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Effect of graphene embedment on fibre-matrix interface and tensile properties of FRCM composites

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Abstract

This paper presents the results of an experimental study to investigate the effects of using two types of graphene, dried graphene (DG) and hydrated graphene (HG), on enhancing the interfacial and tensile mechanical properties of fabric-reinforced cementitious matrix (FRCM) composites. It has been found that inclusion of both DG and HG can produce an improvement in the tensile strength of FRCM composites by increasing the tensile strength of the mortar paste and the amount of fibres participating in loadbearing due to increased penetration of mortar (cement hydrates) into the fibre bundle. The better dispersion of HG produces better results than DG. The maximum increases in overall tensile strengths of the FRCM composites with DG and HG are 18% and 31% respectively, with the majority of these improvements coming from the increase in the amount of fibres participating in loadbearing. Microstructure images indicate increases of up to 20% and 44% in mortar penetration thickness into fibre bundles by using DG and HG, respectively.

Keywords: Bonding mechanism; Graphene enhanced cementitious matrix; Interfacial bond behaviour; Pull-out test; Tensile test.

Introduction

Fabric-reinforced cementitious matrix (FRCM) composites, consisting of bundles of carbon fibres embedded in a cementitious matrix, have emerged as a promising method for strengthening reinforced concrete (RC) structures (Babaeidarabad et al. 2014; Ebead et al. 2017; Pino et al. 2017; Guo et al. 2020). Owing to their light weight-to-high strength ratio and good compatibility with the concrete substrate, FRCM composites can be used to restore RC structures to desirable mechanical and physical properties, while achieving enhanced resistance to aggressive environments (Pino et al. 2017; Raza and Qureshi 2021; Liu et al. 2022).

Fig.1 (a) shows the cross-section of a unit of a FRCM composite, consisting of fibre filaments and a cementitious matrix. Fig.1 (b) illustrates the load transfer mechanism within the FRCM composite when subjected to a tensile load. Under this loading condition, only the fibres directly bonded to the mortar (referred to as "sleeve filaments" in Fig.1 (a)) participate in load bearing. The other fibres (the "core filaments" in Fig.1 (a) and (b)) are not impregnated and their limited loadbearing resistance comes from friction between the core and sleeve filaments, via systematic slipping of the fibres in a load carrying mechanism referred to as "telescopic failure" (Banholzer 2004; Alexandre et al. 2023). However, it should be pointed out that in many practical applications, FRCM composites are attached to RC beams which are subjected to bending. In these cases, due to deflection of the beams, there would exist normal stresses at the FRCM/concrete interface as observed by Li et al. (1990) and Calabrese et al. (2020). The normal stresses force the structure to behave in a mixed fracture mechanics Mode I/II loading condition, and can also increase the interface bond resistance (Calabrese et al. 2020 & 2021). This effect is called snubbing-friction (Li et al. 1990; Fu and Lauke, 1997). Nevertheless, in both the aforementioned loading conditions (pure tension and bending), the strength of the FRCM composite is still mainly provided by the sleeve filaments (Silva et al. 2014; Ascione et al. 2015). This research focuses on the pure tensile loading condition, with the failure mode being fracture mechanics Mode II.

Since the tensile strength and fracture strain of carbon fibres are much greater than that of the mortar, the tensile strength of FRCM composite is dominated by the volume fraction of the loadbearing fibres in the FRCM composite, which is crucially a function of the thickness of the sleeve filaments. This thickness is referred to as the penetration depth (Ozturk and Chung 2021). Therefore, the most important factor in improving the tensile strength of FRCM composite is to increase the penetration thickness of mortar into the fibres.

In FRCM composites, the mechanism to engage fibres in loadbearing is by mortar penetration into fibre filaments. The dissolved cement ions are small enough to diffuse through the space between the fibre filaments, as illustrated in Fig. 2. However, when cement hydrates precipitate on the surface of the unhydrated cement particles, they block further contact between the unhydrated cement particles and water, preventing further dissolution of the unhydrated cement particles. The unhydrated cement particles are large and cannot penetrate into the fibre bundles. While the dissolved ions can move into the space, they are limited within the fibre bundles due to the low hydration degree of cement. Therefore, the penetration of cementitious mortar into fibre bundles is limited in conventional FRCM composites.

Without any measure, the volume of the core filaments is approximately 60-70% of the full bundle (Jesse 2004; Hartig et al. 2008; Aljewifi et al. 2010; Zhu et al. 2021), leaving only 30-40% of the fibres as sleeve filaments to participate in loadbearing. This is not materially and structurally efficient. Consequently, efforts are being made to find means to increase the volume of loadbearing fibres in FRCM composites.

One method is to include short fibres in the cementitious matrix (Zhang and Deng 2022). Short glass fibres could improve the bond between multifilament yarns and the surrounding matrix by means of new cross-links between the cementitious matrix and fabric yarn, and bridging effects that delay the development of cracks (Butler et al. 2011; Barhum and Mechtcherine 2012). However, using short fibres has a number of problems, including fibre concentration to form fibre balls, labour intensive mixing (Emdadi et al. 2015) and reduced workability (Boulekbache et al. 2010). Another method is to change the water/cement ratio to improve flowability of the cement paste to alleviate the aforementioned dissolution problem of large unhydrated cement particles (Peled et al. 2008). However, increasing the water/cement ratio of cementitious materials compromises their mechanical strengths. Although plasticizers are commonly used as an alternative to water to improve the fluidity of cement while preserving mechanical properties, it is important to recognize that this approach does not enhance the penetration thickness of cement into fibre bundles because this method does not reduce the sizes of hydrate particles.

Another alternative method to enhance bonding between the cementitious matrix and the fibre bundles in FRCM composites is silica suspension coating on the fibre yarn (Guo et al. 2022). The absorption of active silica on the fibre yarn surface promotes the formation of C-(A)-S-H and leading to an increase in the composite's properties. However, the complexity of silica suspension treatment hinders its application as a construction material. Similarly, plasma modification (Zhao et al. 2020) or the employment of a peeling process to improve fabric adhesion to the cementitious matrix (Rambo et al. 2021) are only applicable to highly demanding applications where cost is not a key governing issue.

Among the various existing methods, epoxy impregnation of the fabric (Xu et al. 2004; Donnini et al. 2016) is the most feasible. Epoxy coating is an effective method for enhancing the mechanical properties of composites (Signorini et al. 2020). The low viscosity of epoxy allows a thin coating to be deposited on the fibres and improves the bond, thereby reducing the amount of resin and consequently high temperature sensitivity (Signorini et al. 2020). However, reducing the viscosity of epoxy resin often requires the addition of acetone (Signorini et al. 2020). This technique has a number of drawbacks when the material is used in construction, including the risk of fire, health problems associated with acetone usage, and cost. With regard to fire safety, research by the same group (Messori et al. 2019) demonstrated that the post-fire mechanical performance of the materials decreased drastically when the temperature exceeded 200 °C. In fire conditions, the material temperature would be much higher. Thus, a cement-based approach is preferred.

In summary, the penetration thickness of cement paste into fibre bundles in FRCM composites depends on many factors including filament size and spacing, the hydrophilic characteristics of the filaments, mortar flowability, and the size of the cement particles and their degree of hydration. Because of the many influential parameters and the complexity of their effects, effective enhancement of cement paste penetration into fibre bundles is challenging. All the existing methods have significant shortcomings and further research is needed to find easily implementable methods of increasing fibre participation in loadbearing without compromising the mechanical properties of the composite.

A more promising and simpler approach is to improve the degree of cement hydration, which can be enhanced by incorporation of graphene into cement, and this is the focus of the research reported in this paper. The emergence of graphene as a possible additive to construction materials has opened a door to solving the problem of limited bonding between cementitious matrix and fibre bundles. The benefits of graphene in cement mortar go beyond the commonly recognized benefits, such as improved mechanical properties (Du et al. 2016) and acting as bridging elements to delay the initiation and propagation of cracks at the nanoscale (Contamine et al. 2011; Santis et al. 2018; Mahmoudi et al. 2022).

Graphene, a two-dimensional material of carbon atoms, can act as nucleation sites to accelerate hydration of cement (Jing et al. 2017; Baomin and Shuang 2019; Ho et al. 2020; Lin and Du 2020). When graphene nanoparticles are dispersed in the cement paste, they act as nucleation seeding to stimulate the precipitation of cement hydrates on their surfaces away from the unhydrated cement particles, as illustrated in (Fig. 3). This process decreases the diffusion barrier of cement hydrates around the unhydrated cement particles (Thomas et al. 2009; Land and Stephan 2012; Artioli et al. 2014), thereby releasing more dissolved cement ions to the surrounding aqueous medium. The elevated concentration of ions allows a greater number of dissolved cement ions into the fibre bundles than without graphene, which in turn promotes the generation of hydration products within the fibre bundles and increases the penetration thickness of cement in fibre bundles.

Despite the abovementioned potentials of using graphene to enhance various aspects of the mechanical properties of cementitious matrix, as has been comprehensively reviewed by Yang et al. (2017), there is a lack of reported research in the literature that focuses on the fibre/mortar interface, necessitating the research to be reported in this paper. Whilst there have been numerous research studies on using nanoparticles to improve the mechanical properties of composites, such as (Signorini and Nobili 2021) who used carbon nanotubes and silica nano coating to improve the bonding behaviour at the interface, this research explores the effects of graphene as nucleation seedings to enhance the hydration of cement and the penetration thickness of cement into the fibre bundles of FRCM composites.

This paper will investigate the effects of two types of graphene (i.e., dry graphene (DG) and hydrated graphene (HG)). HG is an intermediate-product of DG and is supplied in paste form without drying, in contrast to DG which is supplied in dried form. Consequently, HG is less expensive and has a lower carbon footprint than DG. Furthermore, due to the greater number of free platelets, HG will facilitate SP adsorption more efficiently than DG.

Both the interfacial and tensile properties of FRCM are needed in the design of beams strengthened by FRCM, depending on the type of failure modes. As has been mentioned earlier, the focus of this research is the sleeve filaments in tension. Therefore, in this research, the FRCM composite specimens were subject to pure tensile loading. Therefore, further discussions of this paper will only consider fracture mechanics Mode II behaviour.

The specific objectives of this research are to:

(a) Demonstrate the beneficial effects of using graphene to increase the mechanical properties of FRCM composites and quantify the increases;

(b) Demonstrate the effects of using graphene to increase the penetration thickness of cement mortar into the fibre bundles of FRCM composites and quantify the penetration thickness;

(c) Quantify the increase in mechanical properties of cement mortar due to using graphene;

(d) Quantify the increase in mechanical properties of the participating fibre bundles.

Materials and experimental methodology

To meet the above objectives, tensile mechanical tests were carried out on cement mortar, the interface between mortar and fibres as well as FRCM composites. Scanning electron microscopy (SEM) image analysis was also performed to support understanding. These tests will be described in detail later.

Material components

In total, the test specimens include the following material components: cementitious matrix (mortar), graphene and carbon fibres. This section provides their properties.

Cementitious matrix (mortar)

The cementitious matrix used in this research was made of Portland cement (PC), superplasticizer, water, and river sand. The Portland cement was CEM2 42.5N with a specific surface area of 362 m^2/kg, and its chemical composition is provided in Table 1.

The river sand with particle size less than 1.18 mm was washed to remove any ionic contaminants and dried in ambient air prior to mixing.

A polycarboxylate-based superplasticizer (SP) (ADVA 650) was used to enhance the workability of the cementitious matrix and the effectiveness of dispersion and stabilization of graphene in the mixing water. The ratio of PC: water: sand: SP was 1:0.4:1.4:0.01.

Graphene

Graphene used in this project was in two forms: DG and HG. Except for the drying process, both DG and HG platelets had undergone the same manufacturing process and hence they had the same physical dimensions as shown in Table 2. HG is an intermediate product of DG, consisting of 80% water and 20% DG, and is supplied as a paste form without drying. In contrast, DG is dried. HG is cheaper and more readily available than DG, which makes it advantageous in terms of lower cost and lower environmental impact. Moreover, HG has the potential to be better dispersed in FRCM composites because it can more easily facilitate the adsorption of superplasticizer due to a higher number of free platelets compared to DG.

Carbon fibres

The carbon fabric mesh (C-Mesh 84/84) used in this study was in bundle form. Fig. 4 shows the arrangement and dimensions of longitudinal and transverse fibres. Table 3 lists its geometrical and mechanical properties. The corresponding properties of the carbon fibre filament are also provided in Table 3 for reference.

Test specimens

Cementitious matrix (mortar)

A total of five mortar mixtures were investigated, consisting of the control mixture without graphene, and two dosages (0.035% and 0.07%) each of DG and HG types of graphene, as listed in Table 4. These two dosages were based on a previous preliminary study (Ho et al. 2020) that determined the optimal graphene dosage that would achieve the greatest increase in the mechanical properties of mortar. The tiny amount of graphene had no adverse effect on workability (Ho et al. 2020).

The graphene mortar specimens were made by adding graphene to water and the SP solution, followed by sonication using an RS Pro ultrasonic cleaner (200W) for 30 minutes, as described in (Dung et al. 2023). Afterwards, the graphene suspension was mixed with the dry mix of cement and sand for 2 minutes. As verified by UV-vis tests, this dispersion approach was satisfactory after 1 hour (Dung et al. 2023). The mixture was cast into dogbone-shaped moulds and consolidated on a vibrating table and finished with a trowel. The specimens were demoulded after 24 hours of casting, cured in lime water for 28 days prior to testing for mechanical strengths. The lime water was a saturated solution of calcium hydroxide to provide a highly alkaline environment. For each mix, a set of triplicate specimens were tested.

FRCM composite plate

Each FRCM composite specimen for tensile and pull-out testing consisted of two layers of cementitious matrix and one layer of carbon fabric, as shown in Fig. 5. The FRCM composite plates had a total thickness of 10 mm and planar dimensions of 300 mm × 500 mm. The specimens were prepared on a wooden formwork.

After the fresh cementitious mortar was prepared as described in the previous section, it was poured onto the wooden formwork to form a 5mm thick layer of cementitious mortar, and then the surface was levelled. Immediately afterwards, a layer of carbon fabric was placed on top of the mortar and stretched. Once this was done, the second 5mm thick layer of the same cementitious mortar was cast and levelled with a metal trowel. The FRCM plate was covered with a plastic wrap for 24 hours before demoulding. Afterwards, the FRCM plates were cured in lime water at ambient temperature (20 ± 2 ºC) for 28 days prior to cutting into specimens for pull-out and tensile tests. In order to minimize the effects of vibration on the specimens due to cutting, a high precision diamond blade (Premium Diamond Disc Cutter Blade PD300SEG-CS) from Evolution Power Tools, UK, was used. After cutting, no debonding at the fibre/mortar interface was observed, and neither was there any debonding between layers of mortar after mechanical testing.

The nominal dimensions of the pull-out test specimens were 250 mm × 30 mm × 10 mm to ensure three carbon bundles inside the cross-section. The nominal dimensions of the tensile test specimens were 500 mm × 50 mm × 10 mm so that they had five warp-carbon bundles inside the cross-section.

Since the fabric mesh had fibre bundles in two perpendicular directions, only the fibres in the direction of the applied load should be included when calculating the fibre reinforcement ratio

(Carloni et al. 2018).

Test Methodology

Tensile testing cementitious matrix

Direct tension tests were performed on dogbone-shaped specimens (Fig. 6) with a cross-section of 15 mm × 30 mm (Kamal et al. 2008; Mahmoudi et al. 2022). Each specimen had two LVDTs with a 50 mm gauge length on both sides, as illustrated in Fig. 6. The direct tension test was conducted on a servo-hydraulic test frame with a loading capacity of 5 kN. Displacement control was used for the loading at 0.05 mm/min. Three nominally identical specimens were tested for each mix.

Pull-out tests

The pull-out tests followed the methodology described in (Zhu et al. 2021; Zhang and Deng 2022) and Fig. 7 shows the specimen dimensions. At the middle of the test specimen (i.e., cross-section B), all materials (i.e., mortar and fibre bundles) except the central fibre bundle were cut off. At cross-section A which is 70 mm away from Cross-section B, the central fibre bundle was cut off. This arrangement ensured that the central bundle of fibres would be loaded and pulled out from the upper part, as the embedded length in the upper part (70 mm) was shorter than that in the lower part (130 mm). The clamping parts at both ends were reinforced with FRP wraps to prevent the mortar from crushing. During each test, a linear potentiometer was used to measure the displacement of the specimen. Displacement control was used and the loading rate of the servo-hydraulic test frame was set at 0.2 mm/min. Again, three identical specimens were tested for each mix.

FRCM composite tensile tests

Fig. 8 shows the FRCM specimen dimensions, and Fig. 9 shows how the test was conducted using a clevis-grip system in accordance with AC434 (ICC 2013). At both ends, perforated steel tabs with dimensions of 200 mm × 50 mm × 3 mm were bonded to the mortar surface by epoxy resin to protect the specimen ends from crushing due to gripping. This test arrangement avoids lateral pressure being applied to the specimen and prevents the specimen from being subjected to bending (Contamine et al. 2011). Two linear potentiometers were attached to the two sides of the specimen to measure displacements. The load was applied by displacement control by a servo-hydraulic test frame with a maximum capacity of 10 kN (Fig. 9), and the loading rate was set at 0.2 mm/min. Three identical specimens were tested for each type of FRCM composite.

Microstructure Images

SEM images were taken of the mortar specimens, the pull-out specimens and the FRCM composite tensile test specimens. The SEM samples were 20-30 mm in diameter and were cleaned and stored in isopropanol to avoid further hydration before SEM imaging. To prepare the SEM analysis samples, the test material was vacuum-dried and the surfaces were polished, using sandpapers prior to final polishing with diamond suspensions (6, 3, and 1 μm). After polishing, the samples were cleaned twice with isopropanol in an ultrasonic bath, and then vacuumed for 24 hours. Prior to the SEM analysis, the polished samples were subjected to a compressed air jet to remove any dust from their surfaces before being mounted onto aluminium stubs using double-sided adhesive carbon discs and coated with gold by a Quorum Q150T ES Sputter coater. A Tescan Mira3 SC microscope was utilized for the SEM analysis with magnifications of 5000× and 1000×.

The measurement of penetration thickness was conducted by analysing the SEM images. A total of 60 random points were measured across a single cross section of the test specimens. After discounting the maximum and minimum values, the remaining measured values were used to calculate the statistical data for the penetration thickness. This methodology was used in prior research by others, such as (Aljewifi et al. 2010; Donnini et al. 2016 & 2017; Zamir et al. 2019).

Results and Discussions

Tensile strength and modulus of cementitious matrix

Table 5 lists the average (of three identical specimens) tensile strength and modulus of elasticity of the cementitious matrix. The stress-strain response of cement mortar under uniaxial direct tensile test can be characterized by a linear ascending curve up to the point of failure. Therefore, the tensile modulus of elasticity is determined by curve fitting to the middle third of the linear portion of the tensile stress-strain response curve, as illustrated in Fig. 10. The gradient of the line with the highest R^2 value is the tensile modulus of elasticity.

As expected, inclusion of graphene made some improvement to the mechanical properties of the cementitious matrix. The improvement in the tensile strength of cement mortar due to adding graphene is consistent and noticeable (18-29 % increase at 0.035 and 0.07 % graphene dosage). However, the changes in modulus of elasticity are small and variable (-22 % and 7 % at 0.035 % and 0.07 % graphene dosages, respectively). The limited improvement in Young's modulus due to graphene is because this property is measured at a low stress level, during which the mechanical properties of the cement mortar follow the mixture law of composites where the tiny dosage of graphene makes negligible difference. The inconsistency in modulus of elasticity is within the range of measurement error.

Results of penetration thickness

Fig. 11 shows images of the interfaces between fibre and cementitious matrix of different FRCM composite specimens. A cross-section of a test specimen was measured at 60 random points to determine the penetration thickness. The standard deviation values for the penetration thickness of CS, DG1, DG2, HG1, and HG2 specimens were 4.92 μm, 4.16 μm, 3.87 μm, 4.11 μm, and 3.92

μm, respectively. These results suggest a comparable accuracy level among the different specimens.

Fig. 12 depicts the effects of graphene on increasing the penetration thickness of cementitious matrix into carbon fibre bundles. Both types of graphene increased the penetration thickness but the improved dispersion of HG gave better results than DG (Dung et al. 2023). The percentage increases in penetration thickness are 6% and 20% for 0.035% and 0.07% DG graphene dosage; 35% and 44% for 0.035% and 0.07% HG graphene dosage.

From the penetration thickness values, the volume fractions of fibre in the sleeve filaments of the FRCM composites (i.e., the ratio of sleeve fibre area and total fibre area) can be calculated to be 29%, 32% & 34%, 40% & 43% for CS, DG with 0.035% graphene dosage & 0.07% graphene dosages, and HG with 0.035% graphene dosage & 0.07% graphene dosages respectively.

Pull-out test results

For the pull-out tests, two failure modes are possible: (a) fibre bundle pull out, and (b) fracture of fibres in the sleeve filaments.

For failure mode (a), the pull-out stress can be calculated using the following equation:

$$\sigma_{interface} = \frac{P_{pull-out}}{L_e C_f} \tag{1}$$

where $P_{pull\text{-}out}$ is the peak pull-out force, L_e is the effective embedded length of the fibre fabric (i.e., 70 mm) and C_f is the perimeter of the embedded fabric bundle.

For failure mode (b), the peak load at failure ($P_{pull\text{-}out,u}$) is calculated according to Eq. (2) because only the sleeve filaments are fractured.

$$P_{pull-out,u} = \varepsilon_f E_f A_{fs} \tag{2}$$

in which A_{fs} is the cross-section area of the sleeve filaments in the pull-out specimens, which can be determined from the penetration thickness based on SEM images; E_f is the Elastic modulus of carbon fibre; and ε_f is the fracture strain of the fibres.

Fig. 13 shows the typical failure mode of the pull-out test specimens. Due to high bond strength, the observed failure mode in all the tests was fracture of the sleeve filaments and slippage between core and sleeve filaments (Fig. 13). Therefore, the peak loads in the pull-out tests represent the tensile strength of the sleeve filaments instead of interfacial shear stress.

Fig. 14 presents the experimental load-displacement curves for all pull-out tests. They show three stages of behaviour as illustrated in Fig. 15: Initially, the load-displacement curves exhibit a nearly linear ascending trend, which indicates elastic behaviour, including undamaged and elastic bond between the fibre bundle and the mortar matrix. During this stage, all the filaments remain intact. As the curve becomes non-linear, it marks debonding of the fibres from the mortar in the sleeve filaments, which continues up to the point where the peak load (P_{max}) is reached. During this stage, sleeve filament ruptures occur. Therefore, the peak load is critically affected by the mortar penetration thickness, or the thickness of the sleeve filaments of the fibre bundle. The load drop thereafter is attributed to progressive fracture of the sleeve filaments. Finally, the curve reached a relatively stable plateau. At this stage, the resistance of the plate is provided by friction between the core filaments and the sleeve filaments. This unique failure is known as 'telescopic debonding'. The contribution of the frictional load provides an enhancement to the bond resistance, and helps to increase the ductility and residual adhesive resistance of the plate. Similar findings were reported by Cogen and Peled (2012); Liu et al. (2020); Focacci et al. (2022) and Alexandre et al. (2023).

Table 6 summarises the pull-out test results. It is important to consider the magnitude of the standard deviation compared to the mean value when assessing the significance of these differences. The differences in peak load values between the CS group and the DG1, DG2, HG1, and HG2 groups are significantly greater than their respective standard deviations, indicating statistical significance in the difference between these groups.

When compared with the CS, the ultimate loads of samples DG1 and DG2 increased by ~13% and ~20%, respectively, while for samples HG1 and HG2, the improvements are even more significant, at 17% and 30%, respectively. These improvements are consistent with the results of microstructure images observed in the previous section associated with increased penetration thickness of mortar into fibre bundles. The results can be seen in Fig. 16 which plots increases in strength of the interfacial bonding between carbon fibre bundles and cementitious matrix.

CS, DG, and HG specimens have standard deviations of 32 N (CS), 5 N (DG1), 15 N (DG2), 55 N (HG1), and 12 N (HG2). DG and HG specimens have smaller standard deviations than CS samples, which indicates consistent performance of graphene modified FRCM composites. In comparison, the approach used by (Donnini et al. 2016), which involved impregnation of fibres in epoxy resin at various levels, followed by application of a quartz sand layer to mitigate fibre slippage within the mortar matrix, produced inconsistent results. While some coated samples exhibited noticeable improved tensile strengths (e.g., 10 to 29% for transition tensile strength and 52 to 82% for ultimate tensile strength), some coated samples had notably lower tensile strengths than those of the corresponding untreated samples (e.g., -21% for transition tensile strength and -4% for ultimate tensile strength).

FRCM tensile test results

Three typical failure modes of the FRCM composites were found, including (I) failure at the ends of the gripping area; (II) breakage of fibres in the middle part together with cracks along the length; (III) slippage of fibre together with partial fibre fracture (see Fig. 17). Among the total of 15 test specimens, two failure modes were observed as shown in Fig. 17, including: (I) 2 cases of failure at the ends of the gripping area and (II) 13 cases of breakage of fibres in the middle part together with cracking along the length. Failure Mode I has no relevance to this research and its results were excluded from the subsequent further analysis. Although the failure mode does not change with the addition of graphene, there were more secondary cracks with smaller spacing between them along the length of the test specimens, as explained below.

For a FRCM composite coupon subjected to tensile loading, the first crack will occur when the stress applied to the matrix is equal to the matrix tensile strength. Once the first two cracks are fully open, the axial force is transferred from the gripped parts to the free part of the coupon by the sleeve filaments through the cracked cross sections. A new crack will form if the distance between two adjacent cracks is longer than a critical distance and when the maximum stress in the matrix equals the matrix tensile strength. This process continues until the distance between two cracks is smaller than the critical distance (Focacci et al. 2022). After the formation of sufficient cracks at increasing tensile stress, the bridging capacity of the sleeve filaments at one of the cracked sections is exceeded thereby causing failure of the specimen (Ranade et al. 2014). During the crack formation process, energy is dissipated when a new crack occurs. Owing to the benefit of graphene in enhancing the tensile strength of the matrix in graphene modified FRCM composites, an increased amount of energy is needed to break the matrix when forming a new crack.

Using graphene also increases the penetration thickness of mortar into the fibre bundle. As a result, the maximum force that can be transferred from the matrix to the fibres by the sleeve filaments is increased. The effect of graphene in increasing the penetration thickness is greater than the effect of graphene in increasing the dissipated energy in forming new cracks. Therefore, the critical distance between cracks becomes smaller, resulting in denser cracks in the graphene enhanced FRCM composite. Similar findings have been reported by others (Dvorkin et al. 2013; Trainor et al. 2013; Wu et al. 2021; Guo et al. 2022;) who investigated other means of increasing the penetration thickness. The increases in energy dissipation and penetration thickness of graphene modified FRCM composites are exhibited as larger areas under the stress-strain curves and increased deformation capacity, which was similarly reported by Focacci et al. (2020).

The detailed results in Fig. 18-19 can be used to quantitatively demonstrate the above-mentioned effects. Consider the average values in Fig. 18 of the three nominally identical tests. The first segment of the curves until point A is governed by the tensile properties of the mortar until mortar cracking. Using graphene increased the tensile strength of the mortar, and hence the average peak stresses at point A for the graphene modified samples (DG & HG) are higher than that of the control sample (CS). After mortar cracking, the stress drops during the transitional period for the applied stress in the mortar to transfer to the fibres in the sleeve, before it rises again as the fibres in the sleeve take up load. The higher average peak loads at point B of the curves for graphene modified samples (DG and HG) are a direct result of the increased penetration thickness of mortar in the fibre bundles. Furthermore, due to better dispersion of graphene in the HG sample than in the DG sample, the average peak stress of the HG sample is higher than that of the DG sample. After the peak stress at point B, all the test samples follow a trend of decreasing stress at increasing strain due to progressive breakage of the fibres in the sleeve. The general trend of the stress-strain curves follow that reported in (Donnini et al. 2016), but the key quantities are highly influenced by the presence of graphene. The tensile failure modes of the coupons are shown in Fig. 19.

The obtained results are consistent with the results of previous studies by others (Donnini et al. 2016; Santis et al. 2018), including: (a) a linear ascending part until Point A (the transition point) which marks the occurrence of first cracking of the cementitious matrix; and (b) a strain hardening part where the stress in the fibres keeps increasing until tensile fracture of the sleeve filaments as marked by point B (the ultimate point). Therefore, the load at point A includes contributions of both the mortar and the sleeve fibres at the same strain level as that of the fracture strain of the mortar, whereas the load at Point B is that of the sleeve fibres at fracture only. These two loads can be calculated as follows

For point A:

$$P_{FRCM,1} = \varepsilon_m E_m A_m + \varepsilon_m E_f A_{fs} \tag{3}$$

where A_m, E_m and ε_m are the cross-section area, the Elastic modulus and the fracture strains of the mortar in the FRCM composite, respectively.

For point B:

After cracking, the mortar matrix does not contribute to the resistance of the composite. Therefore, the ultimate load of the FRCM composite comprises of two parts: the tensile strength of the sleeve filaments and the friction force provided by the core filaments. Since the friction force is small, it is reasonable to take the tensile strength of the sleeve filaments as the load carrying capacity of the FRCM composite, as expressed in Eq. (4).

$$P_{FRCM,2} = \varepsilon_f E_f A_{fs} \tag{4}$$

Fig. 20 (a) and (b) show the effects of graphene content on tensile resistances at the transition point (point A in Fig. 18) and the ultimate point (point B in Fig. 18), respectively. The transition point resistance of the FRCM composite specimens is enhanced by 14 to 18% and 17 to 19% when adding DG and HG, respectively, and the ultimate tensile resistances are increased by 3 to 14% and 26 to 31%, respectively.

The observed increases in transition and tensile strengths of FRCM composites can be attributed to graphene's role as a nucleation seeding (Dung et al. 2023). First, graphene facilitated cement hydration, leading to an improvement in cementitious matrix strength (Table 2). Second, this improved hydration process resulted in elevated concentrations of dissolved cement ions, which promoted the ingress of more dissolved cement ions into the fibre bundles (Fig. 11), thereby further strengthening the transition and tensile strengths of the FRCM composites.

The dispersion of HG within the cementitious matrix was better than that of DG graphene (Dung et al. 2023), leading to better performance of the FRCM composites incorporating HG than those incorporating DG.

The improvement in penetration thickness by using the proposed method of adding graphene in this research ranges from 6% to 44%, leading to 13% to 30% increases in interfacial bonding resistances obtained from pull-out tests. Similar improvements in resistances have been achieved by others (these researchers did not measure the penetration thickness) including 19% and 33% improvements by Cohen and Peled (2012) who used 200-nm silica fume coated AR-glass composites produced by dry and wet processes respectively, 18% increase in the tensile strength of composites by Quadflieg et al. (2018) by using potassium silicate-coated AR-glass fabric, 32% increase by Signorini et al. (2019) who employed 150-nm micro silica coated uniaxial high-tenacity carbon fabric (with AR-glass yarns in the weft). However, for similar improvements, the graphene-based approach in this study is considered a promising method for practical applications.

The improvements in Fig. 20 (a) and (b) can be predicted using the mixture law equations for fibre composites, as expressed in Eq. 3 and Eq. 4 for the transition strength and the ultimate strength respectively, as confirmed in Fig. 21 (a) and (b) respectively, which show good agreement between the calculation results using Eq. 3 and Eq. 4 and the test results.

The improvement in the ultimate strength of graphene modified FRCM composites are almost entirely due to the increase in penetration thickness of mortar into fibre bundles enabled by graphene. This can be clearly seen in Fig. 22 which shows the relationship between the increases in the ultimate tensile strength with the increases in penetration thickness.

Fig. 23 shows the comparison of peak loads of each fibre bundle obtained from FRCM tensile tests and pull-out tests. As expected, the utilisation efficiency of fibres in pull-out specimens is generally higher than FRCM composites; this is because the stress distribution among the five bundles in FRCM composites is not uniform. Some of the fibre bundles contribute less than others.

Conclusions

This study has investigated the effects of using two different types of graphene on the mechanical properties of mortar matrix, pull-out test results for the fibre and mortar interfaces, and tensile test results of FRCM composites. The two types of graphene were Dry Graphene powder (DG) and Hydrated Graphene (HG) paste and their dosages were 0.035 and 0.07%. The main conclusions of this study are:

(a) Using DG and HG increased the tensile strength of cementitious matrix by up to 18 and 31%, respectively;

(b) Compared to the FRCM specimens without graphene, the penetration thickness of cement mortar into fibre filaments increased by 6% to 44% due to graphene inclusion.

(c) The increased penetration thickness almost accounts for the entire increase in both transition tensile strength and ultimate tensile strength of the FRCM composites. Increases of 6 - 20% and 35 - 44% in penetration thickness for DG and HG samples resulted in corresponding increases of 14 - 18% and 3 - 14% increases in transition and ultimate tensile strengths of the

DG specimens, and increases of 17 - 19%, and 26 - 31% increases in transition and ultimate tensile strengths of the HG specimens.

(d) The intermediate state graphene product HG appears to give better results than the dry powder state product DG. The intermediate HG state is also less costly. Therefore, HG should be preferred to DG.

Data Availability Statement

All data, models, or code that support the findings of this study are available from the corresponding author upon reasonable request.

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