, associated with limited cracking. This kind of ECC response is referred to hereon as "strain-resilient" and it is deemed as a tensile characteristic of the PP-ECCs.

2 Experimental Program

Materials. The design of the present strain resilient cementitious composites (SRCC) was based on Tastani et al. (2016); unclassified calcareous fly ash of similar composition (Power plant, Prolemaida-Greece, see Tastani et al. 2016) and PP fibers were used in the mixes. This Greek fly ash has relatively low sum of the pozzolanic components (in the order of 50.5% as compared to higher than 70% of fly ash type-F) and high content of CaO and CaO_f (in total 55%). The result is a by-product with more hydraulic rather than pozzolanic behavior (poor production of late hydrates). The significant CaO_f may be responsible for the increase of hydration heat and along with SO_3 (4.7%) are causes of early age cracking (their hydration products have multiple volume thus generating internal expansion). For these reasons Greek fly ash is used in concrete industry as a small substitute of cement (up to 20%). However, in SRCCs the passive confinement provided by PP fibers in inhibiting or arresting cracking effectively controls the volumetric stability of the composite allowing increased contents of fly ash. A note of caution here is that by using high dosage of calcareous fly ash the resulting mortar is more compliant (lower modulus of elasticity) than the one made by cement due to the lower strength of $CaCO_3$ crystals as compared with the cement hydrates. Besides, the abundance of Ca(OH)₂ favors the self-healing behavior of the composite, by feeding the process of late CaCO₃ crystals creation. In the preset program two fly ash contents were studied: two (FA2) and three times (FA3) per weight of cement.

Two volumetric contents of polypropylene (PP) micro fibers (PP1.5 and PP2%) were studied in regards to their effect on both the compressive and tensile response. Fiber properties are: melting/ignition point 160–170 °C/570 °C, diameter $d_f = 25 \,\mu\text{m}$, length $\ell_f = 12 \,\text{mm}$, aspect ratio $\ell_f/d_f = 480$, fracture stress and strain $f_{fu} = 400 \,\text{MPa}/\epsilon_{fu} = 0.25$. A typical stress - strain response is depicted in Fig. 1a (manufacturer ThracePlastics, TMIX-12). Based on the chosen variables six compositions were studied (Table 1): four SRCC and two as a reference (without PP fibers). In all mixes cement type CEM I 52.5 N and dried fine silica sand (100% passing from 0.5 mm sieve) were used. The ratios of sand/cement and effective water/binder were kept constant, see Table 1.



Fig. 1. (a) Tensile stress-strain law of PP fiber (data from manufacturer). Fresh state of (b) FA2/FA3 mixes (flowable) and (c) FA2-PP2 (uniform dispersion of fibers).

The high range water reducer (WR in Table 1, SIKA VISCOCRETE-ULTRA 200) was calculated as per the weight of the binder (i.e. in FA2-PPi the binder ratio is C + FA2/C = 3 and in FA3-PPi is 4) and differentiated among mixes as a consequence of the mechanical requirements introduced both by the two PP contents (1.5 and 2%) and the binder. The mixing procedure was as follows: the solid particles (placing first the fly ash, then cement and last sand) were introduced in the mixer and mixed for 2 min. Then total water and WR that corresponds to the reference mix (i.e. for FA2-PP2, the WR is 1.33) were added. The result at this stage is a flowable mortar where the attraction forces between particles have been attenuated (Fig. 1b). Finally, the fibers and the

rest WR were slowly intro-	Table 1.	Mixes compos	sition. Note: w	* = W/(C + F.	A <i>i</i>).
duced (i.e. for	ID	Cem. FA/C	S/C W/C	w*	PP	WR
FA2-PP2, this is 5.22 ± 1.22	FA2				0.0	1.33
5.33 - 1.33 = 4%)	FA2-PP1.5	2.00	1.10		1.5	4.67
diminishing the	FA2-PP2	1.00	1 10	0.37	2.0	5.33
frictional forces	FA3	- 1.00	1.10	0.57	0.0	1.50
between fibers	FA3-PP1.5	3.00	1.47		1.5	5.00
and mortar and in	FA3-PP2				2.0	6.00
the fibers' effec- tive dispersion						

(Fig. 1c).

Specimens. The experimental program for the definition of the mechanical response of the SRCCs by also considering durability effects comprised 120 specimens: cubes (side 100 mm) were tested in compression, prisms ($40 \times 40 \times 160$ mm) in three point bending (clear span 140 mm) for definition of flexural strength and toughness and beams ($60 \times 70 \times 370$ mm) in four-point bending (clear span 340 mm, constant moment region 100 mm) for assessment of material's deformation capacity. For each mix and test setup three samples were casted (casting occurred without vibration). After 24 h specimens were placed in chamber with RH >95% and temperature 20 °C until the day of testing.

Cubes were tested in compression (a) after 28 days from casting, (b) after 34 freeze-thaw cycles and (c) at age that coincides with the freeze-thaw cycling termination.



Fig. 2. Test setup for (a) compression, (b) 4-point and (c) 3-point bending.

Freeze-thaw occurred as follows: it started on the 28th day from casting, specimens were immersed in tap water at ambient temperature for 24 h and then they placed in freezer at -20 °C for 24 h thus modelling winter conditions in northern Greece. During the freeze-thaw period materials' density was occasionally measured by using ultrasounds aiming to record any alteration of the internal structure. The low water-binder ratio (0.37) is responsible for a limited capillary pores network; considering that the water inside the saturated capillary pores and air voids only freezes below -12 °C whereas inside the gel pores below -78 °C (Yu et al. 2017), only the former will expected to cause freeze-thaw damage due to the volume increase by 9% when frozen.

Prisms were tested after 4 months from casting: they left in the chamber 28 days and the rest period at ambient conditions aiming to capture the least tensile strength development. Tastani et al. (2016) have reported that the fiber to matrix bond is a time-sensitive mechanism, also related to the cracking mode of SRCCs. Bond strengthens with time promoting fiber elongation instead of pullout with result the compromise of multi-cracking. Accompanying prisms were placed in the oven for 3 h at 200 °C (fibers melting point 160–170 °C) and then tested. Additional prisms were tested at the end of the freeze-thaw protocol (same procedure with cubes). Beams were tested in 4-point bending for assessment of material's strain ductility because this setup ensures region of constant moment as compared with the single cross section in the case of 3-point bending test. Conditioning and age of testing for these specimens were identical to prisms except of the heating duration (3 h) as a result of their larger size.

Density was measured by weighting cubes after 28 days from casting. Apparently, higher fiber and fly ash contents reduce the density: for the mixes with FA2 it ranges between 2.09–1.95 and with FA3 1.95–1.88 g/cm³ (bulk densities of the ingredients: cement $\rho_c = 3.1$, fly ash $\rho_{fa} = 2.5$, sand $\rho_s = 2.6$, fibers $\rho_f = 0.91$, in g/cm³).

Tests setup. Figure 2 portrays the equipment for measurement of length change (cubes: axial strain through LVDT, lateral strain through strain gages at the middle of opposite sides, bending deflections through LVDT and digital targets). In the following, curves of stress – strain and load – deflection are average of three samples each time.





Fig. 3. (a) Compression of FA*i* mixes and (b) appearance after failure. (c) Velocity variation versus freeze-thaw cycles for all six mixes.

Curves of axial stress-axial strain of the reference cubes (FAi. without fibers) tested after 28 days and 9 months from casting are depicted in Fig. 3a (index L denotes the later age). The increase of FA had a negative effect on the achieved strength (20% drop, compare FA2 and FA3 curves) and material compliance at both ages; however these indices were significantly improved at later age. Failure of the later tests was more abrupt (absence of descending branch in Fig. 3a). The response of the accompanied freeze-thaw tests is also depicted in Fig. 3a (by dashed lines. index frth). FA2-frth showed a slight drop of strength as compared to the significant deterioration in the case FA3-frth. Due to conditioning these specimens had developed surface micro-cracking promoting a premature and scaled mode of failure due to compression (Fig. 3b). The sensitivity of FA3 against freeze-thaw is also confirmed through the apparent reduction of the ultrasound velocity. Consistent to the mechanical response, velocity of the FA2 showed an imperceptible increase (Fig. 3c).

The compression responses of SRCC mixes with variable the fly ash (FA*i*, i = 2,3) are compared in Figs. 4(a, b). For the lower percentage of the PP fibers, the mixes with FA2 (grey curves, Fig. 4a) presented similar responses up to peak, both at later age and after freeze-thaw conditioning (strength in the order of 54 MPa, higher than the 40 MPa of 28th day). Conditioning impaired the post-strength behavior by weakening the fiber bond mechanism in effectively arresting the widening of multi-cracking. Mixes FA3 (black curves, Fig. 4a) showed an equivalent increase of strength with age (from 38 to 50 MPa), which however wasn't confirmed by the freeze-thaw tests



Fig. 4. SRCCs in compression. (Notes: (a) At the end of freeze-thaw, SRCC cubes with PP2 had intact external surface whereas with PP1.5 some microcracking. (b) Strength increase with time even in the case of conditioning (Fig. 4) was also confirmed by the increase of the ultrasound velocity in all SRCC (Fig. 3c).)

(their strength was similar to the 28th value). dav The response near strength, regardless the FA content and the age of testing, seems to be solely attributed to the fiber-to-matrix bond: the same bond strength was attained at higher fiber slip in the case of FA3 as compared with the FA2, thus placing the corresponding curve FA3-PP1.5 to the right of the FA2-PP1.5; this explains why at both ages these SRCCs had similar gain in strength but different compliance. At the post-strength stage where multicracking was widening, confinement provided by the fibers was controlling the volumetric expansion with result the coincidence of the descending branches regardless the FA content. For the mixes with 2% PP fibers (Fig. 4b) the conclusions differ: the initial response up to peak became stiffer with age and the strength gain was of similar magnitude (about 47%) for both FA contents. The strength of the FA2 is higher than FA3 by only 4 MPa at both ages. The freeze-thaw tests showed a reduction of strength by 10 MPa and 8 MPa for the FA2 and FA3 respectively as compared with the later age strength (compare FA2-PP2frth with FA2-PP2-L and FA3-PP2frth with FA3-PP2-L respectively). Overall, the increase of the FA content apparently reduced the SRCC strength (this is more pronounced in PP2 mixes) and increased the material compliance as a result of the higher lime in the composition.

Figures 4(c, d) depict the influence of fiber content on compression response. For the FA2 mixes at later age (the thicker curves, Fig. 4c) the fiber content didn't induce any change in strength and also in post-peak response. But the higher fiber percentage (PP2) seems to (a) delay the development of stronger links in the matrix, a process that affects material's compliance with time (the thinner and thicker black curves in Fig. 4c) and (b) negatively affect the material resistance to freeze-thaw conditioning (PP2 tests showed a strength reduction of 10 MPa among the late and the freeze-thaw tests, whereas the PP1.5 none). The FA3 mixes (Fig. 4d) were more sensitive to the fiber content by developing lower strength with increased PP at both ages. Conditioning lowered the strength to the same level of the FA2-PP2frth of Fig. 4c.

4 Mechanical Response - Tension

Three-point bending tests. All SRCC prisms were tested up to 50% loss of strength. The experimental measurements of applied load vs mid-deflection are presented in Fig. 5 where indices *frth* (blue curve) and *f* (red curve) correspond to freeze-thaw and elevated temperature conditioning respectively. The black curves refer to specimens that were tested at age of 4 months. All conditioned SRCCs developed more than two cracks at failure while those tested at later age only a single crack near the region of high moment. Exploring the mechanical response in regards to the first cracking load, the ultimate load and the attained deformation capacity up to 15% loss of strength it may be concluded that:

(a) load: In result to the lower content of 1.5PP fibers the first cracking load was similar to the peak load and also to the fracture load of the reference unreinforced matrices (green lines); in the case of the higher PP2 content, the first cracking load was significantly lower than strength. All SRCCs regardless the fly ash and PP contents developed similar tensile strength (1.5 kN). The accompanying heated specimens developed lower strength (FA3 mixes) and impaired stiffness up to peak load (FA2 mixes). Freeze-thaw tests showed an impressive improvement of strength without apparent first cracking stage. These specimens were fully saturated during conditioning (9 months) whereas those tested after 4 months from casting only a month.

(b) Deformation: Material deformability was solely affected by the fiber content by developing similar magnitudes per fiber content up to 15% loss of strength regardless the FA dosage. The more compliant structure of the FA3 resulted in slightly higher toughness (broader area under the curve) denoting that the fiber-to-matrix bond is controlled by fiber pullout rather than elongation (more pronounced in the case of PP1.5 mixes). Temperature didn't deteriorate the deformability; rather in PP2 mixes it changed the curve to a more ductile shape. Freeze-thaw cycling also enhanced this property, especially in the case of the FA3 mixes by altering the curve shape from parabolic to a ductile one (horizontal branch after attainment of peak load).

Four-point bending tests. Aiming to explore if the test setup alters the material response in tension (especially its deformation capacity), 4-point bending tests were performed on beam specimens cured in a same manner as the prisms (black curves, Fig. 6) by also considering temperature effects (red curves, Fig. 6). The similar strength of all SRCCs subjected in 3-point bending (1.5 kN) is not confirmed from the current series; higher PP content resulted in increase of strength in a range of 35-40%



Fig. 5. Plots of applied load - mid deflection of the 3-point bending prisms.



Fig. 6. Plots of applied load - deflection of 4-point bending beams.

regardless the FA dosage. In contrary to what has been already observed, the first cracking load depended on the FA rather than the fiber content (all FA2 mixes showed lower value than the ultimate while all FA3 similar to the ultimate). More deformable were the FA3 mixes with a slower descending branch than the FA2 mixes. Elevated temperature affected all mixes as per strength, more severely the FA2 mixes (contrary to what was observed from the FA2 prisms). In regards to the cracking mode of failure, all specimens developed a single crack in the constant moment region, while the heated specimens multiple cracking (apart from the FA2-PP2 mix) suggesting that temperature influenced mainly the FA3 mixes and secondly the bond mechanism of the less fiber-reinforced mixes. Temperature, as previously, altered the curves' shape to a more ductile manner.

5 Conclusions

An extensive experimental program was conducted in SRCCs (a class of ECCs) aiming to explore the influence of Greek, unclassified calcareous fly ash and the polypropylene fiber contents on the mechanical response (in compression and flexural tension). Effects considered were later age testing, elevated temperature above the fiber melting point and freeze-thaw cycling. From a first assessment of the results it is concluded that the use of high dosage by-product in combination with 2% PP fibers resulted to a durable concrete material with compressive strength appropriate for structural use, which at the same time it is benefited of increased tensile strength and deformation capacity. All cracking-induced agents of FA are diminished due to restrain provided by PP mass reinforcement. Some degradation occurred due to elevated temperatures above the fiber melting point.

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