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Original Article

Synergistic effects of processing and nanofiber reinforcement on the mechanical and ferroelectric performance of geopolymer matrix composites

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ABSTRACT

This study involved the evaluation of mechanical and ferroelectric properties of a new class of nanofiller infused inorganic polymer (geopolymer, GP). To evaluate the mechanical performance, compressive strength and fracture resistance of neat and nanofillers infused GP were studied at various treatment temperatures. It was found that, addition of 5 vol% alumina nanofiber (ANF), increased compressive strength and modulus by over 30% and 60%, respectively, while it increased fracture toughness (K_{IC}) by over 60% compared to the baseline specimens. Simultaneously, ferroelectric properties were investigated at various treatment temperatures (250 °C, 650 °C and 870 °C). Remarkably, higher ferroelectric hysteresis was observed with the GP treated at 870 °C and remnant polarization increased with the addition of alumina nanofiber. Scanning Electron Microscopy confirmed that neat materials are composed of particles embedded into the poly-condensed matrix, where particle nature existed until the treatment temperature reached above 870 °C. X-ray diffraction analysis suggests that, baseline geopolymer started becoming crystalline while the particle nature gradually disappeared with heating at or beyond 870 °C. The bonding between the polymer and alumina nanofiber tends to be stronger with increasing treatment temperature. The increase in K_{IC} with the addition of 2 vol% and 5 vol% alumina nanofibers (ANF) is due to homogeneous dispersion of high interfacial strength nanofillers, which essentially create strong crack bridging and crack deflection effect. The increase in ferroelectric hysteresis is potentially due to the formation of hierarchical order and domain reorientation of the materials.

1. Introduction

Geopolymers are new materials with excellent fire- and heat-resistant properties. The properties and uses of geopolymers are being explored in many scientific and industrial disciplines and engineering process technologies. Various studies have been performed to investigate the performance of geopolymer as the high temperature coatings [1] and the replacement

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of ordinary port-land cements [2]. However, minimum efforts have been made to establish these materials as the matrix of a continuous fiber reinforced composites. This effort has attempted to focus some mechanical and electrical properties as demanded by high temperature continuous fiber reinforced composites.

Conventional ceramic matrix composites require high temperature powder processing, which usually causes lack in wetting and interfacial reactions between constituent materials [3]. Inorganic materials derived geopolymer offer the potential to bridge between mechanical performances as a matrix material for high temperature capable fiber reinforced composites. Simultaneously it has potential to possess dielectric and ferroelectric behaviors upon certain processing and treatment conditions. This polymer can be readily processed at low temperature with similar technique as organic polymers but can be applicable above the upper use temperatures of conventional polymers [4]. Unfortunately, low toughness and high shrinkage often limit the mechanical performance of this material and restricts it from electronics applications. Previous publications have suggested that process optimization have the potential to improve compressive strength and modulus [5]. However, processing itself does not have enough potential to increase fracture toughness and bending strength. Nanofillers reinforcement has proven to increase the toughness of conventional polymer and monolithic ceramic materials [6–8]. Only few literature [9–11] suggested the improvement in toughness of geopolymer with nano-reinforcement.

Nanofillers in the conventional ceramic materials suffer from thermal degradation and mechanical distortion, which impedes the expected improvement in mechanical performance. Low temperature processing of inorganic polymer with nano-reinforcement can overcome this problem and are therefore expected to improve mechanical performance, especially the fracture toughness. Alumina nanofibers (ANF) have superior tensile strength and thermal stability than conventional fillers of organic polymer composites. Due to inorganic frame work, ANF also disperses well into geopolymer. Therefore, ANF can be potentially applied in order to toughen geopolymer.

High temperature capable ferroelectric materials have potential to the energy storage devices. As previously reported [12], several high temperature capable materials have shown their ferroelectric characteristics at various temperatures. The ferroelectric (polarization-electric field) hysteresis, is a defining property of ferroelectric materials [13]. It has been studied intensively due to the potential applications of ferroelectric thin films in nonvolatile memories [14]. The switching of polarization at high electric field is the main idea behind the applications of this type. The value of the switchable polarization (the difference between the positive and negative remnant polarization, $PR-(-PR)$) can be analyzed from the hysteresis. But, the switching characteristics are still not well agreed as the switching depends on the nature of the ferroelectric material itself, types of electrodes used, thickness of the ferroelectric, temperature, field profile, number of field cycles, and many other parameters [14–17]. However, the isolation of dielectric constant is necessary to isolate from the ferroelectric behavior if there is any dielectric properties existent. This can be measured by dielectric loss calculation. Many researchers have found that [18–20] an ideal dielectric material should have homogeneous and constant dielectric constant at the applicable frequency range. Most of the reported materials are crystalline in nature and therefore the extent of ferroelectricity was not necessarily investigated in the amorphous state. The current study highlighted the nature of polarization transformation with the transformation from amorphous to crystalline state. Investigating the polarization versus electric field (*P*–*E*) of a ferroelectric material yields a hysteresis loop, which allows us to estimate the remnant polarization (P_r) and coercive field (E_c). The polarization behavior and the coercive field of the consequent ferroelectric material vary with the applied voltage frequency.

High temperature treatment [12] and nano-reinforcement [21] are suggested to result in transformation to crystalline phases which possess the property of ferroelectricity at the high temperatures. Investigating mechanical as well as ferroelectric properties therefore explores the potential to use this material in high temperature energy storage devices. The current investigation is therefore focused on understanding the underlying processing and toughening mechanism of inorganic polymer through the knowledge developed from some advanced techniques such as phase transformation and ferroelectric hysteresis.

In the current investigation, a systematic study has been performed on this new class of geopolymer to investigate how the changes in compressive strength and fracture toughness with the heat treatment is related to the changes in ferroelectric property, such as hysteresis loop from the viewpoint of domain reorientation. The current investigation explores the scope of annealing of the material at a reasonably high temperature with and without nano-reinforcement on the ferroelectric phase transformation. The synergy between the heat treatment and nanofiller reinforcement were investigated throughout this study. The ferroelectric properties arising from domain reorientation are related to nanoparticles infusion. The electric displacement (polarization) versus electric field (*P*–*E*) hysteresis loops of neat and nano-reinforced geopolymer were studied at 500 Hz frequency. The mechanism of fracture resistance of nano-reinforced geopolymer has also been explored through this study.

2. Experimental

2.1. Materials

Base material of this investigation is MEYEB, which is a polysialate geopolymer in a suspension form containing various chemicals including potassium silicate, aluminum oxide, silicon dioxide, aluminum phosphate and water. These chemicals are mixed up in the proprietary proportions of Si/Al, K_2O/SiO_2 and H_2O/K_2O, which take place in the poly-condensation reaction during cure process. Nanofillers of this investigation are alumina nanofiber (ANF) and alumina nanoparticle (ANP), which are supplied by ANF technology (Estonia). Chemical configuration and hydrophilic nature of these nanomaterials allow them to disperse well into inorganic polymer, geopolymer in a large volume fraction.