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Conductive aggregate prepared using graphite and clay and its use in conductive mortar



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HIGHLIGHTS

• A kind of conductive aggregate was proposed and prepared by calcination of ceramic matrix and dispersed graphite powder.

• The resistivity of conductive aggregate mortar was much lower than that of ordinary conductive mortar.

• Contents of conductive aggregate and carbon fiber are recommended for attaining good conductivity and piezoresistivity.

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ABSTRACT

A new type of conductive aggregate was proposed and prepared for the first time by calcination of ceramic matrix and dispersed graphite powder. The prepared conductive aggregate was used in mortar containing carbon fiber, and the specimens' electrical resistivity and piezoresistivity were studied. Results show that optimal graphite powder content in conductive aggregate is 10 wt.%. The use of conductive aggregate in mortar significantly improved its electrical conductivity. The resistivity of conductive aggregate mortar (CAM) was lower than that of ordinary aggregate mortar (OAM) by one to two orders of magnitude, at the same content of carbon fiber. Volume contents of conductive aggregate and carbon fiber had an important effect on the resistivity and piezoresistivity of mortar. A conductive aggregate volume content of 30% of mortar and a carbon fiber volume content of 0.6% of cement paste portion are recommended for attaining good electrical conductivity (the volume resistivity of mortar is less than 100 Ω cm) and piezoresistivity.

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1. Introduction

Conductive cement-based material is a kind of cementitious composite with stable and relatively high electrical conductivity. It can be used for smart engineering structures, deicing of roads and airport, and electromagnetic shielding [1–5]. The volume resistivity of conventional concrete is usually above than $10^6 \Omega$ cm and for practical purposes it can be regarded nonconductive. In most cases, the conductivity of cement-based materials is attained by adding some conductive components into cement paste to form the electrical conductive network. In general, two kinds of conductive components are used. One is carbon series such as carbon fiber, graphite or carbon particles. Carbon fiber used in conductive concrete is typically 5 mm in length, and fiber contents exceeding

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1.0 vol.% are common [6]. The other is metal series such as steel fiber and steel shavings [7–9]. The size of steel fiber used in conductive concrete is generally 10-30 mm long and 0.2-0.5 mm in diameter, and the particle size of steel shavings is 0.15–4.75 mm. In Christopher Tuan et al.'s work, 15-20 vol.% of steel shaving and 1.5 vol.% of steel fibers were added in conductive concrete used for deicing [10]. Some other materials containing carbon or metal such as high carbon content fly ash and steel slag powder were used in cement-based materials as conductive components [11,12]. These conductive components in conductive cementbased materials are located in cement paste part of the composite materials. However, ordinary aggregates in conductive mortar or concrete are non-conductive, which is unfavorable for the electrical conductivity and piezoresistivity of the composite material. This paper provides the first report of preparation of conductive aggregate using graphite powder and clay. Conductive mortar specimens were prepared with the aggregate, and the electrical resistivity and piezoresistivity (change of the electrical resistivity with stress/strain), were investigated.

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2. Experimental

2.1. Raw materials

Clay acquired from a clay pit was used as the matrix material of conductive aggregate and graphite powder produced by Jingziyuan Carbon & Graphite Materials Co., Ltd. was used as the conductive phase in the aggregate. Chemical compositions of clay are listed in Table 1. The carbon content of graphite is above 99%, and its scanning electron microscope (SEM) image is shown in Fig. 1.

Ordinary Portland cement used was from Huaxin Cement Co., Ltd. Chemical compositions of cement are listed in Table 1. PAN-based carbon fiber (with the length to diameter ratio of about 500), ordinary aggregates manufactured by crushing limestone gravels, and prepared conductive aggregates were used to prepare conductive mortar specimens. The particle size distribution of the ordinary aggregates is shown in Fig. 2, and the specific gravity and water absorption ratio are 2780 kg/m³ and 1.85%, respectively.

To improve the dispersibility of carbon fiber and the flowability of mortar, small amounts of hydroxypropyl methylcellulose and polycarboxylic superplasticizer were also used.

2.2. Sample preparation

2.2.1. Preparation of conductive aggregates

Graphite was added into dried clay powder sieved through 150 µm sieve and mixed in a rolling blender for 10 min. The clay was replaced with graphite from 6% to 20% with an increment of 2%. The mixtures were then formed into tablets with the diameter of 35 mm and the thickness of 5 mm (Fig. 3) using a tablet press machine. After that, the tablets were calcined using a tube furnace filled with N₂ to prevent graphite from oxidation during the heating process. The heating process was pursued in several steps as shown in Fig. 4: (1) The tablets were placed into the furnace and were heated from room temperature to 500 °C in 125 min and kept at 500 °C for 10 min; (2) After that, the tablets were heated from 500 °C to 900 °C in 55 min. (3) And then the tablets were heated from 900 °C to 1200 °C in 90 min and kept at 1200 °C for 20 min to ensure enough reaction. After the heating process, the tablets were cooled to room temperature in the furnace. The calcined tablet specimens were used for necessary measurements as described in 2.3. And the calcined tablet specimen at the graphite content of 10% was crushed to particles with particle size distribution shown in Fig. 2 and used as conductive aggregates for preparing conductive mortar. The specific gravity and water absorption ratio of the conductive aggregate are 1760 kg/m³ and 15.2%, respectively.

2.2.2. Preparation of conductive mortar specimens

The water to cement ratio was 0.3, and the volume content of carbon fiber and aggregate of each mixture was listed in Table 2. Before mixing the components together, carbon fiber was pre-dispersed into the water solution of hydroxypropyl methylcellulose and polycarboxylic superplasticizer at room temperature by high power sonication (Shuanghe SH series sonicator, at 1 kW, 20 kHz for 30 min). The suspension of carbon fiber and water solution was put into a JJ-5 mortar mixer and mixed at 285 r/min for 1 min; and then aggregates were put into the mixer and mixed for 2 min. The fresh mortar was cast into 40 mm \times 40 mm \times 160 mm prism mould, and compacted with a ZS-15 mortar compactor at 60 cycle/min for 1 min. After 1 day curing in room environment, the specimens were demolded and cured at the temperature of 20 \pm 2 °C and relative humidity of 95%. For specimens used for electrical conductivity and piezoresistivity tests, the stainless steel probes were pre-embedded during the casting process.

2.3. Properties tests and analytical techniques

2.3.1. Electrical resistivity of conductive aggregate

DC electrical conductivity measurements were performed on the calcined tablet specimens with the two-probe method with a HP4284A type precision LCR meter. For each mixture, three specimens were tested and the average value was calculated.

2.3.2. Electrical resistivity and piezoresistivity of mortar

The electrical resistivity of the mortar specimens and their piezoresistivity under cyclic loading were measured at 28d age with four-probe method described in Refs. [13,14]. The loading system was Instron 5882 mechanical testing system, and the maximum compressive load was 15 kN and the loading speed was 1 kN/s.



Fig. 1. SEM image of graphite particles.



Fig. 2. Grading of aggregates in conductive mortar.



Fig. 3. Photo of uncalcined tablet specimens.

Table 1

Chemical compositions of clay and cement (wt.%).

Chemical compositions	SiO ₂	Al ₂ O ₃	CaO	SO ₃	Fe ₂ O ₃	K ₂ O, MgO, Na ₂ O	Loss
Clay	66.34	16.50	-	-	6.15	3.41	5.52
Cement	22.18	4.72	61.53	2.31	3.45	2.86	1.65

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