



Full Length Article

Preparation and characteristic of the fly ash cenospheres/mullite composite for high-temperature application



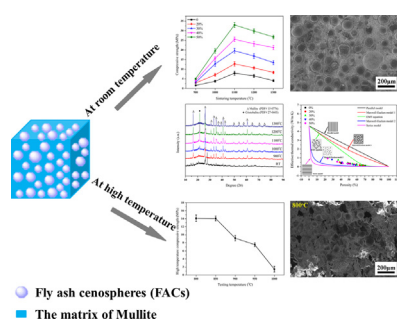
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GRAPHICAL ABSTRACT



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ABSTRACT

Fly ash cenosphere/mullite (FACs/M) composites with excellent high-temperature resistance were prepared through a tert-butyl alcohol gelcasting process, using the mullite and FACs as the matrix and the filler, respectively, and the matrix of mullite was derived from phase transformation of the raw material of kaolin. Results show that the FACs/M exhibited low density and high specific compressive strength and the thermal conductivity was distributed over in the range of values predicted from Maxwell-Eucken model 2 and Effective Medium Theory (EMT) equation. Exponent m describing the relationship between compressive strength and porosity was 2.37. Young's moduli of the composites were lower than that predicted from the Hashin-Shtrikman model. The high-temperature testing results revealed that the FACs/M composite exhibited high compressive strength and the 50% FACs/M composite displayed compressive strength of 14.11 MPa after testing at 800 °C. The FACs/M composites with excellent mechanical properties both at room and high temperatures may possibly be used in the deep sea and other fields in the near future.

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1. Introduction

Syntactic foams (SFs), one type of lightweight composites with closed-pore structure, are prepared by mechanical mixing of the filler of hollow glass microspheres (HGMs) with the matrix of polymeric resin, exhibiting low density, high specific strength, high stiffness, and low moisture absorption [1–3]. They are potentially used as the buoyant materials in deep sea, the sandwich composite in aerospace and other structural materials in a certain field [4,5]. However, as the main component of submarines, the temperature resistance of SFs is greatly affected by the high-temperature invasion during underwater torpedo or missile launch process. Therefore, we need the materials with excellent temperature resistance to reduce the detrimental effects. In the current research, much work concentrated on the mechanical and physical properties of SFs, as studies on improving their temperature resistance were rare. In a typical two-phase system, SFs are composed of the matrix of epoxy resin (EP) and the filler of HGMs, whose capacity of temperature resistance depends mainly on their composition. EP, a type of organic compound with relatively low molecular weight, displays weak temperature resistance, while HGMs, composed of the soda lime-borosilicate glass (BG), exhibits pretty temperature resistance. The previous study showed that the prepared hollow glass microspheres/borosilicate glass (HGMs/BG) composite can only be used at a low or intermediate temperature ($< 600\text{ }^{\circ}\text{C}$) [6]. To improve temperature resistance of the buoyant material and be better used at high temperature, we need to develop a type of temperature resistant materials.

Fly ash cenospheres (FACs) are one type of lightweight particles in fly ash, produced from coal-fired power plants [7–11]. As an industrial solid waste byproduct, its emissions have increased annually with developments in the power industry. In 2015, annual fly ash generation in China was estimated to be 580 million tons [12], a large amount of which were disposed and stacked in many locations, causing serious environmental pollution. Owing to its pozzolanic characteristics, fly ash is used as (i) corrective raw material in the Portland cement industry and (ii) in the design of lightweight composites [13–15]. In recent years, some researchers [16] attempted to use FACs as filler to prepare porous and lightweight materials for high-temperature applications, due to their excellent high-temperature resistance and physical properties.

Kaolin clay is a hydrous aluminosilicate ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) [17] and an essential resource in manufacturing porcelain and ceramics [18,19] and the production of paper, pigments, and filler. Kaolinite is the major mineral component of kaolin, which usually contains quartz

and mica and also, albeit less frequently, feldspar, illite, bauxite, zircon, rutile, kyanite, and halloysite [20]. When the sintering temperature was above $1050\text{ }^{\circ}\text{C}$, the phase of kaolinite transferred to the mullite and cristobalite. It is known that the mullite was usually suitable for high-temperature application, due to a high melting point of $1840\text{ }^{\circ}\text{C}$. Therefore, the prepared composites with the kaolin as the raw materials could be used in high-temperature environment. Chen et al. [21,22] prepared the kaolin ceramic for use as starting materials and discussed the phase and morphology of the ceramics. If the FACs and mullite were served as the filler and the matrix in buoyant materials of the HGMs/BG composite, respectively, then the temperature resistance would be largely improved. What is more, studies on improving temperature resistance of the HGMs/BG composite are rare. Therefore, to improve the temperature resistance of the buoyant materials and be used under high-temperature circumstance, this work is to prepare a fly ash cenosphere/mullite (FACs/M) composite with excellent temperature resistance using FACs and mullite as the filler and matrix, respectively. Moreover, mullite was derived from the phase transformation of the starting materials of kaolin. The effect of FACs content on the mechanical and physical properties of the composites at room and high temperatures will also be discussed.

2. Experimental

2.1. Raw materials

Commercially available FACs (Shijiazhuang Thermal Plant, Hebei, China) and kaolin (Tianjin Kemiou Chemical Reagent Co., Ltd., China) were used as starting materials. Figs. 1 and 2 show the scanning electron microscopy (SEM) images and phase composition of the FACs and kaolin, respectively. It shows that the FACs was composed mainly of the three elements of the Si, Al, and O, and the few elements of Fe, Ca, Ti, and K; while the phase composition of the FACs was mullite (PDF# 15-0776) and quartz (PDF# 46-1045). Similarly, the element of Al, Si, and O formed the main component of the kaolin and still slightly alkali-earth metal element such as K, Ca, Mg, and Na, was also included. XRD pattern indicates that its phase composition was mullite (PDF# 15-0776). Table 1 presents the physical properties of the FACs [23]. Tert-butyl alcohol (TBA), acrylamide (AM), and N,N'-methylenebisacrylamide (MBAM) were used as the solvent, monomer, and crosslinker, respectively. The procedures of preparing premixed solution can be found in a previous study [24] and the ratio of the TBA:AM:MBAM was 100 ml:20 g:2 g. To obtain homogeneous slurry, citric acid (CA) was

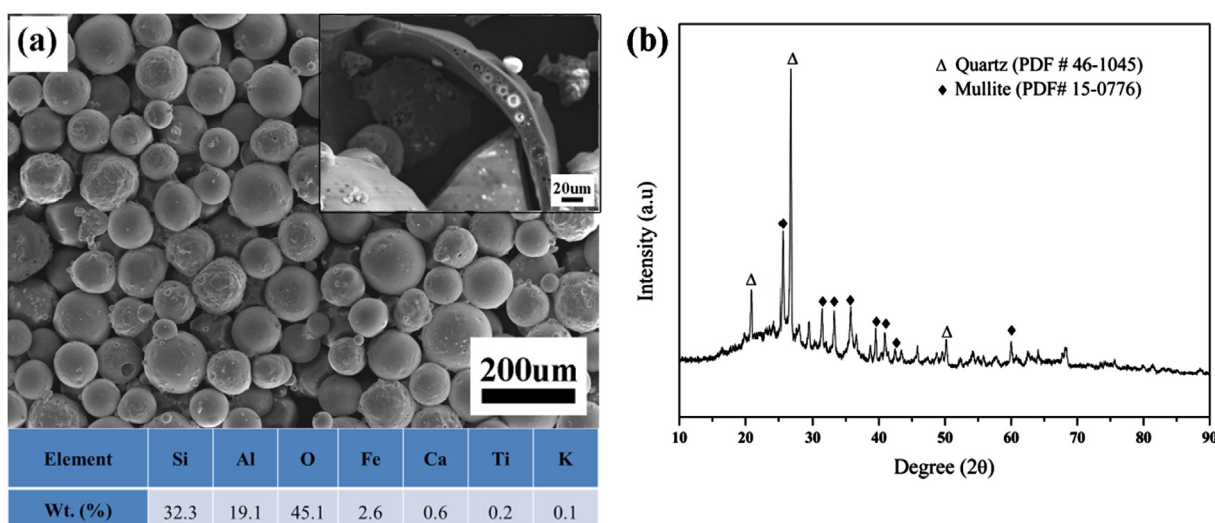


Fig. 1. The scanning electron microscopy images and phase composition of the FACs.

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