Direct Tensile Performance of the Initially Cracked UHPC Subjected to Coupled Chloride Salt and Freeze-Thaw Attacks

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Abstract

The tensile properties of ultra high performance concrete (UHPC) with initial cracking subjected to the coupled chloride salt and freeze-thaw attacks were investigated. A reliable approach to introduce the desired initial cracking under the controlled condition in the laboratory was developed and validated. The method consists of applying direct tension to the dog bone specimens and terminating at a strain at the strain hardening stage. The initially cracked UHPC specimens were then submerged in the sodium chloride solution with a concentration of 4% and subjected to freeze-thaw (F-T) cycles. The results showed that the tensile properties (tensile strength, first cracking strength, strain capacity, energy absorption capacity) of the initially cracked UHPCs were generally improved up to 200 F-T cycles compared with those of the control and then decreased with the further increase of the F-T cycles. The improvement in the mechanical properties up to 200 F-T cycles can be attributed to the secondary hydration of the originally unhydrated cement. This is supported by the higher degree of hydration quantified by the thermogravimetric (TG) analysis. However, the detrimental effect due to the coupled chloride salt and F-T attacks exceeded the beneficial effect of the secondary hydration at the 300 F-T cycles which could explain the reduced tensile properties at 300 F-T cycles.

Keywords: UHPC, freeze-thaw attack, chloride salt attack, secondary hydration

1. Introduction

Ultra-high performance concrete (UHPC) is a special fiber-reinforced cementitious composite with significantly improved mechanical properties and excellent durability. Due to its large amount of cement and supplementary cementitious materials, and the very low water to cementitious material ratio (*w/cm*), UHPC usually has a significantly higher risk of shrinkage cracking compared with normal concrete. In addition, UHPC is an excellent substitute to conventional concrete when the environment is aggressive during the service life such as the cold regions where prolonged freezing days in a year, large temperature difference between day and night, and frequent use of de-icing salt are not uncommon. Therefore, cracking of UHPC during service life is almost inevitable [1].

Despite the extensive research that has been conducted on the performance of UHPC subjected to either freeze-thaw (F-T) attack or chloride salt attack, most of the studies focused on its behavior under the initially intact condition. The investigation on the durability of initially cracked UHPC remains limited. Lv et al. [2] investigated the effect of different degrees of initial cracking on the chloride ion permeability of UHPC. The study showed that the chloride ion diffusion was two-dimensional, and increased with the increase of tensile deformation. Shin and Yoo [3] investigated the tensile behavior of pre-cracked UHPC immersed in sodium chloride solution. The results indicated that the moderate corrosion of steel fibers resulted in improved tensile properties of UHPC, and the less severely pre-cracked UHPC demonstrated imrpoved tensile properties.

Although it has been shown that the self-healing ability of concrete is beneficial to the frost resistance of concrete [4], the research on the self-healing ability of UHPC is still in its initial stage [5]. Hilloulin et al. [6] investigated the self-healing behavior of UHPC and the mechanical properties of the healing products. The results showed that the self-healing of UHPC facilitated partial recovery of its mechanical properties. However, the mechanical properties of the healing products were lower than those of the matrix. Although it has been confirmed that the self-healing ability of UHPC is beneficial to the recovery of its mechanical properties [5], little has been reported for its influence on the tensile performance of initially cracked UHPC subjected to the coupled chloride salt and F-T attacks.

The tensile performance of UHPC with initial cracking subjected to the coupled chloride salt and F-T attacks were investigated in this study. Cracking was introduced to the UHPC specimens under the controlled condition by applying tension and terminating at a point during the strain hardening stage. The pre-cracked specimens were then submerged in the sodium chloride solution with a concentration of 4% and subjected to accelerated F-T cycles.

2. Experimental Program

2.1. Materials and Test Design

The grade 52.5 white cement from Aalborg and the silica fume possessing 92.4% silicon dioxide supplied by Elkem were utilized. The main component of quartz powder was silicon dioxide and has a median particle size of 2.6 μm. Quartz sand was used as fine aggregate with a median particle size of 300 μm. Straight steel fibers were incorporated to improve the ductility of UHPC. The length and diameter are 13 mm and 0.2 mm, respectively. Its tensile strength is 2500 MPa. The powder form polycarboxylate ether-based superplasticizer produced by BASF was utilized to enhance the workability of UHPC. Ordinary tap water was used as the mixing water. The mixture design is shown in Table 1.

Table 1. The mix design of UHPC (unit: kg/m^3)

Cement	Silica fume	Quartz powder	Quartz sand	Steel fiber	Water	Superplasticizer
790	197.5	197.5	907	157	189.6	23.7

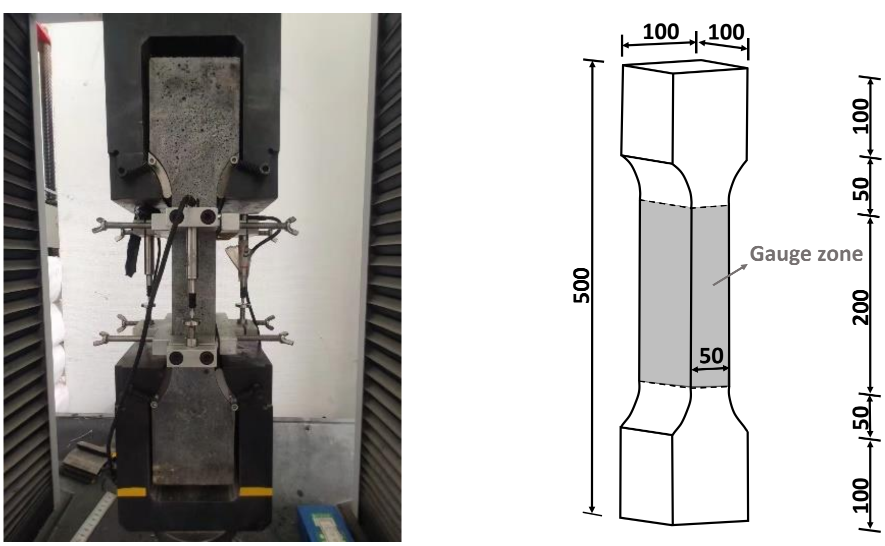
A series of tests was designed to investigate the tensile performance of UHPC subjected to the coupled chloride salt and F-T attack under different initial conditions. The test conditions are shown in Table 2. To facilitate the understanding, the designation of each series starts with the letter of ‘I’ or ‘C’ which represents the initial status of the specimen as intact or cracked. The following letter ‘FT’ and the numbers indicate the number of F-T cycles. For example, C-FT100 is the series of test of specimens with initial cracking subjected to 100 cycles of F-T. The full stress versus strain curve of the UHPC under direct tension was characterized to determine the strain at which the pre-loading process should be terminated to introduce the desired initial cracking. The control specimens (C-FT0) and the initially cracked specimens were then subjected to the coupled chloride salt and F-T attacks.

Table 2. Summary of the test conditions

	C-FT0	C-FT100	C-FT200	C-FT300
Initial cracking	✓	✓	✓	✓
F-T cycles	0	100	200	300

2.2. Characterization of the Direct Tensile Behavior

The dog bone specimen was used to characterize the direct tensile behavior of UHPC. The test setup and dimensions of the specimen are presented in Figure 1. The gauge length is 200 mm and the corresponding cross-section is 50 mm × 100 mm. All specimens were demolded after 24 hours, and then placed in a curing room for 28 days. The temperature in the curing room was 20 ± 2 ℃ and the relative humidity was above 95%. The initial cracking was introduced by pre-applying direct tensile load. The full stress versus strain curve of the UHPC under direct tension was first characterized to determine the strain at which the pre-loading process should be terminated to introduce the desired initial cracking. The strain of 1500 με in the middle of the strain hardening stage was chosen in this study as the point up to which the tensile pre-loading process was terminated. A universal testing machine (WDW-3000) with a capacity of 300KN was used to carry out the test. Two sets of linear variable differential transducers (LVDTs) installed in parallel (four in total) were used to measure the deformation. The load was applied under displacement-controlled mode with a loading rate of 0.3 mm/min. At least three specimens were tested for each series of test.



(a) test set up (b) dimensions of the dog bone specimen (unit: mm)

Figure 1. The setup of the direct tensile test and dimensions of the specimen

2.3. Coupled Chloride Salt and Freeze-thaw Attacks Test

The accelerated freeze-thaw test was conducted in accordance with the ASTM-C666 procedure B [7] with some minor revisions tailored to fulfill the specific purpose of this study. The prismatic specimen was replaced by the dog bone shaped specimen to facilitate the introduction of the desired initial cracking under controlled condition. Instead of using fresh water, specimens were submerged in sodium chloride solution with a concentration of 4% to achieve the coupled chloride salt and F-T attacks.

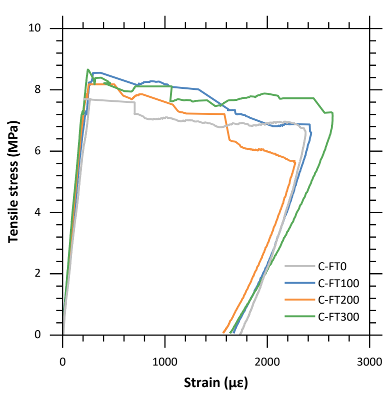
2.4. Thermogravimetric Analysis

Thermogravimetric (TG) analysis was performed to quantify the degree of hydration of UHPC under different conditions. Samples were taken from the fractured surface after the direct tensile test and ground to powder with the particle size smaller than 75 μm. 0.2 g of the ground fine powder sample was dried in a vacuum oven at 60°C for 24 h prior to the TG test. The test was performed using a thermogravimetric analyzer (Mettler STAR, Switzerland) in an inert nitrogen atmosphere with a gas flow rate of 25 mL/min. The samples were heated from the ambient temperature to 1000°C at a constant rate of 10°C/min.

3. Results and Discussion

3.1. Reliable Introduction of the Desired Initial Cracking Under Controlled Condition

The typical stress versus strain curves corresponding to the loading and unloading processes to introduce the desired initial cracking are shown in Figure 2a. It can be seen that all specimens produced similar shapes of tensile stress versus strain curves, comparable peak stresses of about 8 MPa, plastic strains of approximately 1600με, and almost identical unloading modulus. The similarity in the key characteristics and the shape of the tensile stress versus strain curves validate the reliability and repeatability of the proposed approach to introduce the initial cracks. The tensile load was in the range of the strain-hardening stage and multiple fine cracks visible to the naked eyes were formed on the surface of the specimen as shown in Figure 2b. To summarize, these observations indicate that the desired initial cracking was successfully introduced in a reliable and repeatable manner by the proposed approach. Note that the strain actually attained was slightly larger than that have planned. This is expected since it is extremely difficult, if not impossible, to control exactly the strain of the specimen due to the non-homogenous and relatively brittle nature of cement based cementitious composite.



(a) load-unload stress versus strain curves

(b) multiple fine cracks after pre-cracking

Figure 2 Reliable introduction of the desired initial cracking

3.2. Direct Tensile Performance of UHPC

The key characteristics of the tensile behavior, including the tensile strength (f_{tu}), first cracking strength (f_{tc}), strain capacity (ε_t) and energy absorption capacity (g), were extracted from the stress versus strain curve to quantify the effects of the coupled chloride salt and F-T attacks. The first cracking strength is the tensile stress corresponding to the end of the elastic stage in the stress versus strain relationship. The strain capacity is the strain corresponding to the tensile strength. In light of the fluctuation of the tensile stress beyond the peak stress of the UHPCs investigated herein, the strain corresponding to the stress 0.3 MPa lower than the first peak stress was taken as the strain capacity in this investigation. The energy absorption capacity is defined as the area under the tensile stress versus strain curve up to the strain capacity.

Figure 3 compares the aforementioned parameters among the UHPCs subjected to different cycles of the coupled chloride salt and F-T attacks. The general trend is that all of the aforementioned properties increase with the increase of the cycles of the coupled chloride salt and F-T attacks up to 200 cycles beyond which there exists a sharp reduction of these tensile properties. It can be seen from Figure 3a that the first cracking strength of C-FT100 and C-FT200 increased by 14.78% and 20.91%, respectively, compared with the control C-FT0. Since it is mainly dictated by the strength of the matrix [8], the improvement in the first cracking strength implies the occurrence of the secondary hydration. It was validated by the TG test result.

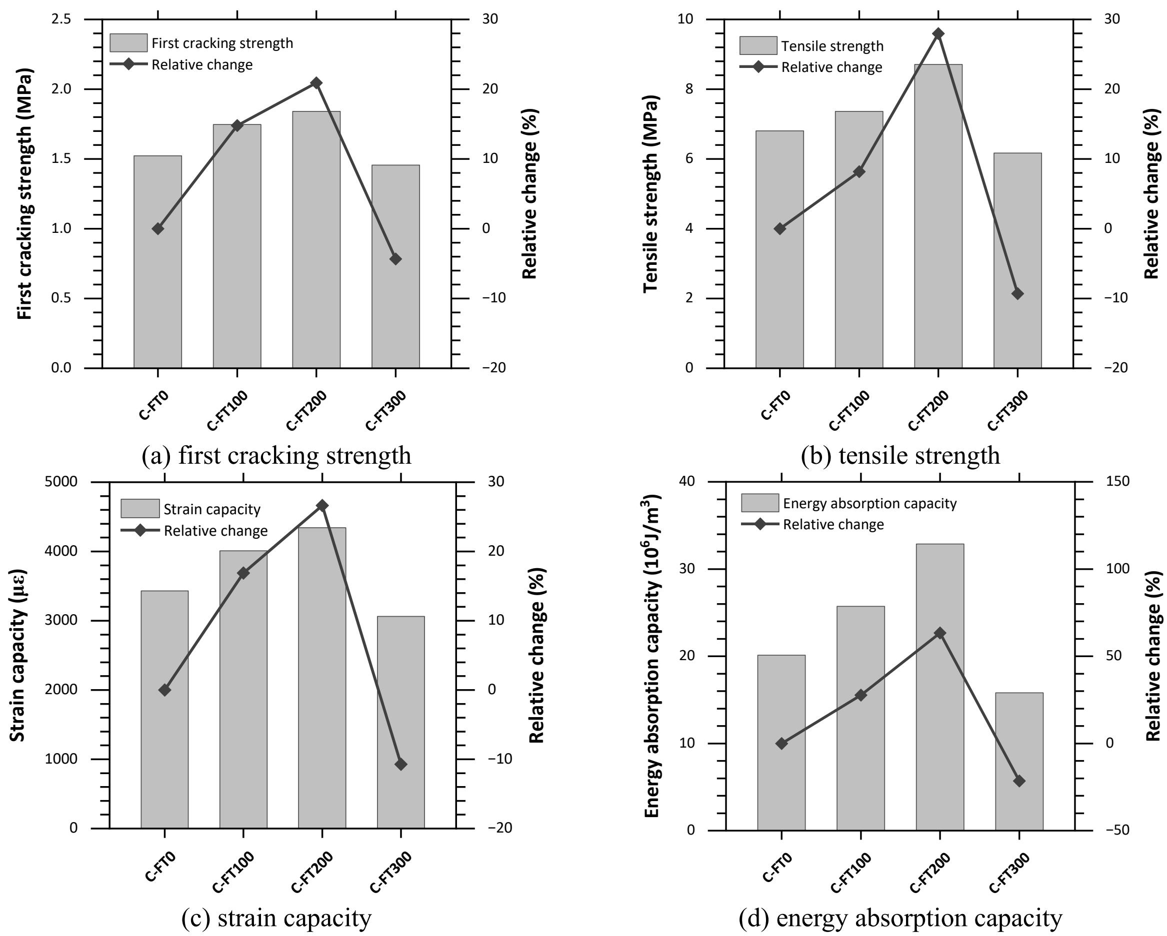


Figure 3. Changes in mechanical properties of UHPC under different test conditions

The differential thermo-gravimetric (DTG) analysis curves are shown in Figure 4. The curves exhibit two peaks at the temperatures before 200°C and around 600°C. The first peak observed before 200°C is due to the evaporation of free water. The other peak around 600°C can be attributable to the decomposition of calcium carbonate [9]. No sign of the major hydration product of portlandite was observed. It could have been converted to calcium carbonate at the presence of carbon dioxide which explains the sharp and broad peak around 600°C. The calculated calcium carbonate contents are shown in Figure 5. It increases with the number of the coupled chloride salt and F-T attacks up to 200 cycles beyond which reduction was observed. The calcium carbonate content increased by 55.86% and 102.70% at 100 and 200 F-T cycles, respectively. The increase in the calcium carbonate content indicates the occurrence of further hydration and the conversion of portlandite to calcium carbonate. The secondary hydration could densify the microstructure and offset the detrimental effect of the coupled chloride salt and F-T attacks which ultimately leads to the observed improvement in the tensile characteristics.

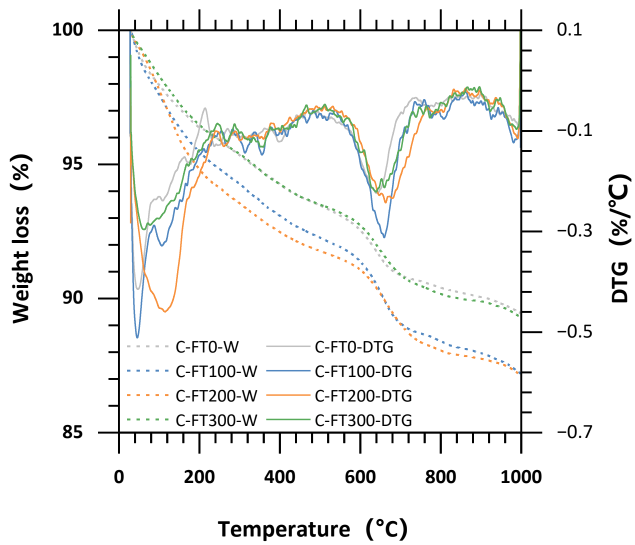


Figure 4 TG results for different test series

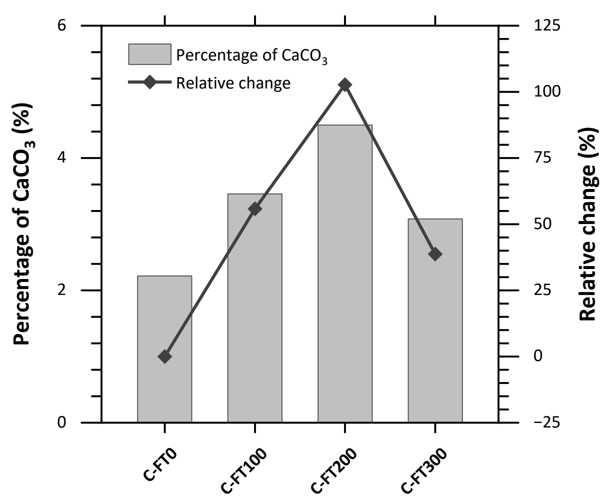


Figure 5 Variation of the calcium carbonate content with F-T cycles

The trends for the variation of the tensile strength and strain capacity are similar as can be observed from Figure 2b and 2c, respectively. Since the energy absorption capacity is dependent on the tensile strength, the first cracking strength, and the strain capacity, its change with the F-T cycles is consistent with that of the other three parameters. The enhancement of energy absorption capacity attained 27.7% and 63.6% at 100 and 200 cycles of the coupled chloride salt and F-T attacks but it turned to a reduction of 21.5% at 300 cycles. The tensile strength of C-FT100 and C-FT200 were improved by 8.2% and 28.0%, respectively, compared with the control specimen C-FT0 without experiencing any coupled chloride salt and F-T attacks. The strain capacity of C-FT100 and C-FT200 were improved by 16.9% and 26.7%, respectively, compared with the control specimen C-FT0. It is interesting to pointing out the non-proportionally higher improvement of energy absorption capacity for C-FT200 (up to 63.6%) compared with the tensile strength (28.0%) and strain capacity (26.7%). It is well established that the fiber-matrix interfacial bonding performance is critical to the tensile strength and corresponding strain capacity of fiber-reinforced cementitious composite [10]. The initial cracking of the UHPCs favored the transport of the external water towards the cracked region where fiber bridging effect was activated. The secondary hydration could be promoted as already confirmed by the first cracking strength (Figure 2a) and TG results (Figure 4). Therefore, the matrix surrounding the fibers could be densified which ultimately resulting in the more pronounced improvement in the energy absorption capacity.

4. Conclusions

A study was carried out on the direct tensile performance of the initially cracked UHPC subjected to the coupled chloride salt and F-T attacks. A reliable method to introduce the desired initial cracking was developed and validated. It was found that the tensile properties including the first cracking strength, tensile strength, strain capacity and the energy absorption capacity increased up to 200 F-T cycles. It is attributable to the secondary hydration of the initially unhydrated cement which was supported by the increased calcium carbonate content. Further increase in the F-T cycles resulted in a moderate decrease of these tensile properties. It could be explained by the dominant detrimental effect of the coupled chloride salt and F-T attacks compared with the beneficial effect of secondary hydration.

Acknowledgement

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References

[1] Bjøntegaard, Ø., Hammer, T.A. and Sellevold, E.J., "On the measurement of free deformation of early age cement paste and concrete," *Cement and Concrete Composites*, Vol. 26, No. 5, July 2004, pp. 427-435.

[2] Lv, L.S., Wang, J.Y., Xiao, R.C., Fang, M.S. and Tan, Y., "Chloride ion transport properties in microcracked ultra-high performance concrete in the marine environment," *Construction and Building Materials*, Vol. 291, July 2021, 123310.

[3] Shin, W. and Yoo, D.Y., "Influence of steel fibers corroded through multiple microcracks on the tensile behavior of ultra-high-performance concrete," *Construction and Building Materials*, Vol. 259, October 2020, 120428.

[4] Jacobsen, S., Marchand, J. and Hornain, H., "SEM observations of the microstructure of frost deteriorated and self-healed concretes," *Cement and Concrete Research*, Vol. 25, No. 5, December 1995,pp. 1781-1790.
[5] Kim, S., Yoo, D.Y., Kim, M.J. and Banthia, N., "Self-healing capability of ultra-high-performance fiber-reinforced concrete after exposure to cryogenic temperature," *Cement and Concrete Composites*, Vol. 104, November 2019, 103335.
[6] Hilloulin, B., Grondin, F., Matallah, M. and Loukili, A., "Modelling of autogenous healing in ultra high performance concrete," *Cement and Concrete Research*, Vol. 61–62, July/August 2014, pp. 64-70.
[7] Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing, ASTM C666, ASTM International, Volume: 04.02, West Conshohocken, PA, 2016.
[8] Niu, Y., Wei, J. and Jiao, C., "Crack propagation behavior of ultra-high-performance concrete (UHPC) reinforced with hybrid steel fibers under flexural loading," *Construction and Building Materials*, Vol. 294, August 2021, 123510.
[9] Goto, S., Suenaga, K., Kado, T. and Fukuhara, M., "Calcium silicate carbonation products," *Journal of the American Ceramic Society*, Vol. 78, No.11, November 1995, pp. 2867-2872.
[10] Kan, L., Wang, F., Zhang, Z., Kabala, W. and Zhao, Y., "Mechanical properties of high ductile alkali-activated fiber reinforced composites with different curing ages," *Construction and Building Materials*, Vol. 306, November 2021, 12483.