The effect of heating rate on the microstructure and thermal diffusivity of carbon foams

Sizhong Li [1,2], Yan Song[1], Quangui Guo[*1], Yu Yang[1,2], Lang Liu[1]

[1]Key Laboratory of Carbon Materials, Institute of Coal Chemistry, Chinese academy of Sciences, Taiyuan 030001, China

[2]Graduate University of the Chinese Academy of Sciences, Beijing 100039, China

Abstract:

In this work, carbon foams were prepared from mesophase pitch. The effect of heating rate during the foaming process on the microstructures and thermal diffusivity of the resultant carbon foams was investigated. Scanning electron microscopy (SEM) ,X-ray diffraction (XRD) and the thermal diffusivity measurement were used to characterize the property of mesophase pitch and as-prepared carbon foams. Results indicated that carbon foam with better microstructure and higher thermal diffusivity could be obtained by choosing appropriate heating rate in the foaming process. The molecule layers arrangement of the carbon foam was more orderly than that of mesophase pitch.

[*] Corresponding author. Tel: +86 3514184106, Fax: +86 3514085506

Email : lisi202001@tom.com (S.Z Li)

1. Introduction

Carbon foam, especially mesophased pitch-based carbon foam, is a sponge like carbon material with certain features, such as low density, large external surface area with open cell structure, and adjustable thermal and electrical conductivity [1-6]. The potential applications of carbon foams include such as high-temperature thermal insulation, high thermally conductive heat sinks, electrodes for energy storage, catalyst supports etc. [1,3,5]. The morphology and properties of carbon foams vary with the different process and materials [4,5]. Typical foam forming processes utilize a blowing technique, or pressure release, to produce foam of the pitch precursor [1-3,11]. In order to avoid the melt of green foam during further heat treatment, stabilization is very necessary to prepare carbon foam. Since a new and less time consuming process for fabricating pitch-based graphitic foams without traditional blowing and stabilization steps was developed by James W. Kleet, it was used widely to prepare carbon foams [1-4].

It is well known that mesophase pitch contained different amount of volatile and non-volatile compounds. With the temperature increase, gases will release from molten pitch. Then the gases would dissolve in the molten pitch, saturate, and then separated from the pitch phase in the end [2,4,7,11]. The bubbles in the molten pitch will grow up. The heating rate will influence on the pyrolysis of mesophase pitch, and the growth of carbon foams. In this paper, the effect of heating rate on the micro structure and thermal diffusivity were investigated.

2. Experimental

Mitsubishi naphthalene based synthetic pitch was used as the precursors of carbon foams. The properties of the pitch were listed in Table 1.

Table 1 Properties of mesophase pitch

	Softening point(K)	Content of mesophase (%)	Volatile constitution (wt.%)	Insoluble of toluene (wt.%)	Insoluble of pyridine (wt.%)
Mesophase pitch	556	100	21.5	78.1	62.0

The mesophase pitch was foamed under 3 MPa in a pressure vessel at a 0.2, 0.5, 1, 2, 4, 5 K/min heating rate. The foaming step consisted of heating under nitrogen atmosphere to at 523 K, applying the foaming pressure, and then heating at the specified heating rate to 733 K, soaking for 2 hrs, and then cooling to room temperature. The green foams were carbonized in 1573K and then graphitized at 2873K. The morphology of foams was examined by using JEOL JSM-6360LV scanning electron microscope (SEM). Room temperature X-ray diffraction measurements (XRD) were conducted using a D8 advance Brucker AXS vertical θ/2 θ goniometer. The thermal diffusivity, α, of the foam was measured on the Netzsch LFA447/2-2 InSb Nano Flash machine.

3. Results and discussion

3.1 The effect of heating rate on the pyrolysis behavior of mesophase pitch

The pyrolysis behavior of mesophase pitch depended on the pressure, the heating rate etc. The curves of the gases quantity as a function of temperature were shown in Fig.1.

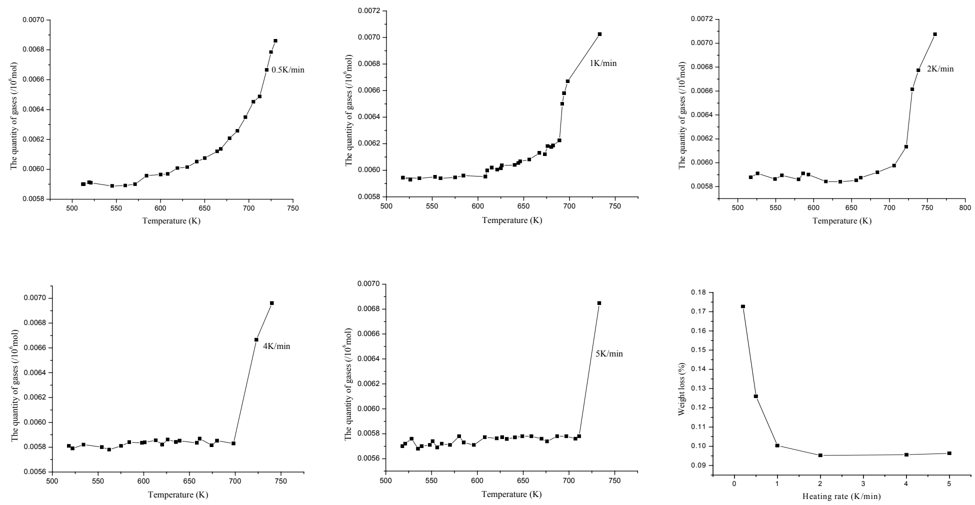

Fig.1. The amount of released gases and the weight loss of pitch as a function of heating rate

From Fig.1 it can be found that with the increase of heating rate, the mesophase pitch began to pyrolysis at higher temperature and the weight loss of pitch was decreased. The relased gases will dissolve, saturate, supersaturate, and nucleate in the molten pitch [2,3]. With the temperature increasing, bubbles in molten pitch will grow up. The growth of bubbles depended on the amount of released gases, the foaming pressure, surface tension, and the viscosity of molten pitch.

3.2 The effect of heating rate on the microstructure of foams

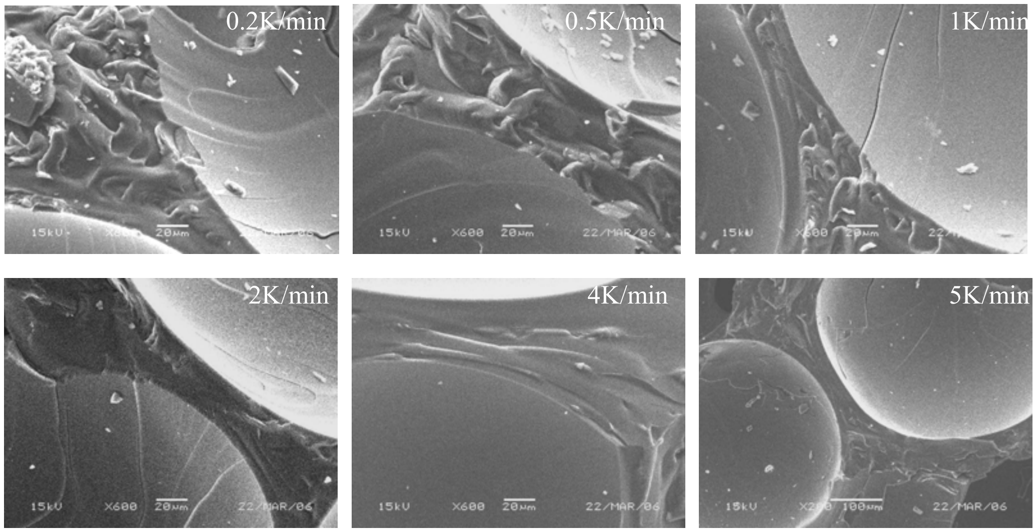

Fig.2. SEM images of carbon foams derived from mesophase pitch

From Fig.2 it can be seen that the ligaments of carbon foams produced at different heating rate were different. The ligaments produced at lower heating rate were better than that at higher heating rate. The mesophase pitch can principally show similar transitions among the isotropic liquid, liquid crystal, and anisotropic glass phases as the conventional liquid crystal substances; however, pyrolysis may take place at a transformation between the isotropic liquid and the liquid crystal phases, giving finally solid anisotropy carbons [8,9]. At low heating rate, the viscosity of molten pitch changed slowly. When bubbles combined under the surface tension driving, the molecule layers will arrange parallel to the axis of ligaments. If the viscosity of molten pitch changed slowly, the molecule layers had enough time to arrange as parallel to the axis of ligaments. So the better ligaments formed.

3.3 X-ray analysis

X-ray diffraction spectra of the mesophase pitch and resultant green foams were given in Fig 3. It can be found that the distance between the molecule layers in green foam was smaller than that in precursor, which meant that the molecule layers arranged in more order. During heat treatment, the molecule layers will dehydrogenate, and then the distance between the molecule layers in green foam decreased. On the other hand, with the bubbles combining, molecule layers were arranged paralleling to the ligament.

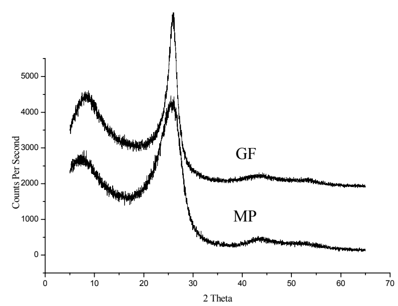

Fig.3. X-ray diffraction patterns of mesophase pitch (MP) and green foam (GF) produced at 733 K

3.4 Thermal diffusivity analysis

Table.1. Thermal diffusivity of graphite foams

Heating rate (K/min)	0.2	0.5	1	2
Thermal diffusivity (mm^2/s)	105.4	99	93.5	91.2

The thermal diffusivity of graphite material was related to the crystal parameters, the bridge of micro graphite structures, and the arrangement of micro graphite structures [5]. The thermal diffusivity of graphite in c axis was lower than that of the other axis. If all the micro graphite pieces arranged paralleling the axis of ligaments, the thermal diffusivity of graphite foams will be higher. The thermal diffusivity of graphite foams were listed in Table 1. The thermal diffusivity of graphite foam prepared at 0.2K/min heating rate during foaming process could reach 105.4mm^2/g. At the same time, the slower the heating rate in the foaming process, the bigger the thermal diffusivity of the resultant graphite foams. The reasons might be as followed: First of all, the molecular layers of graphite foams prepared at relative lower heating rate during the foaming process arranged paralleling to the axis of ligaments, so the thermal diffusivity of them was higher. Secondly, after carbonization and graphitization, the weight loss of resultant foams prepared at lower heating rate during the foaming process was smaller than that with higher heating rate.

4. Conclusion

Carbon foams derived from mesophase pitch were prepared. The effect of heating rate on the microstructures and the thermal diffusivity of resultant carbon foams was discussed. Carbon foam with better microstructure and higher thermal diffusivity (105.4mm^2/g) could be obtained by choosing appropriately heating rate in the foaming process.

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