FABRICATION OF THE CARBON FOAMS DERIVED FROM PHENOLIC RESIN MIXED WITH MESOPHASE PITCH

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Abstract

Carbon foams are developed by pyrolysis with the polymer foam derived from phenolic resin and mesophase pitch. Phenolic resins are widely used as precursors for carbon/carbon composite materials due to their low level of toxic gas emission during pyrolysis and high heat resistance. However, the carbon foams based on the phenolic foams have inferior mechanical strength because of glassy carbon formed during the carbonization procedure. There are many researches about the nano-particles reinforced the phenolic foams to improve the mechanical strength of the carbon foam. When the nano-particles were reinforced in the phenolic foam, it leads to high cell density and cell uniformity affecting the physical properties of carbon foam. However, nano-particles in the carbon foam are easily aggregated, which causes low mechanical properties of the carbon foam.

In this study, the mesophase pitch which has high thermal stability, mechanical properties and carbon yield by intermolecular cyclization after carbonization process was reinforced to improve the mechanical properties of carbon foam. The pitch particle was dissolved into acetone to avoid the aggregation of the particles and coated on the phenolic precursor foam. The pitch solution coated phenolic foam were carbonized to fabricate the carbon foam. The cell morphology of the carbon foam were observed by scanning electron microscope (SEM) images. The physical properties and thermal characteristics of carbon foams were characterized by measuring compressive strength and thermal conductivity.

1. Introduction

Carbon foams are attractive carbon materials due to their advantages such as light weight, high strength, temperature tolerance [1-2]. These advantages of the carbon foams have potential application in industrial field such as energy absorption material, high temperature thermal insulation, electrodes for energy storage and filters [2]. Generally, carbon foams were fabricated by carbonizing the polymeric foams derived from thermoset resins which has the high thermal stability. Among the thermoset resins, the phenolic resin has excellent flame resistance, high self-ignition temperature of 480˚C, and high thermal stability. In addition, the phenolic foam derived from the resin does not generate toxic gases during pyrolysis to fabricate the carbon foam. Accordingly, this has led to a broad usage of the phenolic foam at the application of carbon foams [3-4]. However, the carbon foams based on the pure phenolic foams have inferior mechanical strength because of glassy carbon formed during the carbonization procedure.

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Many studies have focused on the carbonization condition and the content of reinforcement to improve the mechanical properties of carbon foams [Ref]. However, it is difficult to achieve the uniform distribution of the reinforcing materials to the foam precursors without aggregation. Aggregated reinforcements in the polymer foam can caused low mechanical and thermal properties.

In this study, the carbon foams were fabricated via carbonization of the phenolic foams at 800˚C in a nitrogen condition. Mesophase pitch (<35 µm) were dissolved in acetone and the solution was coated on the cell wall of the phenolic precursor foams before carbonization to reinforce the mechanical properties of carbon foam and to increase the carbon yield of the phenolic precursor. The cell morphologies of the carbon foams were observed by SEM images and the compressive strength of the carbon foams were measured by using material testing system. To estimate the insulating performance of the carbon foams, thermal conductivity were measured by a laser flash method.

2. Materials and experimental procedures

2.1. Materials and preparation of phenolic foam

Resole-type phenolic resin (OG-5000, Kangnam Chemical Co., Republic of Korea) was blend with an cure catalyst (PTSA 65%, Kangnam Chemical Co., Korea) in a mixing ratio by weight of 9:1. Before mixing process, the phenolic resin and cure catalyst were kept at 20˚C to control the viscosity change. The mixture was stirred at 500 rpm for 1 min using the impeller. Multi-wall carbon nanotube (724769, Sigma-Aldrich Co. LLC., USA) and graphene (C500, XG Sciences Inc., USA) of 0.5 wt% were added to the phenolic resin and the mixture was foamed by microwave radiation using an effective intensity per unit mass of 12 kW/kg at 2.4 GHz and de-molded.

The mesophase pitch of 0.5 wt% (, OCI Co. LTD., Republic of Korea) was dissolved in acetone at 100˚C using the magnetic stirrer. The dissolved pitch solution was injected into the fabricated phenolic foam using the vacuum bag and then pitch solution coated phenolic foam was heated in a furnace at 80˚C for 1 h to remove the solvent.

2.2. Carbonization procedure

After removing the solvent, the phenolic foam was carbonized at 800˚C for 2 h in a nitrogen atmosphere at a heating rate of 10 ˚C/min. The inert atmosphere was applied to increase the carbon yield and to improve the mechanical properties of carbon foam.(무슨 의미?) After carbonization process, the specimens were naturally cooled to room temperature.

3. Characterizations

3.1. Foam density

The precursor foams (carbon foam 이 아니고??)are dried at 80˚C for 1 h to remove the internal moisture or solvent before measuring the mass. The diameter and length of the foam specimens tested for foam density are 70 mm and 130 mm, respectively. However, the volume and mass of carbon foam was decreased because by-product of phenolic foam was removed during carbonization process. In practice, the density ρ (kg/m^3) of plastic foams is calculated as the ratio of specimen mass m to geometrical volume V as shown in the following equation:

$$\rho = \frac{m}{V} = \frac{m}{\pi r^2 h} \tag{1}$$

3.2. Compressive strength

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To evaluate the load-carrying capacity of the prepared phenolic foams, a uniaxial compression test was conducted to measure the compressive strength. The compressive strength of the each sample with respect to the test variables was measured according to ASTM C 365 using a computer-controlled material testing system (4206, INSTRON, USA). The cross head speed during the test was 0.5 mm/min. To remove the water content, the specimens were dried at 60˚C for 5 h before the test. After the drying process, the specimens were cut to the regular hexahedron-shape of 25 mm in sides and 25 mm in length using a cutting machine.

3.3. Thermal conductivity

Thermal conductivity of the carbon foam was measured at the room temperature by using the laser flash method (Laser PIT-M2, ULVAC. Inc., USA). To measure the thermal conductivity of carbon foam, the specimens of cylindrical shape with diameter of 5 cm were cut to the thickness of 2 cm using the cutting machine.

4. Results and discussion

Figure 1 shows the density of the foams before and after carbonization with respect to reinforcement type. The phenolic precursor foams had different densities one another, so the increasing rate of the density after carbonization are presented in Figure 1. The density of the carbon foam depends on the volume shrinkage and weight loss after carbonization. Accordingly, the carbon yield can be estimated from the increasing rate of the density indirectly. The weight loss of carbonized phenolic foam (PF), MWNCT reinforced phenolic foam (CF0.5) and graphene reinforced phenolic foam (GF0.5) was about 37~40%. However, pitch coated phenolic foam (PAF0.5) has the weight loss of 45% after carbonization, whereas the volume shrinkage rate was about 3 times lower than those of the other specimens. Generally, the density of carbon foam was increased because weight loss was larger than the volume shrinkage. Accordingly, the PAF0.5 showed the minimum increasing rate of the density after carbonization.

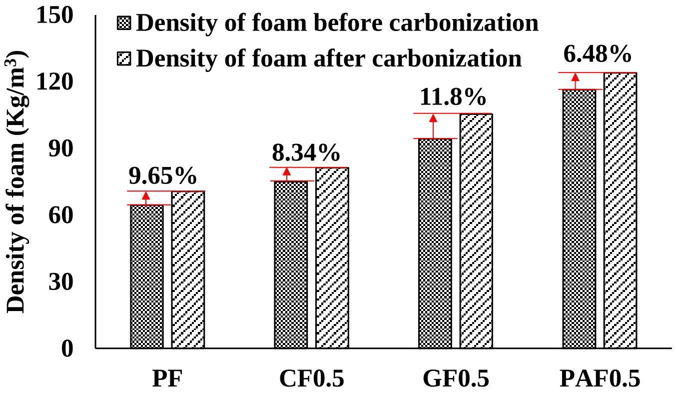

Figure 1. Density of phenolic foam and carbon foam with respect to reinforcement type.

Figure 2 shows cell morphologies of the carbon foam with respect to the reinforcement type. The polymer foams had the anisotropic cell pattern due to different pressure distribution in the mold during the foaming process.. The PF carbon foam showed cell structures composed with nearly round type cells due to large volume shrinkage during carbonization compared to the other foams.

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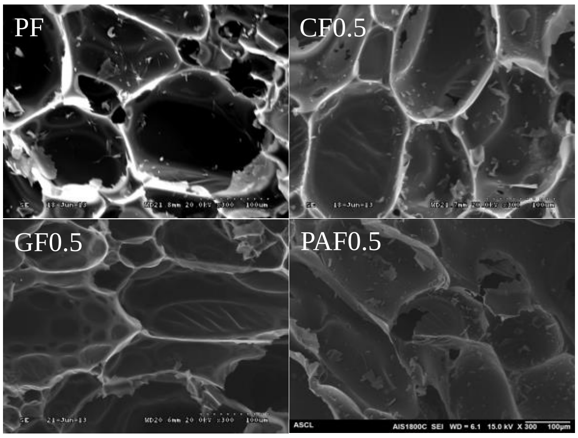

Figure 2. Cell morphologies of carbon foam with respect to reinforcement type.

Figure 3 shows the compressive strength of the carbon foams with respect to the reinforcement type. The CF0.5 showed the smallest compressive strength because of large cell size compared to the other foams and aggregation of the MWNCT. The compressive strength of GF0.5 and PAF0.5 were improved by 101.6% and 77.6%, respectively, compared with that of the PF.

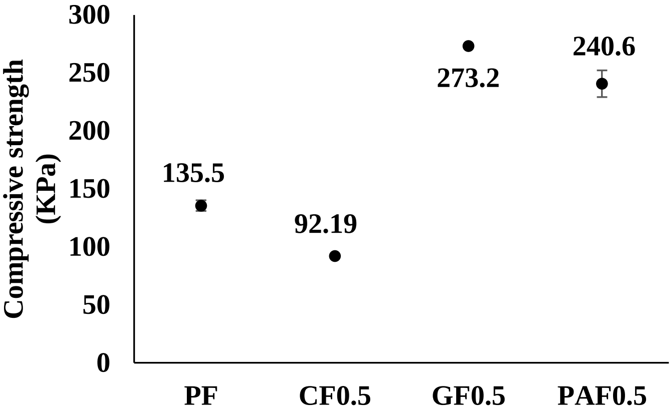

Figure 3. Compressive strength of carbon foam with respect to reinforcement type.

The PF, CF0.5, and GF0.5 showed lower thermal conductivity compared with that of PAF0.5 as shown in Figure 4. The thermal conductivity of PAF0.5 was increased by 269% compared with PF because the carbonized pitch layer having outstanding thermal conductivity was coated on the cell wall of the carbon foam. CF0.5 and GF0.5 showed the similar thermal conductivity to the PF despite high thermal conductivity of MWCNT and graphene. This means that the thermal conductive path by the particles does not exist in the carbon foam.

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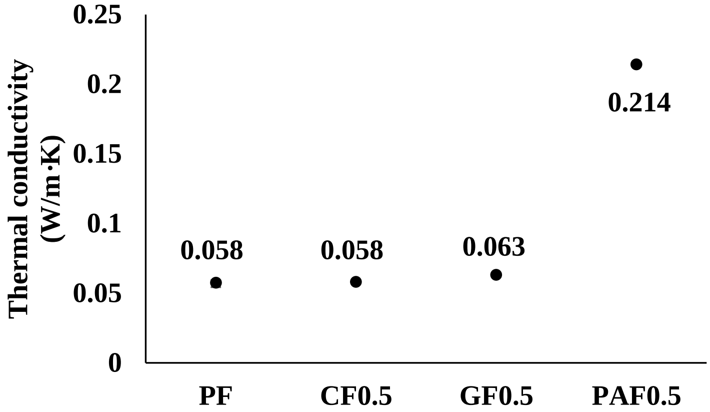

Figure 4. Thermal conductivity of carbon foam with respect to reinforcement type.

5. Conclusion

In this work, the carbon foams were successfully fabricated with respect to reinforcement type. The effect of reinforcement type on the mechanical and thermal properties was explained. Base on the experiments, the following results were obtained:

1. PAF0.5 has weight loss of 45%, whereas volume shrinkage was 2.7 times lower than those of the other specimens.
2. The PF carbon foam showed cell structures composed with nearly round type cells due to large volume shrinkage during carbonization compared to the other foams
3. The compressive strength of GF0.5 and PAF0.5 were improved by 101.6% and 77.6%, respectively, compared with that of the PF.
4. The thermal conductivity of PAF0.5 was increased by 269% compared with PF because the carbonized pitch layer having outstanding thermal conductivity was coated on the cell wall of the carbon foam.

Acknowledgments

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