



Effect of silicon carbide nanowhiskers on hydration and mechanical properties of a Portland cement paste

Nagilla Huerb de Azevedo*, Philippe J.P. Gleize

Laboratory of Application of Nanotechnology in Civil Construction - LabNANOTEC, Civil Engineering Department, Federal University of Santa Catarina, CxP 476, 88040-900 Florianópolis, SC, Brazil



HIGHLIGHTS

- SiC nanowhiskers (SIC NWS) were mixed with Portland cement pastes at 0.25, 0.50, 1.00 and 1.50 wt%.
- The Portland cement pastes showed that SIC NWS act as nucleation sites and accelerate cement hydration.
- The mechanical properties of Portland cement pastes can be modified with small additions of SIC NWS.

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ABSTRACT

This paper investigates the effect of silicon carbide nanowhiskers (SiC NWS) on cement pastes. SiC NWS were used at 0.25–1.50 wt%, significant increases in the compressive and flexural strength were observed (up to 25% and 75%, respectively). However, an optimum effect plateau seems to be reached at content of 0.25–1.00 wt% for compressive and flexural strength, respectively. The microstructural observations also show the ability of the SiC NWS to act as bridges across the matrix cracks. It was concluded that the enhancements observed are mainly due to the SiC NWS (wt%) and the dispersion has a lesser effect.

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1. Introduction

The use of nanomaterials has been the subject of investigation in vast array of diverse areas, mainly to enhance the properties of traditional materials, for example, to make them lighter and more resistant [1–9].

Silicon carbide nanowhiskers (SiC NWS) have been attracting considerable attention as a novel type of reinforcement due to their excellent properties, including high thermal conductivity (50–120 W/mK), and stability, low density (3.13 g/cm³) and excellent oxidation resistance [4]. Values of 610–660 GPa and 53.4 GPa for the elastic modulus and ultimate bending strengths, respectively, of SiC nanorods with several tens of nanometers of thickness have been reported [5].

In recent years the number of investigations on the use of SiC nanomaterials for the reinforcement of monolithic matrices of metals [6,7], polymers [8,9] and even Portland cement [10] has

been increasing, because this nanomaterial promotes a considerable improvement in the mechanical properties of the matrices to which it is added. In a study by Bahari, Berenjjan and Sadeghi-Nik [10], SiC nanoparticles with different weight ratios (1.25, 2.00 and 3.30 wt%) were added to cement mortars. The authors reported that with 2 wt% SiC nanoparticles the values for the 28 day flexural and compressive strength of cement mortars increased by 11.3% and 6%, respectively.

In general, cementitious materials are brittle and characterized by low tensile strength and strain capacity. The use of discrete fibers results in a more uniform distribution of their remarkable mechanical, chemical and thermal properties in reinforcing cementitious materials. Microfibers can delay the growth of cracks at the microscale, whereas nano-reinforcements will delay the growth of cracks at the nanoscale and halt their propagation to the microlevel. In addition, nanofibers can act as stress transfer bridges during the application of load to cementitious materials, increasing their strength and durability over time. In recent years, carbon nanotubes and carbon nanofibers have emerged as

* Corresponding author.

E-mail address: nagillaazevedo@hotmail.com (N.H.d. Azevedo).

promising candidates for the next generation of high-performance additives for cement-based materials [2,3,11].

Despite the excellent results that nano-SiC can provide as a nano-reinforcement for the mechanical reinforcement of matrices [6–10], it is difficult to obtain a uniform distribution in the matrix, and the adequate dispersion of nanomaterials is crucial for the success of applications. Due to its high specific area and surface energy as well as strong van der Waals and electrostatic forces between the nanoparticles, inhibiting their agglomeration remains a challenge. Many researches have addressed this issue, for instance, through studies on physical methods, where the nanomaterials can be appropriately dispersed via external mechanical forces by applying ultrasonication [2,3,11,12] or milling [13]. Another alternative is to apply chemical methods, using various dispersants [14–16].

This paper investigates the effect of SiC NWS on some of the properties of Portland cement pastes, including the cement hydration, Young modulus, compressive and flexural strengths and water absorption.

2. Materials and methods

2.1. Materials

2.1.1. Cement

The cementitious material used in this study was ordinary Portland cement, CII-F-32, conforming to Brazilian standard NBR 11578 with the addition of 6–10% calcareous filler [17]. Its chemical composition and physical properties are shown in Table 1.

2.1.2. SiC nanowhiskers

The β -SiC nanowhiskers used in this study are a commercial product purchased from Nanostructured & Amorphous Materials, Inc., with the properties given in Table 2.

The SiC nanowhiskers were used as received and the size and morphology were examined by scanning electron microscope (SEM, JEOL JSM-6701F). Fig. 1 shows that the material had a mean diameter of $\sim 1 \mu\text{m}$ and length of $\sim 25 \mu\text{m}$. X-ray diffraction (XRD, Philips X-Pert using $\text{CuK}\alpha$, $\lambda = 15418 \text{ \AA}$) was carried out to characterize the nanomaterial (Fig. 2) and indicated that the powder was mainly β -SiC (3C-polytype).

The chemical composition of the NWS was determined by energy dispersive X-ray detection (EDS, JEOL JSM-6390LV) as follows: Si 46.48 wt%; C 50.99 wt% and O 2.53 wt%.

2.1.3. Superplasticizer

A sodium polycarboxylate superplasticizer, with a relative density of 1.10, was incorporated into all mixes. The content was adjusted for each mix to ensure that all of the pastes had the same workability as the reference paste (without SiC NWS).

2.2. Dispersion of SiC NWS in aqueous medium

The SiC NWS were added to 95% of the deionized water to be used in the mixture and the remaining 5% of the water was added to the sodium polycarboxylate superplasticizer (SP). The suspension was sonicated for 6 min. using a Vibra-Cell 750 Watts sonicator (Sonics & Materials, Inc.) with an amplitude of 50% and intervals of 20 s pulse and 20 s standing. To prevent temperature

Table 2
Properties of SiC nanowhiskers (SiC NWS).

| | |
|-----------------------------------|--------------------------------|
| Free Carbon | <0.05% |
| Diameter: | 0.1–2.5 μm |
| Length: | ≥ 2.0 –50.0 μm |
| Crystal Type: | Beta |
| Decomposition Temperature: | 2700 $^{\circ}\text{C}$ |
| Density (15 $^{\circ}\text{C}$): | 3.216 g/cm^3 |
| Hardness (Mohs): | 9.5 |

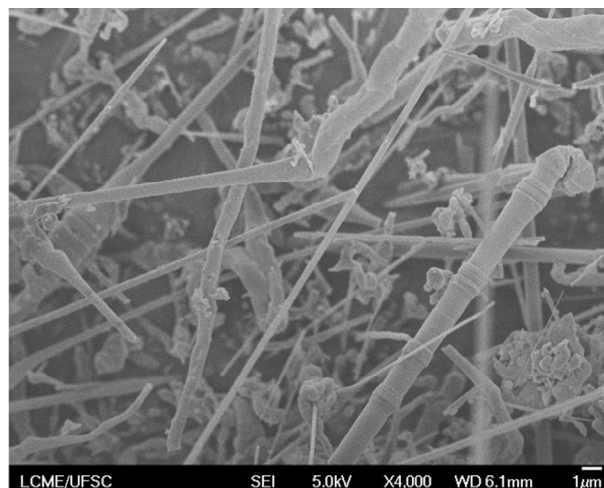


Fig. 1. SEM micrograph of β -SiC nanowhiskers.

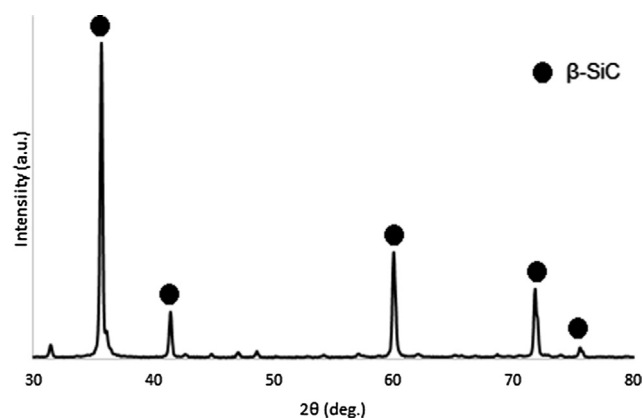


Fig. 2. XRD pattern for as-synthesized β -SiC nanowhiskers.

rising, the suspensions were kept in a water–ice bath during sonication. After this process the solution was allowed to stabilize and was maintained at 23 $^{\circ}\text{C}$ however, it was verified that the SiC NWS started to decant soon after the end of the sonication process.

2.3. Production of the cement pastes

The water–cement ratio (w/c) was 0.4 for all of the mixtures and the mix proportions for the materials are shown in Table 3. For the mixtures with SiC NWS, the appropriate amount of sodium

Table 1
Chemical and physical properties of Portland cement.

| SiO_2 | Al_2O_3 | Fe_2O_3 | CaO | MgO | SO_3 | Specific surface Blaine, $\text{m}^2 \text{kg}^{-1}$ | Compressive strength, 7-day, MPa | Compressive strength, 28-day, MPa |
|----------------|-------------------------|-------------------------|-------|------|---------------|--|----------------------------------|-----------------------------------|
| 18.34 | 4.40 | 2.93 | 60.94 | 4.96 | 2.70 | 3.333 | 35.90 | 42.40 |

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