

Synthesis and characterization of carbon foam by low pressure foaming process using H₂SO₄ modified pitch as precursor

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Carbon foams with anisotropic texture and good mechanical strength were obtained, using coal tar pitch modified by thermal oxidation treatment with H₂SO₄. The investigations on the relation between precursor properties and structure of obtained foam show that the composition and softening point of the pitch precursor significantly affect the foaming process and foam structure. The mechanical strength of carbon foam is found to be related not only to the foam cell structure, but also to the composition of the foaming precursors.

The compositions of the modified and synthetic pitches allow foam formation at relatively low pressure and fast heating of the precursor during foaming process without any stabilization treatment.

Key words: carbon foam, coal tar pitch, optical microscopy, SEM, mechanical properties.

INTRODUCTION

Carbon foam is a sponge-like carbon material, representing cellular ligament microstructure, and it is distinguished by certain features, such as light weight, high temperature tolerance in inert atmosphere, high strength, large external surface area and adjustable thermal and electrical conductivity. As new materials carbon foams have essential advantages, such as low cost, enhanced structural properties, fire resistance, radar cross-section, corrosion susceptibility.

These unique properties, which mainly depend on the precursors' features and synthesis conditions, make carbon foams ultra-high performance engineering materials, and determine their many potential applications in numerous industries [1, 2]: shipbuilding – living space modules, above deck structures, bulkheads; aerospace – aerospace structures, optical benches and lightweight mirrors, rocket nozzles and motors, thermal protection systems, composite tooling, heat transfer systems, radar adsorbing and antennae systems; energy – fuel cells, battery electrodes, nuclear shields, rods for nuclear reactors; automotive catalytic converters, brakes, bumpers; defense-related – insensitive munitions, shields and body lightweight armor; medical – bone surgery material, tooth implants; architecture – insulation, fire-proof blocks, shields and coatings,

safe rooms, heating and cooling units; abrasives – for polishing of glass and metals, for paint removal, in cosmetics; filters for hazardous conditions; electronics – processor radiators, radio frequency shields. Carbon foam is used as a core to build fireproof radio frequency sheltered composite structures with superior lightning protection [2].

Klett et al. [3] have synthesized carbon foam sample with specific thermal conductivity about 6 times greater than that of Cu and approximately 5 times greater than that of Al – it is possible to use it in CPU coolers and heat sinks.

Due to their distinctive physical and high-temperature properties, carbon foams appear to be ideal materials for advanced thermal protection systems. Carbon foams, as well as other carbon and graphite materials, have high emissivity, low coefficient of thermal expansion, and they are self-supporting in low and high temperature regimes [1]. In the absence of oxygen these materials can be heated up to about 3000°C without melting or softening and they have high thermal shock resistance and dimensional stability [2]. These characteristics permit utilization of both thermal insulating and conducting properties, respectively, within a thermal management system [3–6]. Thus specific carbon foam is a component of the thermal protection used on the solid rocket booster of the Space Shuttle [2].

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Carbon foams are excellent materials for acoustic absorbing applications in the shipbuilding, aerospace, and automotive industries. Carbon foam absorbs almost 100% of sound waves, depending on their frequency [2]. Pore structure of carbonized foams had a darkroom effect on microwave absorptivity, which made the carbon foams a promising radar absorber [7, 8]. They are used for advanced radar antenna construction, due to their specific physical (very important - stable towards vibrations) and electrical properties. Carbon foam can be manufactured with desired electrical resistance, dielectric constant, and radar reflection coefficient. The material can be produced with physical dimensions suitable for antenna assembly [2, 7].

Carbon foam, due to its unique combination of high compressive strength and considerable impact absorption capacity, is an attractive material for enhanced performance bumper systems. At the same time, carbon foam is a good material for the construction of advanced electrodes because of its large surface area, high electrical conductivity, and chemically inertness. Additionally, carbon foam can be readily coated with a number of active materials to form either anode or cathode [2, 9].

Carbon foam is relatively inert even at high temperatures and radiation – it is an ideal candidate for use as a filter for filtration of aggressive solvents and molten metals [2], as well as nuclear shields and rods for nuclear reactors [1, 2, 10]. The inertness and mechanical strength of carbon foam makes it also suitable for bone surgery material, prosthetics, tooth implants [1, 2, 11, 12]. Some researchers and engineers consider carbon foam to be one of the next-generation material systems and components – maybe soon carbon foam is going to replace some of the conventional materials such as wood, ceramics, plastics, glass, rubber, metals [2, 13].

Initially, carbon foam was fabricated by carbonization of polymeric foam [14-21]. The development of carbon foam on the basis of coal tar pitch [22-24], petroleum pitch [22, 23, 25], synthetic pitch from organics [3, 4, 23, 26-31] or biomass – melanine [9], cork [32], olive stones [33], and raw coal [22, 34-36] – provides an economical way for the production of lightweight carbon material. The effect of the precursor on structure and properties of the obtained foam is of great importance and it is under extensive investigations [3, 21]. The properties of synthetic pitch can be adjusted to obtain suitable foaming precursor, which can be foamed directly without any pretreatment. The commercial coal tar pitch needs to be pretreated before foaming. The major problem is that its plastic properties usually do not meet the foaming process require-

ments for precursors. The well known pretreatments are air blow and thermal treatment in order to control the viscosity and degree of anisotropy of the foaming precursors [37-39].

When starting material is mesophase pitch, the final product is carbon foam with cellular graphitic ligament microstructure, similar to that in high-tech carbon fibers [1, 3, 26, 27, 40-44], ensuring isotropic material properties – high stiffness and high thermal conductivity graphitic foams [43, 45].

Therefore for preparation of high-strength structural carbon foam the desired foaming precursor should be isotropic in nature. On the contrary for the highly thermal and electrical conductive carbon foam, an anisotropic pitch precursor is required [45-55]. Pitch based carbon foam can be either mechanically strong foam or highly conductive.

The aim of the present investigations is to adjust by appropriate modification the composition and the properties of commercial coal tar pitch. The subject of this paper is developing of carbon foams from modified coal tar pitches using relatively simple and low pressure process without any stabilization step. The foaming process of pitch-based carbon foam, the pretreatment of the precursors, and the properties of resultant foams are discussed in this paper.

EXPERIMENTAL

Foaming precursors and pretreatment

Most petroleum and coal-derived pitches need to be pretreated (under various conditions – with different chemical reagents and temperature regimes) before foaming. The plastic properties of these precursors usually do not meet the foaming process requirements. The pretreatments usually involve the polymerization/condensation of pitch by thermal treatment in order to control the viscosity of the foaming precursors.

The precursors used in this work include pitches obtained after thermal oxidation treatment with H_2SO_4 (at 120°C) of commercial coal tar pitch. Details of the thermal oxidation treatment process are available elsewhere [37].

Precursor characterization

An exhaustive extraction is carried out consecutively by Soxhlet apparatus with petroleum ether to extract maltenes and with toluene to extract asphaltenes. After distilling off the solvent the extracts were dried in vacuum and separated as it is shown elsewhere [37].

The elemental analysis was performed on Carlo Erba 1106-type equipment for C, H and N content. Sulphur content was determined by Eshka's method

[56]. Oxygen was determined by the difference.

Softening point of pitches are determined by the Ring and Ball's method using stainless steel balls of diameter 20 mm. For each measurement an average of three readings is taken. The variation is found to be within $\pm 4^\circ\text{C}$.

Foaming method

Foaming was carried out in a stainless steel pressure vessel by heating the pitch precursor up to 500°C in a N_2 atmosphere at pressure up to 1 MPa. The resultant "green" foams were calcinated at 1000°C in N_2 atmosphere to increase the mechanical strength and to remove further the volatiles.

Foam characterization

Bulk porosity. Both apparent and true densities were measured to determine the bulk porosity of the sample. The apparent density was determined based on the values of the weight and the volume – the latter was calculated using sample dimensions. The true density was measured using a helium gas displacement pycnometer type 1305 Micromeritics®. The porosity was calculated using an expression:

$$P(\%) = 100 \times ((\rho_t - \rho_a) / (\rho_t)) \quad (1)$$

where P is the porosity, ρ_t and ρ_a are the true and apparent densities of the sample respectively.

Ultrasonic velocity and dynamic elastic modulus.

In samples with pores in the 1 nm – 100 μm range, the acoustic wavelengths for frequencies up to about 20 MHz are considerably larger than the pore diameters [57]. Since scattering of ultrasonic waves on pores as defects does not occur at the above frequencies, carbon foams studied may be treated as homogeneous materials in this frequency range and the following equation can be applied to the determination of dynamic elastic moduli [57]:

$$E = \rho v^2 \quad (2)$$

where v is the velocity of the stress wave propagation through a homogeneous material, E – the dynamic elastic modulus and ρ – the density of the material.

The dynamic elastic moduli for three mutually perpendicular directions (along the three coordinate axes of a sample) were determined using an ultrasonic velocity measurements. The velocity of longitudinal ultrasonic wave of 100 kHz frequency was measured along every one of the three basic axes of monolithic cube-formed sample using an ultrasonic tester (Tester CT1, Unipan-Ultrasonic, Poland) based on the pulse transmission method. The ultrasonic tester serves to determine the time interval of transition of ultrasonic wave throughout a sample.

Velocity was derived from the transition time interval (τ) and length of path, i.e., thickness of a sample, measured with a slide caliper. Elastic anisotropy was calculated from a relationship $v_{\text{max}}/v_{\text{min}}$, where v_{max} is the maximal value among the three velocity values determined for the three mutually perpendicular directions of a cube-formed sample.

Optical parameters. Optical texture and reflectance values of carbon foams were determined with a reflected light microscope Axioskop MPM 200 (Opton-Zeiss, Germany), using monochromatic linearly polarized light with $\lambda = 546$ nm, in air. Maximum reflectance (R_{max}) values were automatically measured during rotation of the microscopic stage in several points (15–25), each in various locations on the sample.

RESULTS AND DISCUSSION

Foaming procedure

A lot of factors influence the foaming process, but temperature and pressure are the most critical aspects of the process [3, 13, 27, 29, 54, 55]. Pressure and pressure drop time influence pore structure, density and compressive strength of the carbonized foams. More interconnected open-cellular porous structure is formed for shorter pressure drop times [29].

In this paper the softening point and composition of the pitch precursors, which are connected with the viscosity and volume swelling, are used as important factors influencing the foaming process.

Conventional foaming procedure includes foaming step, oxidation-stabilization step, carbonization step and graphitization step [26]. In order to reduce time and energy consumption, we decided to obtain carbon foam by technology, similar to that of Mehta *et al.* [26], but without stabilization step. Instead of this final stabilization step, a special method for thermal oxidative modification of pitch precursor was developed and carried out as preliminary step.

Foaming precursors and pretreatment

Commercially available pitches are not suitable for producing carbon foam directly. The key problem is that the viscosity is too low to preserve the foam cell shape. Therefore, the pitch properties of these materials were adjusted to meet the foaming requirements. For this aim initial pitch was subjected to thermal oxidative modification with H_2SO_4 . Data in Table 1 show increase of the oxygen content in modified pitches. According to data in Table 1, the thermal oxidation treatment with H_2SO_4 lead to the formation of oxygen-containing struc-

tures. Simultaneously condensation reactions with formation of higher molecular weight substances are occurring and the softening point of the pitch increases.

Table 1. Selected properties of studied pitches.

Sample	C, wt. %	H, wt. %	N, wt. %	S, wt. %	O, diff.	C/H
P	90.60	5.25	0.90	0.50	2.75	1.44
M H ₂ SO ₄	83.90	4.24	0.82	2.62	8.42	1.64
M* H ₂ SO ₄	91.20	3.83	0.80	2.12	2.05	2.01

P - initial pitch; M H₂SO₄ - coal-tar pitch, modified by oxidation treatment with H₂SO₄; M* H₂SO₄ - modified pitch heated up to 350°C.

Heat treatment of pitch up to 350°C in N₂ induces polymerization and condensation reactions through de-hydrogenation of polyaromatic molecules. This treatment results in the formation of larger condensed and more planar molecules in pitch. Thus the viscosity and softening temperature of treated pitch are increased. Table 2 lists the properties of pitch sample before and after thermal treatment at 350°C under N₂ atmosphere.

Table 2. Solubility class separation of the pitches determined by sequential Soxhlet extraction.

Sample	PES, %	PEI-TS %	TI %	QI %	Softening point, °C
P	38.00	34.69	27.31	-	72
M H ₂ SO ₄	33.72	45.04	17.74	3.50	140
M* H ₂ SO ₄	9.19	32.40	45.30	13.11	210

P - initial pitch; M H₂SO₄ - coal-tar pitch, modified by oxidation treatment with H₂SO₄; M* H₂SO₄ - modified pitch heated up to 350°C; PES - petroleum ether soluble; PEI-TS - petroleum ether insoluble-toluene soluble; TI - toluene insoluble; QI - quinoline insoluble fraction.

Thermal treatment significantly increases the softening temperature and the content of quinoline insoluble (QI), which implies the increase of molecular weight and the degree of polymerization and condensation of the pitch. As a result of occurring of condensation reactions with oxygen containing structures, a considerable decrease of oxygen content in heated pitch is observed (Table 1). An increase of softening temperature reflects an increase in viscosity of the pitches. In addition, thermal treatment increases the thermal stability of the pitch, and thus decreases the weight loss in the foaming stage. This is relevant to the formation of bubbles, because the amount of bubbling agent is related to the weight loss, caused by the evolving volatiles in foaming stage. Therefore, the size of foam cells could also be controlled by the amount of volatiles. The properties and composition of modified coal tar pitches can be controlled by the

temperature of thermal oxidation treatment and amount of added acid. In this way, by adjusting the conditions of treatment (temperature and amount of added acid) of tars from agricultural wastes, the properties of the synthetic pitch could be controlled.

Carbon foam properties

Main properties. Table 3 shows the general properties of carbon foam, derived from modified pitch heated up to 350°C. The bulk density of the obtained foam is about 0.5 g/cm³. The prepared carbon foam has over 90% open cell structure with porosity >69% according to helium pycnometry.

Mechanical strength. Table 3 also summarizes the compressive strength of the carbon foam obtained. The strength of pitch-based foam depends not only on the foam structure, but also on the properties of the precursor. Data in Table 2 and Table 3 show that carbon foam, obtained from modified pitch, containing QI and relatively low amount of PES, has good compressive strength. The results confirm that the composition of the modified pitch allows proceeding of polymerization and polycondensation reactions with formation of larger condensed molecules during foaming process.

The cellular structure (thickness of cell-wall, t , and the length of the cell edge, l) affects the foam strength. For a regular foam, t and l are correlated with the relative density ρ/ρ_t , where ρ is the bulk density; ρ_t is the true density of foam [58]. It is difficult to measure t and l of the foam, but the relative density is easy to be determined. Although obtained carbon foams do not have perfect and regular cell structure, as a first approximation, relative density is still used to describe the foam cellular structure.

Table 3. General properties of the carbon foam.

Foam precursor	Bulk density g/cm ³	Porosity %	Open-cell, %	Relative density	Compressive strength, MPa
Modified pitch heated up to 350°C	0.52	73.4	92.1	0.27	11.9

The data show that obtained carbon foam is distinguished by high relative density, and high compressive strength, respectively. High relative density implies presence of thicker cell walls and shorter cell edges, which promote higher compressive strength [4].

Carbon foam texture. Figure 1 shows the optical texture of the carbon foam under polarized light in the optical microscope. Microscopical observations showed that the carbonization product is a distinctly

porous material, where mesopores (visible under optical microscopy) have various, irregular shapes and strongly diversified diameters from less than 1 μm to more than several hundreds μm .

The internal structure of the sample is similar to cokes from coal-tar or petroleum pitch, or from very good coking coals. They look like coarse mosaics, domain or flow type according to classification of optical textures of cokes [59]. Maximum reflectance values of the samples are also similar to cokes. The presence of QI in pitch precursor prevents the coalescence of the mesophase spheres during the thermal treatment, hence optical textures of carbon foam are generally anisotropic.

Highly anisotropic textures are dominant. The possible reason for this is the higher content of QI. Lower anisotropic textures transform into higher anisotropic gradually, but some sharp borders between them also exist.

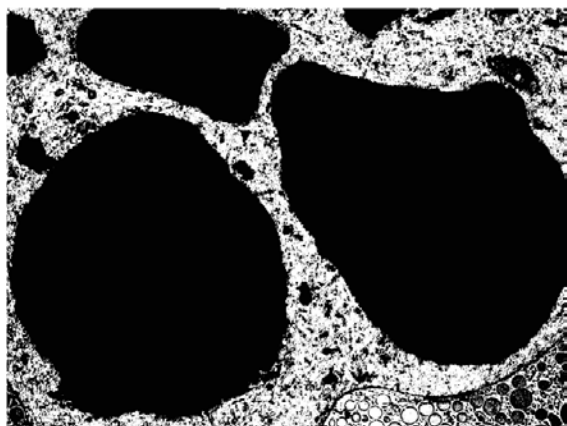


Figure 1. Optical texture of carbon foam derived from pitch, modified with H_2SO_4 (magnification 80x, in air).

SEM observation of carbon foam structure. Figure 2 shows the SEM image of the obtained carbon foam. The foam cell is mainly open with size around 300-350 μm and some cell membranes. The SEM image clearly shows that there are cracks in the samples. These cracks mainly occur between the layers aligned parallel to the cell surfaces, especially in the junction area of the foam cells. However, no major cracks are found on the cell membranes of obtained foams. Carbon foam has small anisotropic domain due to the high content of QI, which prevents the coalescence of the mesophase spheres during thermal treatment.

Ultrasonic velocity, dynamic elastic modulus and elastic anisotropy. The ultrasonic velocity (Table 4) has considerably high values in (a), (b) and (c) directions. High stiffness corresponds to considerably high values of ultrasonic velocities [57]. This confirms suitable arrangement of the layers in carbon foam, due to considerably high

content of QI, which leads to formation of highly orientated structure of the well condensed crystallites, what is indicated by the high ultrasonic velocity.

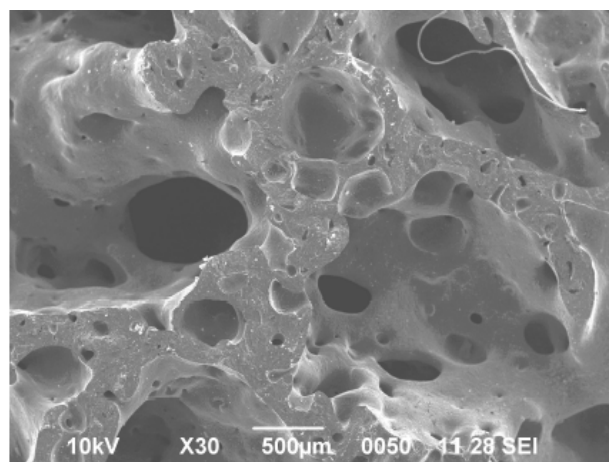


Figure 2. SEM image of carbon foam derived from pitch, modified with H_2SO_4 .

Table 4. Values of ultrasonic velocity of obtained carbon foams.

Physical parameter	Pitch treated with H_2SO_4
Ultrasonic velocity: direction a, v_{max} (m/s)	1948
Ultrasonic velocity: direction b, v_{mid} (m/s)	1931
Ultrasonic velocity: direction c, v_{min} (m/s)	1846
Elastic modulus: direction a, E_{max} (GPa)	1.98
Elastic modulus: direction b, E_{mid} (GPa)	1.95
Elastic modulus: direction c, E_{min} (GPa)	1.78
Elastic anisotropy, $v_{\text{max}}/v_{\text{min}}$	1.06

CONCLUSIONS

Thermal oxidation modification of commercial coal tar pitch with H_2SO_4 is appropriate treatment to adjust its plastic properties before the foaming process. The properties of modified coal tar pitches can be controlled by thermal oxidation treatment conditions - temperature and amount of added acid. The obtained pitch is suitable foaming precursor and it can generate anisotropic carbon foam with good mechanical strength. The low content of PES and high content of QI in the pitch, treated with H_2SO_4 , lead to formation of small crystallites, and respectively carbon foam with high porosity and considerably high strength.

It is important to note that the composition of the obtained modified pitches allows foam formation at relatively low pressure and fast heating of the precursor during foaming process, and without any stabilization treatment.

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СИНТЕЗ И ОХАРАКТЕРИЗИРАНЕ НА ВЪГЛЕРОДНА ПЯНА ПОСРЕДСТВОМ
ПЕНООБРАЗУВАНЕ ПРИ НИСКО НАЛЯГАНЕ С ИЗПОЛЗВАНЕТО НА ПЕК
МОДИФИЦИРАН СЪС H₂SO₄ КАТО ПРЕКУРСОР

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(Резюме)

Получени бяха образци от въглеродни пени с анизотропна текстура и добра механична якост, на базата на каменовъглен пек, модифициран чрез термоокислителна обработка със H₂SO₄. Изследвана бе зависимостта между свойствата на прекурсора и структурата на получената въглеродна пяна и беше показано, че химичния състав и точката на омекване на изходния пек оказват значително влияние върху процеса на пенообразуване и върху порьозната структура на въглеродната пяна. Установено бе, че механичната якост на въглеродната пяна зависи не само от порьозната структура на въглеродната пяна, но и от състава на прекурсора.

Химичния състав на модифицираните и синтетичните пекове позволява образуване на въглеродна пяна при относително ниски налягания и бърз режим на нагрявяне на прекурсора по време на процеса на пенообразуване, като се избягва етапа на стабилизационна обработката.