

Remarkable mechanical enhancement achieved by interfacial strengthening of organic/inorganic/fiber composites



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HIGHLIGHTS

- Organic/inorganic interpenetration network is established in composites.
- Interface of fiber/inorganic matrix is strengthened by continuous organic phase.
- The flexural strength of organic/inorganic/fiber composites is greatly improved.

ARTICLE INFO

Article history:

Received 20 September 2016

Received in revised form 11 February 2017

Accepted 7 March 2017

Available online 19 March 2017

Keywords:

Polymer

Fiber

Interpenetration network

Interfacial strengthening

Flexural strength

ABSTRACT

In this study, a series of organic/inorganic composites were fabricated by melt-mixing ultra-high molecular weight polyethylene (UHMWPE) powder with a mixture of magnesia, potassium di-hydrogen phosphate and water. The microstructure and chemical composition of the composites were characterized by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). Three point bending tests showed that the flexural strength and toughness of the composites were improved with the increase of polymer content. The flexural strength of the composite with 25.0 wt% UHMWPE was 7.7 MPa, and can be dramatically enhanced to 21.1 MPa by incorporating only 0.1 vol% of oriented thermotropic liquid crystalline copolyester (TLCP) fibers. The improvement is due to the enhanced interfacial bonding between the polymer and fibers, which was much stronger than that between the inorganic phase and fibers, making the fibers adequately contribute to the mechanical strength of the three-component composite. Our results demonstrated that the interfacial strengthening between fiber and inorganic matrix achieved by interpenetrated organic phase helps to establish a strong composite system.

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1. Introduction

The scope of organic/inorganic composite applications can be greatly expanded by the enhancement of mechanical properties. Various rigid inorganic particles, such as calcium hydroxide nano-spherulites [1], carbon nanotubes [2,3], graphene [4–6], and clay [7,8] have been implanted into organic polymer matrix to improve the mechanical strength, modulus, electrical and thermal properties. Meanwhile, the toughness of inorganic matrix such as cement can also be modified by adding organic phase [9,10]. Among these composites, structure and interface play important roles in the mechanical enhancement. Well-designed structure enables each component adequately contribute its merits to the composite. For example, as reported in our previous work, with

unique structure and strong interfacial bonding, the toughness of polymer matrix can be enhanced 200 times by adding only 40 ppm calcium hydroxide [1]. For some biological materials, such as abalone shell [11] and nacre [12], although the interfacial bonding between organic and inorganic phase is weak, extremely high mechanical strength can still be achieved due to the interpenetrating organic/inorganic networks in the composites.

The greatest stumbling block to the large-scale production and commercialization of strong composites is the dearth of cost effective methods for controlling the assembling of organic and inorganic phase. Due to the big difference of surface tensions [13–15], phase separation and aggregation usually occur in organic/inorganic composites, which negates any benefits associated with the complementary properties of the two phases. There is a critical need for establishing processing techniques that are effective on the composite structure yet are applicable to macroscopic processing. In this study, we have therefore developed a simple method to

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fabricate organic/inorganic/fiber composites with the interface between fiber and inorganic matrix strengthened by interpenetrated polymer phase. We believe that it could offer innovative insights into the design of strong composite system for industry application.

2. Experimental details

2.1. Raw materials

Dead burned magnesia annealed at 1200 °C for 8 h was obtained from Jinan, Shandong province. The phosphates used here were potassium di-hydrogen phosphate, anhydrous, purchased from Chemical Reagents, UNI-CHEM. Ultra-high molecular weight polyethylene (UHMWPE, with a molecular weight of $M_w = 3,500,000$ and particle size around 50 μm) powder was purchased from Ticona Co. Ltd.. Vectran, a kind of thermotropic liquid crystalline copolyester (TLCP) fiber, with diameter of 23 μm , was synthesized based on the previous work [16,17]. The tensile strength and Young's modulus of the synthesized Vectran fiber were measured to be 3 GPa and 100 GPa, respectively. The Vectran fiber has a density of 1.4 g/cm^3 , and a melting temperature of 300 °C.

2.2. Methods

Magnesium potassium phosphate ceramic (MPC)/UHMWPE composites with UHMWPE content of 15, 20, 25, 30 and 50 wt% were prepared by mixing dead burned magnesia and potassium di-hydrogen phosphate powder (with a mole ratio 4:1) with molten UHMWPE. The specimens with the size of 10 × 30 × 120 mm were fabricated by hot-pressing the composites at 40 MPa and 180 °C. Water was added into the mold before hot-pressing with an approximate water/magnesium potassium phosphate ratio of 0.2. To fabricate the MPC/UHMWPE/Vectran fiber composite containing ordered fibers, 0.1 vol% of Vectran fibers with a length of 120 mm were directionally arranged along the length direction of the specimens before the hot-pressing procedure. Each specimen contained 12 layers of fibers in the mixture of magnesium potassium phosphate/UHMWPE powder, and each layer contained about 8 fibers. The fibers were tiled in the sample mold to form one layer first, covered by the mixture powder. Another fiber layer was fabricated on the surface of the powder, and repeated for 12 times. Flexural tests were conducted at 25 °C using an Instron 3365 testing machine, with a span between the two clamps of 40 mm and a crosshead speed, 0.5 mm/min. For each test, 5 specimens were tested and similar curves were obtained, and a representative curve was selected for further analysis.

3. Results and discussion

In the experiment, we used magnesium potassium phosphate ceramic (MPC) as the continuous inorganic matrix, due to its high early age and long term strengths [18–21]. Ultra-high molecular weight polyethylene (UHMWPE), a hydrophobic polymer with good mechanical strength and toughness [22,23], was selected to improve the interface between inorganic MPC matrix and the implanted fibers. To fabricate the organic/inorganic interpenetration network, a small amount of water was added into the mixture of UHMWPE, magnesia and phosphates in the mold, and the mixture was then hot-pressed immediately. During the melt-mixing process, a continuous polymer phase formed first, giving the initial strength of the specimen. Afterwards, free water was evaporated at such a high processing temperature, but the water trapped in the powder voids is sufficient for the formation of MPC. The reaction

was accelerated by high temperature, which allowed the completion of continuous inorganic phase before the water was fully evaporated.

Fig. 1 shows flexural stress-stain curves of the MPC/UHMWPE composites with different polymer weight content. The relevant mechanical properties are listed in Table 1. It is clear that both the flexural strength and toughness of the composites were improved with increase of the polymer content. The stress-strain curve suddenly drops after cracking occurs in the original MPC specimen. However, with the addition of polymer, the specimen shows strain softening behavior after the first crack, which demonstrates that the polymer phase contributes to the fracture strength and toughness of the composite. After cracking occurred in inorganic phase of the MPC matrix, the polymer phase is still connected the fracture interface, providing the toughness of the composite. A noteworthy increase of flexural strength from 3.2 to 14.1 MPa can be achieved with an indistinct crack point when the polymer content reaches 30.0 wt%. The specimen did not crack even at a strain of 8.0%, and a high toughness value calculated by I_5 index was reached (Table 2). For the specimen containing 50 wt% UHMWPE, the flexural stress remains constant with increase of strain, and the curve had a similar shape to that of many metal materials. The stress-stain curves reveal that the polymer forms continuous interpenetrating phase, and contributes to the mechanical strength and toughness of the composite.

SEM with energy dispersive X-ray spectroscopy (EDX) tests were applied to characterize the morphology and structure of the MPC/UHMWPE composite. As shown in the red boxes of Fig. 2a, the polymer phase with a curved shape is clearly observed in the MPC matrix, indicating the deformation of the polymer during the composite fracturing process. Relevant elemental maps of carbon (green) and calcium (red) by EDX show the distribution of the polymer in the MPC matrix. The polymer distribution is not strictly related to the morphology shown in Fig. 2a due to the rough fracture surface. The polymer phase represented by carbon (green), dispersed irregularly at the fracture surface (Fig. 2b), with the size similar to that of the original polymer powder (particle size around 50 μm). We can infer that during the hot-pressing process, the polymer powder was melted, and aggregated due to surface tension. Thus a continuous polymer phase formed in the channels of the MPC matrix. Although the interaction between the polymer phase and the MPC matrix is weak, the polymer can still contribute to the mechanical strength due to the interpenetrating network formed. Moreover, because of the low surface tension, the polymer

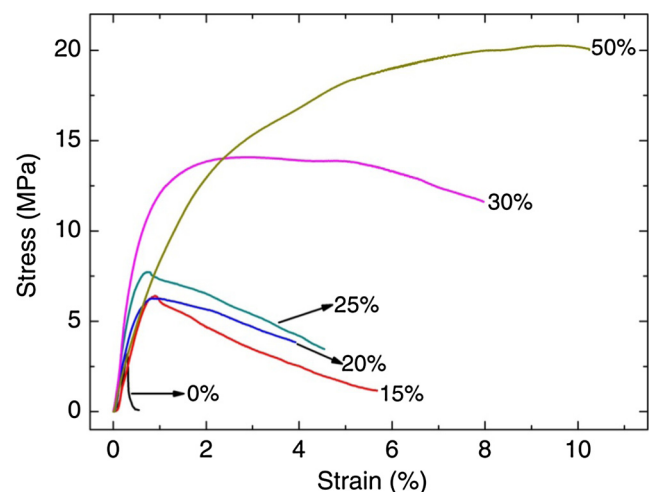


Fig. 1. Flexural stress-stain curves of MPC/UHMWPE composites with different polymer contents.

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