

# Overview of strength aspects of carbon nanotube (CNT)-reinforced cementitious composites

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**Abstract:** Due to the outstanding mechanical, electrical and thermal properties of CNT, many studies have been attempted to develop a CNT-reinforced cementitious composite. It has been reported in the previous studies that the dispersion state of CNT in a cementitious matrix and the bond strength between CNT and hydration products are major factors that affect the mechanical properties of CNT-reinforced cementitious composites. In this paper, the strength aspects of CNT-reinforced cementitious composite considering various CNT contents are reviewed. In addition, the effects of the dispersion technique of CNT in cementitious matrix and bonding characteristics between CNT and hydration products on the mechanical properties of the composites are discussed in detail. Specifically, the correlation between strength enhancement and dispersiveness of CNT is analyzed.

**Keywords:** Carbon nanotube, Cementitious composite, Reinforcement, Dispersion, Mechanical properties.

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## Introductions

Since carbon nanotube (CNT) was discovered by Iijima in 1991, its outstanding mechanical, thermal and electrical properties have been broadly investigated in numerous studies (Salvetat *et al.*, 1999; Zhang *et al.*, 2002; Spitalsky, 2010). In addition, this material exhibits an extraordinary chemical stability resulted from its structural perfection (Tang *et al.*, 2003; Inam *et al.*, 2010). These properties of CNT have broadened the application, i.e., field emission sources, molecular sensors, hydrogen container and composite reinforcement material (Nguyen *et al.*, 2007; De Heer *et al.*, 1995; Liu *et al.*, 1999; Curtin and Sheldon, 2004). In particular, many researches on the fabrication of a CNT-reinforced composite have been extensively conducted, due to its elastic modulus and yield strength of  $10^{12}$  Pa and 20 - 60 GPa, respectively (Nam *et al.*, 2012; Kim *et al.*, 2014).

The studies on the CNT-reinforced composite have started in the field of polymer composite since the late 1990s (Shaffer and Windle, 1999). Attempts to utilize CNT for improving mechanical properties of cementitious composites were initiated in early 2000s (Glenn, 2004). Similar to the fabrication of CNT-reinforced polymer composite, the major challenge to improve the mechanical properties of CNT-reinforced cementitious composite is to ensure appropriate dispersion of CNT in the cementitious matrix (Kim *et al.*, 2014). CNT particles are readily agglomerated due to the van der Waals attraction force (Kim *et al.*, 2016). The CNT agglomerates can act as a defect site in a CNT-reinforced cementitious composite. The applicability of surfactants often used to disperse CNT in polymer composite has been investigated in early

studies on the fabrication of CNT-reinforced cementitious composite (Yazdanbakhsh *et al.*, 2009). However, it was reported that these surfactants inhibited the hydration reaction and deteriorated the composite matrix (Yazdanbakhsh *et al.*, 2009). Furthermore, the characteristics of a cementitious matrix which are clearly different with the polymer matrix can affect the dispersion of CNT (Young, 1988). Therefore, various techniques for the dispersion of CNT in the cementitious matrix have been extensively investigated in previous studies (Konsta-Gdoutos *et al.*, 2010; Collins *et al.*, 2012; Kim *et al.*, 2014; Li *et al.*, 2005).

Other factors that affect the mechanical properties of CNT-reinforced cementitious composite are the added contents of CNT and the bond strength between CNT particles and hydration products (Kim *et al.*, 2014). The CNT contents added to cementitious materials are closely related to the pore characteristics of CNT-reinforced cementitious composite (Nochaiya and Chaipanich, 2011). The bond strength between CNT particles and hydration products is significantly affected by the technique used for CNT dispersion (Li *et al.*, 2005).

In the present study, the strength aspects of CNT-reinforced cementitious composite considering various CNT contents are reviewed. In addition, the effects of the dispersion technique of CNT in a cementitious matrix and the bonding characteristics between CNT and hydration products on the mechanical properties of the composites are discussed in detail. The correlation between strength enhancement and dispersiveness of CNT is also analyzed.

## Dispersion techniques of CNT

### Ultrasonication

Ultrasonication is a representative technique often used to physically disperse CNTs into an aqueous solution (Zou *et al.*, 2015). The line voltage during ultrasonication process is converted to mechanical vibration (Zou *et al.*, 2015). The transfer of the vibration into the aqueous solution is achieved by the probe that creates pressure waves (Zou *et al.*, 2015). During this process, microscopic bubbles are formed and collapsed. This generates countless shock waves which lead local shear on the end of CNT bundles and creates a gap between the particles (Hielscher, 2006; Konsta-Gdoutos *et al.*, 2010; Vaisman *et al.*, 2006). For the fabrication of CNT-reinforced cementitious composite, CNTs are put into water, the solution is then sonicated to disperse them, and the solution is thereafter mixed with cementitious materials (Makar and Beaudoin, 2004). Konsta-Gdoutos *et al.* reported that ultrasonication energy more than 70 Pa was needed to properly disperse CNT (Konsta-Gdoutos *et al.*, 2010). Meanwhile, a serious damage on CNT particles can easily occur, when the period of the treatment is too long (Lu *et al.*, 1996). Therefore, the ultrasonication treatment should be carefully applied.

### Admixtures

Various surfactants have been successfully applied to disperse CNT in a polymer matrix. (Vaisman *et al.*, 2006; Rastogi *et al.*, 2008). However, the surfactants such as anionic sodium dodecyl sulfate (SDS) is not compatible with cementitious matrix. (Sobolkina *et al.*, 2012). Yazdanbakhsh *et al.* reported that the hydration reaction was inhibited by these surfactants, which in turn deteriorated the matrix of the CNT-reinforced cementitious composite (Yazdanbakhsh *et al.* 2009). Furthermore, despite the fact that these surfactants can improve the dispersion state of CNT in aqueous solution, the surfactants can react with the cementitious materials, leading to re-agglomeration of CNT (Yazdanbakhsh *et al.* 2009). Therefore, the applicability of chemical agents often used in cement industry has been investigated to disperse CNT in the cementitious matrix without causing significant adverse effects. Collins *et al.* investigated the applicability of the chemical admixtures such as air entrainer, styrene butadiene rubber, polycarboxylates that have been conventionally used in cement industry (Collins *et al.*, 2012) and reported that polycarboxylate-type superplasticizer was effective to disperse CNT in cementitious matrix, because of its long lateral ether chains leading steric repulsion (Collins *et al.*, 2012). In addition, it was reported that nonionic polyoxyethylene(23) laurylether, the mixture of sodium dodecyl benzene sulfonate (SDBS) and Triton X-100 (TX10), methylcellulose, pluronic F-127 were effective to disperse CNT in the cementitious

matrix without negative effect (Sobolkina *et al.*, 2012; Luo *et al.*, 2009; Li *et al.*, 2005; Parveen *et al.*, 2015).

### Silica fume

Silica fume contributes to the improvement of CNT dispersion in a cementitious matrix (Kim *et al.*, 2014). In a number of studies, the use of silica fume has been adopted to disperse carbon fiber in the cementitious matrix (Fu and Chung, 1996). This material with spherical shape can be inserted between carbon fibers, via ball-bearing effect that can increase the distance between them (Fu and Chung, 1996). Similarly, it has been reported in several studies that silica fume can physically contribute to the improvement of CNT dispersion (Kim *et al.*, 2014). Kim *et al.* observed via SEM image analysis that the added silica fume to CNT-reinforced cementitious composite maintained the spherical shape at 24 hrs and this material introduced into CNT agglomerates, reducing the size of the agglomerates (Kim *et al.*, 2014). It is well known that the pozzolanic reaction of silica fume initiates after 3 days (Poon *et al.*, 2006). That is, the spherical shape of silica fume is maintained and physically contributes to the CNT dispersion, since the dissolution of this material does not occur during mixing and casting process of the composite (Kim *et al.*, 2014).

### Modification of CNT

The surface modification methods for improving CNT dispersion include the addition of polar impurities on the surface of CNT such as OH or COOH end groups, decoration using polymer, wrapping CNT with non-linear polymers, growing polymer chains on the surface of CNT, and annealing (Musso *et al.*, 2009; Szeleifer and Yerushalmi-Rozen, 2005; O'Connell *et al.*, 2001; Bandyopadhyaya *et al.*, 2002; Cui *et al.*, 2004). The formulation of functionalized CNT with acid treatment has been most widely applied in many studies (Li *et al.*, 2005; Cwirzen *et al.*, 2008; Nasibulina *et al.*, 2012; Kang *et al.*, 2015). Li *et al.* modified the surface of CNT using H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub>, thereby creating carboxylic acid groups on the surface of CNT and reported that the modified CNT was effectively dispersed in the cementitious matrix (Li *et al.*, 2005). The carboxylic acid group created on the surface of CNT reduces the contact angle in water, thereby increasing the wettability of CNT. The increase in the wettability of CNT improves its dispersion (Cwirzen *et al.*, 2008). Cwirzen *et al.* reported that the functionalized CNT particles in the aqueous solution with a polyacrylic polymer were well dispersed and remained this condition for 2 months (Cwirzen *et al.*, 2008). However, the dispersion of the functionalized CNT can be affected by the present of Ca<sup>2+</sup> ions. The functionalized groups can react with Ca<sup>2+</sup> ions and lead the re-agglomeration of CNT when

they were mixed with Ca-rich cementitious materials (Nasibulina *et al.*, 2012).

Meanwhile, Musso *et al.* proposed the annealing treatment of CNT to improve the dispersion of CNT in the cementitious matrix (Musso *et al.*, 2009). Chemical vapor deposition (CVD) technique that is most widely used to produce a large amount of CNT with low-cost leads significant lattice defects on CNT particles, which induces curling and agglomeration of them (Musso *et al.*, 2009). This condition of CNT is responsible for poor dispersion of it (Thostenson *et al.*, 2001). Musso *et al.* reported that the annealing treatment reduced the lattice defects on the surface of CNT and straightened it. This led to the mitigation of the agglomeration, improving the dispersion of CNT (Musso *et al.*, 2009).

### Mechanical properties of CNT-reinforced cementitious composites

The mechanical properties of CNT-reinforced cementitious composite are determined by dispersion state of CNT in the matrix and bond strength between CNT and hydration products (Kim *et al.*, 2014). CNT agglomerates entrap a large amount of water due to its high aspect ratio when CNT in a cementitious matrix is poorly dispersed (Kim *et al.*, 2016). The site of the entrapped water forms pores in the composite matrix, after evaporation of the water. These pores are responsible for the reduction in the mechanical properties of CNT-reinforced cementitious composite (Collins *et al.*, 2012).

Meanwhile, the mechanical properties of CNT-reinforced cementitious composite could not be improved, when CNT is well-dispersed in the cementitious matrix. This is attributed to that the techniques applied to disperse CNT can significantly affect the bonding characteristics between CNT and cementitious matrix (Kim *et al.*, 2014). Cwirzen *et al.* reported that the polyacrylic acid polymer surfactant had a negative effect on the bond strength between CNT and hydration products, despite that the use of the surfactant clearly improved the dispersion of CNT in the cementitious matrix (Cwirzen *et al.* 2009). Consequently, the compressive strength of CNT-reinforced cementitious composite in that study was similar to cement paste without CNT (Cwirzen *et al.* 2009). It has been stated in previous studies that the cover of surfactants on the surface of CNT inhibits the bonding between CNT and hydration products (Nasibulina *et al.* 2012; Luo *et al.*, 2009; Konsta-Gdoutos *et al.*, 2010). However, the effect of surfactant on the bond strength can be changed with a type of surfactant used for dispersion of CNT. Luo *et al.* reported that the surfactant mixture of SDBS and Triton X-100 contributed to the dispersion of CNT, which in turn improved the compressive strength of CNT-reinforced cementitious composite by 20.8%

(Luo *et al.*, 2009). Parveen *et al.* also reported that the compressive strength of CNT-reinforced cementitious composite was improved by 19.0 % when Pluronic F-127 surfactant was used for the dispersion of CNT (Parveen *et al.*, 2015).

Up to date, one of the most effective methods for improving the mechanical properties of CNT-reinforced cementitious composite reported in previous studies is to use the functionalized CNT particles, which are formulated with acid treatment (Li *et al.*, 2005; Nasibulina *et al.*, 2012; Musso *et al.*, 2009; Kang *et al.*, 2015; Cwirzen *et al.*, 2008). The surface-modified CNT with functional groups can chemically react with hydration products, improving the bond strength between the CNT and the hydration products. Li *et al.* produced the acid-treated CNT with carboxylic acid group and reported that the chemical interaction between the carboxylic acid group of the CNT and hydration products such as C-S-H and  $\text{Ca}(\text{OH})_2$  occurred (Li *et al.*, 2005). The interaction possibly enhanced bonding between CNT and cementitious matrix, thereby improving the transfer of stress in the composite matrix. Consequently, the mechanical properties of the composite such as flexural and compressive strength increased (Li *et al.*, 2005; Cwirzen *et al.*, 2008; Kang *et al.*, 2015).

Meanwhile, carboxylated carbonaceous fragments (CCFs) that possibly react with hydration products can be formed during acid treatment process (Nasibulina *et al.* 2012). The reaction products between CCFs and hydrates do not contribute to an improvement of the mechanical properties of CNT-reinforced cementitious composites (Nasibulina *et al.* 2012). That is, CCFs on the surface of functionalized CNT inhibits the chemical reaction between the functional group of and hydration products. Nasibulina *et al.* reported that the use of acetone removed the CCFs on the surface of CNT treated with acid. In that study, the compressive strength of the cementitious composite with the functionalized CNT was improved by more than 60% (Nasibulina *et al.* 2012).

The period of ultrasonication can affect the mechanical properties of CNT-reinforced cementitious composites. Sobolkina *et al.* reported that the length of CNT was shortened when the period of ultrasonication is too long, thereby deteriorating bonds between CNT and adjacent C-S-H phases and inhibiting to bridge among void spaces (Sobolkina *et al.* 2012). Konsta-Gdoutos *et al.* reported that long CNT (10-100 nm) was more effective to enhance the mechanical properties of CNT-reinforced cementitious composite compared to short CNT (10-30 nm) (Konsta-Gdoutos *et al.*, 2010).

In some studies, pozzolanic materials of hundreds nm in size were used to improve the bond strength between CNT and hydration products (Kim *et al.*, 2014). Kim *et al.* used a silica fume in the range of 10-500nm as a dispersion agent of CNT (Kim *et al.*, 2014).

In that study, the pozzolanic reaction of the silica fume introduced into CNT agglomerates improved the bond strength. This is possibly attributed to that the silica fume introduced into CNT agglomerates acts as an anchor between the agglomerates and hydration products (Kim *et al.*, 2014).

The added content of CNT can also affect the mechanical properties of CNT-reinforced cementitious composites. It is well known that the percolation threshold value of CNT is in the range of 0.3-0.6 wt%, which significantly decreases the electrical resistivity of CNT-reinforced cementitious composite. In contrast, in previous studies, the appropriate contents of CNT to improve the mechanical properties of CNT-reinforced cementitious composite are approximately 0.05-0.5 wt% (Cwirzen *et al.*, 2008; Nasibulina *et al.*, 2012; Luo *et al.*, 2009; Kumar *et al.*, 2011; Bharj, 2015; Sobolkina *et al.*, 2012; Parveen *et al.*, 2015; Kang *et al.*, 2015; Konsta-Gdoutos *et al.*, 2010; Hamzaoui *et al.*, 2014; Kim *et al.*, 2014; Morsy *et al.*, 2011). Morsy *et al.* stated that CNT wrapped cement grains, leading to the segregation of the cement grains from the reaction process when a large amount of CNT content used. That is, CNT particles attached on the surface of cement grains possibly decreases the bond strength among hydration products, when a large amount of CNT is added (Morsy *et al.*, 2011).

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