Review

Advances in the Study of Carbon Nanotube Fibers for Enhancing the Crack Resistance of Geopolymers

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Abstract: With increasing societal awareness of environmental protection, the production process of traditional cement has become an area in urgent need of innovation because of its significant carbon emission contributions and generation of industrial solid waste. As a new type of low-carbon cementitious material, geopolymers not only consume less energy and produce fewer carbon emissions but also effectively allow for the reutilization of industrial solid waste, demonstrating its immense potential for further development. However, the inherent brittleness and poor crack resistance of geopolymers limit their structural applications. The crack resistance of concrete can be significantly improved by utilizing self-stressing structures to generate internal stress or by taking prestressed concrete with its unique manufacturing methods. Furthermore, incorporating admixtures to enhance the material's inherent crack resistance presents another viable strategy. Owing to their excellent mechanical properties, carbon nanotube fibers offer new possibilities for addressing these limitations of geopolymers. In this review, the use of carbon nanotubes (CNTs) to enhance geopolymer performance is investigated. A comprehensive analysis of existing studies reveals that the incorporation of CNTs significantly improves the crack resistance and mitigates the brittleness of geopolymers. Optimal overall performance is frequently reported at CNT dosages between 0.12 wt.% and 0.14 wt.%. These findings provide a theoretical foundation for the practical engineering of CNT-reinforced geopolymers and contribute to the development of sustainable construction materials.

Keywords: carbon nanotube fibers; CNT; geopolymer; anti-cracking performance; crack control

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1 Introduction

In recent years, the focus of the construction industry has progressively shifted from quantity to quality, with the demand for building materials that are not only stronger but also better for human health and environmental sustainability. Against the backdrop of global efforts to advance sustainable development, in China, "dual carbon" goals were explicitly set in September 2020—with carbon emissions peaking by 2030 and carbon neutrality being achieving by 2060. As a result, the development of low-carbon cementitious materials and the green transformation of cement-based materials have become key issues in civil engineering materials research. Although ordinary Portland cement (OPC) is extensively used in construction, its production process generates significant carbon emissions. Studies indicate that the production of one ton of OPC results in the release of approximately one ton of CO_2 greenhouse gases [1]. Research has further shown that CO_2 emissions from China's cement industry will peak during the mid-14th Five–Year Plan period, reaching 1.38–1.42 billion tons [2]. Moreover, an annual report from China's Ministry of Ecology and Environment [3] revealed that in 2019, 196 large and medium-sized cities generated approximately 1.38 billion tons of general industrial solid waste. Such waste not only contaminates surrounding environments but also poses health risks through atmospheric transmission [4]. Therefore, the rational recycling and utilization of certain solid waste materials to partially replace cement in specific applications has become a major topic of research.

Geopolymer concrete (GC) can be classified into acid-activated and alkali-activated geopolymer materials, with acid-activated geopolymers characterized by a shorter development history and less extensive research than alkali-activated geopolymers [5]. The concept of alkali-activated geopolymer materials was initially introduced by the French researcher J. Davidovits in 1978 [6]. Although geopolymers have been studied for decades, the reaction mechanisms governing their setting and hardening processes remain incompletely elucidated. Glukhovsky et al. [7] described the reaction as a "destruction–condensation" process, whereas contemporary scholars [8-12] propose a three-stage mechanism: "dissolution–reorganization, orientation–condensation, hardening", which ultimately leads to the formation of a more rigid inorganic polymer material. Compared with OPC, geopolymer materials are more cost effective and exhibit superior acid resistance, high-temperature stability, and chloride ion penetration resistance [13,14]. Furthermore, the production of 1.0 kg of geopolymer mortar generates only 0.18 kg of carbon emissions, which is equivalent to 24% of the emissions from the same quantity of OPC [15]. A study [16] indicates that the energy consumption during the production phase of geopolymer materials is significantly lower than that of traditional cement, at approximately 30% of the energy required for OPC production. By enhancing the reactivity of solid waste, the thermal activation demand can be effectively reduced, thereby lowering the energy consumption further to approximately 10% of that of traditional cement. Moreover, as geopolymer concrete is prepared using one or more types of solid waste rich in silicon and aluminum, different materials can synergistically complement each other [5,17,18]. Therefore, the rational application of such materials can reduce production costs and increase the utilization rate of industrial solid waste. Research [19] has demonstrated that the dense internal structure of geopolymer materials improves their flexural load-bearing capacity, thereby enhancing the performance of prestressed structures. These findings indicate that geopolymer mortar has significant potential for advancing low-carbon building materials and has promising prospects for resource recycling, energy conservation, and emission reduction.

However, the inherent brittleness of geopolymer matrices, as well as the development of microcracks induced by shrinkage-related stress concentrations during hardening, significantly compromise their long-term stability and structural safety [13,20,21]. In high-performance structural engineering applications, increased permeability resulting from cracking accelerates chloride ion ingress and carbonation, substantially reducing the service life of structures. Therefore, enhancing the crack resistance and fracture toughness of geopolymer materials has become crucial for promoting their application in engineering. Various methods are currently employed to suppress cracking, such as the use of prestressed concrete structures, self-stressing concrete structures, or the incorporation of one or more new materials into concrete. In prestressed concrete structures, the deliberate introduction of internal stresses enables the material to develop only fine microcracks or remain entirely crack-free. In self-stressing concrete structures, expanding agents are incorporated to generate internal stress, leveraging the expansion of the concrete itself to inhibit crack formation [22]. Moreover, during the preparation of prestressed concrete, shrinkage and creep may reduce compressive stress and cause prestress loss in tendons, which is also considered a self-stress phenomenon. Several studies [23,24] have indicated that the incorporation of nanomaterials and fibrous materials can significantly improve mechanical and physical properties. The addition of materials such as graphene/graphene oxide, nano-SiO_2, nano-Al_2O_3, bamboo fibers, steel fibers, carbon fiber sheets, and carbon nanotube fibers into geopolymer mortar can enhance the geopolymer's mechanical performance, leading to the development of various advanced composite materials (e.g., structural materials such as CFRPs and GFRPs).

Carbon nanotubes (CNTs), which are nanocrystalline carbon fibers that were first discovered by Iijima et al. in 1991 [25], exhibit great potential in materials science because of their unique molecular structure and exceptional mechanical, electromagnetic, and chemical properties, making them ideal reinforcing components for cement-based materials. Multiwalled carbon nanotubes (MWCNTs) consist of multiple—even dozens of—concentrically stacked carbon fiber layers with an interlayer spacing of approximately 0.34 mm, with diameters ranging from several nanometers to tens of nanometers and lengths extending up to several tens of centimeters [26]. Studies [27–33] have demonstrated that carbon nanotubes increase the electrical conductivity, compressive strength, splitting tensile strength, flexural strength, and crack control capability of cement mortar and ordinary concrete.

In this paper, the effects of multiwalled carbon nanotube fibers on the crack resistance and crack control performance of geopolymer mortar and geopolymer concrete after fiber incorporation are compared.

2 Reaction Principles of Alkali–Activated Geopolymers

Although the precise mechanism of alkali-activated geopolymer formation remains incompletely understood, most researchers currently endorse a three-stage reaction process of "dissolution–reorganization, orientation–condensation, hardening" [1]: when an alkaline solution is introduced into solid raw materials, the pH of the system increases, leading to the breakdown of Si–O–Si, Al–O–Si, and Al–O–Al covalent bonds within the material. The original molecular structures are disrupted, resulting in the formation of low-stability structural units, whereas the hardness of the mortar decreases. The degree of this disruption intensifies with increasing pH. The disrupted species subsequently interact and undergo polycondensation, forming an inorganic polymer with a new three-dimensional network structure composed of $[AlO_4]^{5-}$ and $[SiO_4]^{-}$ tetrahedral units, represented by the chemical formula $Mn\{(SiO_2)z–AlO_2\}_n·wH_2O$.

Notably, geopolymer materials produced by alkali activators inherently exhibit a highly alkaline environment (pH 12–14) and possess a unique three-dimensional network structure composed of $[AlO_4]^{5-}$ and $[SiO_4]^{-}$ tetrahedra. These characteristics may induce irreversible chemical reactions with the surface functional groups of carbon nanotube fibers, thereby affecting their dispersion efficiency and stress-transfer effectiveness [34]. Moreover, the interfacial interaction strength between nanogels (such as the N-A-S-H gel) formed during the geopolymerization process and the carbon nanotube fibers directly determines the extent of improvement in the macroscopic fracture properties of the composite material. Currently, a systematic theoretical model and experimental validation framework for quantitatively explaining the effects of carbon nanotube fibers on the evolution of shrinkage stress, crack initiation threshold, and the behavior of subcritical crack growth in geopolymer materials are lacking.

Several studies [35–38] have indicated that geopolymer materials offer several advantages over OPC in terms of environmental impact, mechanical performance, and durability. The table below presents a comparison of crack resistance in various geopolymers and OPC.

Table 1 Comparison of the properties of selected geopolymers with those of OPC

Property	Eliane et al. [39] (GCC-20FA-RHA_40)	Mohammad et al. [40] (C7, 30% GGBFS)	Mo et al. [41] (Standard GCC)	OPC (Data from PCC_40)
Compressive Strength (MPa)	40	25	–	40
Tensile Strength (MPa)	–	2.6	–	–
Elastic Modulus (GPa)	–	14.58	–	–
Fracture Toughness K_{IC}(MPa·mm$^{1/2}$)	61.18±8.06	27.49	Value not provided (initial fracture toughness increased by 27.8%, unstable fracture toughness increased by 12.74 times).	37.01±4.22
Fracture Energy G_F(N/m)	50.21 ± 6.46	156.06	–	40.36±10.73
Fracture Resistance G_f(N/m)	50.23 ± 6.47	51.85	–	40.37±10.73
Maximum J-Integral (J/m^2)	454.15 ± 56.27	–	–	146.93±12.88
Characteristic Length L_ch(mm)	–	336.59	–	–
Fracture Process Zone Length C_f(mm)	–	72.39	–	–
GF/G_f Ratio	–	3.01	–	–
Performance Improvement vs. OPC	K_{IC} increased by 65.3% G_F increased by 24.3%	K_{IC} increased by 74.5% G_F increased by 284%	Fatigue life increased by 96% Fracture toughness significantly improved	Baseline
Main Testing Method	RILEM TC80-FMT Three-Point Bending Test (a/d=0.5)	Work of Fracture Method (WFM) and Size Effect Method (SEM)	Dynamic Cyclic Flexural Fatigue Test	RILEM TC80-FMT Three-Point Bending Test (a/d=0.5)
Curing Condition	–	80 °C Heat Curing	–	–

Note: "–" indicates that no specific value was provided.

As evidenced by the data in the table, compared with ordinary Portland cement concrete, a geopolymer concrete with appropriate composition demonstrates superior crack resistance, with particularly significant increases in fracture toughness and fracture energy, positioning it as a promising alternative for sustainable construction.

3 Advantages of Carbon Nanotube Fibers as Concrete Reinforcement Materials

Currently, comparative studies on the incorporation of fibers into geopolymer materials are relatively limited. Given the similarities between geopolymers and ordinary concrete in terms of macroscopic mechanical behavior, structural application objectives, and certain testing methodologies, the research findings on fiber-reinforced ordinary concrete can serve as valuable reference data. Therefore, in this section, the results of studies involving the addition of various fibers to ordinary concrete are discussed.

To enhance the properties of geopolymer materials, numerous researchers have incorporated one or more types of materials into geopolymers for performance modification. Among these, carbon nanotube fibers have attracted significant research attention because of their excellent performance-enhancing effects and lightweight characteristics. Existing studies [42–44] indicate that the mechanism through which carbon nanotube fibers improve crack resistance is manifested in two key ways: during the material setting and hardening stage, the carbon nanotube fibers effectively restrict crack development and fill pores; under external loading, they restrict crack propagation through the fiber pull-out mechanism, thereby increasing the overall toughness of the material.

In comparative studies with other fibers [45–47], carbon nanotube fibers demonstrated the following advantages: they reduced the number of harmful macropores while increasing the number of transitional pores to minimize microcrack formation; with their high specific surface area, they provided nucleation sites for surrounding materials, accelerating gel formation; and with their nanoscale dimensions, they enabled the filling of nanosized pores, reducing capillary connectivity and enhancing impermeability. Additionally, carbon nanotube fibers possess excellent electrical conductivity. Researchers [48] have utilized them in thin-film sensing coatings sprayed onto concrete samples, in which the correlation between strain and conductivity changes derived from voltage data allows for the detection of crack distribution and morphology. This characteristic provides new perspectives and research directions for intelligent manufacturing applications involving carbon nanotube fiber-reinforced geopolymer concretes, for example, monitoring internal electromagnetic data to assess crack evolution in structural components.

4 Comparative Experimental Studies

Although carbon nanotube fibers were discovered as early as the 1990s, research on their incorporation into geopolymer materials remains limited. As mentioned previously, geopolymers can be composed of different materials, and their properties vary depending on the added components. Therefore, in the following sections, the influence of carbon nanotube fibers on the crack resistance of geopolymers is examined by categorizing them on the basis of the composition of the geopolymer mortar.

4.1 Fly Ash, Granulated Blast Furnace Slag, Metakaolin and Silica Fume Combinations

The geopolymer mortar in this system consists of four components: fly ash (FA), granulated blast furnace slag (GBFS), metakaolin (MK), and silica fume (SF). Li et al. [49–51] incorporated carbon nanotube fibers at 0.05 wt.%, 0.10 wt.%, and 0.15 wt.% into this geopolymer system and measured the mechanical strength and crack width across groups with different fiber contents. Their findings indicate that sample performance initially improves with increasing carbon nanotube fiber content but declines after a certain point, with the optimal performance observed at 0.10 wt.% fiber content by total mass. Similar results were reported in the experiments conducted by Nejib et al. [52].

4.2 Fly Ash and Granulated Blast Furnace Slag Combinations

The geopolymer mortar in this system is composed of FA and GBFS. Li et al. [42] conducted experiments by incorporating carbon nanotube fibers at 0.05 wt.%, 0.10 wt.%, and 0.15 wt.% into the geopolymer material, along with varying proportions of FA and GBFS. A comparative analysis of the results indicates that when the ratio of FA to GBFS is the same, samples with 0.10 wt.% carbon nanotube fiber content exhibit optimal mechanical performance. Specifically, when the GBFS content is 30% and the carbon nanotube fiber content is 0.1 wt.%, the compressive strength and flexural strength of the samples reach their maximum values. Balamurali et al. [53] further refined the study by testing a wider range of carbon nanotube fiber contents: 0.0 wt.%, 0.02 wt.%, 0.04 wt.%, 0.06 wt.%, 0.08 wt.%, 0.10 wt.%, 0.12 wt.%, 0.14 wt.%, and 0.16 wt.%. Their experimental results demonstrate that at a 0.12 wt.% fiber content, the flexural toughness factor (FTF) peaks, which is a 154.7% improvement over that of the baseline group (0.0 wt.%), indicating exceptional plastic deformation capacity under bending loads. Additionally, the energy absorption capacity (EAC) reaches its maximum, with a 155.9% increase over the baseline, highlighting outstanding energy dissipation performance. Scanning electron microscopy (SEM) analysis revealed that samples with 0.12 wt.% fiber content exhibited the densest internal structure, characterized by uniformly distributed calcium aluminosilicate hydrate (C-A-S-H), sodium aluminosilicate hydrate (N-A-S-H), and calcium silicate hydrate (C-S-H) gels, which effectively filled pores and enhanced interfacial bonding within the matrix. This dense microstructure significantly improves the resistance of the material to microcrack initiation and propagation. However, when the carbon nanotube fiber content exceeds 0.1 wt.%, a noticeable decrease in material stiffness occurs, likely because of fiber agglomeration rather than uniform dispersion, which diminishes the effectiveness of the carbon nanotube fibers.

4.3 *Fly Ash and Metakaolin Combinations*

The geopolymer mortar in this system is composed of FA and MK. Xie et al. [43] added carbon nanotube fibers at concentrations of 0.0 wt.%, 0.06 wt.%, 0.10 wt.%, 0.12 wt.%, 0.14 wt.%, and 0.18 wt.% to the geopolymer material and conducted flexural and compressive strength tests. Their results demonstrated that at a carbon nanotube fiber content of 0.14 wt.%, the flexural strength of the mortar samples reached a peak value of 8.0 MPa, representing a 21.21% improvement over the baseline group. This performance was significantly superior to that of the other groups with doses, indicating exceptional crack resistance capability under bending loads. Scanning electron microscopy (SEM) observations of the material microstructure revealed that the carbon nanotube fibers primarily function through “nanonucleation and mechanical interlocking” mechanisms within the geopolymer matrix. During the material setting and hardening stage, the carbon nanotube fibers effectively restrict crack development and fill pores, whereas under external loading, they restrict crack propagation through the fiber pull-out mechanism. When the carbon nanotube fiber content ranged from 0.12 wt.% to 0.14 wt.%, the nanofibers were uniformly dispersed in the matrix, forming an effective three-dimensional network structure that significantly enhanced the nanoscale structural integrity of the material matrix, reduced the formation of interconnected pores, and resulted in a more uniform pore distribution and a denser structure, thereby leading to optimal crack resistance.

4.4 *Calcined Clay and Granulated Blast Furnace Slag Combinations*

The geopolymer mortar in this system consists of calcined clay (CC) and GBFS. Filazi et al. [44] conducted experiments by incorporating carbon nanotube fibers at 0.0 wt.%, 0.25 wt.%, 0.5 wt.%, 0.1 wt.%, and 0.15 wt.% into the geopolymer material. Their results demonstrated that in a matrix containing 10% CC, samples with 0.5 wt.% carbon nanotube fiber content achieved the maximum compressive strength and flexural strength. The compressive strength increased by 15.6% compared with that of the baseline group at 7 days and by 9.1% at 28 days. SEM image analysis revealed that at a 0.5 wt.% dose, the carbon nanotube fibers were uniformly dispersed within the matrix, forming an effective crack-bridging network. Moreover, energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD) analyses confirmed that a 0.5 wt.% carbon nanotube fiber content promoted the formation of calcium aluminosilicate hydrate (C-A-S-H) and sodium aluminosilicate hydrate (N-A-S-H) gels, increasing matrix density. The dense structure reduced stress concentration and suppressed crack initiation.

5 Conclusions

As environmentally friendly and innovative green materials, one of the primary obstacles to the widespread application of geopolymers lies in their inherent drawbacks in flexural strength and crack resistance. Through a comparative analysis of multiple studies, this paper confirms that incorporating carbon nanotube fibers into geopolymers is an effective method for enhancing geopolymer crack resistance, and the following conclusions are drawn.

(1) Carbon nanotube fibers can significantly increase the crack resistance of geopolymer materials through two main mechanisms: "nanonucleation and mechanical interlocking" and "crack resistance through fiber pull-out". During the material setting and hardening stage, the fibers effectively restrict crack development and fill pores; under external loading, they restrict crack propagation through the fiber pull-out mechanism, thereby improving the overall toughness of the material.

(2) The optimal proportion of carbon nanotube fibers in geopolymer matrices is material-dependent. For geopolymer systems without CC (including FA-GBFS-MK-SF combinations, FA-GBFS combinations, and FA-MK combinations), the crack resistance reaches its optimum value when the carbon nanotube fiber content ranges from 0.12 wt.% to 0.14 wt.%. Within this range, the fibers are uniformly dispersed in the matrix, significantly enhancing the nanostructural integrity of the material and resulting in a more uniform pore distribution and a denser structure. However, in systems containing CC, the optimal content increases to 0.5 wt.%, which may be attributed to the influence of CC on the geopolymerization mechanism.

(3) The effectiveness of carbon nanotube fiber addition follows a distinct "increasing-then-decreasing" pattern. When the content is below the optimal proportion, the dispersion of carbon nanotube fibers improves with increasing content, leading to significant enhancing effects. However, once the optimal proportion is exceeded, the fibers tend to agglomerate, resulting in reduced interfacial bonding strength and a noticeable decline in material performance, thereby diminishing the reinforcing effect.

(4) Microstructural analysis indicates that the incorporation of an appropriate amount of carbon nanotube fibers promotes the formation of gels such as calcium aluminosilicate hydrate (C-A-S-H) and sodium aluminosilicate hydrate (N-A-S-H), effectively filling pores and enhancing interfacial bonding within the matrix. This significantly improves the resistance of the material to the initiation and propagation of microcracks.

With the exception of geopolymers containing CC, the positive effects of carbon nanotube fibers remain largely unaffected by variations in the proportions of different silico-aluminous solid waste raw materials. By rationally controlling the dosage of carbon nanotube fibers, the mechanical properties of geopolymers—particularly their ability to control crack propagation—can be effectively enhanced. This provides theoretical support and a practical reference for the future application of geopolymer materials in more demanding engineering fields, such as those involving prestressed structures.

The simultaneous incorporation of different types of fibers into samples can synergistically enhance their mechanical properties. Therefore, in future studies, researchers could explore the effects of adding different fibers within cost-controllable ranges to develop specimens with superior performance. Additionally, the inherent electrical conductivity of carbon nanotube fibers offers a novel direction for future studies: by investigating the electrical property changes of concrete containing this material under electrification, it may be possible to develop methods for measuring parameters such as crack width in samples.

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