Enhanced sensing performance of cement-based composites achieved via magnetically aligned nickel particle network

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Abstract:

Cement-based sensors, consisting of a cementitious matrix filled with electrically conductive fillers, are one of the most promising sensing composites to monitor and track the status of structural systems continuously. This study proposes a new type of cement-based sensor with a unique anisotropic conductive chains activated by magnetically aligned nickel particles in a chain form structure. The piezoresistivity is significantly enhanced along the direction of the chain structure and the gauge factor ranges from 2491 to 3626, providing the sensors with excellent piezoresistivity and sensitivity. In the meantime, the usage of nickel particles drops significantly compared to the composites with randomly dispersed nickel particles. To explore its potential in engineering applications, the developed sensors are embedded in the compression zone of a beam structure. Three-point-bending test result shows that under same condition, the anisotropic sensors exhibits superior piezoresistive performance and higher sensitivity than the isotropic sensors.

Keywords: cement sensors, nickel, magnetic, anisotropic, piezoresistivity, stress monitoring

1. Introduction

Cement-based self-sensing composites/sensors have attracted significant research interest recently [1]. By dispersing conductive fillers in the cement matrix, the composites can convert external loads and environmental changes into electrical signals [2]. Comparing to commercialised sensors, cement-based sensors have the advantages such as long service life, excellent durability and compatibility, high mechanical strength, and low maintenance cost [3].

The sensing ability of cement-based sensor is achieved through the conductive network formed by functional fillers. The most commonly used conductive fillers in the composites are carbon fibre [4], carbon nanotubes [5] and graphite [6]. They are all used as random dispersion in the cement matrix for the development of cement-

based sensors. The gauge factors of these cement sensors with carbon based conductive fillers could range from 30 to 720 [7-11]. The conductive network enables the sensing capacity to detect stress or even damage under external loads and environmental changes [12]. Except the carbon based material, metal such as nickel powder is also used in the development of self-sensing composites. In particular, nickel powder with spiky spherical shape has excellent conductivity due to the field emission effect on the nano tips [13]. Moreover, unlike the fibrous or nano-scale carbon material with tremendous specific surface area and aspect ratio, research result shows that this type of nickel powder does not need special surfactant to remove aggregation and entanglement [14]. However, high demand in particle usage becomes a demerit of the spiky spherical nickel powder. The percolation threshold can be up to 24% in volume fraction [15], leading to the decreasing of mechanical strength and workability of the cement composites.

Due to the ferromagnetic property of nickel powder, this research introduces a method to align the particles into chains using magnetic field during the fresh stage of cement paste. The ferromagnetic particles are driven by the magnetic fields and form chain patterns along the magnetic lines of force. This pattern then is locked in the matrix after the cement paste consolidates and transformed from a spatial 3D network to numerous conductive chains. The electrons then transmit through these tracks unidirectional. The objective of this study is that with the magneto-alignment on the conductive fillers, the piezoresistivity along the chained particle can be greatly enhanced. The proposed method can significantly improve the flowability of the cement past during the sensor development and the mechanical strength of the sensor due to less conductive fillers required. A three-point bending test which aims to put the sensors forward to practical application is then conducted on a beam with both anisotropic and isotropic sensors embedded.

2. Materials and methods

The conductive filler is spiky spherical nickel powder with an average diameter of 5 μ m (model T123, Vale Canada Ltd.). The matrix material is general purpose cement. The fabrication process of the proposed cement sensors is as follows: Firstly, nickel powder is added into water with water reducing agent (a compound of 10% hydroxyethyl non-ionic surfactant and <1 % SDBS anionic surfactant). The mixture is mixed for 10 minutes to ensure homogeneous distribution, followed by cement mixing for 5 minutes (0.4 water to cement ratio). After the paste is well mixed, it is cast into 12.5 mm × 12.5 mm × 25 mm plastic moulds and two copper mesh electrodes in size of 10 mm × 20 mm are vertically placed into the paste. To align the nickel particles into chain structures,

they are transferred to an electromagnet (SBV-130, Yingpu Magnetic Technology Development Co., Ltd, China) immediately for magnetic field treatment. A 0.252 T magnetic field with duration of 30 s is used to align the nickel particles inside the fresh cement. A gauss metre (FH 54, Magnet-physik, Germany) calibrates the magnetic flux density. Samples are cured for 28-day under standard conditions of 20 °C and relative humidity of 95%, followed by a 3-day oven drying at 50 °C. There are various volume contents of nickel powder are developed. For example, 5 vol.% is for piezoresistivity test of cement sensors (the percolation threshold is achieved under this content). 10 vol.% and 15 vol.% are for microstructure observation. 2.5 – 15 vol.% are for resistance measurement and percolation identification. To further understand the spatial distribution of chain pattern, scanning electron microscope (Zeiss EVO LS15, 15 KV) and X-ray computed tomography (HeliScan, microCT) are introduced. The results can provide geometry data of the microstructure of the samples and the visualised images after post-processing.

The piezoresistivity of the sensors is tested by applying cyclic compression with a loading rate of 0.625 MPa/s on Shimadzu AG-X50 universal testing machine. The resistance is recorded synchronously by a multimeter (Siglent SDM3055 5½ Digit Dual-Display Digital Multimeter) while loading using DC and two electrodes method. The change of resistivity (FCR) is calculated according to equation 1. The sensitivity is denoted by gauge factor (GF) which is calculated in equation 2. Two strain gauges are attached to both sides of the samples for balancing strain results and synchronous recorded during loading and unloading.

$$FCR = \Delta \rho / \rho_0 = (\rho_x - \rho_0) / \rho_0 \tag{1}$$

$$GF = (\Delta \rho / \rho) / (\Delta l / l) = FCR / \varepsilon$$
⁽²⁾

Where, ρ_0 is the initial resistivity, ρ_x is the resistivity during loading, ε is the strain.

After the cement sensors are tested, they are embedded in a cement beam for the application of stress monitoring. The sensors with chain pattern configured in the longitude direction of the beam are defined as A1 which are placed far away from loading point. The one near loading point is named as A2. The sensor with chains in the transvers direction of the beam is A3, as shown in Fig. 3a. The isotropic sensors are named accordingly as R1, R2 and R3. Three-point-bending test is carried out using Shimadzu AG-X50 universal testing machine under laboratory condition of 22 °C and 50% relative humidity. In the following sections, the microstructure and sensing performance of the cement sensors with magnetically aligned nickel particles are examined before embedded into

a beam. During the bending test, the piezoresistive performance of these sensors are investigated and the sensing behaviour is evaluated by varying stress magnitude and position.

3. Results and discussions

3.1 Morphological Characterization

As illustrated in Fig. 1a, firstly, nickel powder is aligned by the 0.252 T magnetic field generated from an electromagnet, forming clear short chian structure in the cement matrix. Fig 1b and 1c show that the length of each chain ranges from 20 µm to 30 µm under 5 vol.% particle concentration, obtained from CT and scanning SEM results. With the increase of particle content till 10 vol.%, the particle chain length increases up to 2 - 3 times. The length increases with the rising of magnetic field flux intensity. For instance, the samples with 10 vol.% nickel particles have average chain length of 50.71 µm, 85.29 µm and 105.1 µm under 0.085 T, 0.167 T and 0.252 T magnetic field strength, respectively, as shown in Fig. 1d, 1e and 1f. The discontinuity of the particles in each chain and the gaps between chains provide space to form conductive path under loading. In this way, the electron tunnelling effect appears to be enhanced. The transmission paths of electrons are clear and direct and subsequently the piezoresistive performance. For instance, within an elastic range, the chains are shortened by external compression and recover to the original position after unloading.



Fig. 1. a) Schematic illustration, b) CT scanning, c) 5 vol.%, particle concentration SEM images of ferromagnetic particles alignment under 0.252 T magnetic field and the increment of chain length with the rising of magnetic field strength from d) 0.085 T, e) 0.167 T to f) 0.252 T in the composites with 10 vol.% particle content.

3.2 Anisotropic piezoresistive property of sensors

In Fig. 2b, the FCR of the sensors with magnetically aligned nickel particles are -31.5% and -1.7% when the conductive chains parallel and perpendicular to loading, respectively, showing significant anisotropic

piezoresistivity. The FCR of the sensor with random particles is -5.1%, sitting in-between of the two. The distinction of FCR between the two directions is resulted from the patterned conductive chains. Electron tunnelling effect mainly takes place in the chians and insulated by the cement matrix between the chains. Thus, the electrical resistivity and percolation threshold both decrease significantly (Fig. 2a). The FCR is 6 times higher than the sensors with isotropic network, showing an excellent piezoresistive performance. The anisotropic sensors may achieve similar sensing performance under a much lower filler concentration, which significantly reduces the costs in fabricating such sensor. In the meantime, mechanical strength is is greatly improved due to the drop of nickel content. To be more specific, the compressive strength has a decreasing tend with the rising of particle concentration. As shown in Fig. 2d, samples with 5 vol.% particles present reasonable strength, approximately 50 MPa. On the contrary, 15 vol.% group drops the strength significantly to 40 MPa below and may not have application value. The strength difference between random and patterned is not evident. Therefore, samples with aligned nickel particles in lower content have the advantages of significant piezoresistivity and sensitivity. Simultaneously, the mechanical strength is guaranteed.



Fig. 2. a) Electrical resistivity of nickel powder and cement-based composites with random and patterned conductive network, b) FCR of the composites under cyclic compression, c) The maximum gauge factor in the testing group of the anisotropic sensors. d) Compressive strength of cement and nickel composites with particle

content from 5 vol.% to 15 vol.%. (R-sample with random dispersed fillers, Pa, Pe-sample with chain pattern parallel and perpendicular to the direction of stress, respectively.)

3.3 Sensitivity of the sensors

The sensitivity test is carried out in an elastic range (0 - 6 MPa) to ensure a linear material deformation and reversible change of electrical resistance. Two strain gauges are attached to the opposite sides of the samples to obtain balanced results. The GFs of the sensors with aligned nickel particles range from 2491 to 3626 (Fig. 2c). The GF values of random group are between 364 and 545 against the strain range from 0 to 80 μ ε. The average GF reading of the magnetically aligned sensors is 6.3 times of that of the sensors with random distributed nickel fillers. It is also 5 to 10 times of the samples with carbon based conductive fillers mention in the introduction section, presenting giant factor (GF > 500) feature [16] and high sensitivity. The high elastic modulus of nickel equips the composites with low deformation under compressive loading. The high FCR is a result from the effective particle alignment and conductive paths. It is also proved in previous studies, the aligned nickel chains network induced by magnetic field could improve the sensitivity in the direction of chain structure significantly [14].

The FCR of the sensors exhibits liner property in the elastic range as shown in Fig 2b. When it steps in the end of elastic stage, a slight non-linear relationship can be found between the stress and FCR. This may due to the fragile property of the cementitious material. Micro cracks gradually form and propagate inside the composites and results in irreversible changes. Regarding the repeatability, all the results are from group test. In each group, three copies of the samples presents similar results and has unique piezoresistive performance from other groups with different parameters.

3.4 Piezoresistive performance of the sensors embedded into the beam

In this study, the potential of the proposed sensor in stress monitoring is verified by planting the sensors into compression zone of a cement beam. The experiment results show good applicability of the proposed sensor in monitoring small stress and strain. To be more specific, the FCRs decrease with loading and rise reversibly with unloading in the compression zone of the beam. Although the bending stress is in a low state (< 2 MPa), the piezoresistivity of the sensor is sensitive to detect such low stress. As shown in Fig. 3b, taking sensor A2 as an example, the FCR drops 23 times, from -25% to - 1.1% after embedded into the beam. The reason may due to the decrease on the strain which is from 120 $\mu\epsilon$ to 10 $\mu\epsilon$ under same stress before and after the implant. Despite the

decrement, the sensors reflect the stress correctly under different stress magnitude. For instance, the FCR of sensor near the loading point (A2) is greater than that (A1) away from the centre. Moreover, the FCRs of sensors A1 and A2 with aligned fillers are 10 times higher than the random dispersed group (R1 and R2 in Fig. 3c). While before the implantation, the difference is a lesser 5 times. Notably, isotropic sensor, such as A3, is not subjected to the placement whether it is perpendicular to or parallel to the compressive stress. However, anisotropic piezoresistive behaviour can be observed for the sensors A3 with aligned nickel particles. This may result from the different conductive network inside the composites. The isotropic one is functional regardless the direction of loading stress. On the contrary, the higher FCR values obtained in the anisotropic sensor denote the enhancement along the direction of conductive chains in an anisotropic network. Additionally, for the sensors with anisotropic property, majority of the particles take part in the conductive chains. Same piezoresistivity can be achieved under a much lower filler concentration compared with the isotropic sensors.



Fig. 3. Three-point-bending test of the beam embedded with cement sensors. a) The layout of sensors in the compression zone and piezoresistive performance of sensors with b) Aligned nickel particles and c) Randomly dispersed particles.

4. Conclusions

This paper proposed a novel development of anisotropic cement-based sensor by utilising magnetic field to align the conductive fillers. The sensors with magnetically aligned nickel particles present the features of significant enhanced piezoresistivity and gauge factor in the direction of formed chain-structure. Compared to isotropic sensors, a high sensing performance can be achieved under a much lower particle concentration due to the advancement of percolation threshold intervened by magnetic field. In addition, the usage of filler material is considerably reduced. Subsequently, the mechanical strength can be guaranteed. In the three-point-bending test, much higher FCR value was achieved in the sensor with aligned particles parallel to the direction of stress than the sensor with random particles.

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