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Interface microstructure and compressive behavior of fly ash/phosphate geopolymer hollow sphere structures

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ABSTRACT

In the present work, novel fly ash/phosphate geopolymer hollow sphere structures (FPGHSS) were prepared by the pre-bonding and curing technology. Moreover, the interface microstructure and its role on the compressive behavior were studied. The compressive properties of FPGHSS demonstrated a typical character of cellular materials, with three well defined stages in stress–strain curve. The compressive strength was approximately 5.8 MPa, and the failure of FPGHSS was mainly due to the evolution of the multi-collapsed layers of fly ashes and large macro-cracks during the compression. The microstructure of fly ash was composed of aluminosilicate glass phase, crystalline quartz and mullite. The phosphate geopolymer comprised of aluminum-phosphate phase and α-Al_2O_3, and the nano-pore structure was observed. Moreover, the chemical reaction interface of FPGHSS was generated. It should be noted that the horizontal cracks were mainly produced in fly ashes, and the evolution of horizontal cracks leaded to the clasped layers. Moreover, the large macro-cracks propagated preferentially in the phosphate geopolymer and along the interface region of FPGHSS, due to the dense glass phase of fly ash and the chemical reaction interface. Further, the interface structure between α-Al_2O_3 and amorphous phase could increase the propagation path of cracks in phosphate geopolymer.

1. Introduction

Hollow sphere structures (HSS) are an interesting group of cellular materials, characterized by particular topology, and present a combination of multi-functional and mechanical properties [1]. Due to the open porosity between the bonded/sintered hollow spheres, HSS have excellent multi-functional properties, such as acoustical behavior and thermal conduction [2–4]. Moreover, thanks to the closed porosity inside the hollow spheres, HSS have high compressive strength and energy absorption capacity [5]. In the last decades, metal hollow sphere structures (MHSS) have been studied extensively, however, the applications of MHSS are limited by the problems of high cost and easily oxidized at high temperature [6,7]. Therefore, novel HSS, with low cost and environmental stability, are promising for the widely civil applications.

It is known that the HSS present potential interest as the lightweight structural materials, due to the excellent compressive behavior and energy absorption capability [8]. Recently, much attention has been attracted to reveal the relation between pore structure parameters and mechanical properties of HSS [9–11]. It is found that the packing models of MHSS have significant influence on its mechanical properties, and the FCC packing has superior properties to the BCC or SC packing [9,10]. Interestingly, the geometrical defects could lead to the reduction of compressive strength of HSS [11]. Moreover, the roles of bulk density, pore size, pore morphology and porosity on the mechanical behavior of cellular materials were studied [12–15], and the effect of the loading models on mechanical properties of MHSS was investigated to support the applications in complex loading environment [16]. However, the relations between the interface microstructure and mechanical behavior of HSS have been studied rarely. This issue might be due to the difficulty of specimen preparation for transmission electron microscope (TEM) observation considering the porous materials [17]. Moreover, it is essential to understand the microstructure and interface structure of HSS for the optimization of its mechanical properties [18].

In the present work, a novel fly ash/phosphate geopolymer hollow sphere structures (FPGHSS) was prepared by the pre-bonding and curing technology. The phase composition of FPGHSS was determined by X-ray diffraction (XRD), and the microstructure and interface structures were characterized by TEM. Furthermore, the compressive properties were tested, and the failure mechanisms were analyzed through the observation of fracture

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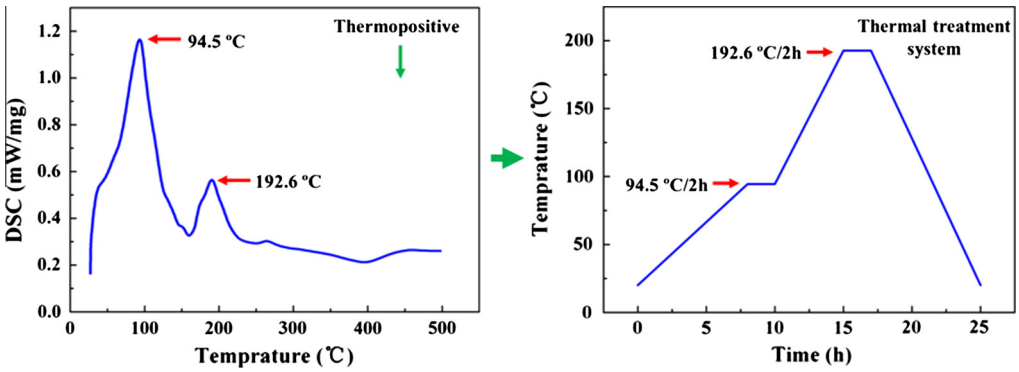

Fig. 1. Thermal treatment system of curing process of FPGHSS determined by DSC curve.

morphology. Finally, the relations between the interface microstructure and mechanical behavior of FPGHSS were discussed, which would be useful for the optimization of the mechanical properties.

2. Experimental details

In this work, the FPGHSS were prepared by the pre-bonding and curing technology using fly ashes (Yaomeng power plant, Henan province, China) and phosphate geopolymer (Institute of Petrochemistry, Heilongjiang Academy of Sciences, China). The preparation procedures are described as follows: first, the fly ash and phosphate geopolymer were added together with mass ratios of 55%:45%, and then mixed uniformly through mechanical stirring; second, the preforms were obtained in graphite molds; at last, the FPGHSS were synthesized after the performs cured according to the thermal treatment system, as shown in Fig. 1.

The morphology and chemical composition of FPGHSS were characterized by scanning electron microscope (SEM) equipped with EDS (Quanta 200FEG, FEI, USA). The phase compositions of fly ash, phosphate geopolymer and FPGHSS were determined by XRD in a scanning angle range of 10–90° (X'pert X-ray diffractometer, Philips, Holland). The microstructure and interface structure were characterized by TEM (Tecnai G2 F30, FEI, USA). In the present work, the dense materials were prepared to achieve the TEM specimens of porous FPGHSS, and the detailed procedures were shown as follows: the fly ash was grinded to be sub-micron sized powder, which was mixed uniformly with phosphate geopolymer binder by Mechanical stirring, and then the dense FPGHSS were prepared by pre-bonding and curing process. Finally, the thin area of FPGHSS specimen was obtained by ion beam thinning (GATAN-691 Ionic Reduced Imager, GATAN, USA).

The compressive tests were performed on square samples with the side-length 20 mm, at a crosshead speed of 2.0 mm/min^{-1} (Instron 5569, Instron, USA), according to ASTM: D1621. In this work, the ratio of the characteristic size of test samples to the largest heterogeneity of FPGHSS (the maximal pore size, about 200 μm) was approximately 100, which was considered to be sufficient to characterize a representative volume of FPGHSS. Moreover, the fracture morphologies were observed by SEM, and the observation samples were acquired at different stages during the compression.

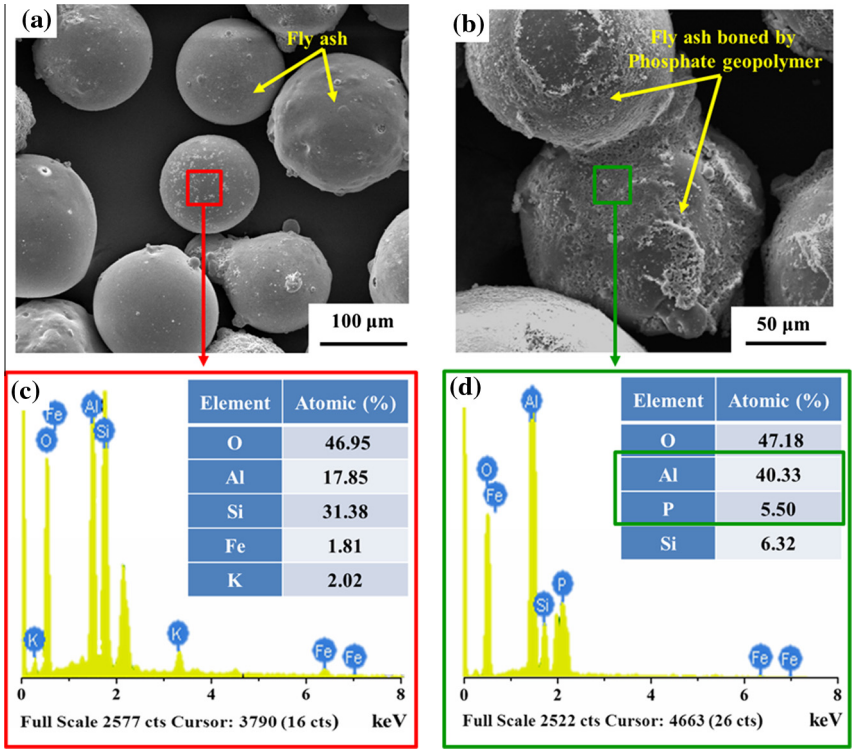

Fig. 2. Chemical composition and morphology of fly ash and fly ash/phosphate geopolymer characterized by SEM–EDS.