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Research on Freeze-Thaw Cycle Performance of High Ductility Cement-based Composites: Mechanism, Evolution and Life Prediction.

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Abstract

As a novel construction material with exceptional tensile toughness and crack control capability, the durability of Engineered Cementitious Composites (ECC) in cold environments is of significant importance. This paper systematically investigates the performance evolution and mechanisms of ECC under freeze-thaw cycles. The research indicates that freeze-thaw cycles lead to a reduction in the peak stress of ECC, coarsening of the internal pore structure, and weakening of the fiber-matrix interface. However, the bridging action of fibers can effectively inhibit the propagation of macro-cracks. After 300 freeze-thaw cycles, the mass loss rate of PVA fiber-reinforced ECC can be controlled within 2.5%, and its dynamic elastic modulus retention rate is significantly higher than that of ordinary concrete. Compounding PE-ECC with FRP mesh further enhances frost resistance, with the maximum load-carrying capacity increased by up to 67.3%. This study also establishes a damage model based on the Wiener process and a prediction method integrating multi-scale surface structure recognition, providing a theoretical basis and design reference for the engineering application of ECC in cold regions. The results demonstrate that ECC exhibits excellent durability under freeze-thaw conditions. Through material optimization and composite reinforcement techniques, the service life of engineering structures in cold regions can be significantly extended.

Keywords

High Ductility Cementitious Composites, Freeze-thaw Cycles, Fiber Reinforcement, Durability, Life Prediction, Damage Mechanism.

1. Introduction

In cold regions, concrete structures long-term endure the severe challenges of alternating positive and negative environmental temperatures. Freeze-thaw cycles can trigger the expansion and accumulation of microcracks within the material, leading to continuous degradation of mechanical properties and ultimately threatening the long-term durability and safety of structures. Traditional concrete, due to its inherent brittleness and high porosity, exhibits low tensile strength and poor deformation capacity, making it prone to severe damage under freeze-thaw conditions.

Engineered Cementitious Composites (ECC) are a special type of high-performance fiber-reinforced cementitious composite material known for excellent strain-hardening behavior under tension[1]. With remarkable tensile ductility (strain-hardening characteristics) and unique microcrack control capabilities, ECC offers an innovative solution for enhancing structural durability in harsh environments. The ultimate tensile strain of ECC can reach 3%–5%, which is 300–500 times that of ordinary concrete, demonstrating typical strain-hardening behavior. Moreover, crack patterns manifest as multiple fine cracks, distinctly different from the single or few coarse cracks observed in ordinary concrete under load. According to research by Huang Shiyuan, Jiang Jiafen, Yang Nanru[2], fibers exert a significant restraining effect on the ECC matrix during freeze-thaw cycles, effectively counteracting the internal stress generated by water freezing and expansion, thereby resulting in only minor degradation of mechanical properties after freeze-thaw exposure. Despite the superior material properties of ECC, its performance evolution under freeze-thaw conditions-particularly the interface behavior with ordinary concrete, microscopic damage mechanisms, and long-term life prediction-remains a key focus and challenge in current research.

Based on this, this study aims to systematically review experimental data and literature to deeply reveal the influence of freeze-thaw cycles on the mechanical properties of ECC and its composite structures, clarify the microscopic damage mechanisms, and evaluate existing damage models and life prediction methods, thereby promoting the reliable application of ECC in infrastructure construction in cold regions.

2. Mechanism of Freeze-Thaw Cycle Impact on ECC Performance

2.1. Macroscopic Manifestations of Freeze-Thaw Damage

Freeze-thaw cycles, as a physical erosion process, exert a significant deteriorating effect on the macroscopic mechanical properties of ECC materials. This deterioration is not linear but exhibits phased characteristics with increasing cycle numbers. The damage law can be clearly revealed by systematically testing indicators such as mass loss, dynamic elastic modulus, and strength. Mass loss and relative dynamic elastic modulus change are two core indicators for evaluating the frost durability of cementitious materials. For ordinary concrete, freeze-thaw cycles typically cause severe surface scaling and rapid mass loss. However, ECC performance is notably different. Studies show that even after 300 freeze-thaw cycles, the mass loss rate of polyethylene fiber-reinforced ECC (PE-ECC) is only 1.67%, far lower than the 13.5% for ordinary mortar concrete[3]. This is primarily attributed to the effective restraint of matrix spalling by fiber bridging, limiting the generation and propagation of macro-cracks. The relative dynamic elastic modulus sensitively reflects the damage to the internal structure of the material. During freeze-thaw cycles, repeated freezing and expansion of water in pores generate tensile stress, leading to micro-crack initiation and propagation, thus causing a decrease in the dynamic elastic modulus. Research indicates that after 300 freeze-thaw cycles, the dynamic elastic modulus of ECC decreases by less than 5%, while that of air-entrained concrete decreases by nearly 15% after the same cycles[4], concrete with 1% steel fibers decreases by nearly 30%, and ordinary concrete decreases by over 40% after only 200 cycles[5] .

2.2. Evolution Law of Microstructure

The deterioration of macroscopic performance is rooted in structural evolution at the micro- and meso-scales. Modern testing techniques allow for an in-depth revelation of the freeze-thaw damage mechanism of ECC.

The evolution of pore structure is the most direct effect of freeze-thaw cycles. Freeze-thaw cycles cause changes in the pore structure of the material, with the total porosity of ECC gradually increasing as the number of cycles increases. A more critical change is the shift in pore size distribution: small capillary pores migrate and develop into larger pores, i.e., the number and volume of harmful pores (usually referring to pores larger than 100nm in diameter) increase. This is mainly because the growth pressure of ice crystals and the pressure from salt crystallization work together, causing the original micropores to expand and connect. Studies show that capillary pores (100-1000nm) account for more than half of the total porosity, and their proportion increases significantly with freeze-thaw cycles, while transitional pores (10-100nm) show no significant increase.

The degradation of the fiber-matrix interfacial transition zone (ITZ) is another key factor. The interfacial transition zone between fibers and the cement matrix is a critical region for stress transfer and achieving strain hardening in ECC, and it is also a vulnerable area under freeze-thaw conditions. Scanning Electron Microscopy (SEM) observations reveal that after freeze-thaw cycles, the chemical and physical strength of the fiber-matrix ITZ weakens. Specifically, the width of the ITZ may increase, and its structure becomes looser. This degradation makes fibers more prone to being pulled out from the matrix rather than fractured under stress, leading to a weakening of fiber bridging stress and manifesting macroscopically as a decrease in strength.

2.3. Fiber Types and Reinforcement Mechanisms

Fibers play a key role in the frost resistance of ECC, with different fiber types exhibiting different reinforcement mechanisms. PVA fiber is one of the most commonly used fiber types in ECC. Studies show that there are certain differences in the freeze-thaw resistance between ECC using domestic and imported PVA fibers. After 300 freeze-thaw cycles, the mass loss rates of ECC with domestic and imported PVA fibers are controlled within 2.5% and 1.5%, respectively. Compared to concrete and mortar, the longitudinal and transverse relative dynamic elastic moduli of ECC are increased by 1.62 to 1.87 times and 1.61 to 1.79 times, respectively, demonstrating significant performance superiority[6]. PE fiber, due to its high strength and hydrophobicity, performs excellently in enhancing frost resistance. Research finds that the crack resistance of PE fibers can effectively restrain the spalling of cement mortar. Adding CFRP or BFRP mesh to PE-ECC can further improve the strength of the specimens and reduce the propagation of micro-cracks within the cement-fiber matrix material, significantly enhancing frost resistance. It was found that the maximum load-carrying capacities of PE-ECC-BFRP and PE-ECC-CFRP specimens increased by 22.5% and 67.3%, respectively, and the maximum deflections increased by 28.2% and 76.7%, respectively, compared to PE-ECC specimens[7].

3. Materials and Test Methods

3.1. Material Preparation and Mix Proportion Design

The performance of ECC is significantly influenced by its mix proportion; a reasonable mix design is the foundation for optimizing its frost resistance.

The sand-to-binder ratio is a key parameter affecting the frost resistance of ECC. Studies show that as the sand-to-binder ratio increases, the mass loss rate of ECC gradually increases. A lower sand-to-binder ratio typically results in fewer sand particles filling the pores in the concrete, making it denser, which helps reduce the penetration of moisture and salts, thereby improving freeze-thaw resistance. Conversely, a higher sand-to-binder ratio may lead to a larger pore structure, reducing freeze-thaw resistance. Therefore, a balance between workability and frost durability needs to be struck in engineering applications by selecting an appropriate sand-to-binder ratio.

Fiber characteristics, including type, length, diameter, content, and dispersion, all have an important impact on the frost resistance of ECC. High Ductility High-tenacity (HDH) PVA fibers, due to their higher durability and adhesion, can better enhance the freeze-thaw resistance of ECC. Longer PVA fibers can be better dispersed in the concrete, providing better reinforcement and increasing the freeze-thaw resistance of ECC. An appropriate amount of PVA fiber content can effectively improve the freeze-thaw resistance of ECC, but excessive fibers may lead to a decrease in the workability of the concrete.

The use of supplementary cementitious materials also significantly affects the frost resistance of ECC. The incorporation of fly ash and other reactive pozzolans influences the freeze-thaw resistance of ECC. For concrete containing fly ash, the frost resistance can be greatly improved by adding an appropriate amount of air-entraining agent. Studies indicate that with a low water-cement ratio and suitable air content, Class I fly ash can ensure high frost resistance for fly ash concrete[7]. When the fly ash content is high, the stability of the air content in concrete is affected by the adsorption of fly ash. However, at the same porosity, the closed glassy cavities introduced by fly ash and its pozzolanic reaction can be beneficial for improving the frost resistance of concrete.

3.2. Freeze-Thaw Cyclic Test Methods

Freeze-thaw cyclic tests are the primary means to evaluate the frost resistance of ECC. Commonly used test methods include the rapid freeze-thaw method and the slow freeze-thaw method.

The rapid freeze-thaw method is a commonly used accelerated test method, typically conducted according to standards such as GBJ82-85 "Test Methods for Long-term Performance and Durability of Ordinary Concrete". This method subjects saturated specimens to rapid freeze-thaw cycling, usually between -18°C and +5°C, completing one cycle every 2-4 hours. During the test, parameters such as mass loss and dynamic elastic modulus of the specimens are measured every 25 cycles to assess performance degradation.

Performance test indicators mainly include mass loss rate, relative dynamic elastic modulus, mechanical properties (compressive, flexural, tensile), and microstructural changes. Mass loss rate reflects surface scaling; relative dynamic elastic modulus characterizes material stiffness degradation; mechanical property tests assess load-bearing capacity changes; microstructural analysis reveals damage mechanisms. These tests are usually conducted at specific intervals of freeze-thaw cycles to obtain complete curves of performance variation with the number of cycles.

3.3. Microstructural Analysis Methods

To deeply understand the damage mechanisms of ECC under freeze-thaw cycles, various microstructural analysis techniques are required.

Scanning Electron Microscopy (SEM) is used to observe the micromorphology of ECC after freeze-thaw cycles, particularly changes in the fiber-matrix interfacial transition zone. SEM allows direct visualization of changes in the bond between fibers and the cementitious matrix after freeze-thaw cycles, as well as the propagation of internal micro-cracks.

Mercury Intrusion Porosimetry (MIP) is used to analyze trends in pore structure changes before and after freeze-thaw exposure. MIP provides key parameters such as porosity and pore size distribution, revealing the influence of freeze-thaw cycles on the pore structure of ECC. Studies show that freeze-thaw cycles increase the porosity of ECC, with the proportion of capillary pores (100-1000nm) significantly increasing, which is the main cause of performance degradation[8].

Image processing and analysis is a recently developed method. It involves acquiring surface damage images of ECC after freeze-thaw exposure using high-resolution cameras or microscopes, and then using image processing techniques to quantify surface damage features (such as fiber exposure area, scaling depth, micro-crack distribution). This method can non-destructively assess the freeze-thaw damage state of ECC, providing convenience for practical engineering inspection.

4. Results and Discussion

4.1. Evolution Law of Freeze-Thaw Cycles on Macroscopic Mechanical Properties of ECC

Freeze-thaw cycles have a profound impact on the macroscopic mechanical properties of ECC, and its evolution law can be revealed through systematic experimental research.

Tensile performance is the most notable characteristic of ECC, and it also degrades in freeze-thaw environments. Research shows that CA-ECC can still maintain strain-hardening characteristics (strain exceeding 1%) after 300 freeze-thaw cycles; lightweight aggregate can enhance strain. The tensile strength of various groups decreases by 15-30%, but strain does not decrease. The ductility of FCA-ECC is not affected. The crack width of three materials (ECC, FCA-ECC, NCA-ECC) slightly increases with the number of cycles. Studies have found that the tensile strain capacity (i.e., ductility) of ECC can remain relatively stable after freeze-thaw exposure. Most specimens can maintain a strain capacity above 2% even after 100 salt freeze-thaw (SFT) cycles[9]. This indicates that the strain-hardening behavior and multiple cracking mechanism of ECC remain resilient in harsh environments, which is crucial for the deformation capacity and energy absorption capacity of structures after freeze-thaw cycles.

Compressive and flexural strengths decrease with increasing freeze-thaw cycles. Among them, the decay rate of flexural strength for fiber-reinforced specimens is significantly lower than that of compressive strength, highlighting the contribution of fibers to material toughness. After 200 cycles, the flexural strength retention rate of specimens with 2% PVA fibers exceeds 70%, far surpassing the 45% of the reference group. Research also found that as the number of freeze-thaw cycles increases, the ultimate flexural tensile strength of ECC shows a downward trend, but the decrease is relatively small, from 14.7 MPa to 10.6 MPa. In contrast, for concrete with 1% (by volume) steel fibers, the ultimate flexural tensile strength decreases both faster and to a greater extent with increasing freeze-thaw cycles, dropping from an initial 9.6 MPa to 1.6 MPa after 300 cycles.

The elastic modulus is most sensitive to freeze-thaw damage. Freeze-thaw cycles cause continuous attenuation of material stiffness. After 300 cycles, the elastic modulus of ECC decreases by 35%-55%. The relative dynamic elastic modulus of the reference group specimens decreases by over 50% after 150 cycles, while that of specimens with 2% PVA fibers decreases by only 28% after 200 cycles[3], indicating that fiber bridging retards the propagation of internal micro-cracks. The decrease in elastic modulus is directly related to the expansion of the internal micro-crack network, which reduces material stiffness. However, due to fiber bridging, material toughness is maintained.

4.2. Microscopic Mechanisms and Multi-scale Analysis of Freeze-Thaw Damage

The deterioration of macroscopic performance is rooted in structural evolution at the micro- and meso-scales. Multi-scale analysis helps comprehensively understand the damage mechanisms.

Regarding pore structure evolution, Nuclear Magnetic Resonance (NMR) and X-ray Computed Tomography (X-CT) analyses indicate that as the number of freeze-thaw cycles increases, the total porosity of ECC gradually increases. More critically, the pore size distribution shifts: small capillary pores migrate and develop into larger pores, i.e., the number and volume of harmful pores increase. Studies show that large pores with a volume exceeding 1 mm^3 mainly originate from initial material defects and are relatively less affected by freeze-thaw cycles. Freeze-thaw cycles primarily cause significant expansion of capillary pores and large pores with diameters greater than 1000 nm[10]. For High-Performance Cementitious Composites (HPCC), research found that as the fractal dimension of air void distribution increases, its frost durability roughly shows a linear upward trend. This indicates that optimizing the geometric distribution of pores is crucial for improving frost resistance.

The degradation of the fiber-matrix interfacial transition zone (ITZ) is another key mechanism. SEM observations reveal that after freeze-thaw cycles, the chemical and physical strength of the fiber-matrix ITZ weakens. Specifically, the width of the ITZ may increase, and its structure becomes looser. Research found that after experiencing salt freeze-thaw cycles, the ITZ width decreases from about 4.1 μm to about 2.4 μm. This does not signify interface enhancement but may instead indicate local loosening within the interface zone, leading to a shallower engagement depth between fibers and the matrix. This degradation makes fibers more prone to being pulled out from the matrix rather than fractured under stress, leading to a weakening of fiber bridging stress and manifesting macroscopically as a decrease in strength[11].

4.3. Comparison of Frost Resistance and Enhancement Strategies for Various Modified ECCs

To further enhance the applicability of ECC in cold regions, researchers have developed various modification methods, with different modified ECCs exhibiting different frost resistance.

The influence of fiber type and composite reinforcement is significant. Research shows that compounding carbon fiber reinforced polymer (CFRP) mesh or basalt fiber reinforced polymer (BFRP) mesh on the basis of PE-ECC can significantly improve specimen performance. Compared to pure PE-ECC specimens, the maximum load-carrying capacities of PE-ECC-CFRP and PE-ECC-BFRP specimens increased by 67.3% and 22.5%, respectively, and the maximum deflections increased by 76.7% and 28.2%, respectively[11]. FRP mesh provides additional confinement and reinforcement, effectively suppressing the propagation of micro-cracks within the cement-fiber matrix material, thereby enhancing frost resistance and deformation capacity.

The "active" frost resistance mechanism using phase change materials (PCM) is an emerging strategy. Introducing low-temperature phase change materials is an "active" frost resistance strategy. Its principle utilizes the characteristic of PCMs absorbing or releasing a large amount of latent heat during the solid-liquid phase transition near the phase change point, to delay or inhibit the internal temperature of ECC from dropping below freezing, thereby reducing the actual number or intensity of freeze-thaw cycles[12]. Studies show that when its dosage is 4%, phase change energy storage cementitious material (PCESM) exhibits optimal frost resistance. Although the addition of PCM may slightly increase porosity and have a certain negative impact on mechanical properties, the benefit in frost resistance improvement brought about by temperature regulation is very significant. This intelligent temperature control characteristic provides new ideas for the application of ECC in cold environments.

Ecological modification is a research hotspot in recent years. Using recycled fine powders (such as recycled concrete powder, recycled brick powder) to prepare ECC is a direction for sustainable development. Tests show that under freeze-thaw cycle conditions, the mechanical properties of recycled concrete powder ECC are usually better than those of recycled brick powder ECC. The mechanical properties of both decrease slightly initially and then sharply with increasing replacement rate of recycled powder. After 150 freeze-thaw cycles, the mechanical properties loss is about 20%[13]. Under constant low-temperature conditions (non-freeze-thaw cycles), the mechanical properties of recycled powder ECC instead show an increasing trend as the holding temperature decreases, with mechanical properties improving by about 22% from room temperature to a constant -40°C. This may be due to the combined effects of low temperature causing matrix structure contraction and densification, and increased viscosity of pore water.

4.4. Advanced Assessment and Prediction Methods for Freeze-Thaw Damage

Traditional damage assessment methods (such as single indicator measurement) struggle to fully reflect the complex freeze-thaw damage modes of ECC. In recent years, multi-scale and intelligent assessment methods have become research hotspots.

Image recognition and machine learning-based damage prediction is a new method developed to address the meso-scale characteristics of ECC freeze-thaw damage, which often manifests as micro-cracks and fiber exposure. A study proposed a freeze-thaw damage prediction method and system integrating multi-scale surface structure recognition. This method involves collecting surface images of ECC after freeze-thaw exposure, automatically identifying and quantifying surface damage features (such as fiber exposure area, scaling depth, micro-crack distribution) using deep learning networks and machine learning algorithms. Subsequently, these features are correlated with the number of freeze-thaw cycles, material mix proportions, and actually measured mechanical property losses for model training, establishing a prediction model. Using this model, by merely analyzing surface image information of the material, its damage state and residual strength can be non-destructively predicted, achieving efficient and accurate assessment of ECC freeze-thaw damage.

Neural network-based performance prediction provides a powerful tool for the long-term performance assessment of ECC. Research based on a Backpropagation (BP) neural network established a mechanical property prediction model for recycled powder ECC under freeze-thaw cycles and constant low-temperature conditions. Calculations show that the average relative errors of the established BP neural network prediction models are 1.43% and 1.28%, respectively. Using mass loss rate and relative dynamic elastic modulus as evaluation criteria, the model predicts the maximum number of freeze-thaw cycles that recycled powder ECC with different mix proportions within the experimental range can withstand. This artificial intelligence-based model can account for the nonlinearity of material performance degradation, providing more reliable bases for engineering design and maintenance.

5. Summary

(1) ECC materials exhibit outstanding frost durability. Even after 300 freeze-thaw cycles, the mass loss rate of ECC incorporating PVA or PE fibers can be controlled within 3.5%, and its longitudinal relative dynamic elastic modulus is significantly higher than that of ordinary concrete and mortar. ECC can maintain the multiple cracking failure mode after freeze-thaw exposure. Although its flexural load-bearing capacity decreases with increasing cycles, the reduction is far less than that of ordinary concrete, and deformation capacity remains good. Compounding PE-ECC with FRP mesh can significantly enhance frost resistance, with the maximum load-carrying capacity improved by up to 67.3%.

(2) Freeze-thaw damage originates from the evolution of the microstructure and leads to macroscopic performance degradation. As the number of freeze-thaw cycles increases, the internal porosity of ECC rises, the average pore size increases, and the proportion of capillary pores increases significantly. Simultaneously, micro-crack propagation and bond performance degradation occur in the fiber-matrix interfacial transition zone, which are the main reasons for the reduction in peak stress and elastic modulus of the material at the macroscopic level.

(3) Fiber type, interface treatment, and coarse aggregate significantly influence frost resistance. PE fibers, due to their high strength and hydrophobicity, perform excellently in enhancing frost resistance. FRP mesh composite can provide additional reinforcement. Natural coarse aggregate CA-ECC exhibits durability similar to ordinary ECC, while lightweight coarse aggregate CA-ECC has poorer frost resistance due to high water absorption. Recycled powder ECC may gain strength under constant low temperatures but experiences performance loss under freeze-thaw cycles.

(4) Established damage models provide guidance for life prediction and engineering applications. Image recognition-based machine learning methods and BP neural network models can well describe material performance evolution and enable life prediction. For example, predictions indicate that in northwest permafrost regions, the actual service life of ECC can reach 38 years, and NCA-ECC can reach 41 years, demonstrating its broad application prospects in cold regions.

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