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DEVELOPMENT OF RETICULATED CARBON FOAM: AN ATTRACTIVE MATERIAL

Satish M. Manocha* and Kalpesh Patel

Department of Materials Science, Sardar Patel University, Vallabh Vidyanagar – 388 120

ABSTRACT

Reticulated vitreous carbon foams are an open pore foam material composed solely of vitreous carbon having highly interconnected porosity. Carbon foams were synthesized using commercially available polymeric foams as templates and thermosetting phenolic resin as carbon source in the present work. The preforms were fabricated by impregnated polymeric foam with resin followed by carbonization at different temperatures up to 1400 °C in inert atmosphere to form carbon foam. The changes in density and porosity of carbon foam were determined. Surface morphological studies were carried out using SEM (Scanning electron microscope) which showed that the pores were uniformly distributed and highly interconnected throughout the structure. At 1400 °C, the foams retained their compactness and the compressive strength of the carbon foam was 0.23 MPa and it was found that the compressive strength and Modulus of carbon foam increases with increase in heat treatment temperature.

Key words: compressive strength, porosity, reticulated carbon foam, resin, template.

INTRODUCTION

Various types of ultra-lightweight porous carbons have been reported in the literature including xerogels [1] to aerogels [2] and carbon foams [3]. Carbon foams have attracted great attention recently because of their light weight and tailorability of their physical properties over a wide range of applications [4–7]. The type of carbon foams depend on the raw material (precursor) used and the manufacturing process employed. The major categories are: reticulated vitreous carbon foam (RVC), graphitic carbon foam and non-graphitic carbon foam. Reticulated carbon foam consists of interconnected voids surrounded by a web of carbon struts.

The attractive applications of carbon foams involve thermal managements, electrodes, catalyst supports and filters owing to their novel features such as adjustable thermal-conductivity and electrical conductivity, high porosity, low thermal expansion Coefficient, high-temperature tolerance, etc. [7–9]. Carbon foams also find their way to act as Radar absorbing materials, mainly due to their well matched impedance, high durability, light-weight, and effective EM wave absorbing capability [10]. Due to the larger pore sizes as well as fully open pore structure, the permeability is usually high in reticulated carbon foams.

One of the current interests in carbon foam applications is in the Power Technology, wherein a battery is comprised of electrical current collectors composed of reticulated vitreous carbon covered with a thin layer of a lead tin alloy. The current collectors create up to four times higher surface area for electrochemical reactions to take place compared to those in a typical lead acid battery, resulting in a battery with higher efficiency and higher capacity meaning more electricity generation [11]. Moreover, the carbon foam can be shaped according to the requirement. The machinability of carbon foam is also convenient. These can be tailored to have low or high thermal conductivity with a low CTE and density. These foams have high modulus but low compression and tensile strength. Foams of C, Si, and SiC are being investigated by various commercial organizations [12,13] for similar applications at higher temperatures.

Carbon foams were first obtained in the late 1960s as reticulated vitreous carbon foams [14]. RVC is synthesized by

carbonizing precursor foam which is impregnated by a thermosetting resin. Open cell polyurethane foams are commonly used as precursors. Open cell foams are those in which the foam lamellae separating neighbouring cells are all ruptured, resulting in a structure comprising a network of struts with continuous air passages. The impregnating resins used are generally phenolic resins, epoxy resins or furfuryl alcohol. The carbonization rate of furfuryl alcohol impregnated polyurethane foams could be higher than that for foams infused with phenolic and epoxy resins [15]. In the present work carbon foam has been synthesized by template route. Open cell polyurethane foams were used as precursors or templates and phenolic resins as carbon source.

MATERIALS AND METHODS

The overview of the template route for making carbon foam is shown in Fig. 1. The cleaned PU foams were impregnated with phenolic resin. Impregnated PU foams were cured at 150 °C and were heat treated in inert atmosphere at different temperature up to 1400°C to study the pyrolysis behaviour of foam. Up to 1000°C the foams were heat treated in N₂ atmosphere at slow heating rate 25°C/hr and those up to 1400°C were heat treated in Argon atmosphere.

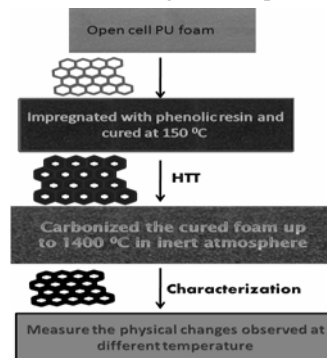


Fig. 1: Overview of the preparation method

The synthesized carbon foams were characterized for different properties like the change in density and the percentage porosity (by kerosene porosity). The compressive strength and modulus of carbon foam were measured by

*Corresponding author: sm_manocha@rediffmail.com

INSTRON 5500R. The main intrinsic properties of carbon foams like cell structure, size and shape of cell, pore wall thickness, etc were evaluated by SEM (HITACHI-S3000N).

RESULTS AND DISCUSSION

Pyrolysis Behaviour of The Resin Impregnated Foam

The coating of polymeric template is the crucial step in the template method. The viscosity of resin plays an important role in the impregnation or coating process because higher viscosity gives dead end in the sample while lower viscosity results into uneven coating through the sample. Moreover, incomplete removal of the excess resin slip before firing leads to the presence of some closed cells in the final carbon foam, altering its mechanical properties and permeability behaviour. Firing must be conducted at an appropriately slow rate during the elimination of the polymeric template, to avoid stresses and macro defects in the final carbon foam structure.

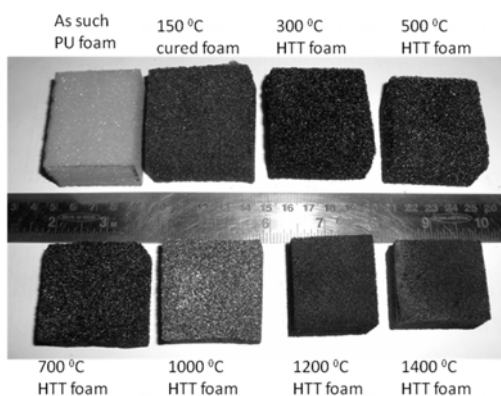


Fig. 2: Photograph of carbon foam at different temperature

Fig. 2 shows photographs of the foams at various stages of formation. It shows that rigidity of the foam is retained at all stages, even during heat treatment [16].

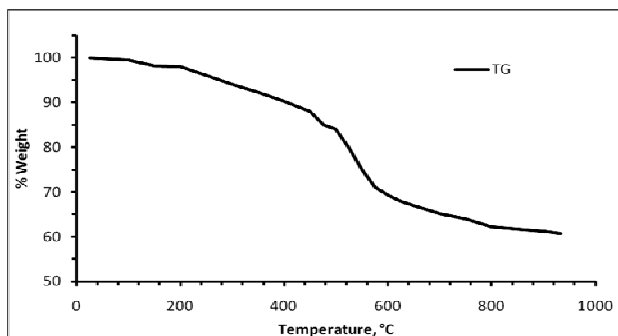


Fig. 3: TGA of cured Phenolic resin in N_2 atmosphere

The structural changes in the resin impregnated foam as well as the final structure depend on the pyrolysis behaviour and ultimate char yield of the phenolic resin, especially in the low temperature range since at around 350°C the decomposition of polyurethane is practically completed by releasing components like diisocyanates and polyols. Other decomposition products are amines, olefins and carbon dioxide because of the destruction of the polymer chains and subsequent secondary reactions [17]. At this time it is the impregnated resin or the pyrolyzed product of the resin which imparts rigidity to the foam structure. Fig. 3 shows TGA of the cured phenolic resin used in the present studies. As seen from

the Fig., in the temperature range up to 300°C , the weight loss is not appreciable. It means it is rigid enough to sustain the structure while the template, polyurethane is decomposing. The phenolic resin gives ultimate yield of 60 % after pyrolysis as shown in Fig. 3. According to Yamashita et al. [18], thermal weight loss below 450°C is due to dehydration reaction of phenolic resin, including thermocuring reaction between hydroxymethyl groups and hydrogen groups within aromatic rings and the condensation reaction between methylene and hydroxyl groups. The condensation aromatic polynuclear structure started to form above 450°C and developed above 500°C [19], releasing small molecular substances such as CH_4 , H_2 , CO , CO_2 , etc. weight loss at approximately 700°C was attributed to further carbonization and dehydrogenation reaction in phenolic resin. As a result, uniting phenol compound advanced and the dehydration and depolymerisation reaction were promoted. Phenolic resin was converted into glassy carbon after carbonization. Weight loss rate reaches maxima at approximately 550°C , and evidently decreases above 600°C , hardly varies above 800°C . To retain the original porous structure of precursor foam, it is necessary to cure the impregnated phenolic resin in PU foam.

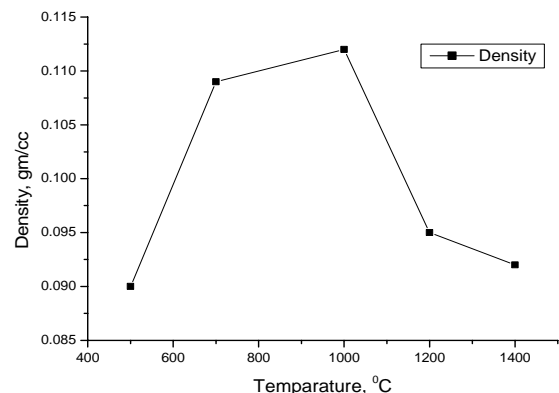


Fig. 4: Changes observed in density at different temperature

The changes observed in density at different temperature are shown in Fig. 4. In the temperature range $500 - 1000^\circ\text{C}$, the density increases gradually with temperature due to the pyrolysis of phenolic resin. Further heat treatment to temperature between $1000 - 1400^\circ\text{C}$, the density decreases due to denitrogenation. It is observed that the weight loss and volume shrinkage observed up to 1000°C was higher. It also leads to shrinkage resulting in strong cell formation.

Physical Properties of The Carbon Foam

At 1000°C , the carbon foam produced has glassy colour. It is also noticed that the sound of carbon foam when it strike with ceramic tiles become more metallic or glassy with increase in heat treatment temperature.

The kerosene porosity method is generally used for the samples having pore diameter larger than that of kerosene molecules. The samples used for analysis must be free of moisture and can react with kerosene. The samples were evacuated in vacuum for 2 hours and impregnated with kerosene. The porosity of carbon foams at different heat treatment temperature was determined by using kerosene porosity method. The porosity was found out by equation 1 as given below:

$$\% \text{ Kerosene porosity} = \frac{\text{Weight absorbed Kerosene}}{\text{Density of Kerosene} \times \text{Volume of sample}} \times 100 \quad (1)$$

Table 1: Kerosene porosity of carbon foam at different temperature

Heat treatment temp, °C	Kerosene porosity, %
150	86.26
300	78.15
500	78.08
700	76.03
1000	75.73
1200	72.32
1400	70.57

As shown in Table 1, the porosity of carbon foam decreases as the heat treatment temperature increases because with increase in temperature the entire foam structure gets shrunk and some of the pores may get blocked with vitreous carbon.

In most solid, rigid foams, whether they are made of carbon or not, the compressive stress-strain curves show a linear elastic region, followed by a stress plateau region [20, 21]. The present carbon foam presents a brittle failure mode undergoing successive cell wall fractures. The compressive strength and modulus of the foam heat treated at different temperature is given in Fig. 5:

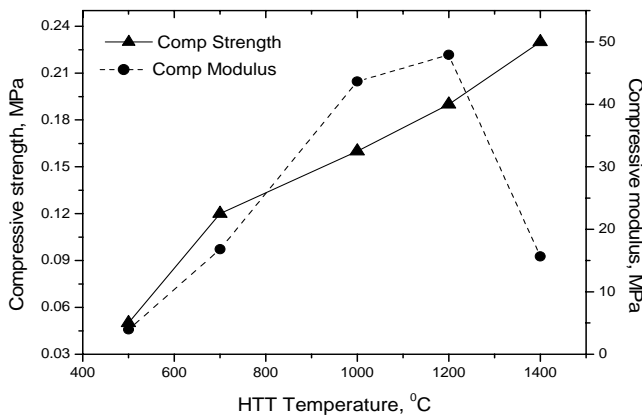


Fig. 5: compressive strength of carbon foam

As shown in Fig. 5, the compressive strength and modulus of carbon foam increases with increase in heat treatment temperature due to more shrinkage resulting in strong cell formation and blocking of some pores. The compressive strength increases with relative density. The strength of carbon foam mainly depends on the type of porosity, open cell to close cell ratio, size and shape of pore, cell wall thickness, length of individual struts, area of pore junction and interconnected network of porous structure. In addition, the strength of carbon foam is also related to the properties of the precursor material, such as the viscosity of resin. However, heating rate is also an important parameter because during heating, the expansion and gas evolution of the polymer lead to significant stresses that can damage the carbon foam surface if not carefully controlled, leading to carbon struts containing macroscopic flaws.

Surface Morphology of the Carbon Foam

The surface morphology of carbon foam is also studied with the scanning electron microscope (SEM). Fig. 6 shows that the pores are uniformly distributed throughout the structure. It indeed shows the open cell and well organized cell structure without any cracks and defects. The pore wall thickness varies between 50 μm to 85 μm. SEM image shows that the foam cell size varies from approximately 200 μm to 500 μm. As shown in Fig. 6 (A), the pores are broken during sample preparation or machining. It also shows that all the open cell pores are highly interconnected with very less closed cell pore. The closed pores are formed in the structure due to excess resin present in foam during impregnation process.

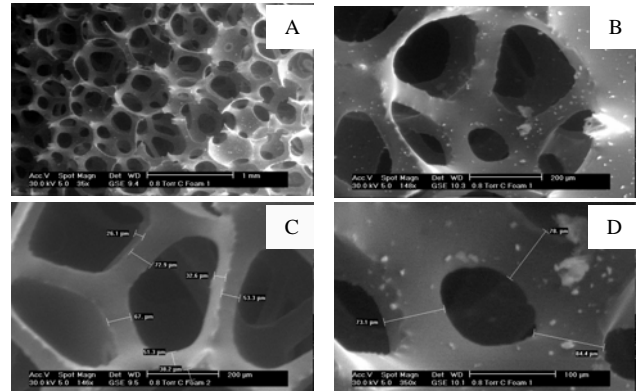


Fig 6: SEM images of carbon foam (A) and (B) shows uniform distribution of porosity, (C) and (D) pore wall or pore boundary

It is clearly seen that the surface of carbon foam cells are not porous. The foam cell size is relatively uniform throughout the structure.

CONCLUSION

Reticulated vitreous carbon foams having high interconnected porosity has been synthesized by carbonizing the open cell polyurethane foams infused with phenolic resin. The pore size and total porosity of carbon foam can be controlled by using appropriate processing conditions.

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