

Fabrication and formation mechanism of carbon foam with two-level cell structure



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ABSTRACT

A novel method was developed for production of carbon foam with two-level cell structure, which possesses two kinds of cell sizes of about 1 μm and 300 μm . Results show a carbothermal reduction occurs between the pore wall of first-level foam and the nano-MgO at high temperatures, which results in further bubbling and forming the second-level foam. The carbon foam prepared exhibits a characteristic of pseudoplastic deformation with a relatively compressive strength of 1.72 MPa, and possesses a low bulk density of 0.11 g/cm^3 and a high porosity of 94.2%.

1. Introduction

Carbon foam is a porous carbon material with novel features such as low density, high porosity, good thermal stability, adjustable thermal and electrical conductivity, and therefore has shown great promise in various applications including thermal management, supercapacitor, battery electrode, catalyst support, water purification, and so on [1–3]. It has been since established that carbon foam usually requires a relatively low density, high porosity and a certain mechanical property when used in these applications. In recent years, reticulated vitreous carbon (RVC) foam from resin has been considered as a kind of carbon foam with low density, high porosity but poor mechanical property [4,5], which greatly influences its application and further development. Hence, it is necessary to develop a simple method for production of carbon foam with lower density and higher porosity as well as improved mechanical property.

Conventional carbon foams, i.e. cellular carbon foam and reticulated carbon foam, only have one-level cell structure; meanwhile, their pore walls generally have a wall thickness of more than 10 μm and exhibit dense structure for achieving a desired mechanical property. In order to further decrease the bulk density of carbon foam and increase its porosity, the internal space of pore walls is hoped to be further utilized while maintaining its mechanical property by specific treatments. Cyanate ester resin had been adopted to prepare carbon foam due to its self-foaming characteristics upon carbonization [6]. In addition, it was reported that the carbothermal reduction between nano-MgO and porous carbons could occur at high temperatures, thus leading to the disappearance of nano-MgO [7]. Therefore, the composite of

cyanate ester resin and nano-MgO may be suitable for achieving carbon foam with relatively low density and high porosity. In this work, we attempt the preparation of carbon foam with two-level cell structure by carbonization of the composite of cyanate ester resin and nano-MgO.

2. Experimental

The preparation process was described as follows: epoxy resin (E-51) and bisphenol A cyanate ester (purity > 99%) with a mass ratio of 1:4 were thoroughly blended at 130 $^{\circ}\text{C}$ for 0.5 h with vigorous stirring to form a prepolymer. 15 wt% of nano-MgO (purity > 99.9%; mean particle size, 50 nm) was dispersed in dichloromethane before adding the prepolymer and the mixture was sonicated for 30 min using a high-power ultrasonic machine, and then the dichloromethane was evaporated with vacuum distillation. The liquid obtained was degassed in a vacuum oven at 140 $^{\circ}\text{C}$ for 30 min, and then cast into molds, followed by curing via the procedures of 160 $^{\circ}\text{C}/1\text{h}$ + 180 $^{\circ}\text{C}/1\text{h}$ + 200 $^{\circ}\text{C}/1\text{h}$ + 220 $^{\circ}\text{C}/2\text{h}$. After that the cured composite was put into a furnace, degassed to remove entrapped air under high vacuum (< 0.01 Pa), and then heated up to 1600 $^{\circ}\text{C}$ at a heating rate of 10 $^{\circ}\text{C}/\text{min}$ and kept for 1 h at vacuum pressures of around 50 Pa. The resulting carbon foam was used for further investigations.

X-ray diffraction (XRD) patterns were recorded on a X-ray diffractometer (Ultima III) using Cu K α radiation at $\lambda = 0.15406\text{ nm}$. Morphological measurements were performed using a field emission scanning electron microscope (FESEM, Carl Zeiss Supra 55). The electrical conductivity, bulk density, porosity and percentage of open cell were determined according to the literature [8]. For the compressive

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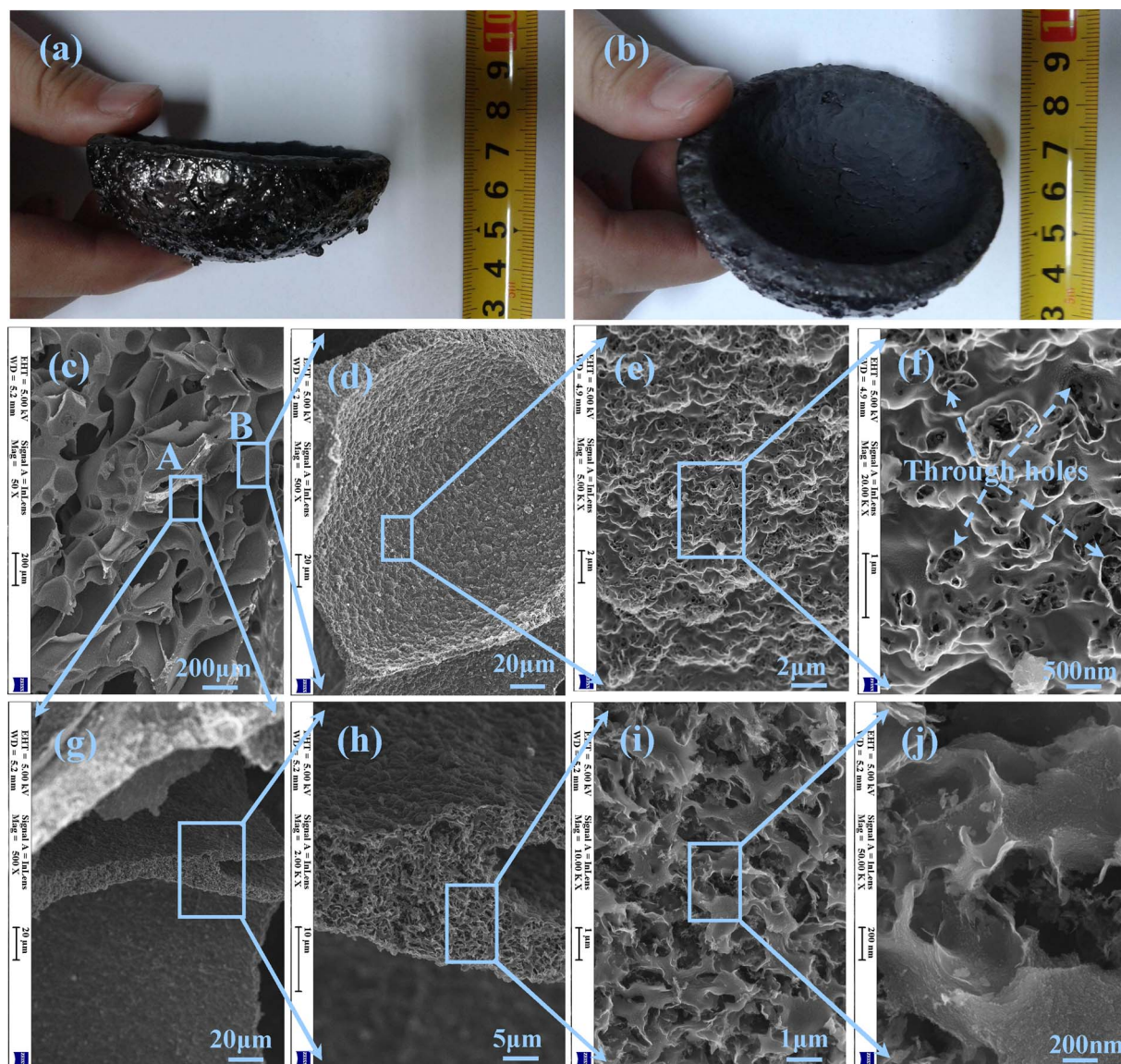


Fig. 1. Pictures of carbon foam with two-level cell structure. (a and b) bowl-shaped carbon foam; (d–f) the FESEM images with different magnifications of the square section B in (c); (g–j) the FESEM images with different magnifications of the square section A in (c).

tests, the samples were machined to cylinders of 12 mm in diameter and 10 mm in height. The tests were carried out at room temperature on an electron universal testing machine (model CMT-5505) with a cross-head speed of 2.5 mm/min, and all the results were calculated based on the average of five tests.

3. Results and discussion

Fig. 1 shows the digital pictures of the carbon foam prepared and the field emission scanning electron microscope (FESEM) images of its fracture surface. Fig. 1(a) and (b) exhibits a bowl-shaped carbon foam. It can be obviously seen that the carbon foam possesses two-level cell structure: the first-level cell size of the carbon foam is about 300 μm (Fig. 1(c)), and the internal space of its cell walls contains many interconnected second-level cells with sizes of about 1 μm (Fig. 1(i)). In addition, numerous bubble-like bulges with through holes can be observed in the surface of cell walls (Fig. 1(e)) whereas the nano-MgO particles disappear in the internal space of the first-level cell walls. The facts reveal that the carbon foam prepared possesses a degree of viscoelasticity at a certain high temperature, which seems to be related to the formation of the two-level cell structure. Moreover, the carbon foam

exhibits a characteristic of light porous material, as indicated by that its porosity, opening rate and bulk density are 94.2%, 91.8% and 0.11 g/cm^3 , respectively.

In order to investigate formation mechanism of the carbon foam, we investigated the microstructural changes in the carbon foam during carbonization. Fig. 2 exhibits X-ray diffraction (XRD) patterns of the carbon foam prepared at different carbonized temperatures. When the temperature is 1000 $^{\circ}\text{C}$, there are three characteristic peaks for MgO. The resulting carbon foam only has a one-level cell structure, and the nano-MgO particles are dispersed in the internal space of its cell walls (see Fig. S1). When the temperature increases from 1000 $^{\circ}\text{C}$ to 1400 $^{\circ}\text{C}$, these characteristic peaks for MgO can be observed in the XRD patterns. With a further increase of temperature up to 1600 $^{\circ}\text{C}$, the characteristic peaks for MgO disappear and there are only (002) and (100) peaks of graphite in the XRD pattern. This can be attributed to a series of carbothermal reduction between MgO and carbons when the temperature arrives at a certain value [7]. As shown in Fig. 1(e), the carbon foam exhibits a degree of viscoelasticity at a certain high temperature and therefore, gas products like magnesium vapor and carbon monoxide can be escaped from the first-level cell walls to develop the second first-level cell. Based on the above discussion, a formation mechanism of the

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