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Effect of graphene oxide on mechanical properties of UHPC and analysis of micro-control mechanism

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Abstract

Recently, graphene oxide (GO) is one of the nanomaterials that enhance the performance of cement-based materials. In this paper, the effects of different mass of GO on the workability and mechanical properties of UHPC is studied, and its mechanism is explored by micro-nano analysis methods such as XRD, SEM, MIP and nano indentation. The results show that, compared with the reference group, the 0.04% GO addition reduces the fluidity of UHPC, reduces the initial and final setting time of slurry, and enhance the compressive strength, flexural strength and tensile strength of the sample at 28d by 15.8%, 14% and 15.3%, respectively. Micro-nano analysis results show that GO promotes cement hydration, reduces the porosity of UHPC, improves the microstructure of the interface transition zone between steel fiber and matrix, and improves the nano-mechanical properties. This is mainly attributed to the nucleation effect of GO and interfacial bonding with C-S-H gel.

1. Introduction

Ultra-high-performance concrete (UHPC) is favored because of its excellent mechanical properties, high ductility and good durability [1, 2]. Previous research results prove that the compressive strength of UHPC is about 3–16 times that of ordinary concrete, while the ductility and energy absorption of UHPC containing steel fiber is about 300 times that of high-performance concrete [3]. UHPC’s excellent durability enables extended service life and reduced maintenance costs because it is almost impermeable to carbon dioxide, chloride and sulfate [4]. Due to the ultra-high compressive strength, the weight of the UHPC structure under the same load is only 1/3 ~ 1/2 of the ordinary concrete structure, which is conducive to improving the height of the building and the utilization of ground space and reducing the construction cost. However, UHPC inevitably has certain weaknesses. Many researchers have confirmed that the water-binder ratio of UHPC is low, and the cementitious material required is about 800–1000 kg m$^{-3}$, resulting in some cementing materials that only play a filling role [5]. Unfortunately, the high cost and low utilization rate of its cementing materials limit its wide application.

With the development of science and technology, nanomaterials are widely used in various industries, such as information, environment, energy and environmental protection, biomedicine, etc. In recent years, great progress has been made in the application of nano-materials in cement-based materials, such as nano-silica [6], nano-calcium carbonate [7], carbon nanotubes [8] and graphene oxide (GO) [9], etc. GO of which is undoubtedly a new type of nano-material, showing excellent mechanical properties and good dispersion. The results declare that the mechanical properties and durability of cement-based materials can be significantly improved by adding appropriate amount of GO [10, 11]. Pan et al point out that the compressive strength and flexural strength of cement slurry with 0.05% GO nanosheets (by weight of cement) increased by 15%–33% and 41%–58%, respectively [12]. Lv et al obtain similar results in the study of cement mortar [13]. Wu et al report that when the content of GO is in the range of 0.02%–0.08%, the compressive strength, flexural strength and splitting tensile strength of ordinary concrete increase with the increase of GO content, but the slump of concrete implies the opposite trend. Ren [15] claims that the addition of GO improves the mechanical properties.
and resistance to chloride ion permeability of self-compacting concrete, but its fluidity decreases to some extent [16]. However, the application of GO in UHPC remains a significant challenge. In this paper, the effect of GO content on the workability and mechanical properties of UHPC is studied, including compressive strength, flexural strength and tensile strength. The phase composition and microstructure evolution of UHPC samples are analyzed by x-ray diffraction (XRD), scanning electron microscope (SEM) and mercury injection (MIP), and the effects of GO on the interfacial transition zone and nano-mechanical properties of UHPC steel fibers are investigated by nano-indentation technique.

2. Raw materials and test methods

2.1. Raw materials
Ordinary Portland cement P.O 52.5 meeting the requirements of GB/T175–2007 specification is selected, its chemical composition of cement and silica fume is shown in table 1. Using natural flake graphite as raw material, GO is synthesized by improved Hummers method [17]. The GO dispersion with a concentration of 10 mg ml$^{-1}$ is prepared by filtering while it is hot, centrifuging and washing for many times, adding a certain amount of deionized water and ultrasonic dispersion for 2 h. The carbon and oxygen contents of GO are 47% and 52%, respectively. As shown in figure 1, the microstructure of cement is mainly block and sheet, while silica fume is flocculent. Figure 2 provides a TEM image of GO, indicating the characteristics of wrinkles and folds. The sand adopts standard sand, the length of steel fiber is 13 mm, the superplasticizer is Subot’s superplasticizer (PCE), and the water is tap water.

2.2. Mix ratio
In order to study the effect of GO content on the mechanical properties and micro-nano structure of UHPC, the UHPC samples are mixed with 0%, 0.02%, 0.04% and 0.06% GO by weight of cement, respectively, which are expressed as UHPC-0, UHPC-2, UHPC-4 and UHPC-6. All mixtures have a water-binder ratio of 0.18 and a sand-binder ratio of 0.8. The GO nanosheets are dissolved in water and mixed into the UHPC mixture as a dispersion, and the mixing ratio of UHPC is shown in table 2. The GO suspension was ultrasonic for 30 minutes to ensure uniform dispersion of GO in water. The mix ratio of UHPC cement paste does not contain standard sand and steel fiber, and the others remain unchanged.

2.3. Testing method
The fluidity of UHPC and setting time of cement paste are determined by referring to GB/T 2419–2005 and GB/T1346–2000.
In order to test the mechanical properties of UHPC at 7d, 28d and 90d, samples with a size of $40 \times 40 \times 160$ mm are prepared. The phase composition of UHPC paste is determined by x-ray diffraction (XRD). The samples are scanned in the range of $10^\circ$ ∼ $90^\circ$ 2θ at a scanning rate of $10^\circ$/min. Scanning electron microscope (SEM) and mercury intrusion (MIP) were used to test the microstructure of the 28d sample. The nano-mechanical properties of the interfacial transition zone of UHPC steel fiber are tested by nano-indentation technique. To meet nanoindentation measurement requirements, the concrete samples were cutted, dried, inlaid in epoxy and carefully polished to achieve a smooth surface, as shown in figure 3. One representative area on the ITZ of the UHPC sample were characterized using nanoindentation. The distance between different indents in the samples was set to $5 \mu$m in the lateral and vertical directions. The loading time is 5 s, the holding time is 2 s, and the unloading time is 5 s. The elastic modulus

![Figure 2. TEM and SEM images of GO.](image)

| Table 2. UHPC mixture proportions. |
|-----------------------------------|
| Sample  | w/b | Cement | SF | Sand | Water | GO | Steel fibre | PCE |
|------ |----- |-------- |--- |------ |------ |--- |------------- |---- |
| UHPC-0 | 0.18 | 1.0     | 0.1 | 0.8  | 0.198 | 0 | 0.1         | 0.03|
| UHPC-2 | 0.18 | 1.0     | 0.1 | 0.8  | 0.198 | 0.02% | 0.1       | 0.03|
| UHPC-4 | 0.18 | 1.0     | 0.1 | 0.8  | 0.198 | 0.04% | 0.1       | 0.03|
| UHPC-6 | 0.18 | 1.0     | 0.1 | 0.8  | 0.198 | 0.06% | 0.1       | 0.03|

In order to test the mechanical properties of UHPC at 7d, 28d and 90d, samples with a size of $40 \times 40 \times 160$ mm are prepared. The phase composition of UHPC paste is determined by x-ray diffraction (XRD). The sample is scanned in the range of $10^\circ$ ∼ $90^\circ$ 2θ at a scanning rate of $10^\circ$/min. Scanning electron microscope (SEM) and mercury intrusion (MIP) were used to test the microstructure of the 28d sample. The nano-mechanical properties of the interfacial transition zone of UHPC steel fiber are tested by nano-indentation technique. To meet nanoindentation measurement requirements, the concrete samples were cutted, dried, inlaid in epoxy and carefully polished to achieve a smooth surface, as shown in figure 3. One representative area on the ITZ of the UHPC sample were characterized using nanoindentation. The distance between different indents in the samples was set to $5 \mu$m in the lateral and vertical directions. The loading time is 5 s, the holding time is 2 s, and the unloading time is 5 s. The elastic modulus
The indentation point is calculated by equation (1).

\[ E = (1 - \nu^2) \times \left[ \frac{1}{E_r} - \frac{(1 - \nu^2)}{E_i} \right]^{-1} \]  

Wherein, \( E_i \) represents the Young’s modulus of the indenter, taking 1140 GPa; \( \nu_i \) represents the Poisson’s ratio, taking 0.07; \( \nu \) represents the Poisson’s ratio of the sample, taking 0.24.

Table 3. Fluidity and setting time of UHPC.

| Sample   | Fluidity, mm | The initial setting time, min | The final setting time, min |
|----------|--------------|------------------------------|-----------------------------|
| UHPC-0   | 266          | 465                          | 525                         |
| UHPC-2   | 254          | 450                          | 489                         |
| UHPC-4   | 250          | 435                          | 480                         |
| UHPC-6   | 235          | 470                          | 550                         |

Figure 3. Flow chart of nanoidentation specimen preparation.

Figure 4. The compressive strength and flexural strength of UHPC with different contents of GO.
3. Macro performance analysis

3.1. Workability

Table 3 provides the fluidity and setting time of UHPC. According to the table, with the increase of GO content, the fluidity of UHPC prompts a downward trend, in which the UHPC-6 sample decreased by 11.7% compared with the benchmark group. This may be due to the fact that GO has a larger specific surface area, which makes its surface absorb more free water, resulting in a decrease in the fluidity of mortar. The initial setting time and final setting time of UHPC decreased at first and then increased with the increase of GO content. When the content of GO is 0.04%, the initial setting time and final setting time of the sample are reduced by 30 min and 45 min respectively compared with the benchmark group. This is due to the nano-size effect of GO, which can provide nucleation sites for hydration product calcium hydroxide (\(\text{CH}\)), promote cement hydration and shorten setting time. However, with the further increase of GO content to 0.06%, the initial setting time and final setting time have increased, which may be due to the excessive GO dissolved in the paste to form flocculation adsorption on the surface of cement particles, resulting in inhibition of further hydration of cement particles and water.

3.2. Compressive strength

Figure 4 shows the effect of GO content on the compressive strength and flexural strength of UHPC. The figure shows that when the content of GO is less than 0.04%, the compressive strength of UHPC increases with the increase of GO content; when the content of GO is more than 0.04%, the compressive strength of UHPC shows a downward trend. Compared with the benchmark group, the compressive strength of UHPC-4 samples at 28d and 90d increased by 15.8% and 14.5%, respectively. The change trend of flexural strength is similar to that of compressive strength, and the flexural strength containing 0.04% GO UHPC is the highest. Compared with the benchmark group, the flexural strength of UHPC-4 samples increased by 14% and 13.5% at 28d and 90d, respectively, indicating that an appropriate amount of GO is beneficial to improve the mechanical properties of UHPC, which is consistent with the results of previous literature [18].

3.3. Tensile strength

Figure 5 shows the effect of GO content on the tensile strength of UHPC. The results prompt that the tensile strength of UHPC samples containing GO increases obviously, and the changing trend of tensile strength is similar to that of compressive strength. When the content of GO is 0.04%, the tensile strength of UHPC reaches the maximum; Compared with the benchmark group, the tensile strength of UHPC-2, UHPC-4 and UHPC-6 samples at 28d increased by 9.3%, 15.3% and 13.9%, respectively, suggesting that the tensile strength of UHPC is not proportional to the content of GO. The tensile strength of UHPC containing GO ranges from 7.55MPa to 8.7MPa, which is consistent with the strength of previous studies [19]. In general, the tensile strength of UHPC is closely related to the hydration degree of cement, the compressive strength of the matrix and the bond strength between the steel fiber and the matrix [5].

Figure 5. The tensile strength of UHPC with different contents of GO.
4. Micro-nano structural analysis

4.1. XRD analysis

Figure 6 provides a XRD diagram of the UHPC paste sample. The results imply that the phase composition of cement paste mainly consists of unhydrated cement clinker (CS), CH and AFt, and CS includes C3S and C2S. With the addition of GO, the characteristic peak value of CS decreases, which implies that GO promotes the hydration of cement. In addition, a small amount of AFt in the paste is mainly produced by the reaction of calcium aluminate and sulfate ion.

4.2. SEM analysis

The SEM images of the interface transition zone (ITZ) between the steel fiber and the matrix of the UHPC sample under different GO contents at 28 d is listed in figure 7. According to the figure, there are flaky or massive CH and a small number of microcracks near the ITZ of the reference group, as well as less gel content on the surface of the steel fiber (figure 7(a)). With the incorporation of GO, the number of microcracks and CH crystals in the interface transition zone of UHPC samples decreased, the hydration products are flocculated and dense, and the gel bonded on the surface of steel fiber gradually increased (figures 7(b) and (c)). However, when the content of GO is further increased to 0.06%, the accumulation of hydration products is poor, and the number of CH crystals increases (figure 7(e)). In contrast, the microstructure of UHPC-4 sample is better, which corresponds to the results of mechanical properties.

Figure 8 shows the SEM images of UHPC-0 and UHPC-4 matrix. It can be seen that there are a small number of microcracks in the matrix of UHPC-0 sample, and some lamellar and massive CH crystals are distributed in C-S-H gel, which has an adverse effect on the mechanical strength of UHPC. Interestingly, the morphology of hydration products in the matrix changed obviously with the incorporation of GO. As shown in figure 8(b), C-S-H gels are evenly distributed in the sample UHPC-4 matrix, and part of needle-like ettringite and C-S-H gels are interconnected together, and a small amount of GO uniformly covers the lamellar CH crystal surface, filling the pores among hydration products. This explains why the sample containing 0.04%GO has good mechanical properties.

4.3. MIP analysis

As we all know, porosity and pore size distribution play an important role in the performance of concrete. MIP is currently the study of porous materials, especially widely used in cementitious materials [20]. Figure 9 prompts the cumulative pore distribution and pore structure distribution of UHPC samples at 28d (table 4), which
indicates that the total porosity of GO UHPC samples is obviously lower than that of reference samples. Compared with the benchmark group, the total porosity of UHPC-2, UHPC-4 and UHPC-6 decreased by 6.2%, 29% and 16.6%, respectively, indicating that the total porosity of UHPC did not decrease linearly with the increase of GO content. Usually, different sizes of pores have different effects on the performance of concrete, in which pores larger than 100 nm are defined as harmful pores [21]. It can be found from the table that the UHPC sample with 0.04% GO content reduces the porosity with a pore diameter of more than 100 nm, but the porosity above 100 nm increases with the increase of GO content to 0.06%, indicating that an appropriate amount of GO can improve the pore structure of UHPC [22]. However, excessive GO causes the pore size distribution to show an opposite trend and reduces its microstructure, which is consistent with the results of the above SEM images.

4.4. Nanoindentation

Figure 10 prompts the nanoindentation image of the ITZ between the steel fiber and the cement paste of the UHPC sample at 28d. Under normal conditions, there are many pores in the matrix and loose gel phase in the ITZ region, which plays an important role in determining the mechanical properties of UHPC. The figure indicates that there is an area with low elastic modulus between steel fiber and cement paste, namely ITZ. The thickness of ITZ is estimated according to the distribution of the elastic modulus of the interface between the steel fiber and the matrix, because the elastic modulus of the interface is lower than that of the steel fiber and the matrix. The ITZ width of UHPC-0 sample is about 45 μm, and the average elastic modulus is about 19.63 GPa. The ITZ width of UHPC-4 sample is about 35 μm, and the average elastic modulus is about 20.68 GPa. Moreover, the elastic moduli of the matrix of UHPC-0 and UHPC-4 samples are 25 GPa and 29.9 GPa, respectively, which implies that, compared with the reference group, the width of ITZ of the sample containing 0.04%GO UHPC decreases, and the elastic modulus of ITZ and matrix also increases. Accordingly, the nano-mechanical properties of UHPC are improved by adding 0.04%GO.
4.5. Mechanism analysis

The mechanical properties of cement composites are considered to be the most important properties in most applications. The results suggest that the mechanical properties of UHPC can be improved by adding appropriate amount of GO (figure 4), and its mechanism is as follows: (1) The excellent mechanical properties of UHPC.
GO play a positive role in improving the mechanical properties of UHPC; (2) The nucleation effect of GO can provide additional nucleation sites in the process of cement hydration, accelerate cement hydration and improve its microstructure (figures 7 and 8). (3) GO can reduce the porosity during crack initiation (figure 9), and the interlock and interaction between microcracks and GO are easy to form dense gel structure and inhibit crack propagation. (4) The C-S-H gel layer of UHPC contains more calcium ions and hydroxides. Calcium ions combine with GO oxygen-containing functional groups, and water molecules form hydrogen bonds between GO functional groups and C-S-H gel. The chemical bond at the interface of GO/matrix enhances the binding of C-S-H and GO, and improves the ductility and tensile strength of the structure (figure 5). It is

**Figure 10.** Distributions of modulus for the grid nanoindentation across the ITZs between steel fiber and cement paste.

**Table 4.** Pore structure distribution of UHPC 28 d with different GO content (ml g⁻¹).

| Sample       | UHPC-0 | UHPC-2 | UHPC-4 | UHPC-6 |
|--------------|--------|--------|--------|--------|
| >200 nm      | 0.04515| 0.0416 | 0.0271 | 0.0572 |
| 100–200 nm   | 0.0117 | 0.0209 | 0.0130 | 0.0128 |
| 50–100 nm    | 0.01745| 0.0199 | 0.0210 | 0.0196 |
| 20–50 nm     | 0.02315| 0.0204 | 0.0143 | 0.0133 |
| 0–20 nm      | 0.01825| 0.0057 | 0.0055 | 0.0044 |
worth noting that when the content of GO exceeds the optimal content, the mechanical properties of UHPC are reduced due to the agglomeration effect. Furthermore, the reinforcement effect and optimum content of GO are very different, which is mainly different from the characteristics (size, oxygen content, layer thickness, number of layers) of GO and the physical properties (tensile strength, elastic modulus) of GO, including cement type, superplasticizer, water–binder ratio, curing conditions and preparation methods, etc. Currently, the application of GO in UHPC remains in the initial stage, and its mechanism is still in the preliminary research stage.

5. Conclusions

In this paper, the effect of different GO content on the mechanical properties of UHPC is studied, and the effect of GO on the micro-nano structure of UHPC is analyzed. According to the research results, the main conclusions are as follows:

(1) With the increase of GO content, the fluidity of UHPC decreases. When the content of GO is 0.04%, the initial setting time and final setting time of the sample are reduced by 30 min and 45 min respectively compared with the benchmark group.

(2) When the content of GO is less than 0.04%, the compressive strength of UHPC increases with the increase of GO content. The change trend of flexural strength and tensile strength is similar to that of compressive strength. The compressive strength of UHPC-4 sample at 28 d and 90 d increased by 15.8% and 14.5%, respectively, and the flexural strength increased by 14% and 13.5%, respectively. Besides, the tensile strength of UHPC samples containing GO ranges from 7.55 MPa to 8.7 MPa.

(3) With the addition of GO, the characteristic peak value of CS decreases, which implies that GO promotes the hydration of cement.

(4) SEM and MIP analysis explain that with the incorporation of GO, the porosity of UHPC decreased by 6.2%–29%. The porosity of harmful pores (pore diameter > 100nm) of UHPC samples with 0.04% GO content decreased, and the number of ITZ microcracks and CH crystals between steel fiber and matrix decreased, indicating that the microstructure of UHPC can be improved by adding appropriate amount of GO into UHPC.

(5) The ITZ width of UHPC-0 sample is about 45 μm, and the average elastic modulus is about 19.63 GPa. The ITZ width of UHPC-4 sample is about 35 μm, and the average elastic modulus is about 20.68 GPa. Additionally, the elastic moduli of the matrix of UHPC-0 and UHPC-4 samples are 25 GPa and 29.9 GPa, respectively. Accordingly, the nano-mechanical properties of UHPC are improved by adding 0.04% GO.

(6) The ITZ width of UHPC-0 and UHPC-4 sample are about 45 μm and 35 μm, respectively. The average elastic modulus are about 19.63 GPa and 20.68 GPa, respectively. Accordingly, the nano-mechanical properties of UHPC are improved by adding 0.04% GO.

(7) The enhancement effect of GO on the mechanical properties of UHPC is mainly attributed to the excellent physical properties of GO itself, the crystal nucleus effect promotes the hydration of cement, improves its microstructure, and the chemical bond formed at the interface of GO/matrix strengthens the bond between C-S-H and GO.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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